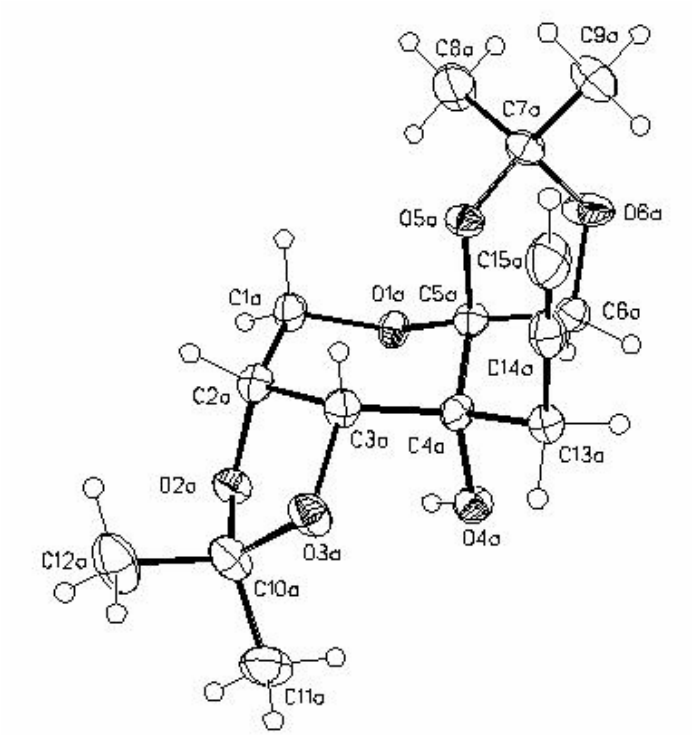
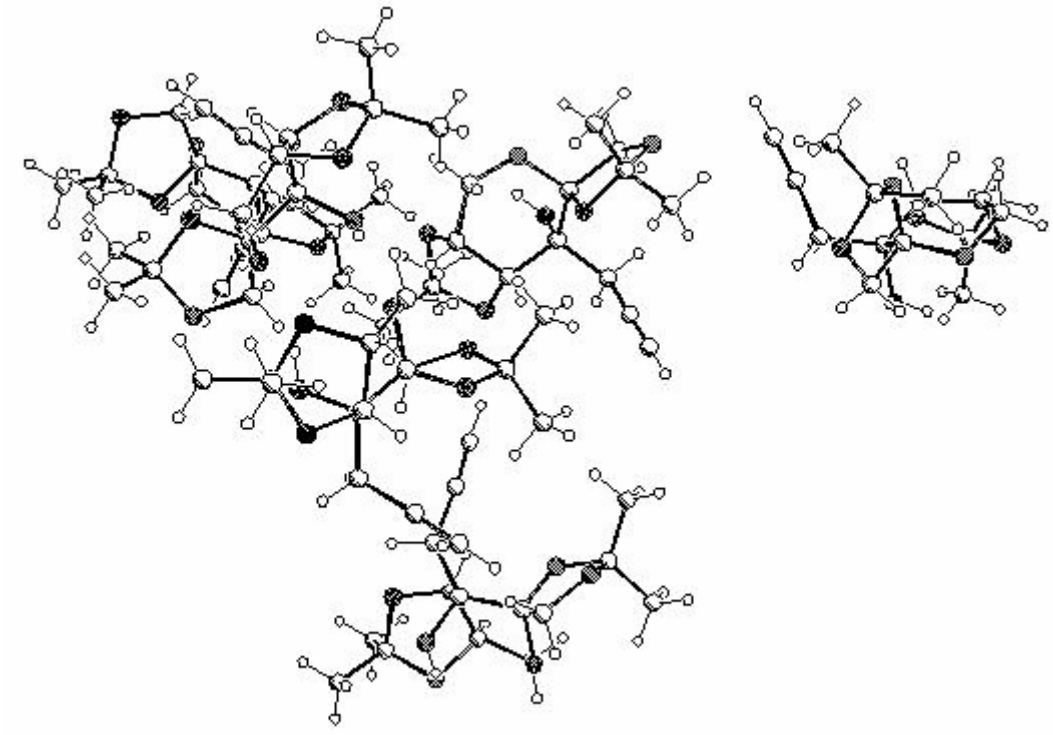
# H Röntgenkristallstrukturanalysen

**1.** **(*1R,4S,5R,6R*)-8,8,2´,2´-Tetramethyl-5-(2´´-propinyl)-spiro-1,3-dioxa-cyclopentyl-4,4´-3,7,9-trioxabicyclo[4.3.0]-nonan** (1,2:4,5-Di-*O*-iso-propyliden-3-(2-propinyl)-α-D-psicopyranose) ***[4]***



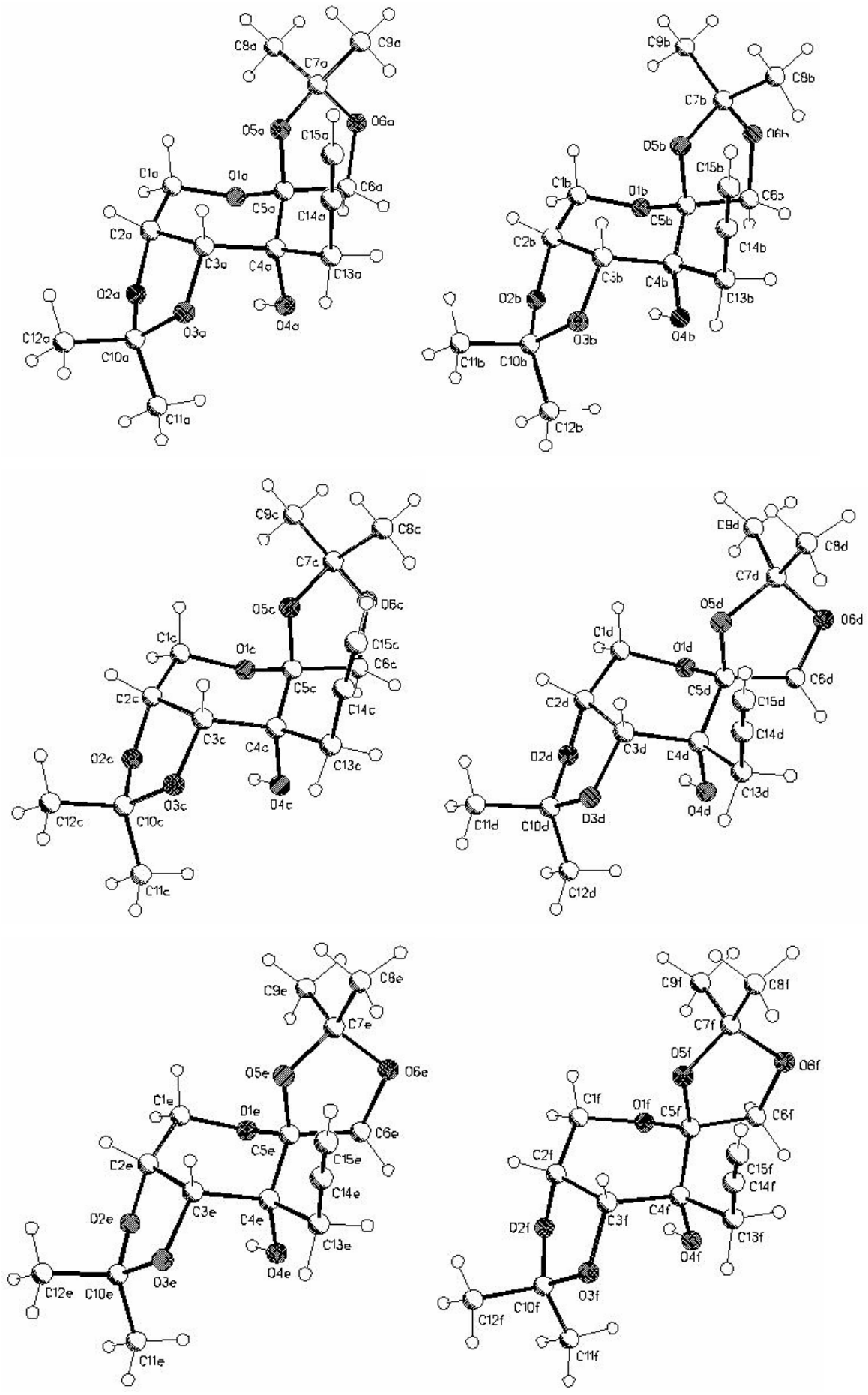

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**Table 1: Crystal data and structure refinements for *[4]***

| | |
|---|---|
| Identification code | ***[4]*** |
| Empirical formula | $C_{15}H_{22}O_6$ |
| Formula weight | 298.33 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 12.8056(1) Å α = 90° |
| | b = 17.1285(2) Å β = 90° |
| | c = 42.3902(6) Å γ = 90° |
| Volume | 9297.90(19) $Å^3$ |
| Z | 24 |
| Calculated density | 1.279 $mg/m^3$ |
| Absorption coefficient | 0.098 $mm^{-1}$ |
| F(000) | 3840 |
| Crystal size | 0.40 x 0.30 x 0.20 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.57 to 25.00° |
| Limiting indices | $-14 \le h \le 15$, $-17 \le k \le 20$, $-45 \le l \le 50$ |
| Reflections collected / unique | 37232 / 15157 [R(int) = 0.0491] |
| Completeness to Θ = 24.99 | 96.9 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 15157 / 0 / 1141 |
| Goodness-of-fit on $F^2$ | 0.854 |
| Final R indices [I>2σ(I)] | R1 = 0.0381, wR2 = 0.0545 |
| R indices (all data) | R1 = 0.0836, wR2 = 0.0620 |
| Absolute structure parameter | 0.3(5), cannot be determined reliabl. |
| Largest diff. peak and hole | 0.391 and -0.196 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[4]*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| O(1A) | 6447(1) | 5761(1) | 4980(1) | 20(1) |
| C(1A) | 7184(2) | 5424(2) | 4762(1) | 24(1) |
| C(2A) | 7972(2) | 4899(1) | 4917(1) | 21(1) |
| O(2A) | 8773(1) | 5325(1) | 5079(1) | 22(1) |
| C(3A) | 7537(2) | 4391(2) | 5182(1) | 22(1) |
| O(3A) | 8429(1) | 4238(1) | 5375(1) | 27(1) |
| C(4A) | 6665(2) | 4777(2) | 5377(1) | 20(1) |
| O(4A) | 7048(1) | 5369(1) | 5585(1) | 24(1) |
| C(5A) | 5885(2) | 5179(2) | 5157(1) | 19(1) |
| O(5A) | 5397(1) | 4636(1) | 4957(1) | 21(1) |
| C(6A) | 4979(2) | 5593(2) | 5314(1) | 23(1) |
| O(6A) | 4151(1) | 5533(1) | 5094(1) | 36(1) |
| C(7A) | 4380(2) | 4940(2) | 4870(1) | 23(1) |
| C(8A) | 4406(2) | 5291(2) | 4545(1) | 38(1) |
| C(9A) | 3615(2) | 4279(2) | 4903(1) | 34(1) |
| C(10A) | 9251(2) | 4779(2) | 5287(1) | 27(1) |
| C(11A) | 9639(2) | 5210(2) | 5574(1) | 38(1) |
| C(12A) | 10112(2) | 4319(2) | 5123(1) | 40(1) |
| C(13A) | 6199(2) | 4166(1) | 5606(1) | 22(1) |
| C(14A) | 5792(2) | 3447(2) | 5458(1) | 25(1) |
| C(15A) | 5492(2) | 2863(2) | 5349(1) | 34(1) |
| | **x** | **y** | **z** | **U(eq)** |
| O(1B) | 2217(1) | 1702(1) | 6233(1) | 23(1) |
| C(1B) | 1311(2) | 2006(2) | 6394(1) | 23(1) |
| C(2B) | 1534(2) | 2725(2) | 6585(1) | 20(1) |
| O(2B) | 2012(1) | 2534(1) | 6881(1) | 22(1) |
| C(3B) | 2334(2) | 3274(1) | 6442(1) | 20(1) |
| O(3B) | 2750(1) | 3678(1) | 6711(1) | 24(1) |
| C(4B) | 3221(2) | 2875(2) | 6259(1) | 21(1) |
| O(4B) | 3928(1) | 2460(1) | 6457(1) | 25(1) |
| C(5B) | 2733(2) | 2276(2) | 6039(1) | 20(1) |
| O(5B) | 2020(1) | 2639(1) | 5832(1) | 23(1) |
| C(6B) | 3431(2) | 1819(2) | 5819(1) | 25(1) |
| O(6B) | 2730(1) | 1588(1) | 5575(1) | 31(1) |
| C(7B) | 2001(2) | 2212(2) | 5537(1) | 23(1) |
| C(8B) | 2356(2) | 2754(1) | 5278(1) | 28(1) |
| C(9B) | 923(2) | 1870(2) | 5486(1) | 33(1) |
| C(10B) | 2453(2) | 3249(2) | 6992(1) | 26(1) |
| C(11B) | 1659(2) | 3737(2) | 7170(1) | 31(1) |
| C(12B) | 3418(2) | 3065(2) | 7185(1) | 29(1) |
| C(13B) | 3925(2) | 3490(2) | 6103(1) | 26(1) |
| C(14B) | 3388(2) | 4066(2) | 5901(1) | 28(1) |
| C(15B) | 2983(2) | 4545(2) | 5746(1) | 36(1) |

| | x | y | z | U(eq) |
|---|---|---|---|---|
| O(1C) | 7585(1) | 6959(1) | 5410(1) | 21(1) |
| C(1C) | 8457(2) | 7219(2) | 5221(1) | 21(1) |
| C(2C) | 8149(2) | 7730(2) | 4948(1) | 20(1) |
| O(2C) | 7726(1) | 7272(1) | 4691(1) | 22(1) |
| C(3C) | 7251(2) | 8293(2) | 5012(1) | 21(1) |
| O(3C) | 6813(1) | 8421(1) | 4705(1) | 25(1) |
| C(4C) | 6427(2) | 7973(2) | 5243(1) | 20(1) |
| O(4C) | 5798(1) | 7372(1) | 5104(1) | 23(1) |
| C(5C) | 6974(2) | 7606(2) | 5526(1) | 19(1) |
| O(5C) | 7615(1) | 8151(1) | 5681(1) | 21(1) |
| C(6C) | 6298(2) | 7286(2) | 5788(1) | 24(1) |
| O(6C) | 6974(1) | 7306(1) | 6056(1) | 28(1) |
| C(7C) | 7707(2) | 7926(2) | 6009(1) | 24(1) |
| C(8C) | 7446(2) | 8622(2) | 6209(1) | 34(1) |
| C(9C) | 8779(2) | 7592(2) | 6076(1) | 32(1) |
| C(10C) | 7192(2) | 7821(2) | 4493(1) | 24(1) |
| C(11C) | 6285(2) | 7413(2) | 4336(1) | 32(1) |
| C(12C) | 7928(2) | 8187(2) | 4257(1) | 32(1) |
| C(13C) | 5621(2) | 8614(1) | 5320(1) | 25(1) |
| C(14C) | 6021(2) | 9310(2) | 5485(1) | 28(1) |
| C(15C) | 6310(2) | 9877(2) | 5610(1) | 38(1) |
| | **x** | **y** | **z** | **U(eq)** |
| O(1D) | 8354(1) | 4854(1) | 6614(1) | 26(1) |
| C(1D) | 7787(2) | 5066(2) | 6335(1) | 26(1) |
| C(2D) | 6794(2) | 5504(1) | 6405(1) | 22(1) |
| O(2D) | 5972(1) | 4988(1) | 6509(1) | 25(1) |
| C(3D) | 6854(2) | 6081(2) | 6677(1) | 22(1) |
| O(3D) | 5782(1) | 6142(1) | 6779(1) | 26(1) |
| C(4D) | 7558(2) | 5801(2) | 6949(1) | 23(1) |
| O(4D) | 7115(1) | 5173(1) | 7123(1) | 26(1) |
| C(5D) | 8596(2) | 5510(2) | 6814(1) | 27(1) |
| O(5D) | 9101(1) | 6104(1) | 6644(1) | 27(1) |
| C(6D) | 9417(2) | 5226(2) | 7050(1) | 34(1) |
| O(6D) | 10337(2) | 5586(2) | 6961(1) | 76(1) |
| C(7D) | 10222(2) | 5987(2) | 6676(1) | 36(1) |
| C(8D) | 10729(2) | 6773(2) | 6702(1) | 62(1) |
| C(9D) | 10653(2) | 5514(2) | 6407(1) | 70(1) |
| C(10D) | 5199(2) | 5487(2) | 6648(1) | 27(1) |
| C(11D) | 4438(2) | 5794(2) | 6401(1) | 37(1) |
| C(12D) | 4662(2) | 5047(2) | 6910(1) | 34(1) |
| C(13D) | 7643(2) | 6444(1) | 7201(1) | 29(1) |
| C(14D) | 8011(2) | 7217(2) | 7094(1) | 29(1) |
| C(15D) | 8270(2) | 7847(2) | 7023(1) | 37(1) |

| | x | y | z | U(eq) |
|---|---|---|---|---|
| O(1E) | 3639(1) | 10853(1) | 6730(1) | 23(1) |
| C(1E) | 2932(2) | 10599(2) | 6971(1) | 27(1) |
| C(2E) | 2219(2) | 9945(1) | 6866(1) | 23(1) |
| O(2E) | 1364(1) | 10246(1) | 6683(1) | 28(1) |
| C(3E) | 2686(2) | 9347(2) | 6645(1) | 23(1) |
| O(3E) | 1801(1) | 9061(1) | 6474(1) | 29(1) |
| C(4E) | 3500(2) | 9697(2) | 6416(1) | 21(1) |
| O(4E) | 3042(1) | 10174(1) | 6176(1) | 26(1) |
| C(5E) | 4250(2) | 10217(2) | 6603(1) | 25(1) |
| O(5E) | 4736(1) | 9787(1) | 6842(1) | 23(1) |
| C(6E) | 5152(2) | 10593(2) | 6420(1) | 29(1) |
| O(6E) | 6065(1) | 10385(1) | 6591(1) | 45(1) |
| C(7E) | 5758(2) | 10119(2) | 6896(1) | 29(1) |
| C(8E) | 6474(2) | 9468(2) | 6992(1) | 41(1) |
| C(9E) | 5710(2) | 10773(2) | 7131(1) | 42(1) |
| C(10E) | 943(2) | 9584(2) | 6524(1) | 31(1) |
| C(11E) | 498(2) | 9853(2) | 6213(1) | 41(1) |
| C(12E) | 129(2) | 9176(2) | 6727(1) | 41(1) |
| C(13E) | 4015(2) | 9045(2) | 6220(1) | 29(1) |
| C(14E) | 4507(2) | 8397(2) | 6395(1) | 29(1) |
| C(15E) | 4901(2) | 7863(2) | 6517(1) | 31(1) |
| | **x** | **y** | **z** | **U(eq)** |
| O(1F) | 13011(1) | 8540(1) | 8025(1) | 22(1) |
| C(1F) | 13671(2) | 8135(2) | 8245(1) | 24(1) |
| C(2F) | 13061(2) | 7629(2) | 8470(1) | 28(1) |
| O(2F) | 12595(1) | 8091(1) | 8715(1) | 31(1) |
| C(3F) | 12116(2) | 7212(2) | 8332(1) | 27(1) |
| O(3F) | 11432(1) | 7119(1) | 8597(1) | 35(1) |
| C(4F) | 11561(2) | 7672(2) | 8068(1) | 24(1) |
| O(4F) | 10969(1) | 8316(1) | 8191(1) | 29(1) |
| C(5F) | 12363(2) | 8027(2) | 7843(1) | 22(1) |
| O(5F) | 12954(1) | 7436(1) | 7697(1) | 24(1) |
| C(6F) | 11920(2) | 8503(2) | 7571(1) | 27(1) |
| O(6F) | 12261(1) | 8113(1) | 7292(1) | 27(1) |
| C(7F) | 13172(2) | 7689(2) | 7379(1) | 25(1) |
| C(8F) | 13259(2) | 6982(2) | 7170(1) | 34(1) |
| C(9F) | 14141(2) | 8210(2) | 7369(1) | 34(1) |
| C(10F) | 11789(2) | 7617(2) | 8851(1) | 40(1) |
| C(11F) | 10929(2) | 8153(2) | 8960(1) | 57(1) |
| C(12F) | 12223(3) | 7098(2) | 9111(1) | 69(1) |
| C(13F) | 10715(2) | 7161(2) | 7913(1) | 29(1) |
| C(14F) | 11051(2) | 6410(2) | 7782(1) | 30(1) |
| C(15F) | 11246(2) | 5805(2) | 7673(1) | 49(1) |

**Tabelle 3: Bond lengths [Å] for *[4]*.**

| | | | |
|---|---|---|---|
| O(1A)-C(5A) | 1.439(3) | C(5A)-O(5A) | 1.406(3) |
| O(1A)-C(1A) | 1.443(3) | C(5A)-C(6A) | 1.513(3) |
| C(1A)-C(2A) | 1.503(3) | O(5A)-C(7A) | 1.451(3) |
| C(2A)-O(2A) | 1.434(3) | C(6A)-O(6A) | 1.414(3) |
| C(2A)-C(3A) | 1.526(3) | O(6A)-C(7A) | 1.422(3) |
| O(2A)-C(10A) | 1.423(3) | C(7A)-C(8A) | 1.504(4) |
| C(3A)-O(3A) | 1.430(3) | C(7A)-C(9A) | 1.504(3) |
| C(3A)-C(4A) | 1.538(3) | C(10A)-C(11A) | 1.507(4) |
| O(3A)-C(10A) | 1.451(3) | C(10A)-C(12A) | 1.522(3) |
| C(4A)-O(4A) | 1.431(3) | C(13A)-C(14A) | 1.478(4) |
| C(4A)-C(5A) | 1.529(3) | C(14A)-C(15A) | 1.167(3) |
| C(4A)-C(13A) | 1.548(3) | | |
| O(1B)-C(5B) | 1.443(3) | C(5B)-O(5B) | 1.409(3) |
| O(1B)-C(1B) | 1.443(3) | C(5B)-C(6B) | 1.511(3) |
| C(1B)-C(2B) | 1.502(3) | O(5B)-C(7B) | 1.450(3) |
| C(2B)-O(2B) | 1.437(3) | C(6B)-O(6B) | 1.424(3) |
| C(2B)-C(3B) | 1.517(3) | O(6B)-C(7B) | 1.429(3) |
| O(2B)-C(10B) | 1.428(3) | C(7B)-C(8B) | 1.507(3) |
| C(3B)-O(3B) | 1.438(3) | C(7B)-C(9B) | 1.516(3) |
| C(3B)-C(4B) | 1.536(3) | C(10B)-C(12B) | 1.514(3) |
| O(3B)-C(10B) | 1.449(3) | C(10B)-C(11B) | 1.518(3) |
| C(4B)-O(4B) | 1.426(3) | C(13B)-C(14B) | 1.475(4) |
| C(4B)-C(5B) | 1.521(3) | C(14B)-C(15B) | 1.173(4) |
| C(4B)-C(13B) | 1.536(3) | | |
| O(1C)-C(5C) | 1.445(3) | C(5C)-O(5C) | 1.406(3) |
| O(1C)-C(1C) | 1.445(3) | C(5C)-C(6C) | 1.510(3) |
| C(1C)-C(2C) | 1.505(3) | O(5C)-C(7C) | 1.446(3) |
| C(2C)-O(2C) | 1.445(3) | C(6C)-O(6C) | 1.429(3) |
| C(2C)-C(3C) | 1.526(3) | O(6C)-C(7C) | 1.430(3) |
| O(2C)-C(10C) | 1.434(3) | C(7C)-C(8C) | 1.501(3) |
| C(3C)-O(3C) | 1.434(3) | C(7C)-C(9C) | 1.515(3) |
| C(3C)-C(4C) | 1.540(3) | C(10C)-C(11C) | 1.509(3) |
| O(3C)-C(10C) | 1.447(3) | C(10C)-C(12C) | 1.511(3) |
| C(4C)-O(4C) | 1.433(3) | C(13C)-C(14C) | 1.473(4) |
| C(4C)-C(5C) | 1.527(3) | C(14C)-C(15C) | 1.168(3) |
| C(4C)-C(13C) | 1.542(3) | | |

| | | | |
|---|---|---|---|
| O(1D)-C(1D) | 1.436(3) | C(5D)-O(5D) | 1.403(3) |
| O(1D)-C(5D) | 1.440(3) | C(5D)-C(6D) | 1.532(3) |
| C(1D)-C(2D) | 1.506(3) | O(5D)-C(7D) | 1.456(3) |
| C(2D)-O(2D) | 1.445(3) | C(6D)-O(6D) | 1.384(3) |
| C(2D)-C(3D) | 1.520(3) | O(6D)-C(7D) | 1.398(3) |
| O(2D)-C(10D) | 1.434(3) | C(7D)-C(8D) | 1.497(4) |
| C(3D)-O(3D) | 1.442(3) | C(7D)-C(9D) | 1.503(4) |
| C(3D)-C(4D) | 1.540(3) | C(10D)-C(12D) | 1.510(4) |
| O(3D)-C(10D) | 1.456(3) | C(10D)-C(11D) | 1.524(3) |
| C(4D)-O(4D) | 1.422(3) | C(13D)-C(14D) | 1.477(4) |
| C(4D)-C(5D) | 1.532(3) | C(14D)-C(15D) | 1.169(3) |
| C(4D)-C(13D) | 1.538(3) | | |
| O(1E)-C(1E) | 1.433(3) | C(5E)-O(5E) | 1.399(3) |
| O(1E)-C(5E) | 1.445(3) | C(5E)-C(6E) | 1.533(3) |
| C(1E)-C(2E) | 1.512(3) | O(5E)-C(7E) | 1.446(3) |
| C(2E)-O(2E) | 1.437(3) | C(6E)-O(6E) | 1.421(3) |
| C(2E)-C(3E) | 1.514(3) | O(6E)-C(7E) | 1.426(3) |
| O(2E)-C(10E) | 1.425(3) | C(7E)-C(8E) | 1.499(3) |
| C(3E)-O(3E) | 1.432(3) | C(7E)-C(9E) | 1.501(4) |
| C(3E)-C(4E) | 1.545(3) | C(10E)-C(11E) | 1.509(4) |
| O(3E)-C(10E) | 1.434(3) | C(10E)-C(12E) | 1.522(3) |
| C(4E)-O(4E) | 1.428(3) | C(13E)-C(14E) | 1.477(4) |
| C(4E)-C(5E) | 1.532(3) | C(14E)-C(15E) | 1.165(3) |
| C(4E)-C(13E) | 1.538(3) | | |
| O(1F)-C(5F) | 1.434(3) | C(5F)-O(5F) | 1.407(3) |
| O(1F)-C(1F) | 1.437(3) | C(5F)-C(6F) | 1.521(3) |
| C(1F)-C(2F) | 1.507(3) | O(5F)-C(7F) | 1.443(3) |
| C(2F)-O(2F) | 1.435(3) | C(6F)-O(6F) | 1.428(3) |
| C(2F)-C(3F) | 1.524(3) | O(6F)-C(7F) | 1.423(3) |
| O(2F)-C(10F) | 1.435(3) | C(7F)-C(8F) | 1.505(3) |
| C(3F)-O(3F) | 1.435(3) | C(7F)-C(9F) | 1.529(3) |
| C(3F)-C(4F) | 1.539(4) | C(10F)-C(11F) | 1.506(4) |
| O(3F)-C(10F) | 1.447(3) | C(10F)-C(12F) | 1.522(4) |
| C(4F)-O(4F) | 1.435(3) | C(13F)-C(14F) | 1.467(4) |
| C(4F)-C(5F) | 1.529(3) | C(14F)-C(15F) | 1.160(4) |
| C(4F)-C(13F) | 1.540(3) | | |

**Tabelle 4: Bond angles [°] for *[4]*.**

| | | | |
|---|---|---|---|
| C(5A)-O(1A)-C(1A) | 112.58(18) | O(1A)-C(5A)-C(4A) | 107.59(19) |
| O(1A)-C(1A)-C(2A) | 113.4(2) | C(6A)-C(5A)-C(4A) | 116.4(2) |
| O(2A)-C(2A)-C(1A) | 112.7(2) | C(5A)-O(5A)-C(7A) | 108.33(19) |
| O(2A)-C(2A)-C(3A) | 101.5(2) | O(6A)-C(6A)-C(5A) | 104.6(2) |
| C(1A)-C(2A)-C(3A) | 114.7(2) | C(6A)-O(6A)-C(7A) | 109.69(18) |
| C(10A)-O(2A)-C(2A) | 105.73(19) | O(6A)-C(7A)-O(5A) | 105.8(2) |
| O(3A)-C(3A)-C(2A) | 103.51(19) | O(6A)-C(7A)-C(8A) | 109.3(2) |
| O(3A)-C(3A)-C(4A) | 110.6(2) | O(5A)-C(7A)-C(8A) | 110.9(2) |
| C(2A)-C(3A)-C(4A) | 114.5(2) | O(6A)-C(7A)-C(9A) | 110.0(2) |
| C(3A)-O(3A)-C(10A) | 108.40(19) | O(5A)-C(7A)-C(9A) | 106.9(2) |
| O(4A)-C(4A)-C(5A) | 106.3(2) | C(8A)-C(7A)-C(9A) | 113.7(2) |
| O(4A)-C(4A)-C(3A) | 112.8(2) | O(2A)-C(10A)-O(3A) | 105.47(19) |
| C(5A)-C(4A)-C(3A) | 109.9(2) | O(2A)-C(10A)-C(11A) | 108.8(2) |
| O(4A)-C(4A)-C(13A) | 102.9(2) | O(3A)-C(10A)-C(11A) | 110.1(2) |
| C(5A)-C(4A)-C(13A) | 115.8(2) | O(2A)-C(10A)-C(12A) | 111.6(2) |
| C(3A)-C(4A)-C(13A) | 109.0(2) | O(3A)-C(10A)-C(12A) | 108.1(2) |
| O(5A)-C(5A)-O(1A) | 111.5(2) | C(11A)-C(10A)-C(12A) | 112.5(2) |
| O(5A)-C(5A)-C(6A) | 103.53(19) | C(14A)-C(13A)-C(4A) | 115.6(2) |
| O(1A)-C(5A)-C(6A) | 106.7(2) | C(15A)-C(14A)-C(13A) | 177.4(3) |
| O(5A)-C(5A)-C(4A) | 111.1(2) | | |
| C(5B)-O(1B)-C(1B) | 113.08(18) | O(1B)-C(5B)-C(4B) | 107.4(2) |
| O(1B)-C(1B)-C(2B) | 113.5(2) | C(6B)-C(5B)-C(4B) | 119.1(2) |
| O(2B)-C(2B)-C(1B) | 111.4(2) | C(5B)-O(5B)-C(7B) | 108.95(19) |
| O(2B)-C(2B)-C(3B) | 101.74(19) | O(6B)-C(6B)-C(5B) | 102.66(19) |
| C(1B)-C(2B)-C(3B) | 115.0(2) | C(6B)-O(6B)-C(7B) | 106.61(19) |
| C(10B)-O(2B)-C(2B) | 105.05(19) | O(6B)-C(7B)-O(5B) | 105.6(2) |
| O(3B)-C(3B)-C(2B) | 103.3(2) | O(6B)-C(7B)-C(8B) | 110.2(2) |
| O(3B)-C(3B)-C(4B) | 109.99(19) | O(5B)-C(7B)-C(8B) | 108.3(2) |
| C(2B)-C(3B)-C(4B) | 115.1(2) | O(6B)-C(7B)-C(9B) | 108.7(2) |
| C(3B)-O(3B)-C(10B) | 108.16(18) | O(5B)-C(7B)-C(9B) | 109.6(2) |
| O(4B)-C(4B)-C(5B) | 106.6(2) | C(8B)-C(7B)-C(9B) | 114.1(2) |
| O(4B)-C(4B)-C(3B) | 113.2(2) | O(2B)-C(10B)-O(3B) | 105.7(2) |
| C(5B)-C(4B)-C(3B) | 107.9(2) | O(2B)-C(10B)-C(12B) | 108.8(2) |
| O(4B)-C(4B)-C(13B) | 102.89(19) | O(3B)-C(10B)-C(12B) | 109.5(2) |
| C(5B)-C(4B)-C(13B) | 116.1(2) | O(2B)-C(10B)-C(11B) | 111.7(2) |
| C(3B)-C(4B)-C(13B) | 110.2(2) | O(3B)-C(10B)-C(11B) | 107.7(2) |
| O(5B)-C(5B)-O(1B) | 110.95(19) | C(12B)-C(10B)-C(11B) | 113.2(2) |
| O(5B)-C(5B)-C(6B) | 103.2(2) | C(14B)-C(13B)-C(4B) | 115.7(2) |
| O(1B)-C(5B)-C(6B) | 105.7(2) | C(15B)-C(14B)-C(13B) | 177.6(3) |
| O(5B)-C(5B)-C(4B) | 110.4(2) | | |

| | | | |
|---|---|---|---|
| C(5C)-O(1C)-C(1C) | 111.76(18) | O(1C)-C(5C)-C(4C) | 107.2(2) |
| O(1C)-C(1C)-C(2C) | 113.79(19) | C(6C)-C(5C)-C(4C) | 117.7(2) |
| O(2C)-C(2C)-C(1C) | 111.2(2) | C(5C)-O(5C)-C(7C) | 108.60(19) |
| O(2C)-C(2C)-C(3C) | 101.28(19) | O(6C)-C(6C)-C(5C) | 103.16(19) |
| C(1C)-C(2C)-C(3C) | 115.3(2) | C(6C)-O(6C)-C(7C) | 107.74(19) |
| C(10C)-O(2C)-C(2C) | 105.24(18) | O(6C)-C(7C)-O(5C) | 106.2(2) |
| O(3C)-C(3C)-C(2C) | 103.2(2) | O(6C)-C(7C)-C(8C) | 111.4(2) |
| O(3C)-C(3C)-C(4C) | 111.2(2) | O(5C)-C(7C)-C(8C) | 108.2(2) |
| C(2C)-C(3C)-C(4C) | 113.9(2) | O(6C)-C(7C)-C(9C) | 106.8(2) |
| C(3C)-O(3C)-C(10C) | 108.84(19) | O(5C)-C(7C)-C(9C) | 110.8(2) |
| O(4C)-C(4C)-C(5C) | 106.6(2) | C(8C)-C(7C)-C(9C) | 113.3(2) |
| O(4C)-C(4C)-C(3C) | 112.4(2) | O(2C)-C(10C)-O(3C) | 105.2(2) |
| C(5C)-C(4C)-C(3C) | 109.4(2) | O(2C)-C(10C)-C(11C) | 108.7(2) |
| O(4C)-C(4C)-C(13C) | 102.83(18) | O(3C)-C(10C)-C(11C) | 110.1(2) |
| C(5C)-C(4C)-C(13C) | 115.6(2) | O(2C)-C(10C)-C(12C) | 111.2(2) |
| C(3C)-C(4C)-C(13C) | 109.9(2) | O(3C)-C(10C)-C(12C) | 109.0(2) |
| O(5C)-C(5C)-O(1C) | 110.61(18) | C(11C)-C(10C)-C(12C) | 112.3(2) |
| O(5C)-C(5C)-C(6C) | 103.4(2) | C(14C)-C(13C)-C(4C) | 116.3(2) |
| O(1C)-C(5C)-C(6C) | 106.4(2) | C(15C)-C(14C)-C(13C) | 177.5(3) |
| O(5C)-C(5C)-C(4C) | 111.3(2) | | |
| C(1D)-O(1D)-C(5D) | 113.33(19) | O(1D)-C(5D)-C(6D) | 106.5(2) |
| O(1D)-C(1D)-C(2D) | 113.0(2) | C(4D)-C(5D)-C(6D) | 117.0(3) |
| O(2D)-C(2D)-C(1D) | 111.8(2) | C(5D)-O(5D)-C(7D) | 107.9(2) |
| O(2D)-C(2D)-C(3D) | 101.6(2) | O(6D)-C(6D)-C(5D) | 105.2(2) |
| C(1D)-C(2D)-C(3D) | 115.5(2) | C(6D)-O(6D)-C(7D) | 111.5(2) |
| C(10D)-O(2D)-C(2D) | 105.27(18) | O(6D)-C(7D)-O(5D) | 104.5(2) |
| O(3D)-C(3D)-C(2D) | 103.08(19) | O(6D)-C(7D)-C(8D) | 109.3(3) |
| O(3D)-C(3D)-C(4D) | 110.8(2) | O(5D)-C(7D)-C(8D) | 108.1(2) |
| C(2D)-C(3D)-C(4D) | 113.3(2) | O(6D)-C(7D)-C(9D) | 110.6(3) |
| C(3D)-O(3D)-C(10D) | 108.53(19) | O(5D)-C(7D)-C(9D) | 111.5(3) |
| O(4D)-C(4D)-C(5D) | 107.1(2) | C(8D)-C(7D)-C(9D) | 112.6(3) |
| O(4D)-C(4D)-C(13D) | 102.1(2) | O(2D)-C(10D)-O(3D) | 105.18(18) |
| C(5D)-C(4D)-C(13D) | 115.5(2) | O(2D)-C(10D)-C(12D) | 108.6(2) |
| O(4D)-C(4D)-C(3D) | 113.0(2) | O(3D)-C(10D)-C(12D) | 109.7(2) |
| C(5D)-C(4D)-C(3D) | 109.2(2) | O(2D)-C(10D)-C(11D) | 111.4(2) |
| C(13D)-C(4D)-C(3D) | 109.8(2) | O(3D)-C(10D)-C(11D) | 108.9(2) |
| O(5D)-C(5D)-O(1D) | 111.3(2) | C(12D)-C(10D)-C(11D) | 112.7(2) |
| O(5D)-C(5D)-C(4D) | 110.9(2) | C(14D)-C(13D)-C(4D) | 116.9(2) |
| O(1D)-C(5D)-C(4D) | 106.6(2) | C(15D)-C(14D)-C(13D) | 176.4(3) |
| O(5D)-C(5D)-C(6D) | 104.5(2) | | |

| | | | |
|---|---|---|---|
| C(1E)-O(1E)-C(5E) | 112.20(19) | O(1E)-C(5E)-C(6E) | 106.3(2) |
| O(1E)-C(1E)-C(2E) | 113.4(2) | C(4E)-C(5E)-C(6E) | 117.0(3) |
| O(2E)-C(2E)-C(1E) | 110.6(2) | C(5E)-O(5E)-C(7E) | 108.0(2) |
| O(2E)-C(2E)-C(3E) | 102.0(2) | O(6E)-C(6E)-C(5E) | 104.9(2) |
| C(1E)-C(2E)-C(3E) | 116.4(2) | C(6E)-O(6E)-C(7E) | 108.4(2) |
| C(10E)-O(2E)-C(2E) | 104.95(19) | O(6E)-C(7E)-O(5E) | 103.5(2) |
| O(3E)-C(3E)-C(2E) | 103.45(19) | O(6E)-C(7E)-C(8E) | 108.5(2) |
| O(3E)-C(3E)-C(4E) | 110.4(2) | O(5E)-C(7E)-C(8E) | 107.7(2) |
| C(2E)-C(3E)-C(4E) | 113.3(2) | O(6E)-C(7E)-C(9E) | 112.0(2) |
| C(3E)-O(3E)-C(10E) | 108.50(19) | O(5E)-C(7E)-C(9E) | 111.1(2) |
| O(4E)-C(4E)-C(5E) | 107.0(2) | C(8E)-C(7E)-C(9E) | 113.5(3) |
| O(4E)-C(4E)-C(13E) | 102.0(2) | O(2E)-C(10E)-O(3E) | 106.1(2) |
| C(5E)-C(4E)-C(13E) | 115.7(2) | O(2E)-C(10E)-C(11E) | 108.3(2) |
| O(4E)-C(4E)-C(3E) | 113.0(2) | O(3E)-C(10E)-C(11E) | 110.5(2) |
| C(5E)-C(4E)-C(3E) | 108.8(2) | O(2E)-C(10E)-C(12E) | 111.0(3) |
| C(13E)-C(4E)-C(3E) | 110.3(2) | O(3E)-C(10E)-C(12E) | 108.8(2) |
| O(5E)-C(5E)-O(1E) | 111.7(2) | C(11E)-C(10E)-C(12E) | 112.1(2) |
| O(5E)-C(5E)-C(4E) | 110.3(2) | C(14E)-C(13E)-C(4E) | 117.1(3) |
| O(1E)-C(5E)-C(4E) | 107.0(2) | C(15E)-C(14E)-C(13E) | 176.0(3) |
| O(5E)-C(5E)-C(6E) | 104.6(2) | | |
| C(5F)-O(1F)-C(1F) | 113.22(19) | O(1F)-C(5F)-C(4F) | 107.2(2) |
| O(1F)-C(1F)-C(2F) | 112.6(2) | C(6F)-C(5F)-C(4F) | 115.9(2) |
| O(2F)-C(2F)-C(1F) | 110.7(2) | C(5F)-O(5F)-C(7F) | 107.4(2) |
| O(2F)-C(2F)-C(3F) | 102.0(2) | O(6F)-C(6F)-C(5F) | 105.3(2) |
| C(1F)-C(2F)-C(3F) | 115.9(3) | C(7F)-O(6F)-C(6F) | 105.9(2) |
| C(2F)-O(2F)-C(10F) | 106.1(2) | O(6F)-C(7F)-O(5F) | 103.7(2) |
| O(3F)-C(3F)-C(2F) | 103.5(2) | O(6F)-C(7F)-C(8F) | 108.6(2) |
| O(3F)-C(3F)-C(4F) | 110.1(2) | O(5F)-C(7F)-C(8F) | 108.9(2) |
| C(2F)-C(3F)-C(4F) | 114.0(2) | O(6F)-C(7F)-C(9F) | 111.2(2) |
| C(3F)-O(3F)-C(10F) | 109.0(2) | O(5F)-C(7F)-C(9F) | 110.9(2) |
| O(4F)-C(4F)-C(5F) | 105.9(2) | C(8F)-C(7F)-C(9F) | 113.2(2) |
| O(4F)-C(4F)-C(3F) | 112.1(2) | O(2F)-C(10F)-O(3F) | 105.2(2) |
| C(5F)-C(4F)-C(3F) | 110.3(2) | O(2F)-C(10F)-C(11F) | 107.7(2) |
| O(4F)-C(4F)-C(13F) | 102.65(19) | O(3F)-C(10F)-C(11F) | 110.9(2) |
| C(5F)-C(4F)-C(13F) | 115.6(2) | O(2F)-C(10F)-C(12F) | 111.1(2) |
| C(3F)-C(4F)-C(13F) | 110.1(2) | O(3F)-C(10F)-C(12F) | 108.0(3) |
| O(5F)-C(5F)-O(1F) | 111.50(19) | C(11F)-C(10F)-C(12F) | 113.7(3) |
| O(5F)-C(5F)-C(6F) | 104.7(2) | C(14F)-C(13F)-C(4F) | 117.0(2) |
| O(1F)-C(5F)-C(6F) | 107.2(2) | C(15F)-C(14F)-C(13F) | 175.4(3) |
| O(5F)-C(5F)-C(4F) | 110.5(2) | | |

**Tabelle 5: Torsion angles [°] for *[4]*.**

| | | | |
|---|---|---|---|
| C(5A)-O(1A)-C(1A)-C(2A) | -57.2(3) | O(4A)-C(4A)-C(5A)-C(6A) | -57.3(3) |
| O(1A)-C(1A)-C(2A)-O(2A) | -76.6(3) | C(3A)-C(4A)-C(5A)-C(6A) | -179.7(2) |
| O(1A)-C(1A)-C(2A)-C(3A) | 38.9(3) | C(13A)-C(4A)-C(5A)-C(6A) | 56.3(3) |
| C(1A)-C(2A)-O(2A)-C(10A) | 162.7(2) | O(1A)-C(5A)-O(5A)-C(7A) | -87.0(2) |
| C(3A)-C(2A)-O(2A)-C(10A) | 39.5(2) | C(6A)-C(5A)-O(5A)-C(7A) | 27.4(3) |
| O(2A)-C(2A)-C(3A)-O(3A) | -32.9(2) | C(4A)-C(5A)-O(5A)-C(7A) | 153.1(2) |
| C(1A)-C(2A)-C(3A)-O(3A) | -154.8(2) | O(5A)-C(5A)-C(6A)-O(6A) | -27.2(3) |
| O(2A)-C(2A)-C(3A)-C(4A) | 87.5(2) | O(1A)-C(5A)-C(6A)-O(6A) | 90.6(2) |
| C(1A)-C(2A)-C(3A)-C(4A) | -34.3(3) | C(4A)-C(5A)-C(6A)-O(6A) | -149.3(2) |
| C(2A)-C(3A)-O(3A)-C(10A) | 14.8(3) | C(5A)-C(6A)-O(6A)-C(7A) | 17.1(3) |
| C(4A)-C(3A)-O(3A)-C(10A) | -108.3(2) | C(6A)-O(6A)-C(7A)-O(5A) | -0.7(3) |
| O(3A)-C(3A)-C(4A)-O(4A) | 42.8(3) | C(6A)-O(6A)-C(7A)-C(8A) | -120.1(2) |
| C(2A)-C(3A)-C(4A)-O(4A) | -73.6(3) | C(6A)-O(6A)-C(7A)-C(9A) | 114.4(2) |
| O(3A)-C(3A)-C(4A)-C(5A) | 161.2(2) | C(5A)-O(5A)-C(7A)-O(6A) | -17.6(3) |
| C(2A)-C(3A)-C(4A)-C(5A) | 44.8(3) | C(5A)-O(5A)-C(7A)-C(8A) | 100.8(3) |
| O(3A)-C(3A)-C(4A)-C(13A) | -70.9(3) | C(5A)-O(5A)-C(7A)-C(9A) | -134.8(2) |
| C(2A)-C(3A)-C(4A)-C(13A) | 172.7(2) | C(2A)-O(2A)-C(10A)-O(3A) | -31.2(3) |
| C(1A)-O(1A)-C(5A)-O(5A) | -54.2(3) | C(2A)-O(2A)-C(10A)-C(11A) | -149.3(2) |
| C(1A)-O(1A)-C(5A)-C(6A) | -166.57(19) | C(2A)-O(2A)-C(10A)-C(12A) | 86.0(2) |
| C(1A)-O(1A)-C(5A)-C(4A) | 67.8(3) | C(3A)-O(3A)-C(10A)-O(2A) | 9.1(3) |
| O(4A)-C(4A)-C(5A)-O(5A) | -175.44(19) | C(3A)-O(3A)-C(10A)-C(11A) | 126.3(2) |
| C(3A)-C(4A)-C(5A)-O(5A) | 62.2(3) | C(3A)-O(3A)-C(10A)-C(12A) | -110.4(2) |
| C(13A)-C(4A)-C(5A)-O(5A) | -61.8(3) | O(4A)-C(4A)-C(13A)-C(14A) | -176.3(2) |
| O(4A)-C(4A)-C(5A)-O(1A) | 62.3(2) | C(5A)-C(4A)-C(13A)-C(14A) | 68.2(3) |
| C(3A)-C(4A)-C(5A)-O(1A) | -60.1(3) | C(3A)-C(4A)-C(13A)-C(14A) | -56.3(3) |
| C(13A)-C(4A)-C(5A)-O(1A) | 175.9(2) | C(4A)-C(13A)-C(14A)-C(15A) | 130(8) |
| C(5B)-O(1B)-C(1B)-C(2B) | -54.0(3) | O(4B)-C(4B)-C(5B)-C(6B) | -60.8(3) |
| O(1B)-C(1B)-C(2B)-O(2B) | -79.5(3) | C(3B)-C(4B)-C(5B)-C(6B) | 177.3(2) |
| O(1B)-C(1B)-C(2B)-C(3B) | 35.6(3) | C(13B)-C(4B)-C(5B)-C(6B) | 53.1(3) |
| C(1B)-C(2B)-O(2B)-C(10B) | 163.3(2) | O(1B)-C(5B)-O(5B)-C(7B) | -91.6(2) |
| C(3B)-C(2B)-O(2B)-C(10B) | 40.3(2) | C(6B)-C(5B)-O(5B)-C(7B) | 21.1(2) |
| O(2B)-C(2B)-C(3B)-O(3B) | -33.9(2) | C(4B)-C(5B)-O(5B)-C(7B) | 149.4(2) |
| C(1B)-C(2B)-C(3B)-O(3B) | -154.4(2) | O(5B)-C(5B)-C(6B)-O(6B) | -34.2(2) |
| O(2B)-C(2B)-C(3B)-C(4B) | 86.1(3) | O(1B)-C(5B)-C(6B)-O(6B) | 82.4(2) |
| C(1B)-C(2B)-C(3B)-C(4B) | -34.5(3) | C(4B)-C(5B)-C(6B)-O(6B) | -156.9(2) |
| C(2B)-C(3B)-O(3B)-C(10B) | 15.4(2) | C(5B)-C(6B)-O(6B)-C(7B) | 34.9(3) |
| C(4B)-C(3B)-O(3B)-C(10B) | -108.0(2) | C(6B)-O(6B)-C(7B)-O(5B) | -22.4(2) |
| O(3B)-C(3B)-C(4B)-O(4B) | 46.1(3) | C(6B)-O(6B)-C(7B)-C(8B) | 94.3(2) |
| C(2B)-C(3B)-C(4B)-O(4B) | -70.2(3) | C(6B)-O(6B)-C(7B)-C(9B) | -139.9(2) |
| O(3B)-C(3B)-C(4B)-C(5B) | 163.8(2) | C(5B)-O(5B)-C(7B)-O(6B) | -0.3(3) |
| C(2B)-C(3B)-C(4B)-C(5B) | 47.6(3) | C(5B)-O(5B)-C(7B)-C(8B) | -118.3(2) |
| O(3B)-C(3B)-C(4B)-C(13B) | -68.5(3) | C(5B)-O(5B)-C(7B)-C(9B) | 116.6(2) |
| C(2B)-C(3B)-C(4B)-C(13B) | 175.2(2) | C(2B)-O(2B)-C(10B)-O(3B) | -31.5(2) |
| C(1B)-O(1B)-C(5B)-O(5B) | -52.2(3) | C(2B)-O(2B)-C(10B)-C(12B) | -149.0(2) |
| C(1B)-O(1B)-C(5B)-C(6B) | -163.3(2) | C(2B)-O(2B)-C(10B)-C(11B) | 85.4(3) |
| C(1B)-O(1B)-C(5B)-C(4B) | 68.6(2) | C(3B)-O(3B)-C(10B)-O(2B) | 9.0(2) |

| | | | |
|---|---|---|---|
| O(4B)-C(4B)-C(5B)-O(5B) | -179.88(18) | C(3B)-O(3B)-C(10B)-C(12B) | 126.0(2) |
| C(3B)-C(4B)-C(5B)-O(5B) | 58.3(3) | C(3B)-O(3B)-C(10B)-C(11B) | -110.5(2) |
| C(13B)-C(4B)-C(5B)-O(5B) | -66.0(3) | O(4B)-C(4B)-C(13B)-C(14B) | -174.6(2) |
| O(4B)-C(4B)-C(5B)-O(1B) | 59.0(2) | C(5B)-C(4B)-C(13B)-C(14B) | 69.3(3) |
| C(3B)-C(4B)-C(5B)-O(1B) | -62.8(3) | C(3B)-C(4B)-C(13B)-C(14B) | -53.7(3) |
| C(13B)-C(4B)-C(5B)-O(1B) | 172.97(19) | C(4B)-C(13B)-C(14B)-C(15B) | 137(8) |
| C(5C)-O(1C)-C(1C)-C(2C) | -55.9(3) | O(4C)-C(4C)-C(5C)-C(6C) | -60.8(3) |
| O(1C)-C(1C)-C(2C)-O(2C) | -77.4(3) | C(3C)-C(4C)-C(5C)-C(6C) | 177.5(2) |
| O(1C)-C(1C)-C(2C)-C(3C) | 37.2(3) | C(13C)-C(4C)-C(5C)-C(6C) | 52.8(3) |
| C(1C)-C(2C)-O(2C)-C(10C) | 163.40(19) | O(1C)-C(5C)-O(5C)-C(7C) | -87.8(2) |
| C(3C)-C(2C)-O(2C)-C(10C) | 40.3(2) | C(6C)-C(5C)-O(5C)-C(7C) | 25.8(2) |
| O(2C)-C(2C)-C(3C)-O(3C) | -34.1(2) | C(4C)-C(5C)-O(5C)-C(7C) | 153.1(2) |
| C(1C)-C(2C)-C(3C)-O(3C) | -154.2(2) | O(5C)-C(5C)-C(6C)-O(6C) | -33.0(2) |
| O(2C)-C(2C)-C(3C)-C(4C) | 86.7(3) | O(1C)-C(5C)-C(6C)-O(6C) | 83.6(2) |
| C(1C)-C(2C)-C(3C)-C(4C) | -33.5(3) | C(4C)-C(5C)-C(6C)-O(6C) | -156.2(2) |
| C(2C)-C(3C)-O(3C)-C(10C) | 15.9(2) | C(5C)-C(6C)-O(6C)-C(7C) | 28.3(3) |
| C(4C)-C(3C)-O(3C)-C(10C) | -106.7(2) | C(6C)-O(6C)-C(7C)-O(5C) | -13.1(3) |
| O(3C)-C(3C)-C(4C)-O(4C) | 43.9(3) | C(6C)-O(6C)-C(7C)-C(8C) | 104.5(2) |
| C(2C)-C(3C)-C(4C)-O(4C) | -72.2(3) | C(6C)-O(6C)-C(7C)-C(9C) | -131.4(2) |
| O(3C)-C(3C)-C(4C)-C(5C) | 162.1(2) | C(5C)-O(5C)-C(7C)-O(6C) | -8.9(2) |
| C(2C)-C(3C)-C(4C)-C(5C) | 46.0(3) | C(5C)-O(5C)-C(7C)-C(8C) | -128.6(2) |
| O(3C)-C(3C)-C(4C)-C(13C) | -69.9(3) | C(5C)-O(5C)-C(7C)-C(9C) | 106.7(2) |
| C(2C)-C(3C)-C(4C)-C(13C) | 173.9(2) | C(2C)-O(2C)-C(10C)-O(3C) | -31.4(2) |
| C(1C)-O(1C)-C(5C)-O(5C) | -52.6(3) | C(2C)-O(2C)-C(10C)-C(11C) | -149.4(2) |
| C(1C)-O(1C)-C(5C)-C(6C) | -164.3(2) | C(2C)-O(2C)-C(10C)-C(12C) | 86.5(3) |
| C(1C)-O(1C)-C(5C)-C(4C) | 68.9(2) | C(3C)-O(3C)-C(10C)-O(2C) | 8.7(2) |
| O(4C)-C(4C)-C(5C)-O(5C) | -179.87(18) | C(3C)-O(3C)-C(10C)-C(11C) | 125.7(2) |
| C(3C)-C(4C)-C(5C)-O(5C) | 58.4(3) | C(3C)-O(3C)-C(10C)-C(12C) | -110.6(2) |
| C(13C)-C(4C)-C(5C)-O(5C) | -66.3(3) | O(4C)-C(4C)-C(13C)-C(14C) | 176.7(2) |
| O(4C)-C(4C)-C(5C)-O(1C) | 59.0(2) | C(5C)-C(4C)-C(13C)-C(14C) | 61.0(3) |
| C(3C)-C(4C)-C(5C)-O(1C) | -62.7(3) | C(3C)-C(4C)-C(13C)-C(14C) | -63.4(3) |
| C(13C)-C(4C)-C(5C)-O(1C) | 172.58(19) | C(4C)-C(13C)-C(14C)-C(15C) | 150(8) |
| C(5D)-O(1D)-C(1D)-C(2D) | -55.6(3) | O(4D)-C(4D)-C(5D)-C(6D) | -58.8(3) |
| O(1D)-C(1D)-C(2D)-O(2D) | -77.6(3) | C(13D)-C(4D)-C(5D)-C(6D) | 54.1(3) |
| O(1D)-C(1D)-C(2D)-C(3D) | 37.9(3) | C(3D)-C(4D)-C(5D)-C(6D) | 178.5(2) |
| C(1D)-C(2D)-O(2D)-C(10D) | 164.4(2) | O(1D)-C(5D)-O(5D)-C(7D) | -93.1(3) |
| C(3D)-C(2D)-O(2D)-C(10D) | 40.6(2) | C(4D)-C(5D)-O(5D)-C(7D) | 148.4(2) |
| O(2D)-C(2D)-C(3D)-O(3D) | -34.5(2) | C(6D)-C(5D)-O(5D)-C(7D) | 21.5(3) |
| C(1D)-C(2D)-C(3D)-O(3D) | -155.7(2) | O(5D)-C(5D)-C(6D)-O(6D) | -9.3(3) |
| O(2D)-C(2D)-C(3D)-C(4D) | 85.3(2) | O(1D)-C(5D)-C(6D)-O(6D) | 108.6(3) |
| C(1D)-C(2D)-C(3D)-C(4D) | -35.8(3) | C(4D)-C(5D)-C(6D)-O(6D) | -132.3(3) |
| C(2D)-C(3D)-O(3D)-C(10D) | 16.4(3) | C(5D)-C(6D)-O(6D)-C(7D) | -7.0(4) |
| C(4D)-C(3D)-O(3D)-C(10D) | -105.2(2) | C(6D)-O(6D)-C(7D)-O(5D) | 20.1(3) |
| O(3D)-C(3D)-C(4D)-O(4D) | 44.1(3) | C(6D)-O(6D)-C(7D)-C(8D) | 135.6(3) |
| C(2D)-C(3D)-C(4D)-O(4D) | -71.2(3) | C(6D)-O(6D)-C(7D)-C(9D) | -100.0(3) |
| O(3D)-C(3D)-C(4D)-C(5D) | 163.2(2) | C(5D)-O(5D)-C(7D)-O(6D) | -26.0(3) |
| C(2D)-C(3D)-C(4D)-C(5D) | 47.9(3) | C(5D)-O(5D)-C(7D)-C(8D) | -142.3(3) |
| O(3D)-C(3D)-C(4D)-C(13D) | -69.2(3) | C(5D)-O(5D)-C(7D)-C(9D) | 93.5(3) |
| C(2D)-C(3D)-C(4D)-C(13D) | 175.5(2) | C(2D)-O(2D)-C(10D)-O(3D) | -31.1(3) |

| | | | |
|---|---|---|---|
| C(1D)-O(1D)-C(5D)-O(5D) | -52.8(3) | C(2D)-O(2D)-C(10D)-C(12D) | -148.5(2) |
| C(1D)-O(1D)-C(5D)-C(4D) | 68.3(3) | C(2D)-O(2D)-C(10D)-C(11D) | 86.8(2) |
| C(1D)-O(1D)-C(5D)-C(6D) | -166.1(2) | C(3D)-O(3D)-C(10D)-O(2D) | 8.2(3) |
| O(4D)-C(4D)-C(5D)-O(5D) | -178.5(2) | C(3D)-O(3D)-C(10D)-C(12D) | 124.8(2) |
| C(13D)-C(4D)-C(5D)-O(5D) | -65.5(3) | C(3D)-O(3D)-C(10D)-C(11D) | -111.3(2) |
| C(3D)-C(4D)-C(5D)-O(5D) | 58.8(3) | O(4D)-C(4D)-C(13D)-C(14D) | -175.7(2) |
| O(4D)-C(4D)-C(5D)-O(1D) | 60.2(3) | C(5D)-C(4D)-C(13D)-C(14D) | 68.5(3) |
| C(13D)-C(4D)-C(5D)-O(1D) | 173.1(2) | C(3D)-C(4D)-C(13D)-C(14D) | -55.5(3) |
| C(3D)-C(4D)-C(5D)-O(1D) | -62.5(3) | C(4D)-C(13D)-C(14D)-C(15D) | 149(5) |
| C(5E)-O(1E)-C(1E)-C(2E) | -54.7(3) | O(4E)-C(4E)-C(5E)-C(6E) | -60.3(3) |
| O(1E)-C(1E)-C(2E)-O(2E) | -79.6(3) | C(13E)-C(4E)-C(5E)-C(6E) | 52.6(3) |
| O(1E)-C(1E)-C(2E)-C(3E) | 36.2(3) | C(3E)-C(4E)-C(5E)-C(6E) | 177.4(2) |
| C(1E)-C(2E)-O(2E)-C(10E) | 163.3(2) | O(1E)-C(5E)-O(5E)-C(7E) | -91.4(2) |
| C(3E)-C(2E)-O(2E)-C(10E) | 38.8(2) | C(4E)-C(5E)-O(5E)-C(7E) | 149.8(2) |
| O(2E)-C(2E)-C(3E)-O(3E) | -32.6(2) | C(6E)-C(5E)-O(5E)-C(7E) | 23.1(3) |
| C(1E)-C(2E)-C(3E)-O(3E) | -153.1(2) | O(5E)-C(5E)-C(6E)-O(6E) | -4.5(3) |
| O(2E)-C(2E)-C(3E)-C(4E) | 87.0(2) | O(1E)-C(5E)-C(6E)-O(6E) | 113.7(2) |
| C(1E)-C(2E)-C(3E)-C(4E) | -33.6(3) | C(4E)-C(5E)-C(6E)-O(6E) | -126.9(2) |
| C(2E)-C(3E)-O(3E)-C(10E) | 14.6(3) | C(5E)-C(6E)-O(6E)-C(7E) | -16.0(3) |
| C(4E)-C(3E)-O(3E)-C(10E) | -106.9(2) | C(6E)-O(6E)-C(7E)-O(5E) | 29.9(3) |
| O(3E)-C(3E)-C(4E)-O(4E) | 43.6(3) | C(6E)-O(6E)-C(7E)-C(8E) | 144.0(2) |
| C(2E)-C(3E)-C(4E)-O(4E) | -71.9(3) | C(6E)-O(6E)-C(7E)-C(9E) | -89.9(3) |
| O(3E)-C(3E)-C(4E)-C(5E) | 162.3(2) | C(5E)-O(5E)-C(7E)-O(6E) | -33.2(3) |
| C(2E)-C(3E)-C(4E)-C(5E) | 46.8(3) | C(5E)-O(5E)-C(7E)-C(8E) | -147.9(2) |
| O(3E)-C(3E)-C(4E)-C(13E) | -69.8(3) | C(5E)-O(5E)-C(7E)-C(9E) | 87.3(3) |
| C(2E)-C(3E)-C(4E)-C(13E) | 174.7(2) | C(2E)-O(2E)-C(10E)-O(3E) | -30.6(3) |
| C(1E)-O(1E)-C(5E)-O(5E) | -51.5(3) | C(2E)-O(2E)-C(10E)-C(11E) | -149.2(2) |
| C(1E)-O(1E)-C(5E)-C(4E) | 69.3(3) | C(2E)-O(2E)-C(10E)-C(12E) | 87.3(2) |
| C(1E)-O(1E)-C(5E)-C(6E) | -165.0(2) | C(3E)-O(3E)-C(10E)-O(2E) | 9.1(3) |
| O(4E)-C(4E)-C(5E)-O(5E) | -179.69(19) | C(3E)-O(3E)-C(10E)-C(11E) | 126.3(2) |
| C(13E)-C(4E)-C(5E)-O(5E) | -66.8(3) | C(3E)-O(3E)-C(10E)-C(12E) | -110.3(2) |
| C(3E)-C(4E)-C(5E)-O(5E) | 58.0(3) | O(4E)-C(4E)-C(13E)-C(14E) | -175.3(2) |
| O(4E)-C(4E)-C(5E)-O(1E) | 58.7(3) | C(5E)-C(4E)-C(13E)-C(14E) | 68.9(3) |
| C(13E)-C(4E)-C(5E)-O(1E) | 171.5(2) | C(3E)-C(4E)-C(13E)-C(14E) | -55.0(3) |
| C(3E)-C(4E)-C(5E)-O(1E) | -63.7(3) | C(4E)-C(13E)-C(14E)-C(15E) | 170(4) |
| C(5F)-O(1F)-C(1F)-C(2F) | -56.9(3) | O(4F)-C(4F)-C(5F)-C(6F) | -58.2(3) |
| O(1F)-C(1F)-C(2F)-O(2F) | -78.1(3) | C(3F)-C(4F)-C(5F)-C(6F) | -179.7(2) |
| O(1F)-C(1F)-C(2F)-C(3F) | 37.4(3) | C(13F)-C(4F)-C(5F)-C(6F) | 54.7(3) |
| C(1F)-C(2F)-O(2F)-C(10F) | 162.1(2) | O(1F)-C(5F)-O(5F)-C(7F) | -96.3(2) |
| C(3F)-C(2F)-O(2F)-C(10F) | 38.1(3) | C(6F)-C(5F)-O(5F)-C(7F) | 19.2(3) |
| O(2F)-C(2F)-C(3F)-O(3F) | -31.9(3) | C(4F)-C(5F)-O(5F)-C(7F) | 144.6(2) |
| C(1F)-C(2F)-C(3F)-O(3F) | -152.3(2) | O(5F)-C(5F)-C(6F)-O(6F) | 2.8(3) |
| O(2F)-C(2F)-C(3F)-C(4F) | 87.7(3) | O(1F)-C(5F)-C(6F)-O(6F) | 121.3(2) |
| C(1F)-C(2F)-C(3F)-C(4F) | -32.6(3) | C(4F)-C(5F)-C(6F)-O(6F) | -119.1(2) |
| C(2F)-C(3F)-O(3F)-C(10F) | 14.5(3) | C(5F)-C(6F)-O(6F)-C(7F) | -23.9(3) |
| C(4F)-C(3F)-O(3F)-C(10F) | -107.7(2) | C(6F)-O(6F)-C(7F)-O(5F) | 35.7(2) |
| O(3F)-C(3F)-C(4F)-O(4F) | 41.7(3) | C(6F)-O(6F)-C(7F)-C(8F) | 151.4(2) |
| C(2F)-C(3F)-C(4F)-O(4F) | -74.1(3) | C(6F)-O(6F)-C(7F)-C(9F) | -83.5(3) |
| O(3F)-C(3F)-C(4F)-C(5F) | 159.5(2) | C(5F)-O(5F)-C(7F)-O(6F) | -34.4(2) |

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|---|---|---|---|
| C(2F)-C(3F)-C(4F)-C(5F) | 43.6(3) | C(5F)-O(5F)-C(7F)-C(8F) | -149.9(2) |
| O(3F)-C(3F)-C(4F)-C(13F) | -71.8(3) | C(5F)-O(5F)-C(7F)-C(9F) | 85.0(2) |
| C(2F)-C(3F)-C(4F)-C(13F) | 172.4(2) | C(2F)-O(2F)-C(10F)-O(3F) | -29.9(3) |
| C(1F)-O(1F)-C(5F)-O(5F) | -52.3(3) | C(2F)-O(2F)-C(10F)-C(11F) | -148.2(2) |
| C(1F)-O(1F)-C(5F)-C(6F) | -166.3(2) | C(2F)-O(2F)-C(10F)-C(12F) | 86.7(3) |
| C(1F)-O(1F)-C(5F)-C(4F) | 68.7(2) | C(3F)-O(3F)-C(10F)-O(2F) | 8.5(3) |
| O(4F)-C(4F)-C(5F)-O(5F) | -177.00(19) | C(3F)-O(3F)-C(10F)-C(11F) | 124.7(3) |
| C(3F)-C(4F)-C(5F)-O(5F) | 61.5(3) | C(3F)-O(3F)-C(10F)-C(12F) | -110.2(3) |
| C(13F)-C(4F)-C(5F)-O(5F) | -64.1(3) | O(4F)-C(4F)-C(13F)-C(14F) | -175.8(2) |
| O(4F)-C(4F)-C(5F)-O(1F) | 61.3(3) | C(5F)-C(4F)-C(13F)-C(14F) | 69.4(3) |
| C(3F)-C(4F)-C(5F)-O(1F) | -60.1(3) | C(3F)-C(4F)-C(13F)-C(14F) | -56.3(3) |
| C(13F)-C(4F)-C(5F)-O(1F) | 174.2(2) | C(4F)-C(13F)-C(14F)-C(15F) | 175(100) |

**Tabelle 6: Hydrogen bonds for *[4]* [Å and °].**

| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| O(4A)-H(4A)...O(1C) | 0.84 | 2.15 | 2.904(2) | 149.9 |
| O(4B)-H(4B)...O(1E)[a] | 0.84 | 2.27 | 3.008(2) | 147.1 |
| O(4C)-H(4C)...O(1A) | 0.84 | 2.16 | 2.930(2) | 152.9 |
| O(4D)-H(4D)...O(1F)[b] | 0.84 | 2.14 | 2.871(2) | 144.9 |
| O(4E)-H(4E)...O(1B)[c] | 0.84 | 2.09 | 2.832(2) | 147.8 |
| O(4F)-H(4F)...O(1D)[d] | 0.84 | 2.15 | 2.896(2) | 148.4 |
| C(2C)-H(2C)...O(6A)[e] | 1.00 | 2.52 | 3.245(3) | 129.5 |
| C(3C)-H(3C)...O(6A)[e] | 1.00 | 2.50 | 3.188(3) | 125.8 |
| C(1A)-H(1A1)...O(2C) | 0.99 | 2.45 | 3.254(3) | 138.0 |
| C(2A)-H(2A)...O(6B)[f] | 1.00 | 2.51 | 3.307(3) | 136.0 |
| C(1C)-H(1C1)...O(2A) | 0.99 | 2.47 | 3.325(3) | 144.9 |
| C(11F)-H(11R)...O(3A)[d] | 0.98 | 2.53 | 3.476(3) | 162.8 |
| C(9B)-H(9B3)...O(5A)[g] | 0.98 | 2.38 | 3.258(3) | 149.0 |
| C(6A)-H(6A2)...O(4C) | 0.99 | 2.50 | 3.343(3) | 143.4 |
| C(2B)-H(2B)...O(4F)[h] | 1.00 | 2.53 | 3.493(3) | 160.6 |

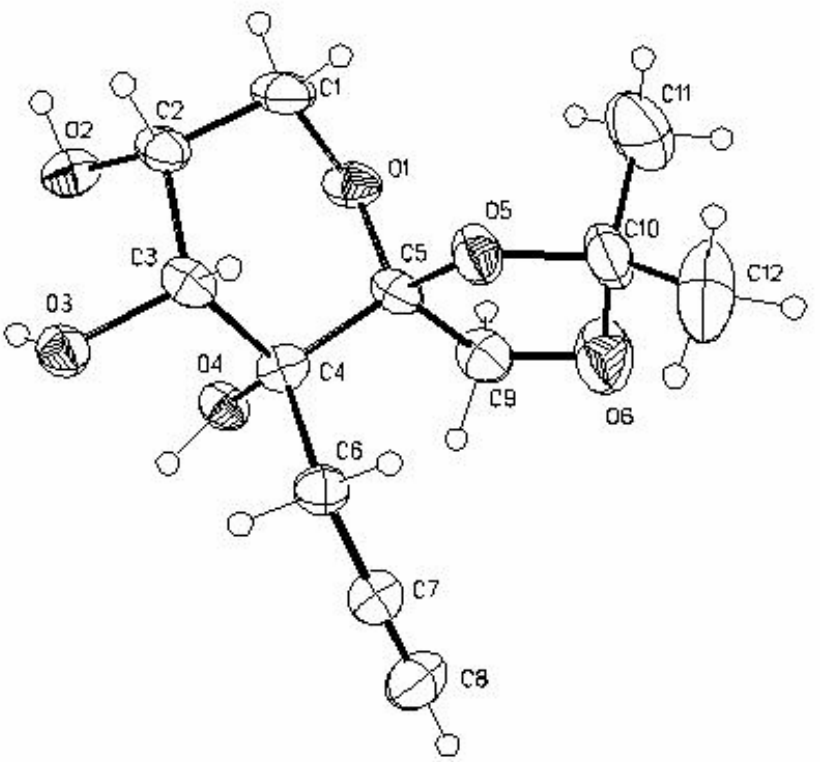
Symmetry transformations used to generate equivalent atoms:

[a] x,y-1,z [b] -x+2,y-1/2,-z+3/2 [c] x,y+1,z

[d] -x+2,y+1/2,-z+3/2 [e] x+1/2,-y+3/2,-z+1 [f] x+1/2,-y+1/2,-z+1

[g] x-1/2,-y+1/2,-z+1 [h] -x+1,y-1/2,-z+3/2

**2. (*5S,8R,9R,10R*)-2,2-Dimethyl-10-prop-2-inyl-1,3,6-trioxa-spiro[4.5]decan-8,9,10-triol** (1,2-*O*-Isopropyliden-3-(2-propinyl)-α-D-psicopyranose) ***[6]***



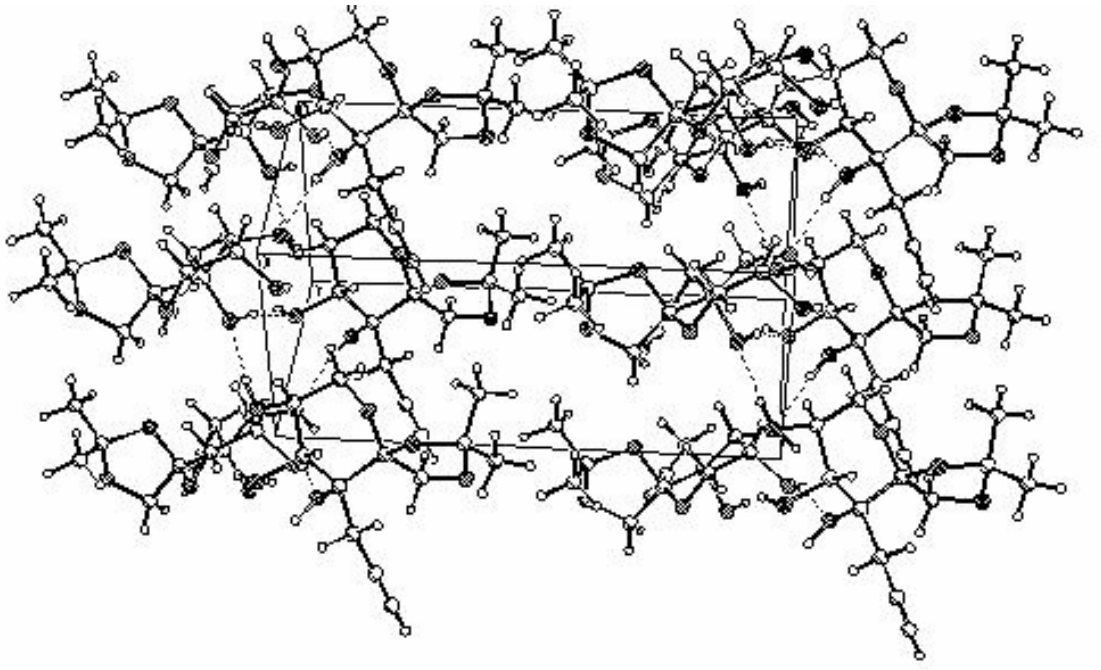



**Table 1: Crystal data and structure refinements for *[6]***

| | |
|---|---|
| Identification code | ***[6]*** |
| Empirical formula | $C_{12}H_{18}O_6$ |
| Formula weight | 258.26 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Monoclinic |
| Space group | P2(1) (No.4) |
| Unit cell dimensions | a = 6.7184(6) Å α = 90° |
| | b = 6.6463(6) Å β = 91.007(4)° |
| | c = 13.9978(15) Å γ = 90° |
| Volume | 624.94(10) $Å^3$ |
| Z | 2 |
| Calculated density | 1.372 $mg/m^3$ |
| Absorption coefficient | 0.110 $mm^{-1}$ |
| F(000) | 276 |
| Crystal size | 0.40 x 0.20 x 0.10 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.91 to 24.99° |
| Limiting indices | $-7 \le h \le 7, -7 \le k \le 7, -16 \le l \le 16$ |
| Reflections collected / unique | 5533 / 2060 [R(int) = 0.0749] |
| Completeness to Θ = 24.99 | 97.4 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 2060 / 4 / 172 |
| Goodness-of-fit on $F^2$ | 0.855 |
| Final R indices [I>2σ(I)] | R1 = 0.0429, wR2 = 0.0660 |
| R indices (all data) | R1 = 0.0998, wR2 = 0.0750 |
| Absolute structure parameter | -1.2(15), cannot be determined reliabl. |
| Largest diff. peak and hole | 0.187 and -0.174 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[6]*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| O(1) | 65(3) | 3200(3) | 1884(2) | 33(1) |
| C(1) | 240(5) | 1291(4) | 1395(2) | 38(1) |
| C(2) | 1700(5) | 1379(4) | 590(2) | 30(1) |
| O(2) | 913(3) | 2511(3) | -209(2) | 33(1) |
| C(3) | 3627(5) | 2424(5) | 890(2) | 27(1) |
| O(3) | 4699(3) | 2943(3) | 51(2) | 30(1) |
| C(4) | 3387(5) | 4282(5) | 1518(2) | 25(1) |
| O(4) | 2548(3) | 5910(3) | 981(2) | 28(1) |
| C(5) | 1895(5) | 3890(5) | 2304(2) | 28(1) |
| O(5) | 2715(3) | 2415(3) | 2932(2) | 35(1) |
| C(6) | 5475(5) | 4854(4) | 1910(2) | 30(1) |
| C(7) | 5709(5) | 6905(5) | 2298(2) | 30(1) |
| C(8) | 5933(6) | 8537(6) | 2603(3) | 43(1) |
| C(9) | 1365(5) | 5638(5) | 2962(2) | 37(1) |
| O(6) | 1985(4) | 4999(3) | 3893(2) | 59(1) |
| C(10) | 2065(6) | 2879(5) | 3888(3) | 47(1) |
| C(11) | 50(7) | 1937(6) | 4074(3) | 77(2) |
| C(12) | 3660(7) | 2201(7) | 4574(3) | 74(1) |

**Tabelle 3: Bond lengths [Å] for *[6]*.**

| | | | |
|---|---|---|---|
| O(1)-C(5) | 1.429(4) | C(4)-C(6) | 1.545(5) |
| O(1)-C(1) | 1.448(3) | O(4)-H(4O) | 0.888(17) |
| C(1)-C(2) | 1.508(4) | C(5)-O(5) | 1.421(4) |
| C(2)-O(2) | 1.441(4) | C(5)-C(9) | 1.529(4) |
| C(2)-C(3) | 1.522(4) | O(5)-C(10) | 1.447(4) |
| O(2)-H(2O) | 0.832(18) | C(6)-C(7) | 1.475(5) |
| C(3)-O(3) | 1.431(3) | C(7)-C(8) | 1.174(4) |
| C(3)-C(4) | 1.526(4) | C(9)-O(6) | 1.426(4) |
| O(3)-H(3O) | 0.802(18) | O(6)-C(10) | 1.410(4) |
| C(4)-O(4) | 1.428(4) | C(10)-C(12) | 1.496(5) |
| C(4)-C(5) | 1.523(4) | C(10)-C(11) | 1.519(5) |

**Tabelle 4: Bond angles [°] for *[6]*.**

| | | | |
|---|---|---|---|
| C(5)-O(1)-C(1) | 113.5(2) | O(5)-C(5)-O(1) | 110.7(3) |
| O(1)-C(1)-C(2) | 112.3(2) | O(5)-C(5)-C(4) | 108.2(3) |
| O(2)-C(2)-C(1) | 111.4(3) | O(1)-C(5)-C(4) | 109.3(3) |
| O(2)-C(2)-C(3) | 105.9(2) | O(5)-C(5)-C(9) | 104.1(3) |
| C(1)-C(2)-C(3) | 111.9(3) | O(1)-C(5)-C(9) | 106.5(3) |
| C(2)-O(2)-H(2O) | 105(2) | C(4)-C(5)-C(9) | 117.9(3) |
| O(3)-C(3)-C(2) | 108.8(3) | C(5)-O(5)-C(10) | 107.8(3) |
| O(3)-C(3)-C(4) | 109.8(3) | C(7)-C(6)-C(4) | 116.6(3) |
| C(2)-C(3)-C(4) | 115.5(2) | C(8)-C(7)-C(6) | 178.7(4) |
| C(3)-O(3)-H(3O) | 111(2) | O(6)-C(9)-C(5) | 104.9(3) |
| O(4)-C(4)-C(5) | 104.6(3) | C(10)-O(6)-C(9) | 107.7(3) |
| O(4)-C(4)-C(3) | 110.7(3) | O(6)-C(10)-O(5) | 103.3(2) |
| C(5)-C(4)-C(3) | 110.8(2) | O(6)-C(10)-C(12) | 108.9(4) |
| O(4)-C(4)-C(6) | 110.3(2) | O(5)-C(10)-C(12) | 107.8(3) |
| C(5)-C(4)-C(6) | 113.0(3) | O(6)-C(10)-C(11) | 112.1(3) |
| C(3)-C(4)-C(6) | 107.4(3) | O(5)-C(10)-C(11) | 110.8(3) |
| C(4)-O(4)-H(4O) | 106.0(19) | C(12)-C(10)-C(11) | 113.3(3) |

**Tabelle 5: Torsion angles [°] for *[6]*.**

| | | | |
|---|---|---|---|
| C(5)-O(1)-C(1)-C(2) | -60.1(4) | C(6)-C(4)-C(5)-O(1) | -174.9(3) |
| O(1)-C(1)-C(2)-O(2) | -72.4(4) | O(4)-C(4)-C(5)-C(9) | -56.6(4) |
| O(1)-C(1)-C(2)-C(3) | 46.0(4) | C(3)-C(4)-C(5)-C(9) | -176.0(3) |
| O(2)-C(2)-C(3)-O(3) | -42.8(3) | C(6)-C(4)-C(5)-C(9) | 63.4(4) |
| C(1)-C(2)-C(3)-O(3) | -164.4(2) | O(1)-C(5)-O(5)-C(10) | -94.4(3) |
| O(2)-C(2)-C(3)-C(4) | 81.1(3) | C(4)-C(5)-O(5)-C(10) | 145.8(3) |
| C(1)-C(2)-C(3)-C(4) | -40.5(4) | C(9)-C(5)-O(5)-C(10) | 19.6(3) |
| O(3)-C(3)-C(4)-O(4) | 52.5(3) | O(4)-C(4)-C(6)-C(7) | 41.8(4) |
| C(2)-C(3)-C(4)-O(4) | -70.8(3) | C(5)-C(4)-C(6)-C(7) | -74.8(3) |
| O(3)-C(3)-C(4)-C(5) | 168.1(3) | C(3)-C(4)-C(6)-C(7) | 162.7(3) |
| C(2)-C(3)-C(4)-C(5) | 44.8(4) | C(4)-C(6)-C(7)-C(8) | -154(17) |
| O(3)-C(3)-C(4)-C(6) | -68.0(3) | O(5)-C(5)-C(9)-O(6) | 1.5(4) |
| C(2)-C(3)-C(4)-C(6) | 168.6(3) | O(1)-C(5)-C(9)-O(6) | 118.6(3) |
| C(1)-O(1)-C(5)-O(5) | -55.4(3) | C(4)-C(5)-C(9)-O(6) | -118.3(3) |
| C(1)-O(1)-C(5)-C(4) | 63.7(3) | C(5)-C(9)-O(6)-C(10) | -22.7(4) |
| C(1)-O(1)-C(5)-C(9) | -167.9(2) | C(9)-O(6)-C(10)-O(5) | 34.7(4) |
| O(4)-C(4)-C(5)-O(5) | -174.3(3) | C(9)-O(6)-C(10)-C(12) | 149.1(3) |
| C(3)-C(4)-C(5)-O(5) | 66.4(4) | C(9)-O(6)-C(10)-C(11) | -84.7(4) |
| C(6)-C(4)-C(5)-O(5) | -54.3(3) | C(5)-O(5)-C(10)-O(6) | -33.8(4) |
| O(4)-C(4)-C(5)-O(1) | 65.1(3) | C(5)-O(5)-C(10)-C(12) | -149.0(3) |
| C(3)-C(4)-C(5)-O(1) | -54.3(3) | C(5)-O(5)-C(10)-C(11) | 86.5(4) |

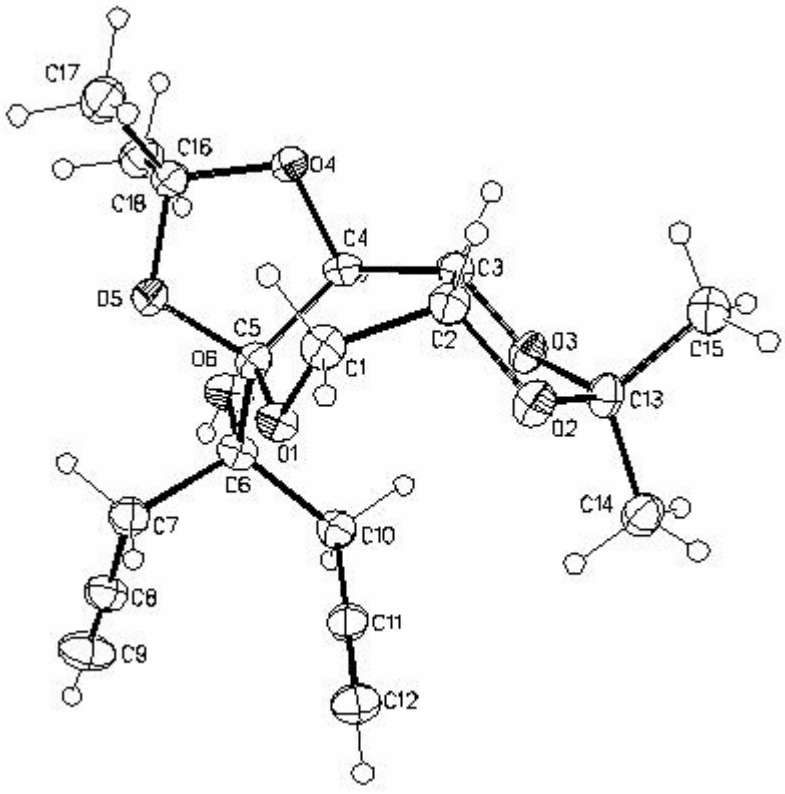
**Tabelle 6: Hydrogen bonds for *[6]* [Å and °].**

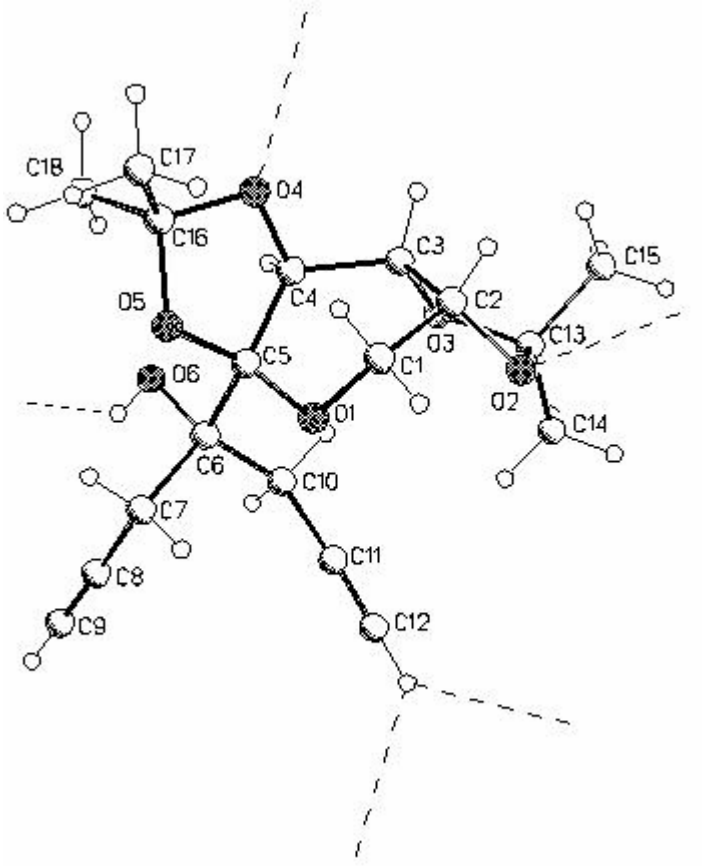
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| O(2)-H(2O).....O(4)[a] | 0.832(18) | 1.98(2) | 2.761(3) | 156(3) |
| C(1)-H(1A).....O(2)[a] | 0.99 | 2.60 | 3.102(4) | 111.6 |
| C(2)-H(2).....O(2)[a] | 1.00 | 2.57 | 3.153(4) | 117.4 |
| O(3)-H(3O).....O(2) | 0.802(18) | 2.13(3) | 2.579(3) | 116(3) |
| O(4)-H(4O).....O(3)[b] | 0.888(17) | 1.863(19) | 2.725(3) | 163(3) |
| C(2)-H(2).....O(3)[c] | 1.00 | 2.67 | 3.456(4) | 135.6 |

Symmetry transformations used to generate equivalent atoms:

[a] -x,y-1/2,-z [b] -x+1,y+1/2,-z [c] -x+1,y-1/2,-z

3. **(*1´R,2´S,6´S,9´R*)-4-(4´,4´,11´,11´-Tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´-yl)-hepta-1,6-diin-4-ol** (2,3:4,5-Di-*O*-isopropyliden-1,1-di(prop-2-inyl)-α-D-fructopyranose) ***[7]***




C18
C17
O4
C16
C3
C15
O5
C4
C2
O3
C13
C5
C1
O6
O1
O2
C14
C6
C10
C7
C11
C8
C9
C12

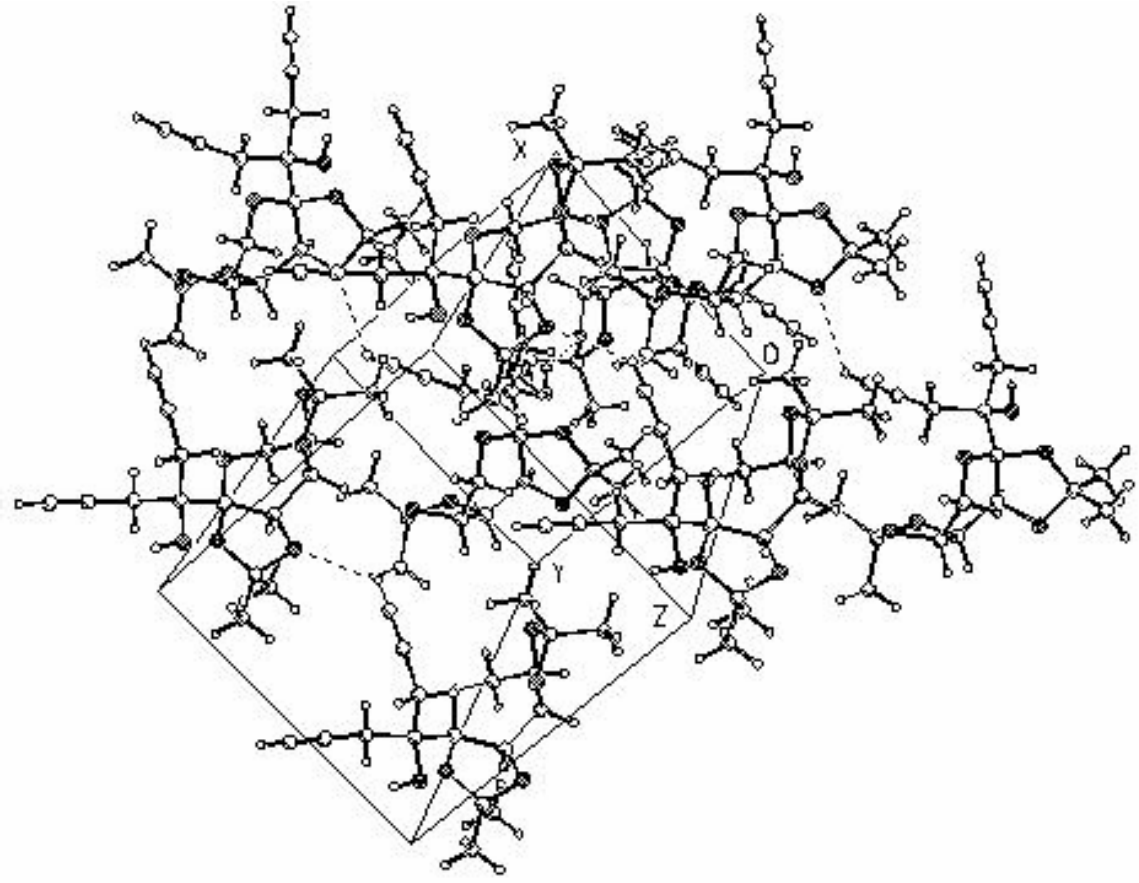

X
O
Y
Z

**Table 1: Crystal data and structure refinements for *[7]***

| | |
|---|---|
| Identification code | ***[7]*** |
| Empirical formula | $C_{18}H_{24}O_6$ |
| Formula weight | 336.37 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 9.3412(1) Å α = 90° |
| | b = 9.9768(2) Å β = 90° |
| | c = 19.3810(4) Å γ = 90° |
| Volume | 1806.22(6) $Å^3$ |
| Z | 4 |
| Calculated density | 1. 237mg/$m^3$ |
| Absorption coefficient | 0.092 $mm^{-1}$ |
| F(000) | 720 |
| Crystal size | 0.60 x 0.35 x 0.30 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.99 to 25.00° |
| Limiting indices | $-11 \le h \le 11$, $-11 \le k \le 11$, $-23 \le l \le 23$ |
| Reflections collected / unique | 15790 / 3159 [R(int) = 0.0533] |
| Completeness to Θ = 24.99 | 99.8 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 3159 / 0 / 220 |
| Goodness-of-fit on $F^2$ | 1.009 |
| Final R indices [I>2σ(I)] | R1 = 0.0287, wR2 = 0.0616 |
| R indices (all data) | R1 = 0.0344, wR2 = 0.0634 |
| Absolute structure parameter | -0.4(7), cannot be determined reliabl. |
| Largest diff. peak and hole | 0.159 and -0.165 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[7]*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| C(1) | 5006(2) | 5526(1) | 4961(1) | 21(1) |
| O(1) | 5773(1) | 4890(1) | 4413(1) | 19(1) |
| C(2) | 4424(2) | 6867(1) | 4741(1) | 21(1) |
| O(2) | 5584(1) | 7809(1) | 4684(1) | 23(1) |
| C(3) | 3775(2) | 6832(1) | 4007(1) | 19(1) |
| O(3) | 4705(1) | 7655(1) | 3605(1) | 21(1) |
| C(4) | 3681(1) | 5444(1) | 3689(1) | 19(1) |
| O(4) | 2440(1) | 4859(1) | 4000(1) | 24(1) |
| C(5) | 4902(2) | 4451(1) | 3867(1) | 19(1) |
| O(5) | 4153(1) | 3267(1) | 4069(1) | 21(1) |
| C(6) | 5949(2) | 4106(1) | 3275(1) | 20(1) |
| O(6) | 5050(1) | 3807(1) | 2703(1) | 26(1) |
| H(6) | 5551(19) | 3425(15) | 2393(9) | 40 |
| C(7) | 6836(2) | 2871(1) | 3484(1) | 24(1) |
| C(8) | 7793(2) | 2399(1) | 2936(1) | 26(1) |
| C(9) | 8569(2) | 1997(2) | 2498(1) | 37(1) |
| C(10) | 6920(2) | 5291(1) | 3068(1) | 23(1) |
| C(11) | 8144(2) | 5551(1) | 3524(1) | 23(1) |
| C(12) | 9162(2) | 5738(1) | 3875(1) | 28(1) |
| C(13) | 5351(2) | 8587(1) | 4077(1) | 22(1) |
| C(14) | 6770(2) | 9022(1) | 3787(1) | 26(1) |
| C(15) | 4338(2) | 9743(1) | 4216(1) | 31(1) |
| C(16) | 2626(2) | 3442(1) | 3998(1) | 23(1) |
| C(17) | 1903(2) | 2865(1) | 4625(1) | 32(1) |
| C(18) | 2080(2) | 2837(2) | 3335(1) | 30(1) |

**Tabelle 3: Bond lengths [Å] for *[7]*.**

| | | | |
|---|---|---|---|
| C(1)-O(1) | 1.4292(15) | O(5)-C(16) | 1.4437(17) |
| C(1)-C(2) | 1.5057(19) | C(6)-O(6) | 1.4216(16) |
| O(1)-C(5) | 1.4056(16) | C(6)-C(7) | 1.5395(19) |
| C(2)-O(2) | 1.4384(17) | C(6)-C(10) | 1.5421(19) |
| C(2)-C(3) | 1.5465(19) | O(6)-H(6) | 0.851(17) |
| O(2)-C(13) | 1.4268(16) | C(7)-C(8) | 1.466(2) |
| C(3)-O(3) | 1.4265(16) | C(8)-C(9) | 1.187(2) |
| C(3)-C(4) | 1.5183(18) | C(10)-C(11) | 1.468(2) |
| O(3)-C(13) | 1.4370(16) | C(11)-C(12) | 1.185(2) |
| C(4)-O(4) | 1.4301(16) | C(13)-C(14) | 1.504(2) |
| C(4)-C(5) | 1.5496(19) | C(13)-C(15) | 1.516(2) |
| O(4)-C(16) | 1.4235(18) | C(16)-C(17) | 1.505(2) |
| C(5)-O(5) | 1.4266(15) | C(16)-C(18) | 1.509(2) |
| C(5)-C(6) | 1.5471(19) | | |

**Tabelle 4: Bond angles [°] for *[7]*.**

| | | | |
|---|---|---|---|
| O(1)-C(1)-C(2) | 111.44(11) | O(6)-C(6)-C(10) | 107.82(11) |
| C(5)-O(1)-C(1) | 114.03(10) | C(7)-C(6)-C(10) | 111.46(11) |
| O(2)-C(2)-C(1) | 109.28(11) | O(6)-C(6)-C(5) | 104.57(11) |
| O(2)-C(2)-C(3) | 103.87(10) | C(7)-C(6)-C(5) | 108.78(11) |
| C(1)-C(2)-C(3) | 112.42(11) | C(10)-C(6)-C(5) | 113.20(11) |
| C(13)-O(2)-C(2) | 107.67(10) | C(6)-O(6)-H(6) | 108.6(12) |
| O(3)-C(3)-C(4) | 109.80(11) | C(8)-C(7)-C(6) | 113.17(12) |
| O(3)-C(3)-C(2) | 104.51(10) | C(9)-C(8)-C(7) | 178.90(17) |
| C(4)-C(3)-C(2) | 114.62(11) | C(11)-C(10)-C(6) | 115.93(12) |
| C(3)-O(3)-C(13) | 106.31(10) | C(12)-C(11)-C(10) | 177.69(16) |
| O(4)-C(4)-C(3) | 104.40(11) | O(2)-C(13)-O(3) | 103.68(9) |
| O(4)-C(4)-C(5) | 104.00(10) | O(2)-C(13)-C(14) | 109.34(12) |
| C(3)-C(4)-C(5) | 116.76(11) | O(3)-C(13)-C(14) | 108.60(11) |
| C(16)-O(4)-C(4) | 107.79(10) | O(2)-C(13)-C(15) | 111.26(12) |
| O(1)-C(5)-O(5) | 109.60(10) | O(3)-C(13)-C(15) | 110.08(12) |
| O(1)-C(5)-C(6) | 105.20(10) | C(14)-C(13)-C(15) | 113.40(12) |
| O(5)-C(5)-C(6) | 109.22(10) | O(4)-C(16)-O(5) | 103.90(11) |
| O(1)-C(5)-C(4) | 113.22(10) | O(4)-C(16)-C(17) | 108.87(11) |
| O(5)-C(5)-C(4) | 103.24(11) | O(5)-C(16)-C(17) | 108.70(12) |
| C(6)-C(5)-C(4) | 116.29(11) | O(4)-C(16)-C(18) | 110.98(12) |
| C(5)-O(5)-C(16) | 111.01(10) | O(5)-C(16)-C(18) | 111.47(12) |
| O(6)-C(6)-C(7) | 110.80(11) | C(17)-C(16)-C(18) | 112.53(12) |

**Tabelle 5: Torsion angles [°] for *[7]*.**

| | | | |
|---|---|---|---|
| C(2)-C(1)-O(1)-C(5) | -69.79(14) | O(1)-C(5)-C(6)-O(6) | -173.79(9) |
| O(1)-C(1)-C(2)-O(2) | -72.40(13) | O(5)-C(5)-C(6)-O(6) | 68.64(13) |
| O(1)-C(1)-C(2)-C(3) | 42.37(16) | C(4)-C(5)-C(6)-O(6) | -47.64(14) |
| C(1)-C(2)-O(2)-C(13) | 138.33(11) | O(1)-C(5)-C(6)-C(7) | 67.79(13) |
| C(3)-C(2)-O(2)-C(13) | 18.16(13) | O(5)-C(5)-C(6)-C(7) | -49.78(14) |
| O(2)-C(2)-C(3)-O(3) | 4.96(12) | C(4)-C(5)-C(6)-C(7) | -166.07(11) |
| C(1)-C(2)-C(3)-O(3) | -113.07(12) | O(1)-C(5)-C(6)-C(10) | -56.69(14) |
| O(2)-C(2)-C(3)-C(4) | 125.18(12) | O(5)-C(5)-C(6)-C(10) | -174.27(11) |
| C(1)-C(2)-C(3)-C(4) | 7.16(17) | C(4)-C(5)-C(6)-C(10) | 69.45(15) |
| C(4)-C(3)-O(3)-C(13) | -149.46(10) | O(6)-C(6)-C(7)-C(8) | 61.40(15) |
| C(2)-C(3)-O(3)-C(13) | -26.06(12) | C(10)-C(6)-C(7)-C(8) | -58.68(16) |
| O(3)-C(3)-C(4)-O(4) | -163.47(10) | C(5)-C(6)-C(7)-C(8) | 175.82(12) |
| C(2)-C(3)-C(4)-O(4) | 79.28(13) | C(6)-C(7)-C(8)-C(9) | -157(9) |
| O(3)-C(3)-C(4)-C(5) | 82.37(14) | O(6)-C(6)-C(10)-C(11) | -166.37(12) |
| C(2)-C(3)-C(4)-C(5) | -34.88(17) | C(7)-C(6)-C(10)-C(11) | -44.55(16) |
| C(3)-C(4)-O(4)-C(16) | -153.20(11) | C(5)-C(6)-C(10)-C(11) | 78.46(15) |
| C(5)-C(4)-O(4)-C(16) | -30.30(13) | C(6)-C(10)-C(11)-C(12) | 100(4) |
| C(1)-O(1)-C(5)-O(5) | -75.28(12) | C(2)-O(2)-C(13)-O(3) | -34.57(13) |
| C(1)-O(1)-C(5)-C(6) | 167.40(10) | C(2)-O(2)-C(13)-C(14) | -150.25(11) |
| C(1)-O(1)-C(5)-C(4) | 39.39(14) | C(2)-O(2)-C(13)-C(15) | 83.71(13) |
| O(4)-C(4)-C(5)-O(1) | -101.91(12) | C(3)-O(3)-C(13)-O(2) | 37.77(13) |
| C(3)-C(4)-C(5)-O(1) | 12.47(17) | C(3)-O(3)-C(13)-C(14) | 153.98(10) |
| O(4)-C(4)-C(5)-O(5) | 16.51(13) | C(3)-O(3)-C(13)-C(15) | -81.33(13) |
| C(3)-C(4)-C(5)-O(5) | 130.89(12) | C(4)-O(4)-C(16)-O(5) | 31.88(14) |
| O(4)-C(4)-C(5)-C(6) | 136.08(11) | C(4)-O(4)-C(16)-C(17) | 147.59(12) |
| C(3)-C(4)-C(5)-C(6) | -109.54(14) | C(4)-O(4)-C(16)-C(18) | -88.03(14) |
| O(1)-C(5)-O(5)-C(16) | 123.50(12) | C(5)-O(5)-C(16)-O(4) | -20.81(15) |
| C(6)-C(5)-O(5)-C(16) | -121.74(12) | C(5)-O(5)-C(16)-C(17) | -136.64(11) |
| C(4)-C(5)-O(5)-C(16) | 2.59(14) | C(5)-O(5)-C(16)-C(18) | 98.76(13) |

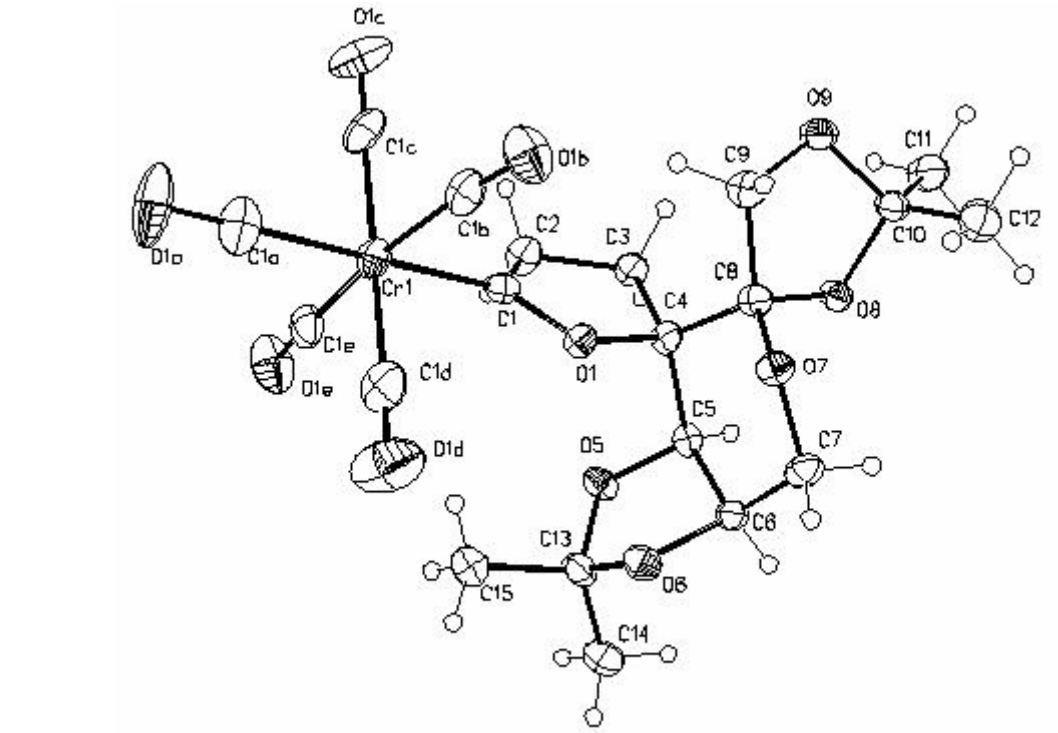
**Tabelle 6: Hydrogen bonds for *[7]* [Å and °].**

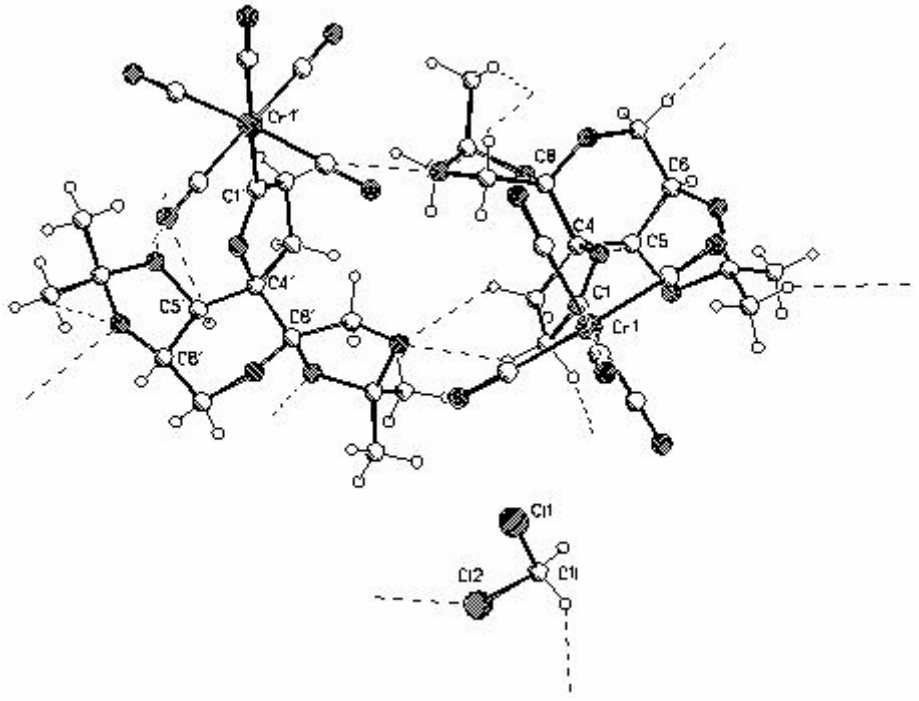
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(12)-H(12)...O(2)[a] | 0.95 | 2.66 | 3.4148(19) | 137.2 |
| C(12)-H(12)...O(4)[b] | 0.95 | 2.54 | 3.1945(18) | 126.5 |
| O(6)-H(6)...O(3)[c] | 0.851(17) | 2.096(17) | 2.7935(14) | 138.8(16) |

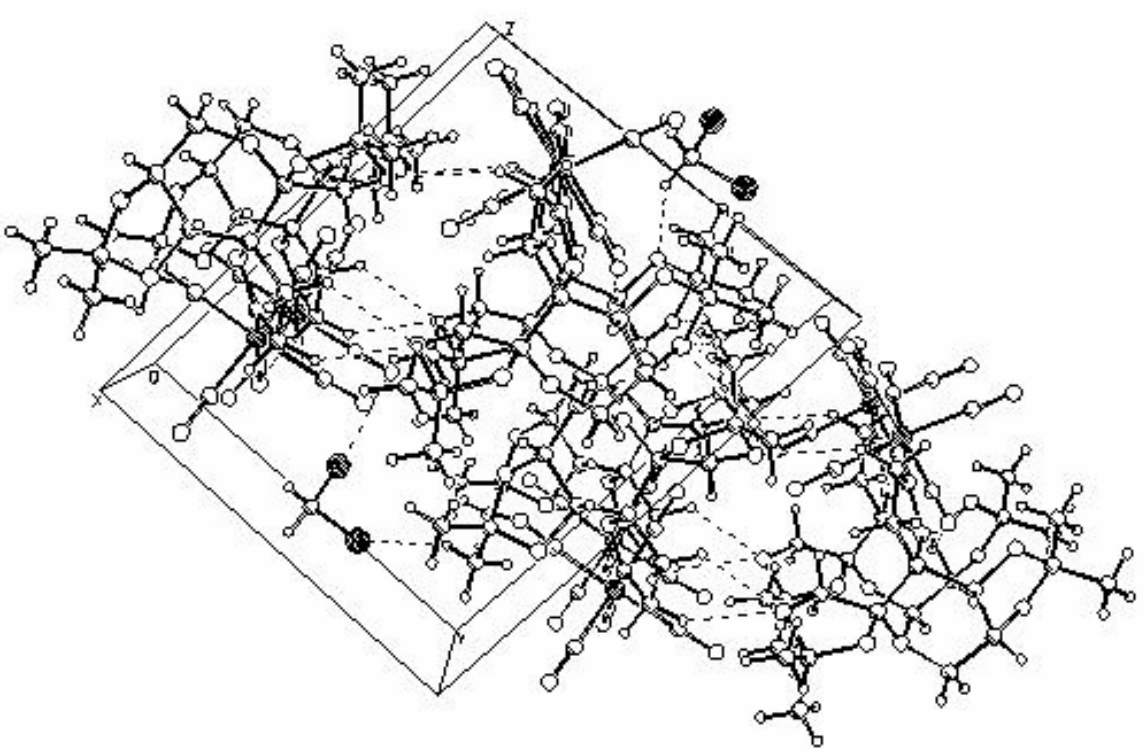
Symmetry transformations used to generate equivalent atoms:

[a] x+1/2,-y+3/2,-z+1 [b] x+1,y,z [c] -x+1,y-1/2,-z+1/2

**4. (*3R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-dispiro[3,7,9-trioxa-bicyclo[4.3.0]nonan-4´,4´´-1,3-dioxa-cyclopentan-5´,3-2-oxacyclopent]-1-yliden}chrom(0) *[14]***





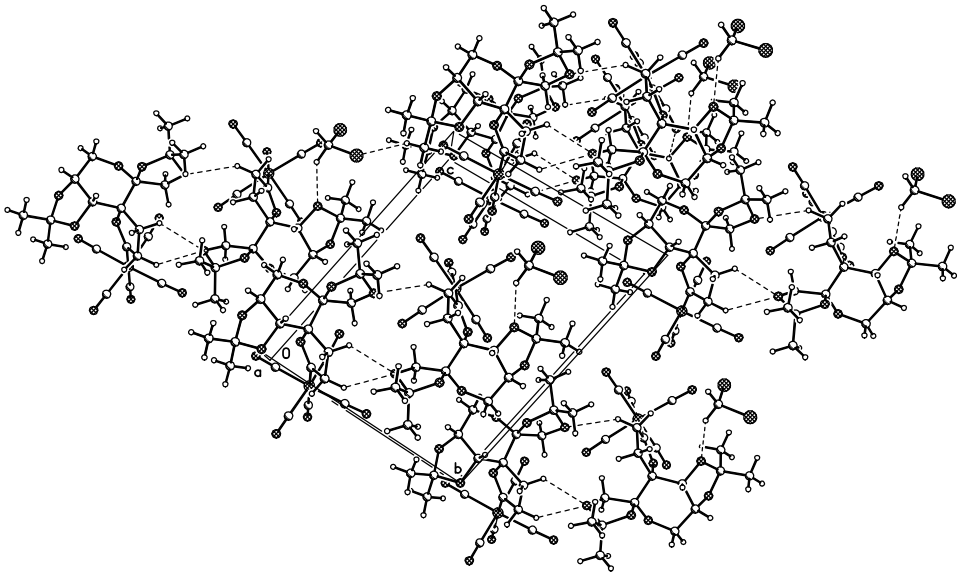


**Table 1: Crystal data and structure refinements for *[14]***

| | |
|---|---|
| Identification code | ***[14]*** |
| Empirical formula | $C_{20.5}H_{23}ClCrO_{11}$ |
| Formula weight | 532.84 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Triclinic |
| Space group | P1 (No.1) |
| Unit cell dimensions | a = 8.4928(2) Å α = 78.639(1)° |
| | b = 11.7130(2) Å β = 80.126(1)° |
| | c = 13.4098(4) Å γ = 69.017(1) ° |
| Volume | 1213.65(5) $Å^3$ |
| Z | 2 |
| Calculated density | 1.458/$m^3$ |
| Absorption coefficient | $0.637^{-1}$ |
| F(000) | 550 |
| Crystal size | 0.70 x 0.50 x 0.30 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.58 to 27.46° |
| Limiting indices | $-11 \leq h \leq 11$, $-15 \leq k \leq 15$, $-17 \leq l \leq 17$ |
| Reflections collected / unique | 17942 / 10005 [R(int) = 0.0317] |
| Completeness to Θ = 24.99 | 97.2 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 10005 / 3 / 604 |
| Goodness-of-fit on $F^2$ | 0.985 |
| Final R indices [I>2σ(I)] | R1 = 0.0322, wR2 = 0.0703 |
| R indices (all data) | R1 = 0.0388, wR2 = 0.0726 |
| Absolute structure parameter | 0.004(10) |
| Largest diff. peak and hole | 0.733 and -0.656 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[14]*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 3188(1) | 4311(1) | 5816(1) | 21(1) |
| C(1A) | 1429(3) | 3634(2) | 6350(2) | 27(1) |
| O(1A) | 403(3) | 3211(2) | 6653(2) | 42(1) |
| C(1B) | 3353(3) | 3845(2) | 4515(2) | 32(1) |
| O(1B) | 3478(3) | 3593(2) | 3712(2) | 52(1) |
| C(1C) | 3150(3) | 4870(2) | 7077(2) | 23(1) |
| O(1C) | 3168(2) | 5207(2) | 7805(1) | 35(1) |
| C(1D) | 1606(3) | 5863(2) | 5338(2) | 26(1) |
| O(1D) | 634(2) | 6777(2) | 5037(2) | 36(1) |
| C(1E) | 4741(3) | 2730(2) | 6272(2) | 30(1) |
| O(1E) | 5623(3) | 1759(2) | 6546(2) | 45(1) |
| O(1) | 4895(2) | 6012(1) | 4634(1) | 20(1) |
| C(1) | 5113(3) | 4946(2) | 5233(2) | 18(1) |
| C(2) | 6967(3) | 4332(2) | 5324(2) | 26(1) |
| C(3) | 7896(3) | 5048(2) | 4523(2) | 26(1) |
| C(4) | 6488(3) | 6236(2) | 4152(2) | 18(1) |
| C(5) | 6579(3) | 7391(2) | 4490(2) | 20(1) |
| O(5) | 6090(2) | 7365(2) | 5567(1) | 27(1) |
| C(6) | 5360(3) | 8610(2) | 4021(2) | 22(1) |
| O(6) | 3893(2) | 8776(1) | 4747(1) | 21(1) |
| C(7) | 5001(3) | 8644(2) | 2954(2) | 24(1) |
| O(7) | 4898(2) | 7508(1) | 2771(1) | 22(1) |
| C(8) | 6343(3) | 6459(2) | 3005(2) | 19(1) |
| O(8) | 7879(2) | 6601(1) | 2508(1) | 20(1) |
| C(9) | 6183(3) | 5395(2) | 2568(2) | 28(1) |
| O(9) | 7734(2) | 4954(2) | 1940(1) | 35(1) |
| C(10) | 8390(3) | 5948(2) | 1628(2) | 27(1) |
| C(11) | 7637(4) | 6800(3) | 697(2) | 42(1) |
| C(12) | 10297(4) | 5408(3) | 1490(2) | 43(1) |
| C(13) | 4497(3) | 8377(2) | 5722(2) | 25(1) |
| C(14) | 3247(3) | 7884(2) | 6451(2) | 29(1) |
| C(15) | 4845(4) | 9387(3) | 6105(2) | 40(1) |
| Cr(1') | 5110(1) | 2434(1) | -34(1) | 21(1) |
| C(1A') | 3062(3) | 2881(2) | -624(2) | 33(1) |
| O(1A') | 1839(2) | 3163(2) | -1000(2) | 52(1) |
| C(1B') | 5501(3) | 756(2) | -143(2) | 33(1) |
| O(1B') | 5640(3) | -236(2) | -195(2) | 60(1) |
| C(1C') | 4545(3) | 4106(2) | 139(2) | 29(1) |
| O(1C') | 4201(2) | 5109(2) | 268(2) | 45(1) |
| C(1D') | 4184(3) | 2074(2) | 1334(2) | 26(1) |
| O(1D') | 3704(2) | 1806(2) | 2166(1) | 38(1) |
| C(1E') | 6337(3) | 2691(2) | -1344(2) | 29(1) |

| | | | | |
|---|---|---|---|---|
| O(1E') | 7110(3) | 2846(2) | -2105(2) | 45(1) |
| O(1') | 8075(2) | 995(1) | 1076(1) | 18(1) |
| C(1') | 7327(3) | 2053(2) | 528(2) | 19(1) |
| C(2') | 8375(3) | 2867(2) | 449(2) | 24(1) |
| C(3') | 9580(3) | 2283(2) | 1271(2) | 21(1) |
| C(4') | 9542(3) | 971(2) | 1561(2) | 18(1) |
| C(5') | 11180(3) | 7(2) | 1163(2) | 18(1) |
| O(5') | 11346(2) | 152(1) | 75(1) | 23(1) |
| C(6') | 11230(3) | -1327(2) | 1503(2) | 20(1) |
| O(6') | 10508(2) | -1523(1) | 694(1) | 23(1) |
| C(7') | 10337(3) | -1571(2) | 2557(2) | 22(1) |
| O(7') | 8825(2) | -571(1) | 2831(1) | 21(1) |
| C(8') | 9081(3) | 583(2) | 2693(2) | 18(1) |
| O(8') | 10417(2) | 550(1) | 3209(1) | 19(1) |
| C(9') | 7488(3) | 1489(2) | 3205(2) | 24(1) |
| O(9') | 8106(2) | 1972(1) | 3888(1) | 25(1) |
| C(10') | 9700(3) | 1062(2) | 4140(2) | 22(1) |
| C(11') | 10812(3) | 1709(2) | 4364(2) | 31(1) |
| C(12') | 9438(3) | 63(2) | 5003(2) | 33(1) |
| C(13') | 11186(3) | -920(2) | -219(2) | 25(1) |
| C(14') | 12918(3) | -1737(2) | -626(2) | 32(1) |
| C(15') | 9919(3) | -488(2) | -1003(2) | 33(1) |
| C(1S) | 8421(4) | 6111(3) | 7485(2) | 48(1) |
| Cl(1) | 6700(1) | 5870(1) | 8304(1) | 52(1) |
| Cl(2) | 8811(2) | 7416(1) | 7650(1) | 82(1) |

**Tabelle 3: Bond lengths [Å] for *[14]*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1B) | 1.895(3) | Cr(1')-C(1C') | 1.894(3) |
| Cr(1)-C(1D) | 1.900(3) | Cr(1')-C(1D') | 1.897(3) |
| Cr(1)-C(1E) | 1.904(3) | Cr(1')-C(1B') | 1.904(3) |
| Cr(1)-C(1A) | 1.905(3) | Cr(1')-C(1E') | 1.911(3) |
| Cr(1)-C(1C) | 1.923(3) | Cr(1')-C(1') | 2.016(2) |
| Cr(1)-C(1) | 2.010(2) | C(1A')-O(1A') | 1.145(3) |
| C(1A)-O(1A) | 1.131(3) | C(1B')-O(1B') | 1.140(3) |
| C(1B)-O(1B) | 1.150(3) | C(1C')-O(1C') | 1.145(3) |
| C(1C)-O(1C) | 1.127(3) | C(1D')-O(1D') | 1.143(3) |
| C(1D)-O(1D) | 1.141(3) | C(1E')-O(1E') | 1.135(3) |
| C(1E)-O(1E) | 1.144(3) | O(1')-C(1') | 1.310(3) |
| O(1)-C(1) | 1.315(2) | O(1')-C(4') | 1.491(3) |
| O(1)-C(4) | 1.485(3) | C(1')-C(2') | 1.500(3) |
| C(1)-C(2) | 1.494(3) | C(2')-C(3') | 1.522(3) |
| C(2)-C(3) | 1.519(3) | C(3')-C(4') | 1.520(3) |
| C(3)-C(4) | 1.535(3) | C(4')-C(8') | 1.522(3) |
| C(4)-C(8) | 1.527(3) | C(4')-C(5') | 1.534(3) |
| C(4)-C(5) | 1.539(3) | C(5')-O(5') | 1.423(3) |
| C(5)-O(5) | 1.431(3) | C(5')-C(6') | 1.527(3) |
| C(5)-C(6) | 1.522(3) | O(5')-C(13') | 1.445(3) |
| O(5)-C(13) | 1.459(3) | C(6')-O(6') | 1.425(3) |
| C(6)-O(6) | 1.423(3) | C(6')-C(7') | 1.511(3) |
| C(6)-C(7) | 1.503(3) | O(6')-C(13') | 1.428(3) |
| O(6)-C(13) | 1.418(3) | C(7')-O(7') | 1.444(3) |
| C(7)-O(7) | 1.435(3) | O(7')-C(8') | 1.416(3) |
| O(7)-C(8) | 1.420(3) | C(8')-O(8') | 1.413(3) |
| C(8)-O(8) | 1.410(3) | C(8')-C(9') | 1.539(3) |
| C(8)-C(9) | 1.533(3) | O(8')-C(10') | 1.436(3) |
| O(8)-C(10) | 1.452(3) | C(9')-O(9') | 1.419(3) |
| C(9)-O(9) | 1.419(3) | O(9')-C(10') | 1.437(3) |
| O(9)-C(10) | 1.426(3) | C(10')-C(11') | 1.503(4) |
| C(10)-C(12) | 1.506(4) | C(10')-C(12') | 1.523(3) |
| C(10)-C(11) | 1.514(4) | C(13')-C(14') | 1.516(3) |
| C(13)-C(14) | 1.509(3) | C(13')-C(15') | 1.519(4) |
| C(13)-C(15) | 1.513(3) | C(1S)-Cl(2) | 1.737(3) |
| Cr(1')-C(1A') | 1.893(3) | C(1S)-Cl(1) | 1.746(3) |

**Tabelle 4: Bond angles [°] for *[14]*.**

| | | | |
|---|---|---|---|
| C(1B)-Cr(1)-C(1D) | 88.06(11) | O(7)-C(8)-C(9) | 106.74(19) |
| C(1B)-Cr(1)-C(1E) | 90.53(12) | C(4)-C(8)-C(9) | 116.15(18) |
| C(1D)-Cr(1)-C(1E) | 178.17(12) | C(8)-O(8)-C(10) | 108.23(18) |
| C(1B)-Cr(1)-C(1A) | 91.84(11) | O(9)-C(9)-C(8) | 104.62(18) |
| C(1D)-Cr(1)-C(1A) | 92.05(11) | C(9)-O(9)-C(10) | 107.41(17) |
| C(1E)-Cr(1)-C(1A) | 86.83(11) | O(9)-C(10)-O(8) | 103.40(18) |
| C(1B)-Cr(1)-C(1C) | 174.78(11) | O(9)-C(10)-C(12) | 108.2(2) |
| C(1D)-Cr(1)-C(1C) | 90.75(10) | O(8)-C(10)-C(12) | 108.6(2) |
| C(1E)-Cr(1)-C(1C) | 90.75(10) | O(9)-C(10)-C(11) | 112.3(2) |
| C(1A)-Cr(1)-C(1C) | 93.28(10) | O(8)-C(10)-C(11) | 110.1(2) |
| C(1B)-Cr(1)-C(1) | 86.62(10) | C(12)-C(10)-C(11) | 113.8(2) |
| C(1D)-Cr(1)-C(1) | 90.15(9) | O(6)-C(13)-O(5) | 104.86(17) |
| C(1E)-Cr(1)-C(1) | 90.93(10) | O(6)-C(13)-C(14) | 108.6(2) |
| C(1A)-Cr(1)-C(1) | 177.27(10) | O(5)-C(13)-C(14) | 109.15(19) |
| C(1C)-Cr(1)-C(1) | 88.30(10) | O(6)-C(13)-C(15) | 112.1(2) |
| O(1A)-C(1A)-Cr(1) | 178.4(2) | O(5)-C(13)-C(15) | 109.1(2) |
| O(1B)-C(1B)-Cr(1) | 177.7(2) | C(14)-C(13)-C(15) | 112.7(2) |
| O(1C)-C(1C)-Cr(1) | 178.0(2) | C(1A')-Cr(1')-C(1C') | 88.71(12) |
| O(1D)-C(1D)-Cr(1) | 177.9(2) | C(1A')-Cr(1')-C(1D') | 96.03(11) |
| O(1E)-C(1E)-Cr(1) | 177.1(2) | C(1C')-Cr(1')-C(1D') | 89.96(11) |
| C(1)-O(1)-C(4) | 114.78(16) | C(1A')-Cr(1')-C(1B') | 88.64(12) |
| O(1)-C(1)-C(2) | 108.15(19) | C(1C')-Cr(1')-C(1B') | 175.24(12) |
| O(1)-C(1)-Cr(1) | 122.63(15) | C(1D')-Cr(1')-C(1B') | 86.39(11) |
| C(2)-C(1)-Cr(1) | 129.13(15) | C(1A')-Cr(1')-C(1E') | 91.60(11) |
| C(1)-C(2)-C(3) | 107.38(19) | C(1C')-Cr(1')-C(1E') | 91.86(11) |
| C(2)-C(3)-C(4) | 104.12(18) | C(1D')-Cr(1')-C(1E') | 172.19(11) |
| O(1)-C(4)-C(8) | 104.18(17) | C(1B')-Cr(1')-C(1E') | 92.17(12) |
| O(1)-C(4)-C(3) | 104.07(16) | C(1A')-Cr(1')-C(1') | 176.08(10) |
| C(8)-C(4)-C(3) | 114.74(18) | C(1C')-Cr(1')-C(1') | 88.86(10) |
| O(1)-C(4)-C(5) | 109.89(17) | C(1D')-Cr(1')-C(1') | 87.05(10) |
| C(8)-C(4)-C(5) | 110.50(17) | C(1B')-Cr(1')-C(1') | 93.99(10) |
| C(3)-C(4)-C(5) | 112.81(19) | C(1E')-Cr(1')-C(1') | 85.40(10) |
| O(5)-C(5)-C(6) | 103.94(17) | O(1A')-C(1A')-Cr(1') | 178.6(2) |
| O(5)-C(5)-C(4) | 109.85(18) | O(1B')-C(1B')-Cr(1') | 176.2(2) |
| C(6)-C(5)-C(4) | 114.58(19) | O(1C')-C(1C')-Cr(1') | 178.4(2) |
| C(5)-O(5)-C(13) | 108.17(17) | O(1D')-C(1D')-Cr(1') | 176.4(2) |
| O(6)-C(6)-C(7) | 112.98(19) | O(1E')-C(1E')-Cr(1') | 177.6(2) |
| O(6)-C(6)-C(5) | 102.20(17) | C(1')-O(1')-C(4') | 114.46(16) |
| C(7)-C(6)-C(5) | 115.41(19) | O(1')-C(1')-C(2') | 108.04(19) |
| C(13)-O(6)-C(6) | 105.91(17) | O(1')-C(1')-Cr(1') | 122.95(15) |
| O(7)-C(7)-C(6) | 113.98(18) | C(2')-C(1')-Cr(1') | 128.98(15) |
| C(8)-O(7)-C(7) | 113.91(17) | C(1')-C(2')-C(3') | 106.37(18) |
| O(8)-C(8)-O(7) | 113.15(17) | C(4')-C(3')-C(2') | 104.21(18) |
| O(8)-C(8)-C(4) | 106.39(18) | O(1')-C(4')-C(3') | 104.05(16) |
| O(7)-C(8)-C(4) | 109.61(17) | O(1')-C(4')-C(8') | 103.42(17) |
| O(8)-C(8)-C(9) | 104.91(17) | C(3')-C(4')-C(8') | 115.45(19) |

| | | | |
|---|---|---|---|
| O(1')-C(4')-C(5') | 110.42(17) | C(4')-C(8')-C(9') | 115.76(18) |
| C(3')-C(4')-C(5') | 112.75(19) | C(8')-O(8')-C(10') | 108.51(17) |
| C(8')-C(4')-C(5') | 110.08(17) | O(9')-C(9')-C(8') | 104.90(18) |
| O(5')-C(5')-C(6') | 104.30(17) | C(9')-O(9')-C(10') | 106.53(17) |
| O(5')-C(5')-C(4') | 110.30(17) | O(8')-C(10')-O(9') | 103.61(18) |
| C(6')-C(5')-C(4') | 113.99(18) | O(8')-C(10')-C(11') | 108.5(2) |
| C(5')-O(5')-C(13') | 108.24(16) | O(9')-C(10')-C(11') | 108.67(19) |
| O(6')-C(6')-C(7') | 114.34(19) | O(8')-C(10')-C(12') | 110.51(19) |
| O(6')-C(6')-C(5') | 101.51(17) | O(9')-C(10')-C(12') | 111.2(2) |
| C(7')-C(6')-C(5') | 114.88(18) | C(11')-C(10')-C(12') | 113.9(2) |
| C(6')-O(6')-C(13') | 105.33(17) | O(6')-C(13')-O(5') | 105.09(17) |
| O(7')-C(7')-C(6') | 114.86(17) | O(6')-C(13')-C(14') | 111.90(19) |
| C(8')-O(7')-C(7') | 113.51(16) | O(5')-C(13')-C(14') | 109.3(2) |
| O(8')-C(8')-O(7') | 113.21(16) | O(6')-C(13')-C(15') | 109.1(2) |
| O(8')-C(8')-C(4') | 106.73(17) | O(5')-C(13')-C(15') | 108.64(19) |
| O(7')-C(8')-C(4') | 109.38(17) | C(14')-C(13')-C(15') | 112.5(2) |
| O(8')-C(8')-C(9') | 104.35(18) | Cl(2)-C(1S)-Cl(1) | 112.98(17) |
| O(7')-C(8')-C(9') | 107.49(18) | | |

**Tabelle 5: Torsion angles [°] for *[14]*.**

| | | | |
|---|---|---|---|
| C(1B)-Cr(1)-C(1A)-O(1A) | 36(9) | C(1D)-Cr(1)-C(1E)-O(1E) | -30(8) |
| C(1D)-Cr(1)-C(1A)-O(1A) | 124(9) | C(1A)-Cr(1)-C(1E)-O(1E) | 22(5) |
| C(1E)-Cr(1)-C(1A)-O(1A) | -55(9) | C(1C)-Cr(1)-C(1E)-O(1E) | 116(5) |
| C(1C)-Cr(1)-C(1A)-O(1A) | -145(9) | C(1)-Cr(1)-C(1E)-O(1E) | -156(5) |
| C(1)-Cr(1)-C(1A)-O(1A) | -20(10) | C(4)-O(1)-C(1)-C(2) | 5.9(3) |
| C(1D)-Cr(1)-C(1B)-O(1B) | 67(7) | C(4)-O(1)-C(1)-Cr(1) | -171.01(15) |
| C(1E)-Cr(1)-C(1B)-O(1B) | -115(7) | C(1B)-Cr(1)-C(1)-O(1) | 78.82(19) |
| C(1A)-Cr(1)-C(1B)-O(1B) | 159(7) | C(1D)-Cr(1)-C(1)-O(1) | -9.23(19) |
| C(1C)-Cr(1)-C(1B)-O(1B) | -10(7) | C(1E)-Cr(1)-C(1)-O(1) | 169.30(19) |
| C(1)-Cr(1)-C(1B)-O(1B) | -24(7) | C(1A)-Cr(1)-C(1)-O(1) | 134(2) |
| C(1B)-Cr(1)-C(1C)-O(1C) | -19(7) | C(1C)-Cr(1)-C(1)-O(1) | -99.98(19) |
| C(1D)-Cr(1)-C(1C)-O(1C) | -96(6) | C(1B)-Cr(1)-C(1)-C(2) | -97.4(2) |
| C(1E)-Cr(1)-C(1C)-O(1C) | 85(6) | C(1D)-Cr(1)-C(1)-C(2) | 174.5(2) |
| C(1A)-Cr(1)-C(1C)-O(1C) | 172(6) | C(1E)-Cr(1)-C(1)-C(2) | -6.9(2) |
| C(1)-Cr(1)-C(1C)-O(1C) | -5(6) | C(1A)-Cr(1)-C(1)-C(2) | -42(2) |
| C(1B)-Cr(1)-C(1D)-O(1D) | 42(6) | C(1C)-Cr(1)-C(1)-C(2) | 83.8(2) |
| C(1E)-Cr(1)-C(1D)-O(1D) | 2(9) | O(1)-C(1)-C(2)-C(3) | -11.5(3) |
| C(1A)-Cr(1)-C(1D)-O(1D) | -50(6) | Cr(1)-C(1)-C(2)-C(3) | 165.16(18) |
| C(1C)-Cr(1)-C(1D)-O(1D) | -143(6) | C(1)-C(2)-C(3)-C(4) | 12.2(3) |
| C(1)-Cr(1)-C(1D)-O(1D) | 128(6) | C(1)-O(1)-C(4)-C(8) | 122.52(18) |
| C(1B)-Cr(1)-C(1E)-O(1E) | -69(5) | C(1)-O(1)-C(4)-C(3) | 2.0(2) |

| | | | |
|---|---|---|---|
| C(1)-O(1)-C(4)-C(5) | -119.08(19) | C(5)-O(5)-C(13)-O(6) | 13.7(2) |
| C(2)-C(3)-C(4)-O(1) | -8.7(2) | C(5)-O(5)-C(13)-C(14) | 130.0(2) |
| C(2)-C(3)-C(4)-C(8) | -121.8(2) | C(5)-O(5)-C(13)-C(15) | -106.5(2) |
| C(2)-C(3)-C(4)-C(5) | 110.4(2) | C(1C')-Cr(1')-C(1A')-O(1A') | 76(12) |
| O(1)-C(4)-C(5)-O(5) | 44.3(2) | C(1D')-Cr(1')-C(1A')-O(1A') | 165(12) |
| C(8)-C(4)-C(5)-O(5) | 158.72(18) | C(1B')-Cr(1')-C(1A')-O(1A') | -108(12) |
| C(3)-C(4)-C(5)-O(5) | -71.3(2) | C(1E')-Cr(1')-C(1A')-O(1A') | -16(12) |
| O(1)-C(4)-C(5)-C(6) | -72.2(2) | C(1')-Cr(1')-C(1A')-O(1A') | 24(13) |
| C(8)-C(4)-C(5)-C(6) | 42.2(2) | C(1A')-Cr(1')-C(1B')-O(1B') | -37(4) |
| C(3)-C(4)-C(5)-C(6) | 172.13(18) | C(1C')-Cr(1')-C(1B')-O(1B') | 19(5) |
| C(6)-C(5)-O(5)-C(13) | 9.8(2) | C(1D')-Cr(1')-C(1B')-O(1B') | 59(4) |
| C(4)-C(5)-O(5)-C(13) | -113.21(19) | C(1E')-Cr(1')-C(1B')-O(1B') | -129(4) |
| O(5)-C(5)-C(6)-O(6) | -29.6(2) | C(1')-Cr(1')-C(1B')-O(1B') | 146(4) |
| C(4)-C(5)-C(6)-O(6) | 90.2(2) | C(1A')-Cr(1')-C(1C')-O(1C') | 115(8) |
| O(5)-C(5)-C(6)-C(7) | -152.65(19) | C(1D')-Cr(1')-C(1C')-O(1C') | 19(8) |
| C(4)-C(5)-C(6)-C(7) | -32.8(3) | C(1B')-Cr(1')-C(1C')-O(1C') | 59(9) |
| C(7)-C(6)-O(6)-C(13) | 163.84(18) | C(1E')-Cr(1')-C(1C')-O(1C') | -154(8) |
| C(5)-C(6)-O(6)-C(13) | 39.2(2) | C(1')-Cr(1')-C(1C')-O(1C') | -68(8) |
| O(6)-C(6)-C(7)-O(7) | -80.1(2) | C(1A')-Cr(1')-C(1D')-O(1D') | 142(4) |
| C(5)-C(6)-C(7)-O(7) | 37.0(3) | C(1C')-Cr(1')-C(1D')-O(1D') | -130(4) |
| C(6)-C(7)-O(7)-C(8) | -54.2(2) | C(1B')-Cr(1')-C(1D')-O(1D') | 53(4) |
| C(7)-O(7)-C(8)-O(8) | -54.0(2) | C(1E')-Cr(1')-C(1D')-O(1D') | -26(4) |
| C(7)-O(7)-C(8)-C(4) | 64.6(2) | C(1')-Cr(1')-C(1D')-O(1D') | -41(4) |
| C(7)-O(7)-C(8)-C(9) | -168.85(18) | C(1A')-Cr(1')-C(1E')-O(1E') | 160(6) |
| O(1)-C(4)-C(8)-O(8) | -176.46(15) | C(1C')-Cr(1')-C(1E')-O(1E') | 72(6) |
| C(3)-C(4)-C(8)-O(8) | -63.3(2) | C(1D')-Cr(1')-C(1E')-O(1E') | -32(6) |
| C(5)-C(4)-C(8)-O(8) | 65.6(2) | C(1B')-Cr(1')-C(1E')-O(1E') | -111(6) |
| O(1)-C(4)-C(8)-O(7) | 60.9(2) | C(1')-Cr(1')-C(1E')-O(1E') | -17(6) |
| C(3)-C(4)-C(8)-O(7) | 173.99(18) | C(4')-O(1')-C(1')-C(2') | 7.8(2) |
| C(5)-C(4)-C(8)-O(7) | -57.1(2) | C(4')-O(1')-C(1')-Cr(1') | -170.32(14) |
| O(1)-C(4)-C(8)-C(9) | -60.2(2) | C(1A')-Cr(1')-C(1')-O(1') | -161.6(17) |
| C(3)-C(4)-C(8)-C(9) | 53.0(3) | C(1C')-Cr(1')-C(1')-O(1') | 146.60(19) |
| C(5)-C(4)-C(8)-C(9) | -178.13(19) | C(1D')-Cr(1')-C(1')-O(1') | 56.58(19) |
| O(7)-C(8)-O(8)-C(10) | -100.7(2) | C(1B')-Cr(1')-C(1')-O(1') | -29.6(2) |
| C(4)-C(8)-O(8)-C(10) | 138.86(17) | C(1E')-Cr(1')-C(1')-O(1') | -121.45(19) |
| C(9)-C(8)-O(8)-C(10) | 15.2(2) | C(1A')-Cr(1')-C(1')-C(2') | 20.6(19) |
| O(8)-C(8)-C(9)-O(9) | 5.7(2) | C(1C')-Cr(1')-C(1')-C(2') | -31.1(2) |
| O(7)-C(8)-C(9)-O(9) | 126.04(19) | C(1D')-Cr(1')-C(1')-C(2') | -121.2(2) |
| C(4)-C(8)-C(9)-O(9) | -111.4(2) | C(1B')-Cr(1')-C(1')-C(2') | 152.7(2) |
| C(8)-C(9)-O(9)-C(10) | -25.0(3) | C(1E')-Cr(1')-C(1')-C(2') | 60.8(2) |
| C(9)-O(9)-C(10)-O(8) | 34.3(3) | O(1')-C(1')-C(2')-C(3') | -15.6(3) |
| C(9)-O(9)-C(10)-C(12) | 149.4(2) | Cr(1')-C(1')-C(2')-C(3') | 162.43(17) |
| C(9)-O(9)-C(10)-C(11) | -84.3(3) | C(1')-C(2')-C(3')-C(4') | 16.8(2) |
| C(8)-O(8)-C(10)-O(9) | -30.6(2) | C(1')-O(1')-C(4')-C(3') | 3.1(2) |
| C(8)-O(8)-C(10)-C(12) | -145.3(2) | C(1')-O(1')-C(4')-C(8') | 124.09(18) |
| C(8)-O(8)-C(10)-C(11) | 89.6(2) | C(1')-O(1')-C(4')-C(5') | -118.17(19) |
| C(6)-O(6)-C(13)-O(5) | -33.7(2) | C(2')-C(3')-C(4')-O(1') | -12.2(2) |
| C(6)-O(6)-C(13)-C(14) | -150.27(18) | C(2')-C(3')-C(4')-C(8') | -124.8(2) |
| C(6)-O(6)-C(13)-C(15) | 84.6(2) | C(2')-C(3')-C(4')-C(5') | 107.5(2) |

| | | | |
|---|---|---|---|
| O(1')-C(4')-C(5')-O(5') | 48.9(2) | C(3')-C(4')-C(8')-O(7') | 170.62(18) |
| C(3')-C(4')-C(5')-O(5') | -67.0(2) | C(5')-C(4')-C(8')-O(7') | -60.3(2) |
| C(8')-C(4')-C(5')-O(5') | 162.46(18) | O(1')-C(4')-C(8')-C(9') | -63.9(2) |
| O(1')-C(4')-C(5')-C(6') | -68.0(2) | C(3')-C(4')-C(8')-C(9') | 49.1(3) |
| C(3')-C(4')-C(5')-C(6') | 176.04(19) | C(5')-C(4')-C(8')-C(9') | 178.1(2) |
| C(8')-C(4')-C(5')-C(6') | 45.5(3) | O(7')-C(8')-O(8')-C(10') | -100.4(2) |
| C(6')-C(5')-O(5')-C(13') | 9.9(2) | C(4')-C(8')-O(8')-C(10') | 139.16(17) |
| C(4')-C(5')-O(5')-C(13') | -112.86(19) | C(9')-C(8')-O(8')-C(10') | 16.1(2) |
| O(5')-C(5')-C(6')-O(6') | -30.2(2) | O(8')-C(8')-C(9')-O(9') | 5.7(2) |
| C(4')-C(5')-C(6')-O(6') | 90.2(2) | O(7')-C(8')-C(9')-O(9') | 126.13(18) |
| O(5')-C(5')-C(6')-C(7') | -154.13(19) | C(4')-C(8')-C(9')-O(9') | -111.3(2) |
| C(4')-C(5')-C(6')-C(7') | -33.8(3) | C(8')-C(9')-O(9')-C(10') | -25.2(2) |
| C(7')-C(6')-O(6')-C(13') | 163.88(18) | C(8')-O(8')-C(10')-O(9') | -31.8(2) |
| C(5')-C(6')-O(6')-C(13') | 39.6(2) | C(8')-O(8')-C(10')-C(11') | -147.14(18) |
| O(6')-C(6')-C(7')-O(7') | -81.3(2) | C(8')-O(8')-C(10')-C(12') | 87.4(2) |
| C(5')-C(6')-C(7')-O(7') | 35.5(3) | C(9')-O(9')-C(10')-O(8') | 35.2(2) |
| C(6')-C(7')-O(7')-C(8') | -52.0(3) | C(9')-O(9')-C(10')-C(11') | 150.4(2) |
| C(7')-O(7')-C(8')-O(8') | -54.5(2) | C(9')-O(9')-C(10')-C(12') | -83.5(2) |
| C(7')-O(7')-C(8')-C(4') | 64.4(2) | C(6')-O(6')-C(13')-O(5') | -34.5(2) |
| C(7')-O(7')-C(8')-C(9') | -169.16(18) | C(6')-O(6')-C(13')-C(14') | 84.0(2) |
| O(1')-C(4')-C(8')-O(8') | -179.52(15) | C(6')-O(6')-C(13')-C(15') | -150.85(18) |
| C(3')-C(4')-C(8')-O(8') | -66.6(2) | C(5')-O(5')-C(13')-O(6') | 14.2(2) |
| C(5')-C(4')-C(8')-O(8') | 62.5(2) | C(5')-O(5')-C(13')-C(14') | -106.1(2) |
| O(1')-C(4')-C(8')-O(7') | 57.7(2) | C(5')-O(5')-C(13')-C(15') | 130.9(2) |

**Tabelle 6: Hydrogen bonds for *[14]* [Å and °].**

| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(1S)-H(1S2)...O(5) | 0.99 | 2.46 | 3.297(4) | 142.1 |
| C(2)-H(2A)...O(9') | 0.99 | 2.57 | 3.440(3) | 146.8 |
| C(3')-H(3'2)...O(9) | 0.99 | 2.46 | 3.187(3) | 129.8 |
| C(5)-H(5)...O(1D)[a] | 1.00 | 2.57 | 3.434(3) | 144.5 |
| C(2')-H(2'2)...O(1A')[a] | 0.99 | 2.53 | 3.324(3) | 137.2 |
| C(15')-H(15F)...Cl(2)[b] | 0.98 | 2.84 | 3.763(3) | 156.4 |

Symmetry transformations used to generate equivalent atoms:

[a]1 x+1,y,z [2] x,y-1,z-1

**5.** **(*3R,1´R,2´S,6´S,9´R*)-Pentacarbonyl{3-(4´,4´,11´,11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]-dodec-6´-yl)-2-oxacyclopentyliden}chrom(0) *[16]R***

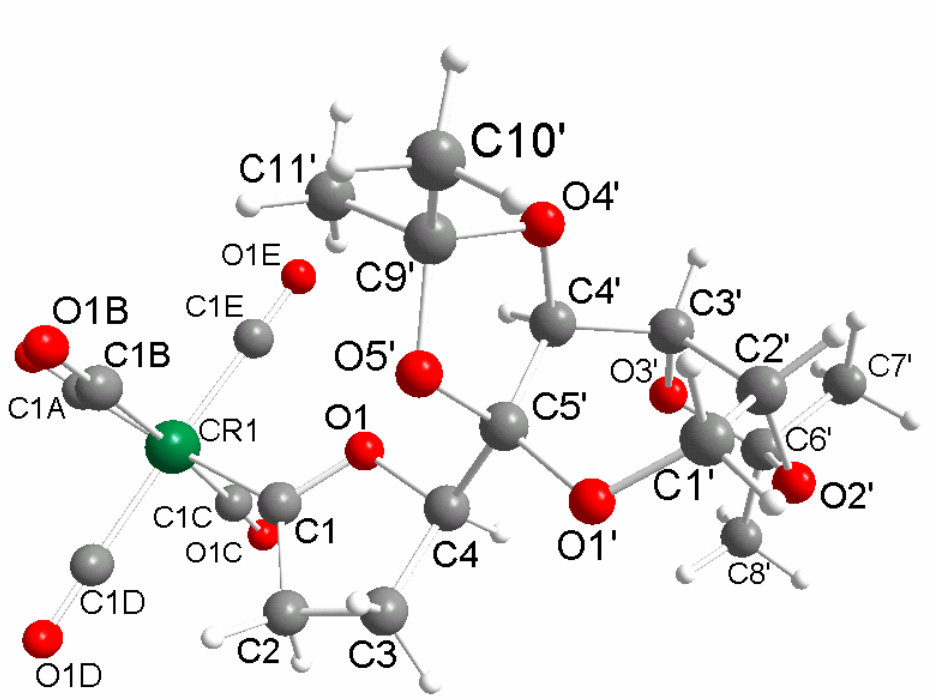



**Table 1:** **Crystal data and structure refinements for *[16]R***

| | |
|---|---|
| Identification code | ***[16]R*** |
| Empirical formula | $C_{20}H_{22}CrO_{11}$ |
| Formula weight | 490.38 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 10.3469(1) Å α = 90° |
| | b = 12.0963(1) Å β = 90° |
| | c = 17.7733(2) Å γ = 90° |
| Volume | 2224.49(4) Å$^3$ |
| Z | 4 |
| Calculated density | 1.464 mg/m$^3$ |

| | |
|---|---|
| Absorption coefficient | 0.572 $mm^{-1}$ |
| F(000) | 1016 |
| Crystal size | 0.50 x 0.30 x 0.10 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.83 to 25.00° |
| Limiting indices | $-12 \leq h \leq 12$, $-14 \leq k \leq 14$, $-21 \leq l \leq 21$ |
| Reflections collected / unique | 41802 / 3910 [R(int) = 0.0443] |
| Completeness to $\Theta = 25.00$ | 99.5 % |
| Absorption correction | Empirical from multiple refl |
| Max. and min. transmission | 0.8476 and 0.7611 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 3910 / 0 / 289 |
| Goodness-of-fit on $F^2$ | 1.053 |
| Final R indices [$I>2\sigma(I)$] | R1 = 0.0185, wR2 = 0.0496 |
| R indices (all data) | R1 = 0.0195, wR2 = 0.0500 |
| Absolute structure parameter | -0.011(10) |
| Largest diff. peak and hole | 0.198 and -0.205 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[16]R*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 6869(1) | 3185(1) | 5715(1) | 17(1) |
| C(1A) | 7458(1) | 1895(1) | 5221(1) | 19(1) |
| O(1A) | 7836(1) | 1134(1) | 4904(1) | 28(1) |
| C(1B) | 6061(2) | 3661(1) | 4809(1) | 23(1) |
| O(1B) | 5560(1) | 3944(1) | 4272(1) | 34(1) |
| C(1C) | 7566(2) | 2744(1) | 6663(1) | 25(1) |
| O(1C) | 7939(1) | 2473(1) | 7236(1) | 43(1) |
| C(1D) | 5323(2) | 2470(1) | 6028(1) | 23(1) |
| O(1D) | 4422(1) | 2018(1) | 6234(1) | 33(1) |
| C(1E) | 8457(2) | 3889(1) | 5456(1) | 21(1) |
| O(1E) | 9423(1) | 4288(1) | 5300(1) | 30(1) |
| O(1) | 6966(1) | 5528(1) | 6120(1) | 22(1) |
| C(1) | 6263(1) | 4634(1) | 6157(1) | 18(1) |
| C(2) | 5034(2) | 4901(1) | 6558(1) | 36(1) |
| C(3) | 4948(2) | 6160(1) | 6577(1) | 25(1) |
| C(4) | 6353(1) | 6509(1) | 6466(1) | 20(1) |
| O(1') | 5862(1) | 8334(1) | 6339(1) | 21(1) |
| C(1') | 6274(2) | 9440(1) | 6190(1) | 21(1) |
| C(2') | 7630(1) | 9627(1) | 6485(1) | 19(1) |
| C(3') | 8566(1) | 8690(1) | 6246(1) | 18(1) |
| C(4') | 7963(1) | 7794(1) | 5771(1) | 17(1) |
| C(5') | 6542(1) | 7503(1) | 5949(1) | 17(1) |
| O(2') | 7619(1) | 9576(1) | 7289(1) | 23(1) |
| O(3') | 8965(1) | 8204(1) | 6941(1) | 21(1) |
| C(6') | 8777(2) | 9026(1) | 7508(1) | 21(1) |
| C(7') | 9910(2) | 9826(1) | 7529(1) | 26(1) |
| C(8') | 8555(2) | 8446(1) | 8247(1) | 30(1) |
| O(4') | 7895(1) | 8206(1) | 5021(1) | 19(1) |
| O(5') | 5997(1) | 7311(1) | 5230(1) | 21(1) |
| C(9') | 6928(1) | 7583(1) | 4646(1) | 19(1) |
| C(10') | 6309(1) | 8319(1) | 4064(1) | 24(1) |
| C(11') | 7480(1) | 6526(1) | 4316(1) | 24(1) |

**Tabelle 3: Bond lengths [Å] for *[16]R*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1A) | 1.8920(16) | O(1')-C(1') | 1.4296(17) |
| Cr(1)-C(1D) | 1.9019(16) | C(1')-C(2') | 1.515(2) |
| Cr(1)-C(1B) | 1.9040(16) | C(2')-O(2') | 1.4299(18) |
| Cr(1)-C(1E) | 1.9070(16) | C(2')-C(3') | 1.550(2) |
| Cr(1)-C(1C) | 1.9089(17) | C(3')-O(3') | 1.4286(17) |
| Cr(1)-C(1) | 2.0198(14) | C(3')-C(4') | 1.5084(19) |
| C(1A)-O(1A) | 1.1470(18) | C(4')-O(4') | 1.4254(17) |
| C(1B)-O(1B) | 1.1376(19) | C(4')-C(5') | 1.5450(19) |
| C(1C)-O(1C) | 1.1376(19) | C(5')-O(5') | 1.4166(17) |
| C(1D)-O(1D) | 1.1406(19) | O(2')-C(6') | 1.4255(18) |
| C(1E)-O(1E) | 1.1436(18) | O(3')-C(6') | 1.4297(18) |
| O(1)-C(1) | 1.3051(17) | C(6')-C(8') | 1.506(2) |
| O(1)-C(4) | 1.4801(17) | C(6')-C(7') | 1.521(2) |
| C(1)-C(2) | 1.494(2) | O(4')-C(9') | 1.4190(17) |
| C(2)-C(3) | 1.525(2) | O(5')-C(9') | 1.4538(17) |
| C(3)-C(4) | 1.526(2) | C(9')-C(10') | 1.507(2) |
| C(4)-C(5') | 1.526(2) | C(9')-C(11') | 1.5188(19) |
| O(1')-C(5') | 1.4086(17) | | |

**Tabelle 4: Bond angles [°] for *[16]R*.**

| | | | |
|---|---|---|---|
| C(1A)-Cr(1)-C(1D) | 91.82(6) | O(2')-C(2')-C(1') | 109.40(12) |
| C(1A)-Cr(1)-C(1B) | 89.87(6) | O(2')-C(2')-C(3') | 104.30(11) |
| C(1D)-Cr(1)-C(1B) | 90.89(7) | C(1')-C(2')-C(3') | 111.99(12) |
| C(1A)-Cr(1)-C(1E) | 88.78(6) | O(3')-C(3')-C(4') | 107.93(11) |
| C(1D)-Cr(1)-C(1E) | 176.83(7) | O(3')-C(3')-C(2') | 104.19(11) |
| C(1B)-Cr(1)-C(1E) | 92.22(6) | C(4')-C(3')-C(2') | 114.88(12) |
| C(1A)-Cr(1)-C(1C) | 93.29(6) | O(4')-C(4')-C(3') | 107.04(11) |
| C(1D)-Cr(1)-C(1C) | 86.14(7) | O(4')-C(4')-C(5') | 102.97(10) |
| C(1B)-Cr(1)-C(1C) | 175.73(7) | C(3')-C(4')-C(5') | 116.22(12) |
| C(1E)-Cr(1)-C(1C) | 90.71(6) | O(1')-C(5')-O(5') | 111.21(11) |
| C(1A)-Cr(1)-C(1) | 174.98(6) | O(1')-C(5')-C(4) | 101.62(11) |
| C(1D)-Cr(1)-C(1) | 91.13(6) | O(5')-C(5')-C(4) | 111.30(11) |
| C(1B)-Cr(1)-C(1) | 86.03(6) | O(1')-C(5')-C(4') | 114.48(11) |
| C(1E)-Cr(1)-C(1) | 88.49(6) | O(5')-C(5')-C(4') | 103.36(11) |
| C(1C)-Cr(1)-C(1) | 90.95(6) | C(4)-C(5')-C(4') | 115.16(11) |
| O(1A)-C(1A)-Cr(1) | 177.74(13) | C(6')-O(2')-C(2') | 106.69(11) |
| O(1B)-C(1B)-Cr(1) | 178.95(14) | C(3')-O(3')-C(6') | 106.52(10) |
| O(1C)-C(1C)-Cr(1) | 177.59(15) | O(2')-C(6')-O(3') | 104.22(11) |
| O(1D)-C(1D)-Cr(1) | 177.42(14) | O(2')-C(6')-C(8') | 109.10(12) |
| O(1E)-C(1E)-Cr(1) | 178.39(12) | O(3')-C(6')-C(8') | 108.21(12) |
| C(1)-O(1)-C(4) | 113.89(11) | O(2')-C(6')-C(7') | 110.93(12) |
| O(1)-C(1)-C(2) | 108.61(12) | O(3')-C(6')-C(7') | 110.82(12) |
| O(1)-C(1)-Cr(1) | 121.78(10) | C(8')-C(6')-C(7') | 113.15(13) |
| C(2)-C(1)-Cr(1) | 129.61(11) | C(9')-O(4')-C(4') | 106.76(10) |
| C(1)-C(2)-C(3) | 106.02(13) | C(5')-O(5')-C(9') | 110.10(10) |
| C(2)-C(3)-C(4) | 102.62(13) | O(4')-C(9')-O(5') | 104.62(11) |
| O(1)-C(4)-C(5') | 109.01(11) | O(4')-C(9')-C(10') | 107.96(11) |
| O(1)-C(4)-C(3) | 103.85(11) | O(5')-C(9')-C(10') | 110.03(11) |
| C(5')-C(4)-C(3) | 114.72(12) | O(4')-C(9')-C(11') | 111.26(11) |
| C(5')-O(1')-C(1') | 115.28(11) | O(5')-C(9')-C(11') | 109.52(11) |
| O(1')-C(1')-C(2') | 110.60(12) | C(10')-C(9')-C(11') | 113.10(13) |

**Tabelle 5: Torsion angles [°] for *[16]R*.**

| | | | |
|---|---|---|---|
| C(1D)-Cr(1)-C(1A)-O(1A) | 149(3) | O(1')-C(1')-C(2')-C(3') | 48.14(16) |
| C(1B)-Cr(1)-C(1A)-O(1A) | 58(3) | O(2')-C(2')-C(3')-O(3') | 0.76(14) |
| C(1E)-Cr(1)-C(1A)-O(1A) | -34(3) | C(1')-C(2')-C(3')-O(3') | -117.44(13) |
| C(1C)-Cr(1)-C(1A)-O(1A) | -125(3) | O(2')-C(2')-C(3')-C(4') | 118.62(13) |
| C(1)-Cr(1)-C(1A)-O(1A) | 23(4) | C(1')-C(2')-C(3')-C(4') | 0.42(18) |
| C(1A)-Cr(1)-C(1B)-O(1B) | 118(7) | O(3')-C(3')-C(4')-O(4') | -164.48(10) |
| C(1D)-Cr(1)-C(1B)-O(1B) | 26(7) | C(2')-C(3')-C(4')-O(4') | 79.79(14) |
| C(1E)-Cr(1)-C(1B)-O(1B) | -153(7) | O(3')-C(3')-C(4')-C(5') | 81.14(14) |
| C(1C)-Cr(1)-C(1B)-O(1B) | -20(8) | C(2')-C(3')-C(4')-C(5') | -34.58(17) |
| C(1)-Cr(1)-C(1B)-O(1B) | -65(7) | C(1')-O(1')-C(5')-O(5') | -86.62(13) |
| C(1A)-Cr(1)-C(1C)-O(1C) | -106(3) | C(1')-O(1')-C(5')-C(4) | 154.86(12) |
| C(1D)-Cr(1)-C(1C)-O(1C) | -14(3) | C(1')-O(1')-C(5')-C(4') | 30.07(16) |
| C(1B)-Cr(1)-C(1C)-O(1C) | 32(4) | O(1)-C(4)-C(5')-O(1') | 177.35(10) |
| C(1E)-Cr(1)-C(1C)-O(1C) | 165(3) | C(3)-C(4)-C(5')-O(1') | 61.41(15) |
| C(1)-Cr(1)-C(1C)-O(1C) | 77(3) | O(1)-C(4)-C(5')-O(5') | 58.89(15) |
| C(1A)-Cr(1)-C(1D)-O(1D) | 69(3) | C(3)-C(4)-C(5')-O(5') | -57.05(16) |
| C(1B)-Cr(1)-C(1D)-O(1D) | 159(3) | O(1)-C(4)-C(5')-C(4') | -58.32(16) |
| C(1E)-Cr(1)-C(1D)-O(1D) | -32(4) | C(3)-C(4)-C(5')-C(4') | -174.26(12) |
| C(1C)-Cr(1)-C(1D)-O(1D) | -24(3) | O(4')-C(4')-C(5')-O(1') | -96.02(13) |
| C(1)-Cr(1)-C(1D)-O(1D) | -115(3) | C(3')-C(4')-C(5')-O(1') | 20.63(17) |
| C(1A)-Cr(1)-C(1E)-O(1E) | -28(5) | O(4')-C(4')-C(5')-O(5') | 25.10(13) |
| C(1D)-Cr(1)-C(1E)-O(1E) | 73(5) | C(3')-C(4')-C(5')-O(5') | 141.75(12) |
| C(1B)-Cr(1)-C(1E)-O(1E) | -118(5) | O(4')-C(4')-C(5')-C(4) | 146.70(11) |
| C(1C)-Cr(1)-C(1E)-O(1E) | 65(5) | C(3')-C(4')-C(5')-C(4) | -96.64(14) |
| C(1)-Cr(1)-C(1E)-O(1E) | 156(5) | C(1')-C(2')-O(2')-C(6') | 141.66(12) |
| C(4)-O(1)-C(1)-C(2) | -0.57(17) | C(3')-C(2')-O(2')-C(6') | 21.69(14) |
| C(4)-O(1)-C(1)-Cr(1) | 179.29(10) | C(4')-C(3')-O(3')-C(6') | -145.40(11) |
| C(1A)-Cr(1)-C(1)-O(1) | -53.9(8) | C(2')-C(3')-O(3')-C(6') | -22.86(13) |
| C(1D)-Cr(1)-C(1)-O(1) | -179.96(12) | C(2')-O(2')-C(6')-O(3') | -36.49(14) |
| C(1B)-Cr(1)-C(1)-O(1) | -89.15(12) | C(2')-O(2')-C(6')-C(8') | -151.88(12) |
| C(1E)-Cr(1)-C(1)-O(1) | 3.19(12) | C(2')-O(2')-C(6')-C(7') | 82.82(14) |
| C(1C)-Cr(1)-C(1)-O(1) | 93.88(12) | C(3')-O(3')-C(6')-O(2') | 37.01(13) |
| C(1A)-Cr(1)-C(1)-C(2) | 126.0(7) | C(3')-O(3')-C(6')-C(8') | 153.03(12) |
| C(1D)-Cr(1)-C(1)-C(2) | -0.13(15) | C(3')-O(3')-C(6')-C(7') | -82.37(14) |
| C(1B)-Cr(1)-C(1)-C(2) | 90.68(15) | C(3')-C(4')-O(4')-C(9') | -158.38(11) |
| C(1E)-Cr(1)-C(1)-C(2) | -176.98(15) | C(5')-C(4')-O(4')-C(9') | -35.37(13) |
| C(1C)-Cr(1)-C(1)-C(2) | -86.29(15) | O(1')-C(5')-O(5')-C(9') | 116.93(11) |
| O(1)-C(1)-C(2)-C(3) | 14.49(19) | C(4)-C(5')-O(5')-C(9') | -130.54(12) |
| Cr(1)-C(1)-C(2)-C(3) | -165.36(12) | C(4')-C(5')-O(5')-C(9') | -6.37(13) |
| C(1)-C(2)-C(3)-C(4) | -21.53(18) | C(4')-O(4')-C(9')-O(5') | 31.82(13) |
| C(1)-O(1)-C(4)-C(5') | -136.15(12) | C(4')-O(4')-C(9')-C(10') | 148.99(11) |
| C(1)-O(1)-C(4)-C(3) | -13.43(16) | C(4')-O(4')-C(9')-C(11') | -86.35(14) |
| C(2)-C(3)-C(4)-O(1) | 20.66(16) | C(5')-O(5')-C(9')-O(4') | -14.85(14) |
| C(2)-C(3)-C(4)-C(5') | 139.54(14) | C(5')-O(5')-C(9')-C(10') | -130.59(12) |
| C(5')-O(1')-C(1')-C(2') | -66.59(15) | C(5')-O(5')-C(9')-C(11') | 104.50(13) |
| O(1')-C(1')-C(2')-O(2') | -66.98(14) | | |

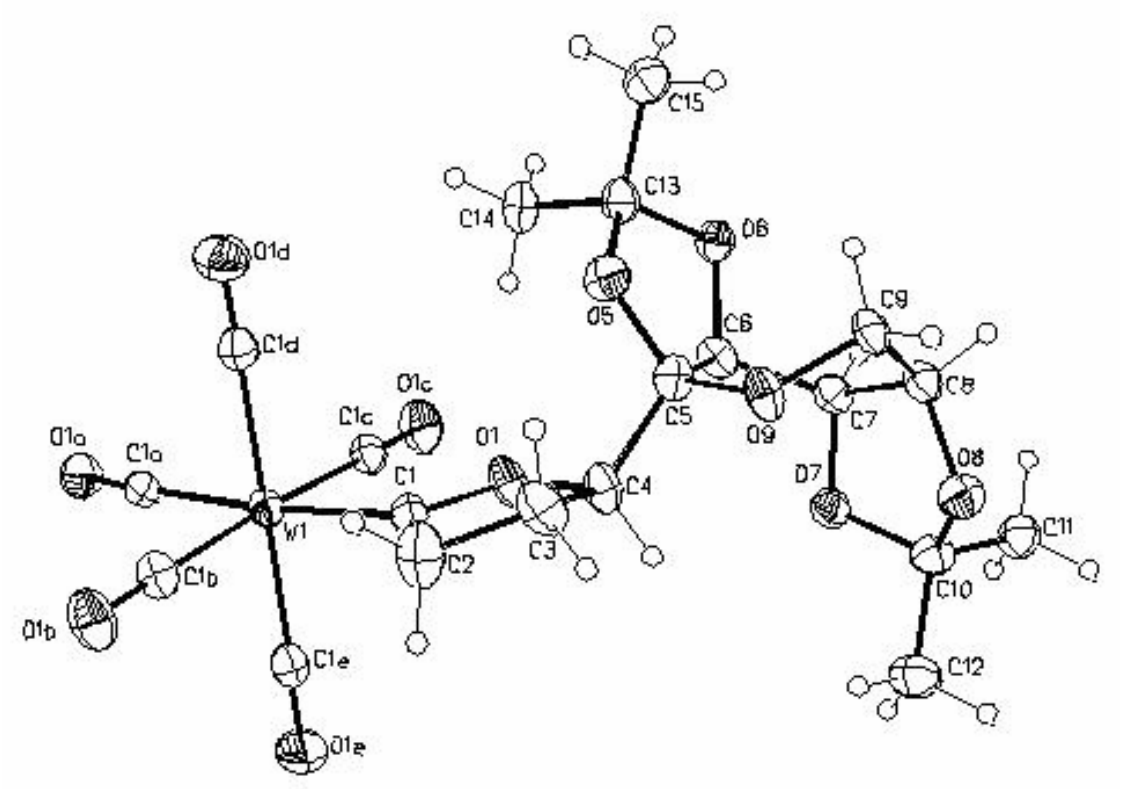
**Tabelle 6: Hydrogen bonds for *[16]R* [Å and °].**

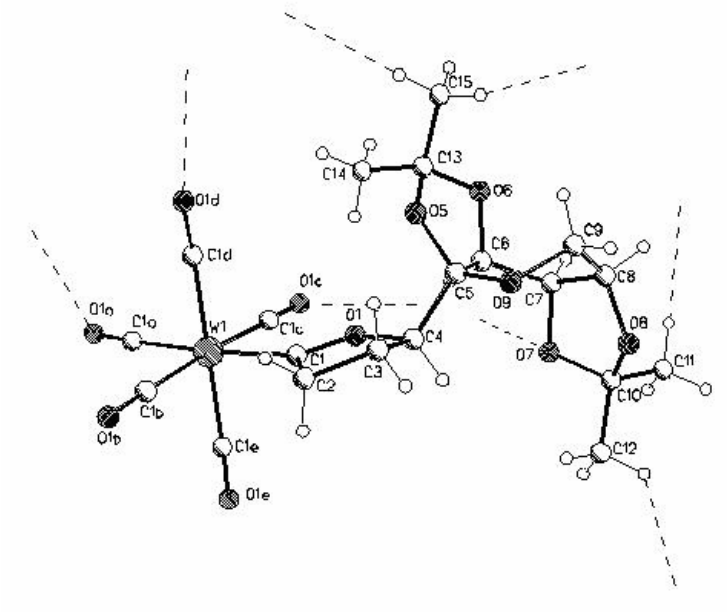
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(10')-H(10A)...O(3')[a] | 0.98 | 2.55 | 3.5299(18) | 177.3 |
| C(8')-H(8'3)...O(1A)[b] | 0.98 | 2.56 | 3.3173(19) | 134.0 |

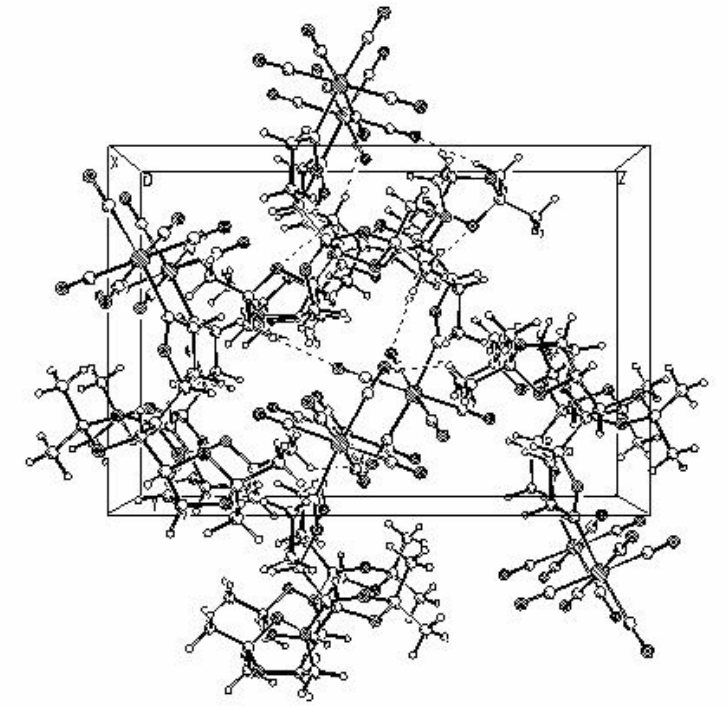
Symmetry transformations used to generate equivalent atoms:

[a] x-1/2,-y+3/2,-z+1 [b] -x+3/2,-y+1,z+1/2

**6. (*3R,1´R,2´S,6´S,9´R*)-Pentacarbonyl{3-(4´,4´,11´,11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]-dodec-6´-yl)-2-oxacyclopentyliden}wolfram(0) *[17]R***




C15
C13
C14
O6
O5
C9
C6
C8
C5
O9
C7
O8
O7
C11
C10
C12
O1d
C1d
O1c
O1a
C1a
W1
C1c
O1
C4
C1
C3
C2
C1b
O1b
C1e
O1e



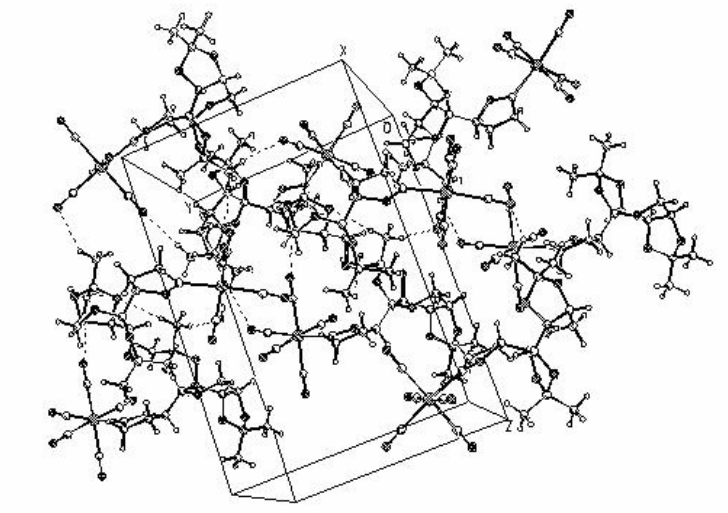


**Table 1: Crystal data and structure refinements for *[17]R***

| | |
|---|---|
| Identification code | ***[17]R*** |
| Empirical formula | $C_{20}H_{22}O_{11}W$ |
| Formula weight | 622.23 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 10. 3662 (2) Å α = 90° |
| | b = 12.2269(2) Å β = 90° |
| | c = 17.8766(4) Å γ = 90° |
| Volume | 2265.80(8) $Å^3$ |
| Z | 4 |
| Calculated density | 1.824 $mg/m^3$ |
| Absorption coefficient | 5.155 $mm^{-1}$ |
| F(000) | 1216 |
| Crystal size | 0.60 x 0.20 x 0.10 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.82 to 27.47° |
| Limiting indices | $-13 \le h \le 13$, $-15 \le k \le 15$, $-23 \le l \le 23$ |
| Reflections collected / unique | 44306 / 5185 [R(int) = 0.0711] |
| Completeness to Θ = 27.47 | 99.6 % |
| Absorption correction | Empirical from multiple refl |
| Max. and min. transmission | 0.3000 and 0.2023 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 5185 / 0 / 289 |
| Goodness-of-fit on $F^2$ | 1.005 |
| Final R indices [I>2σ(I)] | R1 = 0.0255, wR2 = 0.0567 |
| R indices (all data) | R1 = 0.0289, wR2 = 0.0577 |
| Absolute structure parameter | -0.028(7) |
| Largest diff. peak and hole | 1.228 and -1.687 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[17]R*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| W(1) | 3092(1) | 6840(1) | 5724(1) | 21(1) |
| C(1A) | 2462(4) | 8223(3) | 5191(2) | 24(1) |
| O(1A) | 2073(3) | 8958(2) | 4880(2) | 33(1) |
| C(1B) | 4763(5) | 7584(3) | 6064(3) | 28(1) |
| O(1B) | 5661(3) | 8022(2) | 6272(2) | 38(1) |
| C(1C) | 1393(4) | 6077(3) | 5438(2) | 25(1) |
| O(1C) | 464(3) | 5656(3) | 5278(2) | 37(1) |
| C(1D) | 3945(5) | 6352(3) | 4748(3) | 27(1) |
| O(1D) | 4441(3) | 6084(2) | 4213(2) | 41(1) |
| C(1E) | 2320(5) | 7340(4) | 6726(3) | 31(1) |
| O(1E) | 1938(4) | 7626(3) | 7287(2) | 49(1) |
| O(1) | 3023(3) | 4425(2) | 6164(2) | 27(1) |
| C(1) | 3733(4) | 5305(3) | 6199(2) | 23(1) |
| C(2) | 4955(5) | 5038(4) | 6587(3) | 39(1) |
| C(3) | 5040(4) | 3791(3) | 6601(3) | 31(1) |
| C(4) | 3643(4) | 3448(3) | 6495(3) | 25(1) |
| C(5) | 3440(4) | 2478(3) | 5974(2) | 23(1) |
| O(5) | 3981(3) | 2677(2) | 5263(2) | 27(1) |
| C(6) | 2019(4) | 2196(3) | 5796(2) | 22(1) |
| O(6) | 2080(3) | 1807(2) | 5051(2) | 24(1) |
| C(7) | 1413(4) | 1302(3) | 6263(2) | 23(1) |
| O(7) | 1018(3) | 1774(2) | 6962(2) | 26(1) |
| C(8) | 2357(4) | 372(3) | 6497(2) | 24(1) |
| O(8) | 2373(3) | 415(2) | 7292(2) | 28(1) |
| C(9) | 3697(4) | 558(3) | 6198(2) | 24(1) |
| O(9) | 4120(3) | 1643(2) | 6357(2) | 26(1) |
| C(10) | 1217(4) | 961(3) | 7523(2) | 27(1) |
| C(11) | 85(4) | 170(3) | 7554(3) | 30(1) |
| C(12) | 1457(5) | 1526(4) | 8253(3) | 36(1) |
| C(13) | 3054(5) | 2429(3) | 4682(2) | 25(1) |
| C(14) | 2500(5) | 3478(3) | 4369(3) | 31(1) |
| C(15) | 3664(4) | 1707(3) | 4097(2) | 29(1) |

**Tabelle 3: Bond lengths [Å] for *[17]R*.**

| | | | |
|---|---|---|---|
| W(1)-C(1D) | 2.045(5) | C(5)-O(9) | 1.417(5) |
| W(1)-C(1A) | 2.048(4) | C(5)-C(6) | 1.546(6) |
| W(1)-C(1B) | 2.049(5) | O(5)-C(13) | 1.447(5) |
| W(1)-C(1E) | 2.055(5) | C(6)-O(6) | 1.416(5) |
| W(1)-C(1C) | 2.057(5) | C(6)-C(7) | 1.512(6) |
| W(1)-C(1) | 2.165(4) | O(6)-C(13) | 1.425(5) |
| C(1A)-O(1A) | 1.131(5) | C(7)-O(7) | 1.434(5) |
| C(1B)-O(1B) | 1.137(5) | C(7)-C(8) | 1.557(5) |
| C(1C)-O(1C) | 1.128(5) | O(7)-C(10) | 1.427(5) |
| C(1D)-O(1D) | 1.134(5) | C(8)-O(8) | 1.422(5) |
| C(1E)-O(1E) | 1.134(5) | C(8)-C(9) | 1.506(6) |
| O(1)-C(1) | 1.305(5) | O(8)-C(10) | 1.432(5) |
| O(1)-C(4) | 1.480(5) | C(9)-O(9) | 1.425(5) |
| C(1)-C(2) | 1.481(6) | C(10)-C(12) | 1.497(6) |
| C(2)-C(3) | 1.526(6) | C(10)-C(11) | 1.521(6) |
| C(3)-C(4) | 1.519(6) | C(13)-C(15) | 1.508(6) |
| C(4)-C(5) | 1.522(6) | C(13)-C(14) | 1.513(6) |
| C(5)-O(5) | 1.411(5) | | |

**Tabelle 4: Bond angles [°] for *[17]R*.**

| | | | |
|---|---|---|---|
| C(1D)-W(1)-C(1A) | 88.97(17) | O(9)-C(5)-C(4) | 101.4(3) |
| C(1D)-W(1)-C(1B) | 91.00(19) | O(5)-C(5)-C(6) | 103.4(3) |
| C(1A)-W(1)-C(1B) | 92.37(16) | O(9)-C(5)-C(6) | 114.3(3) |
| C(1D)-W(1)-C(1E) | 177.3(2) | C(4)-C(5)-C(6) | 115.6(3) |
| C(1A)-W(1)-C(1E) | 92.04(17) | C(5)-O(5)-C(13) | 110.3(3) |
| C(1B)-W(1)-C(1E) | 86.43(18) | O(6)-C(6)-C(7) | 107.2(3) |
| C(1D)-W(1)-C(1C) | 91.52(18) | O(6)-C(6)-C(5) | 103.0(3) |
| C(1A)-W(1)-C(1C) | 89.20(16) | C(7)-C(6)-C(5) | 116.3(3) |
| C(1B)-W(1)-C(1C) | 177.05(18) | C(6)-O(6)-C(13) | 106.8(3) |
| C(1E)-W(1)-C(1C) | 91.02(18) | O(7)-C(7)-C(6) | 108.0(3) |
| C(1D)-W(1)-C(1) | 87.07(16) | O(7)-C(7)-C(8) | 103.9(3) |
| C(1A)-W(1)-C(1) | 175.15(16) | C(6)-C(7)-C(8) | 114.5(3) |
| C(1B)-W(1)-C(1) | 90.50(16) | C(10)-O(7)-C(7) | 106.9(3) |
| C(1E)-W(1)-C(1) | 92.04(17) | O(8)-C(8)-C(9) | 109.7(3) |
| C(1C)-W(1)-C(1) | 88.10(16) | O(8)-C(8)-C(7) | 104.3(3) |
| O(1A)-C(1A)-W(1) | 176.9(4) | C(9)-C(8)-C(7) | 112.0(3) |
| O(1B)-C(1B)-W(1) | 177.2(4) | C(8)-O(8)-C(10) | 107.2(3) |
| O(1C)-C(1C)-W(1) | 179.5(4) | O(9)-C(9)-C(8) | 110.7(3) |
| O(1D)-C(1D)-W(1) | 178.7(4) | C(5)-O(9)-C(9) | 114.9(3) |
| O(1E)-C(1E)-W(1) | 177.6(4) | O(7)-C(10)-O(8) | 104.0(3) |
| C(1)-O(1)-C(4) | 113.6(3) | O(7)-C(10)-C(12) | 108.4(3) |
| O(1)-C(1)-C(2) | 108.9(3) | O(8)-C(10)-C(12) | 109.1(4) |
| O(1)-C(1)-W(1) | 121.5(3) | O(7)-C(10)-C(11) | 110.9(4) |
| C(2)-C(1)-W(1) | 129.6(3) | O(8)-C(10)-C(11) | 111.1(3) |
| C(1)-C(2)-C(3) | 106.1(4) | C(12)-C(10)-C(11) | 112.9(4) |
| C(4)-C(3)-C(2) | 102.6(4) | O(6)-C(13)-O(5) | 104.5(3) |
| O(1)-C(4)-C(3) | 104.0(3) | O(6)-C(13)-C(15) | 107.8(3) |
| O(1)-C(4)-C(5) | 108.9(3) | O(5)-C(13)-C(15) | 110.0(4) |
| C(3)-C(4)-C(5) | 115.1(4) | O(6)-C(13)-C(14) | 110.8(4) |
| O(5)-C(5)-O(9) | 111.3(3) | O(5)-C(13)-C(14) | 109.9(3) |
| O(5)-C(5)-C(4) | 111.2(3) | C(15)-C(13)-C(14) | 113.5(4) |

**Tabelle 5: Torsion angles [°] for *[17]R*.**

| | | | |
|---|---|---|---|
| C(1D)-W(1)-C(1A)-O(1A) | 74(7) | O(1)-C(4)-C(5)-O(9) | 178.1(3) |
| C(1B)-W(1)-C(1A)-O(1A) | 165(7) | C(3)-C(4)-C(5)-O(9) | 61.9(4) |
| C(1E)-W(1)-C(1A)-O(1A) | -108(7) | O(1)-C(4)-C(5)-C(6) | -57.7(5) |
| C(1C)-W(1)-C(1A)-O(1A) | -17(7) | C(3)-C(4)-C(5)-C(6) | -173.9(4) |
| C(1)-W(1)-C(1A)-O(1A) | 39(8) | O(9)-C(5)-O(5)-C(13) | 117.2(3) |
| C(1D)-W(1)-C(1B)-O(1B) | 158(8) | C(4)-C(5)-O(5)-C(13) | -130.5(3) |
| C(1A)-W(1)-C(1B)-O(1B) | 69(8) | C(6)-C(5)-O(5)-C(13) | -5.9(4) |
| C(1E)-W(1)-C(1B)-O(1B) | -23(8) | O(5)-C(5)-C(6)-O(6) | 24.8(4) |
| C(1C)-W(1)-C(1B)-O(1B) | -53(10) | O(9)-C(5)-C(6)-O(6) | -96.3(4) |
| C(1)-W(1)-C(1B)-O(1B) | -115(8) | C(4)-C(5)-C(6)-O(6) | 146.5(3) |
| C(1D)-W(1)-C(1C)-O(1C) | 2(66) | O(5)-C(5)-C(6)-C(7) | 141.8(3) |
| C(1A)-W(1)-C(1C)-O(1C) | 91(66) | O(9)-C(5)-C(6)-C(7) | 20.6(5) |
| C(1B)-W(1)-C(1C)-O(1C) | -146(64) | C(4)-C(5)-C(6)-C(7) | -96.5(4) |
| C(1E)-W(1)-C(1C)-O(1C) | -177(100) | C(7)-C(6)-O(6)-C(13) | -158.3(3) |
| C(1)-W(1)-C(1C)-O(1C) | -85(66) | C(5)-C(6)-O(6)-C(13) | -35.0(4) |
| C(1A)-W(1)-C(1D)-O(1D) | 112(16) | O(6)-C(6)-C(7)-O(7) | -164.4(3) |
| C(1B)-W(1)-C(1D)-O(1D) | 20(16) | C(5)-C(6)-C(7)-O(7) | 81.0(4) |
| C(1E)-W(1)-C(1D)-O(1D) | 0(18) | O(6)-C(6)-C(7)-C(8) | 80.5(4) |
| C(1C)-W(1)-C(1D)-O(1D) | -159(16) | C(5)-C(6)-C(7)-C(8) | -34.2(5) |
| C(1)-W(1)-C(1D)-O(1D) | -71(16) | C(6)-C(7)-O(7)-C(10) | -144.6(3) |
| C(1D)-W(1)-C(1E)-O(1E) | 7(12) | C(8)-C(7)-O(7)-C(10) | -22.6(4) |
| C(1A)-W(1)-C(1E)-O(1E) | -104(9) | O(7)-C(7)-C(8)-O(8) | 0.7(4) |
| C(1B)-W(1)-C(1E)-O(1E) | -12(9) | C(6)-C(7)-C(8)-O(8) | 118.2(4) |
| C(1C)-W(1)-C(1E)-O(1E) | 166(9) | O(7)-C(7)-C(8)-C(9) | -117.9(4) |
| C(1)-W(1)-C(1E)-O(1E) | 78(9) | C(6)-C(7)-C(8)-C(9) | -0.4(5) |
| C(4)-O(1)-C(1)-C(2) | -1.4(5) | C(9)-C(8)-O(8)-C(10) | 141.5(3) |
| C(4)-O(1)-C(1)-W(1) | 177.9(3) | C(7)-C(8)-O(8)-C(10) | 21.4(4) |
| C(1D)-W(1)-C(1)-O(1) | -89.3(4) | O(8)-C(8)-C(9)-O(9) | -66.0(4) |
| C(1A)-W(1)-C(1)-O(1) | -54(2) | C(7)-C(8)-C(9)-O(9) | 49.4(5) |
| C(1B)-W(1)-C(1)-O(1) | 179.8(4) | O(5)-C(5)-O(9)-C(9) | -86.6(4) |
| C(1E)-W(1)-C(1)-O(1) | 93.3(4) | C(4)-C(5)-O(9)-C(9) | 155.1(3) |
| C(1C)-W(1)-C(1)-O(1) | 2.4(4) | C(6)-C(5)-O(9)-C(9) | 30.1(5) |
| C(1D)-W(1)-C(1)-C(2) | 89.8(4) | C(8)-C(9)-O(9)-C(5) | -67.3(4) |
| C(1A)-W(1)-C(1)-C(2) | 125.2(19) | C(7)-O(7)-C(10)-O(8) | 36.4(4) |
| C(1B)-W(1)-C(1)-C(2) | -1.1(4) | C(7)-O(7)-C(10)-C(12) | 152.4(4) |
| C(1E)-W(1)-C(1)-C(2) | -87.6(4) | C(7)-O(7)-C(10)-C(11) | -83.1(4) |
| C(1C)-W(1)-C(1)-C(2) | -178.5(4) | C(8)-O(8)-C(10)-O(7) | -36.0(4) |
| O(1)-C(1)-C(2)-C(3) | 14.8(5) | C(8)-O(8)-C(10)-C(12) | -151.5(3) |
| W(1)-C(1)-C(2)-C(3) | -164.4(3) | C(8)-O(8)-C(10)-C(11) | 83.4(4) |
| C(1)-C(2)-C(3)-C(4) | -21.3(5) | C(6)-O(6)-C(13)-O(5) | 31.8(4) |
| C(1)-O(1)-C(4)-C(3) | -12.5(5) | C(6)-O(6)-C(13)-C(15) | 148.8(3) |
| C(1)-O(1)-C(4)-C(5) | -135.7(4) | C(6)-O(6)-C(13)-C(14) | -86.5(4) |
| C(2)-C(3)-C(4)-O(1) | 20.0(5) | C(5)-O(5)-C(13)-O(6) | -15.0(4) |
| C(2)-C(3)-C(4)-C(5) | 139.0(4) | C(5)-O(5)-C(13)-C(15) | -130.5(3) |
| O(1)-C(4)-C(5)-O(5) | 59.8(4) | C(5)-O(5)-C(13)-C(14) | 103.9(4) |
| C(3)-C(4)-C(5)-O(5) | -56.5(5) | | |

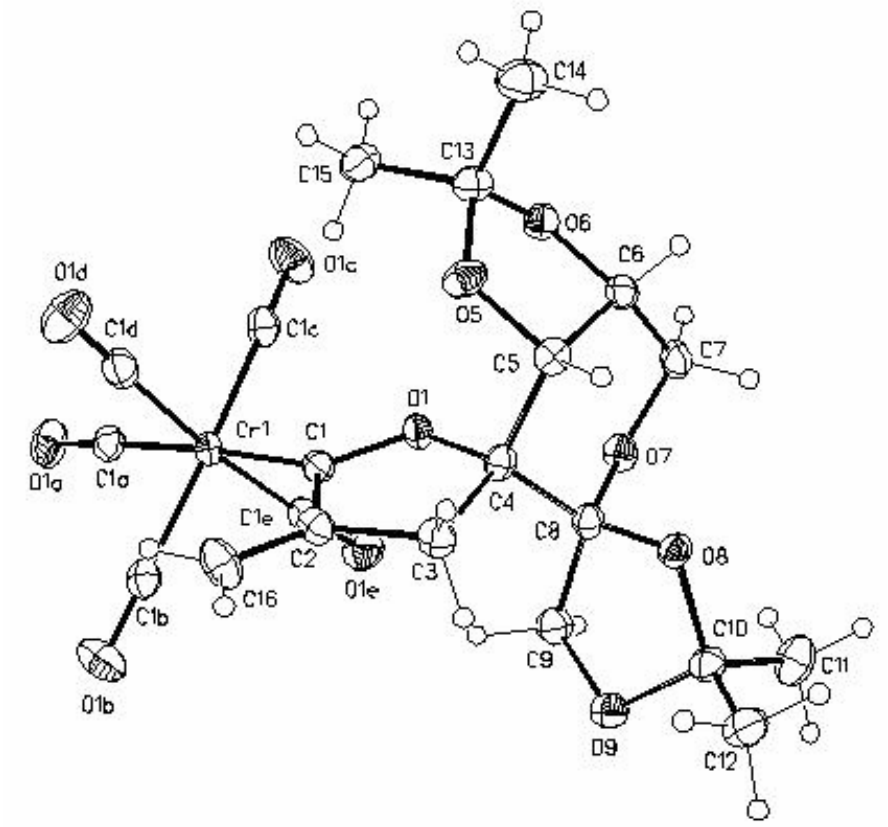
**Tabelle 6: Hydrogen bonds for *[17]R* [Å and °].**

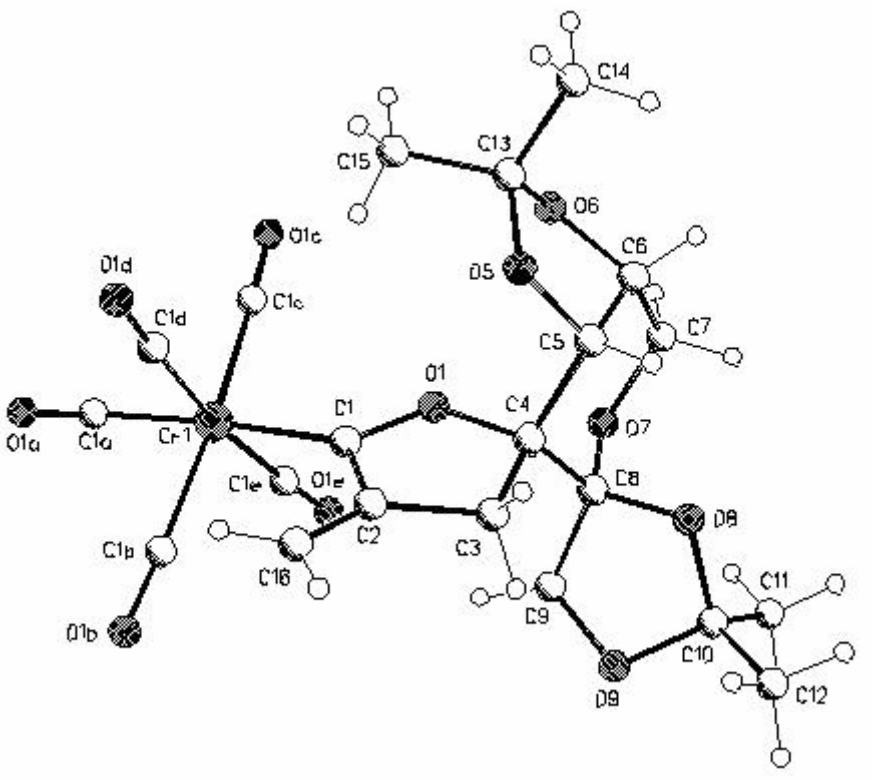
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(12)-H(12A)...O(1A)[a] | 0.98 | 2.60 | 3.337(6) | 131.6 |
| C(15)-H(15C)...O(1C)[b] | 0.98 | 2.66 | 3.616(5) | 166.4 |
| C(15)-H(15A)...O(7)[b] | 0.98 | 2.63 | 3.604(5) | 175.4 |
| C(11)-H(11B)...O(1D)[b] | 0.98 | 2.60 | 3.574(6) | 172.8 |

Symmetry transformations used to generate equivalent atoms:

[a] -x+1/2,-y+1,z+1/2 [b] x+1/2,-y+1/2,-z+1 [c] x-1/2,-y+1/2,-z+1

**7. (*3R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-5-methylen-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,3-2-oxacyclopent]-1-yliden}chrom(0) *[28]***




C14
C13
C15
O6
O1c
C6
O1d
O5
C1c
C1d
C7
C5
O1
C1
C4
O1a
C1a
Cr1
O7
C1e
O1e
C8
O8
C2
C3
C1b
C16
C11
C9
O1b
C10
O9
C12

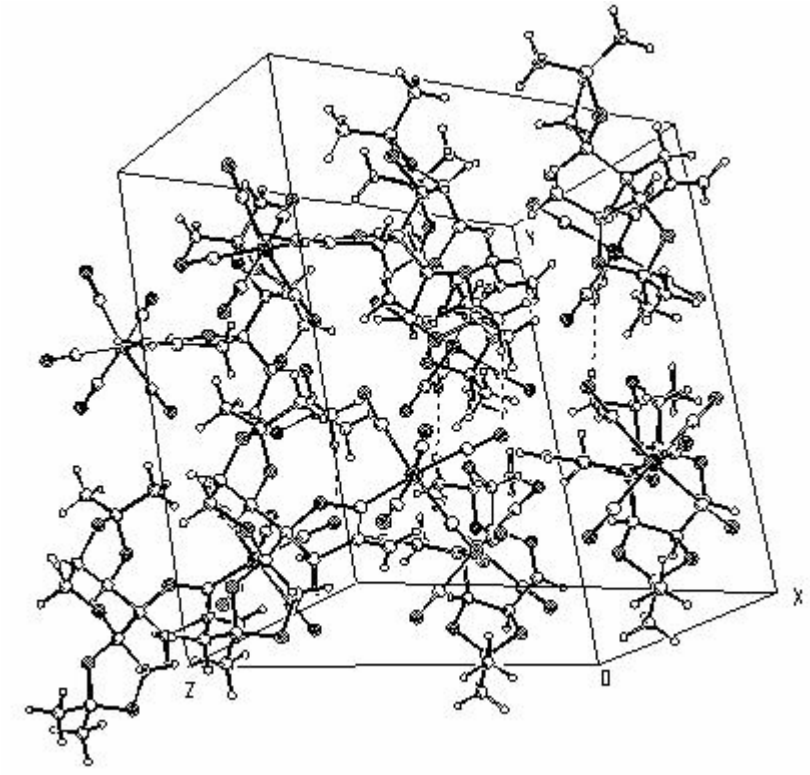

Y
X
Z
0

**Table 1: Crystal data and structure refinements for *[28]***

| | |
|---|---|
| Identification code | ***[28]*** |
| Empirical formula | $C_{21}H_{22}CrO_{11}$ |
| Formula weight | 502.39 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 8.5484(1) Å α = 90° |
| | b = 16.0671(3) Å β = 90° |
| | c = 16.3630(4) Å γ = 90° |
| Volume | 2247.43(7) $Å^3$ |
| Z | 4 |
| Calculated density | 1.485 $mg/m^3$ |
| Absorption coefficient | 0.568 $mm^{-1}$ |
| F(000) | 1040 |
| Crystal size | 0.55 x 0.45 x 0.25 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.69 to 24.99° |
| Limiting indices | $-9 \le h \le 10$, $-19 \le k \le 19$, $-19 \le l \le 19$ |
| Reflections collected / unique | 19752 / 3938 [R(int) = 0.0345] |
| Completeness to Θ = 25.00 | 99.6 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 3938 / 0 / 298 |
| Goodness-of-fit on $F^2$ | 1.037 |
| Final R indices [I>2σ(I)] | R1 = 0.0207, wR2 = 0.0536 |
| R indices (all data) | R1 = 0.0222, wR2 = 0.0541 |
| Absolute structure parameter | -0.023(11) |
| Largest diff. peak and hole | 0.152 and -0.215 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[28]*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 3713(1) | 7737(1) | 8254(1) | 16(1) |
| C(1A) | 1734(2) | 7916(1) | 8731(1) | 22(1) |
| O(1A) | 558(2) | 8001(1) | 9050(1) | 33(1) |
| C(1B) | 3840(2) | 6843(1) | 9009(1) | 22(1) |
| O(1B) | 3806(2) | 6302(1) | 9463(1) | 33(1) |
| C(1C) | 3236(2) | 8617(1) | 7500(1) | 24(1) |
| O(1C) | 2826(2) | 9117(1) | 7061(1) | 38(1) |
| C(1D) | 4684(2) | 8513(1) | 8988(1) | 25(1) |
| O(1D) | 5285(2) | 8971(1) | 9413(1) | 40(1) |
| C(1E) | 2977(2) | 7014(1) | 7429(1) | 21(1) |
| O(1E) | 2584(2) | 6595(1) | 6899(1) | 32(1) |
| O(1) | 6206(1) | 7576(1) | 7029(1) | 17(1) |
| C(1) | 5877(2) | 7472(1) | 7814(1) | 18(1) |
| C(2) | 7251(2) | 7085(1) | 8204(1) | 19(1) |
| C(3) | 8419(2) | 6853(1) | 7551(1) | 22(1) |
| C(4) | 7760(2) | 7255(1) | 6781(1) | 17(1) |
| C(5) | 8774(2) | 7970(1) | 6458(1) | 19(1) |
| O(5) | 8725(1) | 8656(1) | 7008(1) | 23(1) |
| C(6) | 8234(2) | 8328(1) | 5642(1) | 20(1) |
| O(6) | 7198(1) | 8981(1) | 5908(1) | 21(1) |
| C(7) | 7455(2) | 7722(1) | 5066(1) | 23(1) |
| O(7) | 6585(1) | 7066(1) | 5457(1) | 20(1) |
| C(8) | 7431(2) | 6648(1) | 6076(1) | 18(1) |
| O(8) | 8906(1) | 6361(1) | 5817(1) | 19(1) |
| C(9) | 6521(2) | 5859(1) | 6293(1) | 23(1) |
| O(9) | 7663(1) | 5211(1) | 6275(1) | 25(1) |
| C(10) | 8835(2) | 5464(1) | 5712(1) | 21(1) |
| C(11) | 8382(2) | 5265(1) | 4834(1) | 31(1) |
| C(12) | 10379(2) | 5103(1) | 5965(1) | 27(1) |
| C(13) | 7942(2) | 9343(1) | 6603(1) | 22(1) |
| C(14) | 9150(2) | 9986(1) | 6367(1) | 32(1) |
| C(15) | 6685(2) | 9683(1) | 7164(1) | 26(1) |
| C(16) | 7453(2) | 6991(1) | 9005(1) | 28(1) |

**Tabelle 3: Bond lengths [Å] for *[28]*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1A) | 1.8856(19) | C(4)-C(8) | 1.538(2) |
| Cr(1)-C(1E) | 1.8897(19) | C(5)-O(5) | 1.4227(19) |
| Cr(1)-C(1B) | 1.8971(18) | C(5)-C(6) | 1.525(2) |
| Cr(1)-C(1D) | 1.919(2) | O(5)-C(13) | 1.452(2) |
| Cr(1)-C(1C) | 1.921(2) | C(6)-O(6) | 1.440(2) |
| Cr(1)-C(1) | 2.0298(17) | C(6)-C(7) | 1.510(2) |
| C(1A)-O(1A) | 1.140(2) | O(6)-C(13) | 1.427(2) |
| C(1B)-O(1B) | 1.143(2) | C(7)-O(7) | 1.439(2) |
| C(1C)-O(1C) | 1.133(2) | O(7)-C(8) | 1.414(2) |
| C(1D)-O(1D) | 1.136(2) | C(8)-O(8) | 1.407(2) |
| C(1E)-O(1E) | 1.148(2) | C(8)-C(9) | 1.529(2) |
| O(1)-C(1) | 1.3246(19) | O(8)-C(10) | 1.453(2) |
| O(1)-C(4) | 1.4823(18) | C(9)-O(9) | 1.427(2) |
| C(1)-C(2) | 1.475(2) | O(9)-C(10) | 1.420(2) |
| C(2)-C(16) | 1.330(2) | C(10)-C(12) | 1.500(3) |
| C(2)-C(3) | 1.508(2) | C(10)-C(11) | 1.521(2) |
| C(3)-C(4) | 1.524(2) | C(13)-C(14) | 1.510(3) |
| C(4)-C(5) | 1.533(2) | C(13)-C(15) | 1.515(2) |

**Tabelle 4: Bond angles [°] for *[28]*.**

| | | | |
|---|---|---|---|
| C(1A)-Cr(1)-C(1E | 95.20(8) | C(5)-C(4)-C(8 | 108.69(13) |
| C(1A)-Cr(1)-C(1B | 84.10(8) | O(5)-C(5)-C(6 | 104.63(13) |
| C(1E)-Cr(1)-C(1B | 91.09(7) | O(5)-C(5)-C(4 | 110.21(13) |
| C(1A)-Cr(1)-C(1D | 91.73(8) | C(6)-C(5)-C(4 | 114.41(14) |
| C(1E)-Cr(1)-C(1D | 172.07(7) | C(5)-O(5)-C(13 | 108.30(12) |
| C(1B)-Cr(1)-C(1D | 93.46(8) | O(6)-C(6)-C(7 | 112.76(14) |
| C(1A)-Cr(1)-C(1C | 87.89(7) | O(6)-C(6)-C(5 | 101.35(13) |
| C(1E)-Cr(1)-C(1C | 85.54(7) | C(7)-C(6)-C(5 | 115.88(14) |
| C(1B)-Cr(1)-C(1C | 171.00(8) | C(13)-O(6)-C(6 | 105.28(12) |
| C(1D)-Cr(1)-C(1C | 90.88(8) | O(7)-C(7)-C(6 | 115.00(13) |
| C(1A)-Cr(1)-C(1 | 175.26(7) | C(8)-O(7)-C(7 | 113.72(12) |
| C(1E)-Cr(1)-C(1 | 85.47(7) | O(8)-C(8)-O(7 | 113.47(13) |
| C(1B)-Cr(1)-C(1 | 91.20(7) | O(8)-C(8)-C(9 | 104.74(13) |
| C(1D)-Cr(1)-C(1 | 87.95(7) | O(7)-C(8)-C(9 | 107.53(13) |
| C(1C)-Cr(1)-C(1 | 96.84(7) | O(8)-C(8)-C(4 | 105.60(12) |
| O(1A)-C(1A)-Cr(1 | 176.78(16) | O(7)-C(8)-C(4 | 109.21(13) |
| O(1B)-C(1B)-Cr(1 | 175.29(17) | C(9)-C(8)-C(4 | 116.41(14) |
| O(1C)-C(1C)-Cr(1 | 174.18(16) | C(8)-O(8)-C(10 | 108.81(13) |
| O(1D)-C(1D)-Cr(1 | 178.67(16) | O(9)-C(9)-C(8 | 104.58(13) |
| O(1E)-C(1E)-Cr(1 | 176.34(15) | C(10)-O(9)-C(9 | 106.71(12) |
| C(1)-O(1)-C(4 | 114.35(12) | O(9)-C(10)-O(8 | 103.70(13) |
| O(1)-C(1)-C(2 | 107.67(14) | O(9)-C(10)-C(12 | 109.32(14) |
| O(1)-C(1)-Cr(1 | 120.77(11) | O(8)-C(10)-C(12 | 108.32(15) |
| C(2)-C(1)-Cr(1 | 131.30(12) | O(9)-C(10)-C(11 | 111.87(14) |
| C(16)-C(2)-C(1 | 125.32(16) | O(8)-C(10)-C(11 | 109.33(14) |
| C(16)-C(2)-C(3 | 125.69(16) | C(12)-C(10)-C(11 | 113.76(15) |
| C(1)-C(2)-C(3 | 108.93(14) | O(6)-C(13)-O(5 | 104.99(13) |
| C(2)-C(3)-C(4 | 103.71(13) | O(6)-C(13)-C(14 | 112.36(15) |
| O(1)-C(4)-C(3 | 104.58(12) | O(5)-C(13)-C(14 | 108.77(14) |
| O(1)-C(4)-C(5 | 109.91(12) | O(6)-C(13)-C(15 | 108.26(14) |
| C(3)-C(4)-C(5 | 113.20(13) | O(5)-C(13)-C(15 | 108.94(14) |
| O(1)-C(4)-C(8 | 105.18(12) | C(14)-C(13)-C(15 | 113.16(15) |
| C(3)-C(4)-C(8 | 114.84(14) | | |

**Tabelle 5: Torsion angles [°] for *[28]*.**

| | | | |
|---|---|---|---|
| C(1E)-Cr(1)-C(1A)-O(1A) | 107(3) | C(2)-C(3)-C(4)-O(1) | -7.25(16) |
| C(1B)-Cr(1)-C(1A)-O(1A) | 16(3) | C(2)-C(3)-C(4)-C(5) | 112.37(15) |
| C(1D)-Cr(1)-C(1A)-O(1A) | -77(3) | C(2)-C(3)-C(4)-C(8) | -122.00(14) |
| C(1C)-Cr(1)-C(1A)-O(1A) | -168(3) | O(1)-C(4)-C(5)-O(5) | 48.34(17) |
| C(1)-Cr(1)-C(1A)-O(1A) | 9(4) | C(3)-C(4)-C(5)-O(5) | -68.18(17) |
| C(1A)-Cr(1)-C(1B)-O(1B) | 27.0(18) | C(8)-C(4)-C(5)-O(5) | 162.96(12) |
| C(1E)-Cr(1)-C(1B)-O(1B) | -68.1(18) | O(1)-C(4)-C(5)-C(6) | -69.21(17) |
| C(1D)-Cr(1)-C(1B)-O(1B) | 118.4(18) | C(3)-C(4)-C(5)-C(6) | 174.27(14) |
| C(1C)-Cr(1)-C(1B)-O(1B) | 0(2) | C(8)-C(4)-C(5)-C(6) | 45.41(17) |
| C(1)-Cr(1)-C(1B)-O(1B) | -153.6(18) | C(6)-C(5)-O(5)-C(13) | 9.71(17) |
| C(1A)-Cr(1)-C(1C)-O(1C) | -30.6(16) | C(4)-C(5)-O(5)-C(13) | -113.74(14) |
| C(1E)-Cr(1)-C(1C)-O(1C) | 64.8(16) | O(5)-C(5)-C(6)-O(6) | -29.90(16) |
| C(1B)-Cr(1)-C(1C)-O(1C) | -3(2) | C(4)-C(5)-C(6)-O(6) | 90.80(15) |
| C(1D)-Cr(1)-C(1C)-O(1C) | -122.3(16) | O(5)-C(5)-C(6)-C(7) | -152.30(14) |
| C(1)-Cr(1)-C(1C)-O(1C) | 149.7(16) | C(4)-C(5)-C(6)-C(7) | -31.6(2) |
| C(1A)-Cr(1)-C(1D)-O(1D) | -173(100) | C(7)-C(6)-O(6)-C(13) | 164.02(13) |
| C(1E)-Cr(1)-C(1D)-O(1D) | -22(8) | C(5)-C(6)-O(6)-C(13) | 39.48(15) |
| C(1B)-Cr(1)-C(1D)-O(1D) | 102(8) | O(6)-C(6)-C(7)-O(7) | -84.34(17) |
| C(1C)-Cr(1)-C(1D)-O(1D) | -85(8) | C(5)-C(6)-C(7)-O(7) | 31.8(2) |
| C(1)-Cr(1)-C(1D)-O(1D) | 11(8) | C(6)-C(7)-O(7)-C(8) | -49.83(18) |
| C(1A)-Cr(1)-C(1E)-O(1E) | 141(2) | C(7)-O(7)-C(8)-O(8) | -51.98(17) |
| C(1B)-Cr(1)-C(1E)-O(1E) | -135(2) | C(7)-O(7)-C(8)-C(9) | -167.32(13) |
| C(1D)-Cr(1)-C(1E)-O(1E) | -10(3) | C(7)-O(7)-C(8)-C(4) | 65.52(16) |
| C(1C)-Cr(1)-C(1E)-O(1E) | 53(2) | O(1)-C(4)-C(8)-O(8) | 178.03(12) |
| C(1)-Cr(1)-C(1E)-O(1E) | -44(2) | C(3)-C(4)-C(8)-O(8) | -67.58(17) |
| C(4)-O(1)-C(1)-C(2) | 2.27(17) | C(5)-C(4)-C(8)-O(8) | 60.36(16) |
| C(4)-O(1)-C(1)-Cr(1) | -172.47(10) | O(1)-C(4)-C(8)-O(7) | 55.67(15) |
| C(1A)-Cr(1)-C(1)-O(1) | 152.2(8) | C(3)-C(4)-C(8)-O(7) | 170.07(13) |
| C(1E)-Cr(1)-C(1)-O(1) | 53.91(12) | C(5)-C(4)-C(8)-O(7) | -62.00(16) |
| C(1B)-Cr(1)-C(1)-O(1) | 144.90(13) | O(1)-C(4)-C(8)-C(9) | -66.27(17) |
| C(1D)-Cr(1)-C(1)-O(1) | -121.68(13) | C(3)-C(4)-C(8)-C(9) | 48.13(19) |
| C(1C)-Cr(1)-C(1)-O(1) | -31.04(13) | C(5)-C(4)-C(8)-C(9) | 176.06(13) |
| C(1A)-Cr(1)-C(1)-C(2) | -21.1(10) | O(7)-C(8)-O(8)-C(10) | -105.89(14) |
| C(1E)-Cr(1)-C(1)-C(2) | -119.41(16) | C(9)-C(8)-O(8)-C(10) | 11.09(16) |
| C(1B)-Cr(1)-C(1)-C(2) | -28.42(16) | C(4)-C(8)-O(8)-C(10) | 134.53(13) |
| C(1D)-Cr(1)-C(1)-C(2) | 65.00(16) | O(8)-C(8)-C(9)-O(9) | 10.03(17) |
| C(1C)-Cr(1)-C(1)-C(2) | 155.64(15) | O(7)-C(8)-C(9)-O(9) | 131.03(13) |
| O(1)-C(1)-C(2)-C(16) | 170.06(17) | C(4)-C(8)-C(9)-O(9) | -106.15(15) |
| Cr(1)-C(1)-C(2)-C(16) | -16.0(3) | C(8)-C(9)-O(9)-C(10) | -27.89(17) |
| O(1)-C(1)-C(2)-C(3) | -7.17(18) | C(9)-O(9)-C(10)-O(8) | 34.66(16) |
| Cr(1)-C(1)-C(2)-C(3) | 166.81(12) | C(9)-O(9)-C(10)-C(12) | 150.02(14) |
| C(16)-C(2)-C(3)-C(4) | -168.34(17) | C(9)-O(9)-C(10)-C(11) | -83.05(16) |
| C(1)-C(2)-C(3)-C(4) | 8.88(18) | C(8)-O(8)-C(10)-O(9) | -28.29(16) |
| C(1)-O(1)-C(4)-C(3) | 3.38(17) | C(8)-O(8)-C(10)-C(12) | -144.35(14) |
| C(1)-O(1)-C(4)-C(5) | -118.42(14) | C(8)-O(8)-C(10)-C(11) | 91.18(16) |
| C(1)-O(1)-C(4)-C(8) | 124.74(13) | C(6)-O(6)-C(13)-O(5) | -34.50(16) |

| | | | |
|---|---|---|---|
| C(6)-O(6)-C(13)-C(14) | 83.56(16) | C(5)-O(5)-C(13)-C(14) | -105.91(16) |
| C(6)-O(6)-C(13)-C(15) | -150.74(14) | C(5)-O(5)-C(13)-C(15) | 130.31(15) |
| C(5)-O(5)-C(13)-O(6) | 14.54(17) | | |

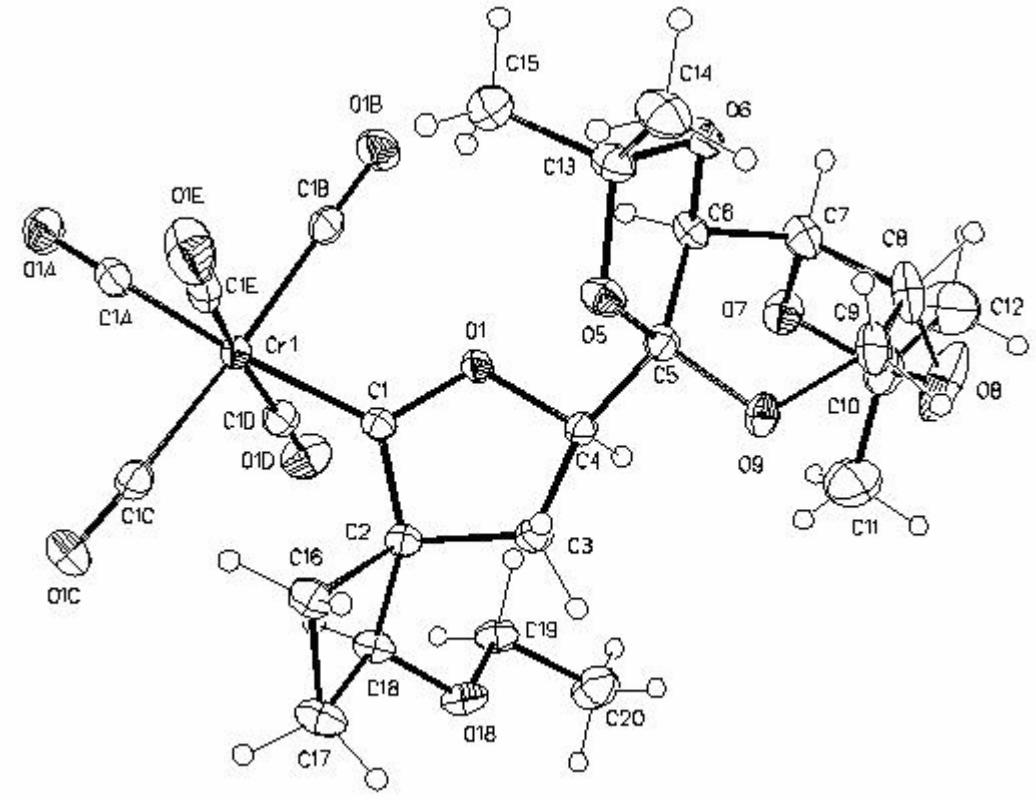
**Tabelle 6: Hydrogen bonds for *[28]* [Å and °].**

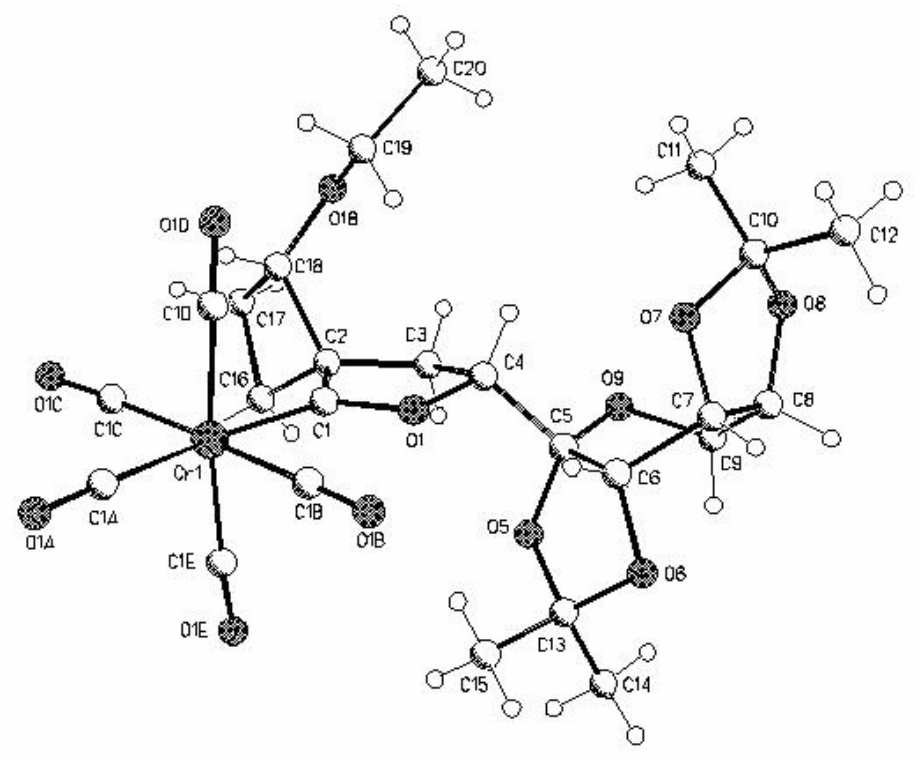
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(12)-H(12B)...O(1A)[a] | 0.98 | 2.51 | 3.471(2) | 166.4 |

Symmetry transformations used to generate equivalent atoms:

[a] -x+1,y-1/2,-z+3/2

**8. (*1S,4R,7R,1´R,2´S,6´S,9´R*)-Pentacarbonyl{1-ethoxy-7-(4´,4´,11´,-11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´-yl)-6-oxaspiro[3.4]oct-5-yliden}chrom(0) *[42]a***




C20
C19
O1B
C18
O1D
C17
C16
C2
C3
C4
O1C
C1C
C1
O1
C5
O9
C7
C8
C11
C10
C12
O7
O8
C6
C9
Cr1
C1A
C1B
O1A
O1B
O5
O6
C1E
O1E
C13
C15
C14

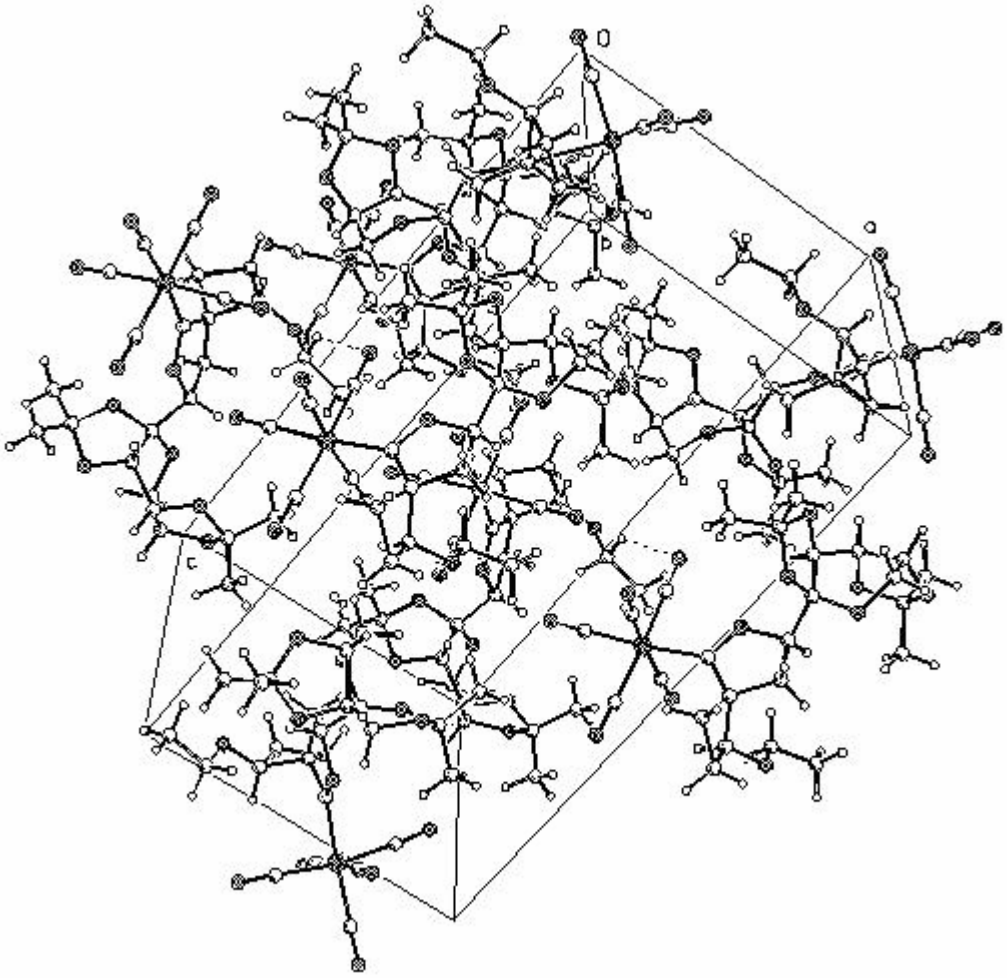

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**Table 1: Crystal data and structure refinements for *[42]a***

| | |
|---|---|
| Identification code | ***[42]a*** |
| Empirical formula | $C_{25}H_{30}CrO_{12}$ |
| Formula weight | 574.49 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 11.1732(1) Å α = 90° |
| | b = 11.4373(1) Å β = 90° |
| | c = 20.9274(2) Å γ = 90° |
| Volume | 2674.34(4) $Å^3$ |
| Z | 4 |
| Calculated density | 1.427 $mg/m^3$ |
| Absorption coefficient | 0.572 $mm^{-1}$ |
| F(000) | 1200 |
| Crystal size | 0.40 x 0.30 x 0.20 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 3.21 to 27.48 ° |
| Limiting indices | $-11 \le h \le 14$, $-14 \le k \le 14$, $-27 \le l \le 27$ |
| Reflections collected / unique | 25872 / 6081 [R(int) = 0.0315] |
| Completeness to Θ = 25.00 | 99.6 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 6081 / 0 / 343 |
| Goodness-of-fit on $F^2$ | 1.024 |
| Final R indices [I>2σ(I)] | R1 = 0.0224, wR2 = 0.0596 |
| R indices (all data) | R1 = 0.0234, wR2 = 0.0601 |
| Absolute structure parameter | -0.005(9) |
| Largest diff. peak and hole | 0.251 and -0.279 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[42]a*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 3952(1) | 5861(1) | 5103(1) | 17(1) |
| C(1A) | 2797(1) | 6648(1) | 4599(1) | 26(1) |
| O(1A) | 2106(1) | 7154(1) | 4304(1) | 38(1) |
| C(1B) | 3938(1) | 7164(1) | 5670(1) | 21(1) |
| O(1B) | 3918(1) | 7945(1) | 6003(1) | 31(1) |
| C(1C) | 3913(1) | 4623(1) | 4489(1) | 22(1) |
| O(1C) | 3851(1) | 3942(1) | 4095(1) | 33(1) |
| C(1D) | 5263(1) | 6523(1) | 4652(1) | 23(1) |
| O(1D) | 6065(1) | 6900(1) | 4383(1) | 33(1) |
| C(1E) | 2794(1) | 5100(1) | 5626(1) | 25(1) |
| O(1E) | 2153(1) | 4633(1) | 5962(1) | 43(1) |
| O(1) | 5544(1) | 5730(1) | 6192(1) | 16(1) |
| C(1) | 5164(1) | 5115(1) | 5698(1) | 15(1) |
| C(2) | 5796(1) | 3959(1) | 5694(1) | 16(1) |
| C(3) | 6469(1) | 3887(1) | 6334(1) | 18(1) |
| C(4) | 6477(1) | 5149(1) | 6570(1) | 16(1) |
| C(5) | 6185(1) | 5309(1) | 7277(1) | 16(1) |
| O(5) | 5074(1) | 4780(1) | 7426(1) | 21(1) |
| C(6) | 6007(1) | 6591(1) | 7490(1) | 18(1) |
| O(6) | 5120(1) | 6494(1) | 7977(1) | 22(1) |
| C(7) | 7110(1) | 7151(1) | 7784(1) | 23(1) |
| O(7) | 7929(1) | 7372(1) | 7277(1) | 25(1) |
| C(8) | 7807(1) | 6338(2) | 8239(1) | 32(1) |
| O(8) | 9007(1) | 6351(1) | 8001(1) | 54(1) |
| C(9) | 7351(1) | 5096(1) | 8223(1) | 27(1) |
| O(9) | 7138(1) | 4730(1) | 7579(1) | 23(1) |
| C(10) | 9113(1) | 7211(1) | 7516(1) | 31(1) |
| C(11) | 9865(2) | 6709(2) | 6986(1) | 54(1) |
| C(12) | 9603(2) | 8341(2) | 7782(1) | 53(1) |
| C(13) | 4333(1) | 5577(1) | 7785(1) | 22(1) |
| C(14) | 3867(1) | 4966(1) | 8375(1) | 29(1) |
| C(15) | 3343(1) | 6041(1) | 7363(1) | 30(1) |
| C(16) | 5105(1) | 2828(1) | 5506(1) | 21(1) |
| C(17) | 6154(1) | 2468(1) | 5061(1) | 26(1) |
| C(18) | 6594(1) | 3730(1) | 5077(1) | 19(1) |
| O(18) | 7833(1) | 3860(1) | 5134(1) | 22(1) |
| C(19) | 8193(1) | 5062(1) | 5093(1) | 21(1) |
| C(20) | 9512(1) | 5136(1) | 5238(1) | 33(1) |

**Tabelle 3: Bond lengths [Å] for *[42]a*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1A) | 1.8948(14) | C(5)-C(6) | 1.5455(16) |
| Cr(1)-C(1D) | 1.8999(15) | O(5)-C(13) | 1.4428(16) |
| Cr(1)-C(1E) | 1.9048(15) | C(6)-O(6) | 1.4258(15) |
| Cr(1)-C(1B) | 1.9050(13) | C(6)-C(7) | 1.5191(18) |
| Cr(1)-C(1C) | 1.9124(13) | O(6)-C(13) | 1.4268(16) |
| Cr(1)-C(1) | 2.0273(12) | C(7)-O(7) | 1.4241(16) |
| C(1A)-O(1A) | 1.1455(18) | C(7)-C(8) | 1.542(2) |
| C(1B)-O(1B) | 1.1327(16) | O(7)-C(10) | 1.4270(18) |
| C(1C)-O(1C) | 1.1366(16) | C(8)-O(8) | 1.430(2) |
| C(1D)-O(1D) | 1.1424(18) | C(8)-C(9) | 1.509(2) |
| C(1E)-O(1E) | 1.1362(18) | O(8)-C(10) | 1.4180(19) |
| O(1)-C(1) | 1.3208(15) | C(9)-O(9) | 1.4318(16) |
| O(1)-C(4) | 1.4682(14) | C(10)-C(11) | 1.505(2) |
| C(1)-C(2) | 1.4991(16) | C(10)-C(12) | 1.510(2) |
| C(2)-C(3) | 1.5367(16) | C(13)-C(14) | 1.5110(18) |
| C(2)-C(16) | 1.5565(17) | C(13)-C(15) | 1.512(2) |
| C(2)-C(18) | 1.5922(16) | C(16)-C(17) | 1.5524(19) |
| C(3)-C(4) | 1.5260(17) | C(17)-C(18) | 1.5249(17) |
| C(4)-C(5) | 1.5265(16) | C(18)-O(18) | 1.3977(16) |
| C(5)-O(9) | 1.4037(15) | O(18)-C(19) | 1.4350(15) |
| C(5)-O(5) | 1.4152(16) | C(19)-C(20) | 1.507(2) |

**Tabelle 4: Bond angles [°] for *[42]a*.**

| | | | |
|---|---|---|---|
| C(1A)-Cr(1)-C(1D) | 93.37(6) | O(9)-C(5)-C(6) | 114.54(11) |
| C(1A)-Cr(1)-C(1E) | 94.22(7) | O(5)-C(5)-C(6) | 103.25(10) |
| C(1D)-Cr(1)-C(1E) | 172.40(6) | C(4)-C(5)-C(6) | 114.91(10) |
| C(1A)-Cr(1)-C(1B) | 88.24(6) | C(5)-O(5)-C(13) | 110.39(9) |
| C(1D)-Cr(1)-C(1B) | 90.21(6) | O(6)-C(6)-C(7) | 107.92(9) |
| C(1E)-Cr(1)-C(1B) | 89.67(6) | O(6)-C(6)-C(5) | 102.80(9) |
| C(1A)-Cr(1)-C(1C) | 87.82(6) | C(7)-C(6)-C(5) | 114.36(11) |
| C(1D)-Cr(1)-C(1C) | 88.79(6) | C(6)-O(6)-C(13) | 106.54(9) |
| C(1E)-Cr(1)-C(1C) | 91.85(6) | O(7)-C(7)-C(6) | 107.10(10) |
| C(1B)-Cr(1)-C(1C) | 175.88(5) | O(7)-C(7)-C(8) | 104.07(11) |
| C(1A)-Cr(1)-C(1) | 175.40(6) | C(6)-C(7)-C(8) | 113.90(11) |
| C(1D)-Cr(1)-C(1) | 87.60(5) | C(7)-O(7)-C(10) | 108.11(10) |
| C(1E)-Cr(1)-C(1) | 84.80(5) | O(8)-C(8)-C(9) | 108.57(14) |
| C(1B)-Cr(1)-C(1) | 87.26(5) | O(8)-C(8)-C(7) | 104.58(11) |
| C(1C)-Cr(1)-C(1) | 96.69(5) | C(9)-C(8)-C(7) | 112.55(11) |
| O(1A)-C(1A)-Cr(1) | 177.95(13) | C(10)-O(8)-C(8) | 109.58(12) |
| O(1B)-C(1B)-Cr(1) | 179.08(12) | O(9)-C(9)-C(8) | 110.62(11) |
| O(1C)-C(1C)-Cr(1) | 175.11(12) | C(5)-O(9)-C(9) | 114.32(10) |
| O(1D)-C(1D)-Cr(1) | 178.52(12) | O(8)-C(10)-O(7) | 105.25(11) |
| O(1E)-C(1E)-Cr(1) | 176.07(13) | O(8)-C(10)-C(11) | 108.07(15) |
| C(1)-O(1)-C(4) | 114.12(9) | O(7)-C(10)-C(11) | 107.92(13) |
| O(1)-C(1)-C(2) | 108.81(10) | O(8)-C(10)-C(12) | 111.10(15) |
| O(1)-C(1)-Cr(1) | 118.09(8) | O(7)-C(10)-C(12) | 110.77(13) |
| C(2)-C(1)-Cr(1) | 133.07(9) | C(11)-C(10)-C(12) | 113.33(16) |
| C(1)-C(2)-C(3) | 105.88(9) | O(6)-C(13)-O(5) | 104.88(10) |
| C(1)-C(2)-C(16) | 120.02(10) | O(6)-C(13)-C(14) | 108.79(11) |
| C(3)-C(2)-C(16) | 114.75(10) | O(5)-C(13)-C(14) | 109.38(11) |
| C(1)-C(2)-C(18) | 114.37(10) | O(6)-C(13)-C(15) | 110.93(11) |
| C(3)-C(2)-C(18) | 115.09(10) | O(5)-C(13)-C(15) | 109.71(11) |
| C(16)-C(2)-C(18) | 86.29(9) | C(14)-C(13)-C(15) | 112.84(12) |
| C(4)-C(3)-C(2) | 103.55(9) | C(17)-C(16)-C(2) | 89.89(9) |
| O(1)-C(4)-C(3) | 104.45(9) | C(18)-C(17)-C(16) | 88.81(9) |
| O(1)-C(4)-C(5) | 108.44(9) | O(18)-C(18)-C(17) | 114.97(11) |
| C(3)-C(4)-C(5) | 115.25(10) | O(18)-C(18)-C(2) | 117.86(11) |
| O(9)-C(5)-O(5) | 111.36(10) | C(17)-C(18)-C(2) | 89.57(9) |
| O(9)-C(5)-C(4) | 102.56(9) | C(18)-O(18)-C(19) | 112.01(9) |
| O(5)-C(5)-C(4) | 110.45(10) | O(18)-C(19)-C(20) | 108.41(11) |

**Tabelle 5: Torsion angles [°] for *[42]a*.**

| | | | |
|---|---|---|---|
| C(1D)-Cr(1)-C(1A)-O(1A) | -75(4) | C(1)-O(1)-C(4)-C(3) | -12.84(13) |
| C(1E)-Cr(1)-C(1A)-O(1A) | 105(4) | C(1)-O(1)-C(4)-C(5) | -136.22(10) |
| C(1B)-Cr(1)-C(1A)-O(1A) | 15(4) | C(2)-C(3)-C(4)-O(1) | 17.19(12) |
| C(1C)-Cr(1)-C(1A)-O(1A) | -164(4) | C(2)-C(3)-C(4)-C(5) | 136.05(10) |
| C(1)-Cr(1)-C(1A)-O(1A) | 27(5) | O(1)-C(4)-C(5)-O(9) | -179.52(9) |
| C(1A)-Cr(1)-C(1B)-O(1B) | 6(9) | C(3)-C(4)-C(5)-O(9) | 63.86(12) |
| C(1D)-Cr(1)-C(1B)-O(1B) | 100(9) | O(1)-C(4)-C(5)-O(5) | 61.71(12) |
| C(1E)-Cr(1)-C(1B)-O(1B) | -88(9) | C(3)-C(4)-C(5)-O(5) | -54.91(13) |
| C(1C)-Cr(1)-C(1B)-O(1B) | 24(10) | O(1)-C(4)-C(5)-C(6) | -54.58(14) |
| C(1)-Cr(1)-C(1B)-O(1B) | -173(100) | C(3)-C(4)-C(5)-C(6) | -171.20(11) |
| C(1A)-Cr(1)-C(1C)-O(1C) | 16.0(15) | O(9)-C(5)-O(5)-C(13) | 115.23(11) |
| C(1D)-Cr(1)-C(1C)-O(1C) | -77.5(15) | C(4)-C(5)-O(5)-C(13) | -131.49(10) |
| C(1E)-Cr(1)-C(1C)-O(1C) | 110.1(15) | C(6)-C(5)-O(5)-C(13) | -8.16(13) |
| C(1B)-Cr(1)-C(1C)-O(1C) | -1(2) | O(9)-C(5)-C(6)-O(6) | -94.95(12) |
| C(1)-Cr(1)-C(1C)-O(1C) | -164.9(15) | O(5)-C(5)-C(6)-O(6) | 26.30(12) |
| C(1A)-Cr(1)-C(1D)-O(1D) | -149(5) | C(4)-C(5)-C(6)-O(6) | 146.64(10) |
| C(1E)-Cr(1)-C(1D)-O(1D) | 34(5) | O(9)-C(5)-C(6)-C(7) | 21.75(14) |
| C(1B)-Cr(1)-C(1D)-O(1D) | 123(5) | O(5)-C(5)-C(6)-C(7) | 143.01(10) |
| C(1C)-Cr(1)-C(1D)-O(1D) | -61(5) | C(4)-C(5)-C(6)-C(7) | -96.66(13) |
| C(1)-Cr(1)-C(1D)-O(1D) | 36(5) | C(7)-C(6)-O(6)-C(13) | -156.55(10) |
| C(1A)-Cr(1)-C(1E)-O(1E) | -164(2) | C(5)-C(6)-O(6)-C(13) | -35.35(12) |
| C(1D)-Cr(1)-C(1E)-O(1E) | 14(2) | O(6)-C(6)-C(7)-O(7) | -173.05(10) |
| C(1B)-Cr(1)-C(1E)-O(1E) | -75(2) | C(5)-C(6)-C(7)-O(7) | 73.24(13) |
| C(1C)-Cr(1)-C(1E)-O(1E) | 109(2) | O(6)-C(6)-C(7)-C(8) | 72.46(14) |
| C(1)-Cr(1)-C(1E)-O(1E) | 12(2) | C(5)-C(6)-C(7)-C(8) | -41.25(15) |
| C(4)-O(1)-C(1)-C(2) | 2.21(13) | C(6)-C(7)-O(7)-C(10) | -145.50(11) |
| C(4)-O(1)-C(1)-Cr(1) | -176.14(7) | C(8)-C(7)-O(7)-C(10) | -24.57(13) |
| C(1A)-Cr(1)-C(1)-O(1) | -15.7(8) | O(7)-C(7)-C(8)-O(8) | 9.86(15) |
| C(1D)-Cr(1)-C(1)-O(1) | 86.67(10) | C(6)-C(7)-C(8)-O(8) | 126.13(13) |
| C(1E)-Cr(1)-C(1)-O(1) | -93.58(10) | O(7)-C(7)-C(8)-C(9) | -107.80(14) |
| C(1B)-Cr(1)-C(1)-O(1) | -3.66(10) | C(6)-C(7)-C(8)-C(9) | 8.47(17) |
| C(1C)-Cr(1)-C(1)-O(1) | 175.16(9) | C(9)-C(8)-O(8)-C(10) | 128.70(13) |
| C(1A)-Cr(1)-C(1)-C(2) | 166.5(7) | C(7)-C(8)-O(8)-C(10) | 8.35(17) |
| C(1D)-Cr(1)-C(1)-C(2) | -91.19(12) | O(8)-C(8)-C(9)-O(9) | -71.50(14) |
| C(1E)-Cr(1)-C(1)-C(2) | 88.57(12) | C(7)-C(8)-C(9)-O(9) | 43.78(17) |
| C(1B)-Cr(1)-C(1)-C(2) | 178.48(12) | O(5)-C(5)-O(9)-C(9) | -84.17(13) |
| C(1C)-Cr(1)-C(1)-C(2) | -2.69(12) | C(4)-C(5)-O(9)-C(9) | 157.70(11) |
| O(1)-C(1)-C(2)-C(3) | 9.36(13) | C(6)-C(5)-O(9)-C(9) | 32.52(15) |
| Cr(1)-C(1)-C(2)-C(3) | -172.64(9) | C(8)-C(9)-O(9)-C(5) | -67.74(15) |
| O(1)-C(1)-C(2)-C(16) | 141.20(11) | C(8)-O(8)-C(10)-O(7) | -23.50(17) |
| Cr(1)-C(1)-C(2)-C(16) | -40.80(16) | C(8)-O(8)-C(10)-C(11) | -138.63(15) |
| O(1)-C(1)-C(2)-C(18) | -118.44(11) | C(8)-O(8)-C(10)-C(12) | 96.45(16) |
| Cr(1)-C(1)-C(2)-C(18) | 59.56(15) | C(7)-O(7)-C(10)-O(8) | 30.26(15) |
| C(1)-C(2)-C(3)-C(4) | -16.32(12) | C(7)-O(7)-C(10)-C(11) | 145.48(14) |
| C(16)-C(2)-C(3)-C(4) | -151.07(10) | C(7)-O(7)-C(10)-C(12) | -89.92(16) |
| C(18)-C(2)-C(3)-C(4) | 111.05(11) | C(6)-O(6)-C(13)-O(5) | 30.86(12) |

| | | | |
|---|---|---|---|
| C(6)-O(6)-C(13)-C(14) | 147.80(11) | C(16)-C(17)-C(18)-C(2) | 17.49(10) |
| C(6)-O(6)-C(13)-C(15) | -87.52(12) | C(1)-C(2)-C(18)-O(18) | 102.86(12) |
| C(5)-O(5)-C(13)-O(6) | -13.09(13) | C(3)-C(2)-C(18)-O(18) | -20.09(15) |
| C(5)-O(5)-C(13)-C(14) | -129.62(11) | C(16)-C(2)-C(18)-O(18) | -135.74(11) |
| C(5)-O(5)-C(13)-C(15) | 106.11(12) | C(1)-C(2)-C(18)-C(17) | -138.88(11) |
| C(1)-C(2)-C(16)-C(17) | 133.27(11) | C(3)-C(2)-C(18)-C(17) | 98.18(11) |
| C(3)-C(2)-C(16)-C(17) | -98.83(11) | C(16)-C(2)-C(18)-C(17) | -17.47(10) |
| C(18)-C(2)-C(16)-C(17) | 17.15(9) | C(17)-C(18)-O(18)-C(19) | 175.27(11) |
| C(2)-C(16)-C(17)-C(18) | -17.90(10) | C(2)-C(18)-O(18)-C(19) | -81.03(13) |
| C(16)-C(17)-C(18)-O(18) | 138.29(11) | C(18)-O(18)-C(19)-C(20) | 173.32(11) |

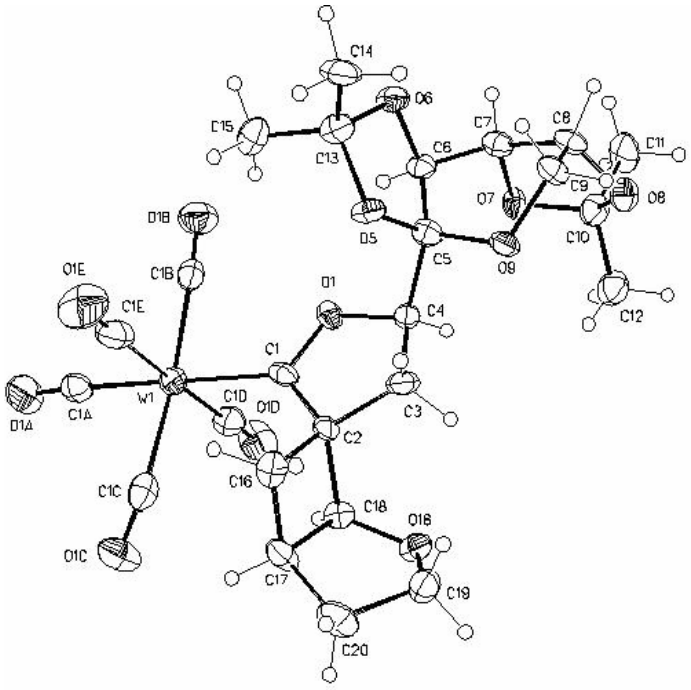
**Tabelle 6: Hydrogen bonds for *[42]a* [Å and °].**

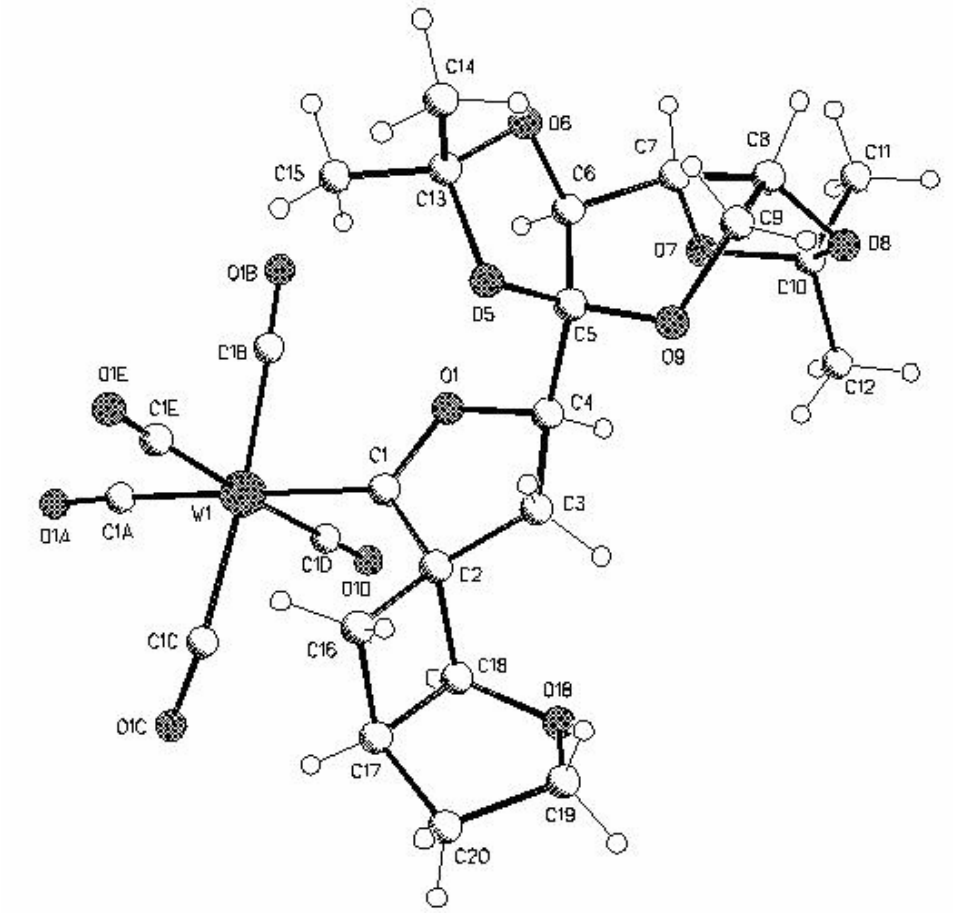
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(19)-H(19B)...O(1B)[a] | 0.99 | 2.57 | 3.3338(16) | 133.6 |

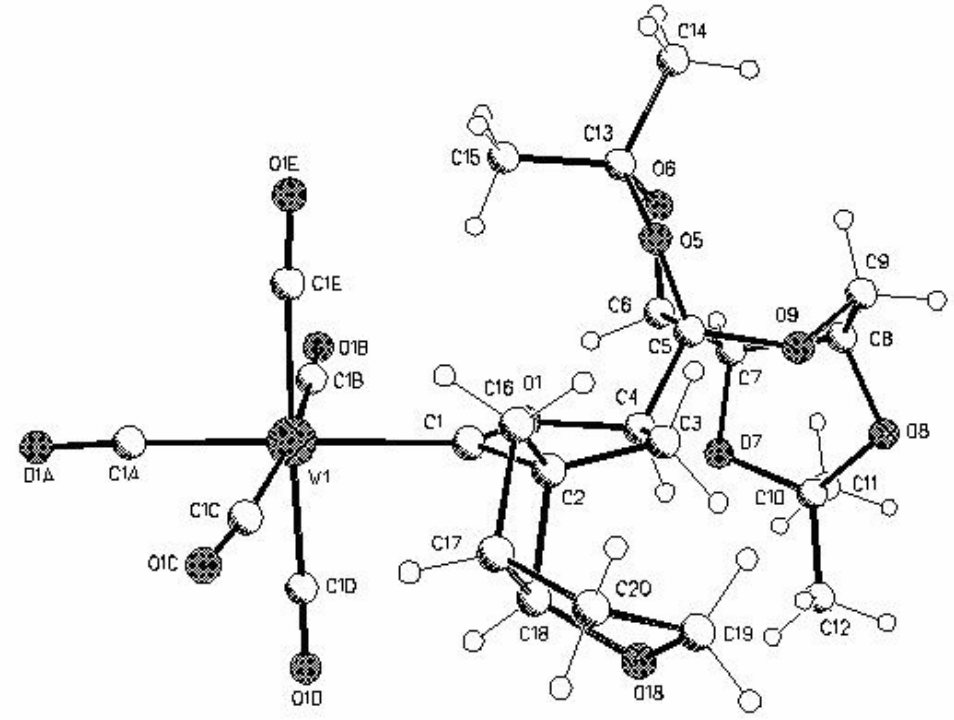
Symmetry transformations used to generate equivalent atoms:

[a] x+1/2,-y+3/2,-z+1

**9. (*3R,5R,1´S,5´R,1´´R,2´´S,6´´S,9´´R*)-Pentacarbonyl{3-(4´´,4´´,-11´´,11´´-tetramethyl-3,5,7,10,12-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´´-yl)-spiro[2-oxabicyclo[3.2.0]heptan-7´,5-2-oxacyclopent]-1-yliden}wolfram(0) *[45]a***




C14
O6
C7
C8
C11
C15
C13
C6
C9
O7
O8
O1B
O5
C10
C1B
C5
O9
O1E
O1
C12
C1E
C4
C1
C3
O1A
C1A
W1
C1D
O1D
C2
C1C
C16
C18
O1C
O18
C17
C19
C20


C14
C13
C15
O6
O1E
O5
C9
C1E
C6
O9
C5
C8
O1B
C7
C1B
C16
O1
C4
C3
O8
C1
O7
O1A
C1A
W1
C2
C10
C11
C1C
C17
O1C
C20
C1D
C18
C19
C12
O1D
O18

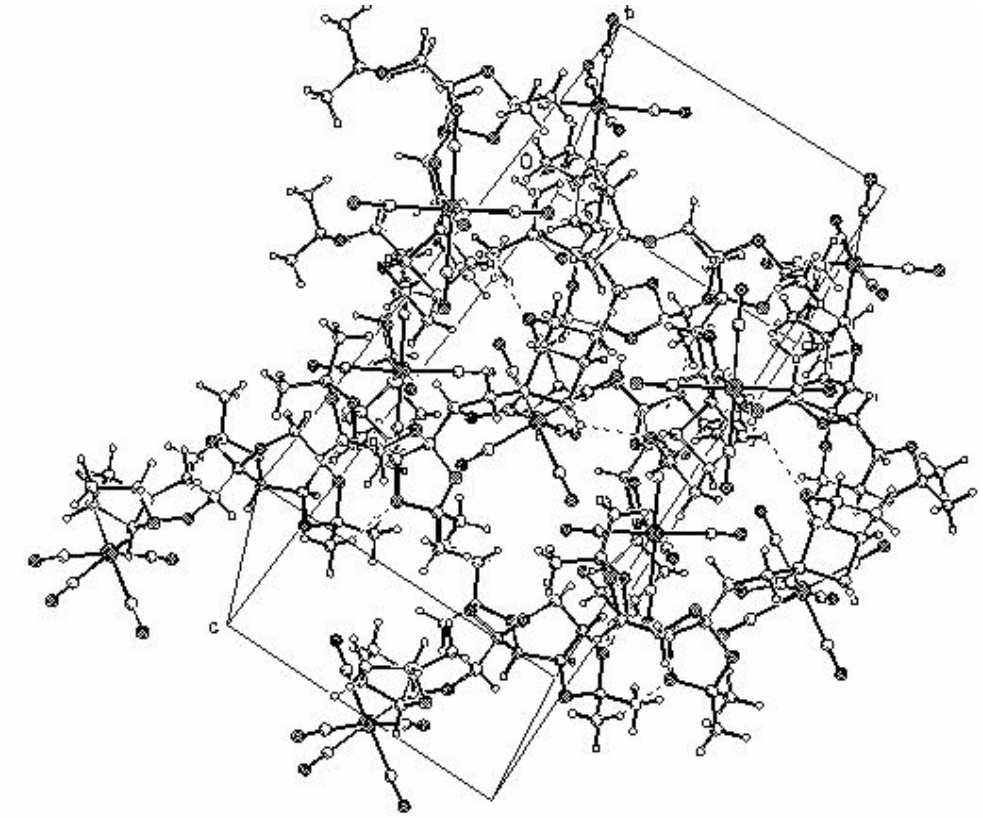

b
c

**Table 1: Crystal data and structure refinements for *[45]a***

| | |
|---|---|
| Identification code | ***[45]a*** |
| Empirical formula | $C_{25}H_{28}O_{12}W$ |
| Formula weight | 704.32 |
| Temperature | 173(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 10.7820(2) Å α = 90° |
| | b = 10.9415(2) Å β = 90° |
| | c = 22.1986(4) Å γ = 90° |
| Volume | 2618.80(8) $Å^3$ |
| Z | 4 |
| Calculated density | 1.786 $mg/m^3$ |
| Absorption coefficient | 4.475 $mm^{-1}$ |
| F(000) | 1392 |
| Crystal size | 0.15 x 0.10 x 0.05 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 3.23 to 25.00 ° |
| Limiting indices | $-8 \le h \le 12$, $-13 \le k \le 12$, $-26 \le l \le 24$ |
| Reflections collected / unique | 13716 / 4576 [R(int) = 0.0662] |
| Completeness to Θ = 25.00 | 99.4 % |
| Absorption correction | Empirical from multiple refl |
| Max. and min. transmission | 0.71918 and 0.52449 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 4576 / 0 / 343 |
| Goodness-of-fit on $F^2$ | 0.855 |
| Final R indices [I>2σ(I)] | R1 = 0.0304, wR2 = 0.0480 |
| R indices (all data) | R1 = 0.0404, wR2 = 0.0505 |
| Absolute structure parameter | -0.035(8) |
| Largest diff. peak and hole | 1.000 and -0.938 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[45]a*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| W(1) | 4640(1) | 3966(1) | 5122(1) | 25(1) |
| C(1A) | 5292(7) | 2549(5) | 4633(2) | 31(2) |
| O(1A) | 5652(5) | 1769(4) | 4334(2) | 56(2) |
| C(1B) | 3764(6) | 2766(6) | 5695(3) | 29(2) |
| O(1B) | 3294(5) | 2065(4) | 5997(2) | 46(1) |
| C(1C) | 5520(7) | 4931(6) | 4468(3) | 32(2) |
| O(1C) | 6032(4) | 5350(4) | 4062(2) | 45(1) |
| C(1D) | 3076(6) | 3998(7) | 4613(2) | 37(2) |
| O(1D) | 2186(4) | 3988(6) | 4323(2) | 57(1) |
| C(1E) | 6196(6) | 3964(7) | 5672(2) | 39(2) |
| O(1E) | 7026(5) | 4002(5) | 5979(2) | 58(1) |
| O(1) | 3062(4) | 5270(4) | 6058(2) | 23(1) |
| C(1) | 3916(6) | 5499(6) | 5641(2) | 23(2) |
| C(2) | 4136(5) | 6855(5) | 5616(2) | 19(2) |
| C(3) | 3443(6) | 7390(5) | 6154(2) | 28(2) |
| C(4) | 2610(6) | 6365(5) | 6366(2) | 22(2) |
| C(5) | 2625(5) | 6159(6) | 7051(2) | 23(1) |
| O(5) | 3869(3) | 5953(4) | 7232(1) | 24(1) |
| C(6) | 1919(6) | 5048(5) | 7279(2) | 22(2) |
| O(6) | 2667(4) | 4655(4) | 7777(2) | 26(1) |
| C(7) | 623(6) | 5305(6) | 7512(2) | 26(2) |
| O(7) | -143(4) | 5484(3) | 6996(1) | 24(1) |
| C(8) | 504(7) | 6513(5) | 7866(2) | 27(2) |
| O(8) | -437(5) | 7188(3) | 7560(2) | 29(1) |
| C(9) | 1696(6) | 7246(5) | 7868(2) | 26(2) |
| O(9) | 2201(4) | 7291(4) | 7272(2) | 25(1) |
| C(10) | -1058(6) | 6370(5) | 7157(3) | 31(2) |
| C(11) | -2144(6) | 5762(6) | 7476(3) | 41(2) |
| C(12) | -1437(6) | 7059(6) | 6604(2) | 41(2) |
| C(13) | 3928(6) | 4882(6) | 7622(3) | 27(2) |
| C(14) | 4638(7) | 5224(5) | 8186(2) | 35(2) |
| C(15) | 4507(6) | 3837(6) | 7283(2) | 34(2) |
| C(16) | 5501(6) | 7257(5) | 5508(2) | 27(2) |
| C(17) | 5192(6) | 7766(5) | 4875(3) | 30(2) |
| C(18) | 3799(5) | 7426(5) | 4974(2) | 22(1) |
| O(18) | 3128(3) | 8531(3) | 5000(2) | 27(1) |
| C(19) | 3975(5) | 9526(5) | 5117(3) | 31(1) |
| C(20) | 5179(6) | 9146(5) | 4807(2) | 38(2) |

**Tabelle 3: Bond lengths [Å] for *[45]a*.**

| | | | |
|---|---|---|---|
| W(1)-C(1A) | 2.018(6) | O(5)-C(13) | 1.458(6) |
| W(1)-C(1D) | 2.030(7) | C(6)-O(6) | 1.434(6) |
| W(1)-C(1C) | 2.031(7) | C(6)-C(7) | 1.516(8) |
| W(1)-C(1B) | 2.058(7) | O(6)-C(13) | 1.424(7) |
| W(1)-C(1E) | 2.075(7) | C(7)-O(7) | 1.425(6) |
| W(1)-C(1) | 2.181(6) | C(7)-C(8) | 1.544(7) |
| C(1A)-O(1A) | 1.149(6) | O(7)-C(10) | 1.428(7) |
| C(1B)-O(1B) | 1.137(7) | C(8)-O(8) | 1.427(7) |
| C(1C)-O(1C) | 1.151(7) | C(8)-C(9) | 1.514(8) |
| C(1D)-O(1D) | 1.155(6) | O(8)-C(10) | 1.431(7) |
| C(1E)-O(1E) | 1.126(6) | C(9)-O(9) | 1.431(6) |
| O(1)-C(1) | 1.329(6) | C(10)-C(12) | 1.497(8) |
| O(1)-C(4) | 1.463(6) | C(10)-C(11) | 1.522(8) |
| C(1)-C(2) | 1.503(7) | C(13)-C(15) | 1.504(8) |
| C(2)-C(3) | 1.524(7) | C(13)-C(14) | 1.514(7) |
| C(2)-C(16) | 1.555(8) | C(16)-C(17) | 1.548(7) |
| C(2)-C(18) | 1.598(7) | C(17)-C(20) | 1.518(7) |
| C(3)-C(4) | 1.511(7) | C(17)-C(18) | 1.564(7) |
| C(4)-C(5) | 1.538(6) | C(18)-O(18) | 1.410(6) |
| C(5)-O(9) | 1.409(7) | O(18)-C(19) | 1.444(6) |
| C(5)-O(5) | 1.418(6) | C(19)-C(20) | 1.526(7) |
| C(5)-C(6) | 1.522(8) | | |

**Tabelle 4: Bond angles [°] for *[45]a*.**

| | | | |
|---|---|---|---|
| C(1A)-W(1)-C(1D) | 90.2(3) | C(6)-C(5)-C(4) | 116.2(5) |
| C(1A)-W(1)-C(1C) | 81.5(2) | C(5)-O(5)-C(13) | 109.7(4) |
| C(1D)-W(1)-C(1C) | 88.9(3) | O(6)-C(6)-C(7) | 108.2(4) |
| C(1A)-W(1)-C(1B) | 90.1(2) | O(6)-C(6)-C(5) | 102.4(4) |
| C(1D)-W(1)-C(1B) | 88.6(3) | C(7)-C(6)-C(5) | 115.2(5) |
| C(1C)-W(1)-C(1B) | 171.3(2) | C(13)-O(6)-C(6) | 107.4(4) |
| C(1A)-W(1)-C(1E) | 91.9(3) | O(7)-C(7)-C(6) | 106.6(4) |
| C(1D)-W(1)-C(1E) | 177.6(2) | O(7)-C(7)-C(8) | 104.1(5) |
| C(1C)-W(1)-C(1E) | 92.5(3) | C(6)-C(7)-C(8) | 114.1(5) |
| C(1B)-W(1)-C(1E) | 90.3(2) | C(7)-O(7)-C(10) | 107.0(4) |
| C(1A)-W(1)-C(1) | 179.3(2) | O(8)-C(8)-C(9) | 109.4(5) |
| C(1D)-W(1)-C(1) | 89.1(2) | O(8)-C(8)-C(7) | 105.0(4) |
| C(1C)-W(1)-C(1) | 98.3(2) | C(9)-C(8)-C(7) | 112.6(5) |
| C(1B)-W(1)-C(1) | 90.0(2) | C(8)-O(8)-C(10) | 107.9(4) |
| C(1E)-W(1)-C(1) | 88.8(2) | O(9)-C(9)-C(8) | 109.8(5) |
| O(1A)-C(1A)-W(1) | 177.2(5) | C(5)-O(9)-C(9) | 114.6(4) |
| O(1B)-C(1B)-W(1) | 177.2(6) | O(7)-C(10)-O(8) | 104.9(5) |
| O(1C)-C(1C)-W(1) | 171.9(5) | O(7)-C(10)-C(12) | 109.0(5) |
| O(1D)-C(1D)-W(1) | 178.5(7) | O(8)-C(10)-C(12) | 109.0(5) |
| O(1E)-C(1E)-W(1) | 177.5(7) | O(7)-C(10)-C(11) | 110.5(5) |
| C(1)-O(1)-C(4) | 113.6(4) | O(8)-C(10)-C(11) | 110.0(5) |
| O(1)-C(1)-C(2) | 108.8(5) | C(12)-C(10)-C(11) | 113.1(6) |
| O(1)-C(1)-W(1) | 118.1(4) | O(6)-C(13)-O(5) | 104.0(4) |
| C(2)-C(1)-W(1) | 133.1(4) | O(6)-C(13)-C(15) | 112.6(5) |
| C(1)-C(2)-C(3) | 105.8(5) | O(5)-C(13)-C(15) | 109.4(5) |
| C(1)-C(2)-C(16) | 115.8(5) | O(6)-C(13)-C(14) | 109.0(5) |
| C(3)-C(2)-C(16) | 118.4(5) | O(5)-C(13)-C(14) | 108.4(5) |
| C(1)-C(2)-C(18) | 112.5(5) | C(15)-C(13)-C(14) | 113.0(5) |
| C(3)-C(2)-C(18) | 115.8(5) | C(17)-C(16)-C(2) | 92.2(5) |
| C(16)-C(2)-C(18) | 88.1(4) | C(20)-C(17)-C(16) | 116.7(5) |
| C(4)-C(3)-C(2) | 104.5(5) | C(20)-C(17)-C(18) | 103.9(5) |
| O(1)-C(4)-C(3) | 105.3(4) | C(16)-C(17)-C(18) | 89.6(5) |
| O(1)-C(4)-C(5) | 109.7(4) | O(18)-C(18)-C(17) | 107.1(4) |
| C(3)-C(4)-C(5) | 114.2(5) | O(18)-C(18)-C(2) | 114.6(4) |
| O(9)-C(5)-O(5) | 110.3(4) | C(17)-C(18)-C(2) | 90.0(4) |
| O(9)-C(5)-C(6) | 115.1(4) | C(18)-O(18)-C(19) | 109.2(4) |
| O(5)-C(5)-C(6) | 104.6(5) | O(18)-C(19)-C(20) | 104.6(4) |
| O(9)-C(5)-C(4) | 102.3(4) | C(17)-C(20)-C(19) | 103.6(5) |
| O(5)-C(5)-C(4) | 108.3(4) | | |

**Tabelle 5: Torsion angles [°] for *[45]a.***

| | | | |
|---|---|---|---|
| C(1D)-W(1)-C(1A)-O(1A) | 49(13) | C(1)-O(1)-C(4)-C(3) | -8.2(6) |
| C(1C)-W(1)-C(1A)-O(1A) | -40(13) | C(1)-O(1)-C(4)-C(5) | -131.6(5) |
| C(1B)-W(1)-C(1A)-O(1A) | 137(13) | C(2)-C(3)-C(4)-O(1) | 13.2(6) |
| C(1E)-W(1)-C(1A)-O(1A) | -132(13) | C(2)-C(3)-C(4)-C(5) | 133.7(5) |
| C(1)-W(1)-C(1A)-O(1A) | 38(30) | O(1)-C(4)-C(5)-O(9) | 178.6(4) |
| C(1A)-W(1)-C(1B)-O(1B) | -6(12) | C(3)-C(4)-C(5)-O(9) | 60.6(6) |
| C(1D)-W(1)-C(1B)-O(1B) | 84(12) | O(1)-C(4)-C(5)-O(5) | 62.0(6) |
| C(1C)-W(1)-C(1B)-O(1B) | 11(13) | C(3)-C(4)-C(5)-O(5) | -55.9(6) |
| C(1E)-W(1)-C(1B)-O(1B) | -98(12) | O(1)-C(4)-C(5)-C(6) | -55.2(6) |
| C(1)-W(1)-C(1B)-O(1B) | 173(12) | C(3)-C(4)-C(5)-C(6) | -173.2(5) |
| C(1A)-W(1)-C(1C)-O(1C) | 11(4) | O(9)-C(5)-O(5)-C(13) | 116.6(5) |
| C(1D)-W(1)-C(1C)-O(1C) | -80(4) | C(6)-C(5)-O(5)-C(13) | -7.7(5) |
| C(1B)-W(1)-C(1C)-O(1C) | -6(6) | C(4)-C(5)-O(5)-C(13) | -132.2(4) |
| C(1E)-W(1)-C(1C)-O(1C) | 102(4) | O(9)-C(5)-C(6)-O(6) | -95.5(5) |
| C(1)-W(1)-C(1C)-O(1C) | -169(4) | O(5)-C(5)-C(6)-O(6) | 25.7(5) |
| C(1A)-W(1)-C(1D)-O(1D) | 40(19) | C(4)-C(5)-C(6)-O(6) | 145.0(5) |
| C(1C)-W(1)-C(1D)-O(1D) | 121(19) | O(9)-C(5)-C(6)-C(7) | 21.6(6) |
| C(1B)-W(1)-C(1D)-O(1D) | -50(19) | O(5)-C(5)-C(6)-C(7) | 142.9(4) |
| C(1E)-W(1)-C(1D)-O(1D) | -113(19) | C(4)-C(5)-C(6)-C(7) | -97.9(6) |
| C(1)-W(1)-C(1D)-O(1D) | -140(19) | C(7)-C(6)-O(6)-C(13) | -157.3(5) |
| C(1A)-W(1)-C(1E)-O(1E) | 171(14) | C(5)-C(6)-O(6)-C(13) | -35.2(5) |
| C(1D)-W(1)-C(1E)-O(1E) | -36(19) | O(6)-C(6)-C(7)-O(7) | -170.1(4) |
| C(1C)-W(1)-C(1E)-O(1E) | 89(14) | C(5)-C(6)-C(7)-O(7) | 76.0(6) |
| C(1B)-W(1)-C(1E)-O(1E) | -99(14) | O(6)-C(6)-C(7)-C(8) | 75.5(6) |
| C(1)-W(1)-C(1E)-O(1E) | -9(14) | C(5)-C(6)-C(7)-C(8) | -38.4(6) |
| C(4)-O(1)-C(1)-C(2) | -0.7(6) | C(6)-C(7)-O(7)-C(10) | -146.5(5) |
| C(4)-O(1)-C(1)-W(1) | -178.0(3) | C(8)-C(7)-O(7)-C(10) | -25.5(6) |
| C(1A)-W(1)-C(1)-O(1) | 89(21) | O(7)-C(7)-C(8)-O(8) | 7.4(6) |
| C(1D)-W(1)-C(1)-O(1) | 78.4(4) | C(6)-C(7)-C(8)-O(8) | 123.3(5) |
| C(1C)-W(1)-C(1)-O(1) | 167.2(4) | O(7)-C(7)-C(8)-C(9) | -111.5(5) |
| C(1B)-W(1)-C(1)-O(1) | -10.2(4) | C(6)-C(7)-C(8)-C(9) | 4.4(7) |
| C(1E)-W(1)-C(1)-O(1) | -100.5(4) | C(9)-C(8)-O(8)-C(10) | 134.4(5) |
| C(1A)-W(1)-C(1)-C(2) | -87(21) | C(7)-C(8)-O(8)-C(10) | 13.3(6) |
| C(1D)-W(1)-C(1)-C(2) | -98.2(6) | O(8)-C(8)-C(9)-O(9) | -70.3(6) |
| C(1C)-W(1)-C(1)-C(2) | -9.4(6) | C(7)-C(8)-C(9)-O(9) | 46.0(6) |
| C(1B)-W(1)-C(1)-C(2) | 173.2(6) | O(5)-C(5)-O(9)-C(9) | -86.6(6) |
| C(1E)-W(1)-C(1)-C(2) | 82.9(6) | C(6)-C(5)-O(9)-C(9) | 31.5(6) |
| O(1)-C(1)-C(2)-C(3) | 9.3(6) | C(4)-C(5)-O(9)-C(9) | 158.4(5) |
| W(1)-C(1)-C(2)-C(3) | -173.9(4) | C(8)-C(9)-O(9)-C(5) | -67.2(6) |
| O(1)-C(1)-C(2)-C(16) | 142.6(5) | C(7)-O(7)-C(10)-O(8) | 34.4(5) |
| W(1)-C(1)-C(2)-C(16) | -40.6(8) | C(7)-O(7)-C(10)-C(12) | 151.0(5) |
| O(1)-C(1)-C(2)-C(18) | -118.2(5) | C(7)-O(7)-C(10)-C(11) | -84.2(6) |
| W(1)-C(1)-C(2)-C(18) | 58.6(7) | C(8)-O(8)-C(10)-O(7) | -29.3(6) |
| C(1)-C(2)-C(3)-C(4) | -13.7(6) | C(8)-O(8)-C(10)-C(12) | -145.9(5) |
| C(16)-C(2)-C(3)-C(4) | -145.5(5) | C(8)-O(8)-C(10)-C(11) | 89.6(6) |
| C(18)-C(2)-C(3)-C(4) | 111.8(5) | C(6)-O(6)-C(13)-O(5) | 30.8(5) |

| | | | |
|---|---|---|---|
| C(6)-O(6)-C(13)-C(15) | -87.5(5) | C(16)-C(17)-C(18)-C(2) | -2.2(4) |
| C(6)-O(6)-C(13)-C(14) | 146.3(5) | C(1)-C(2)-C(18)-O(18) | 136.0(5) |
| C(5)-O(5)-C(13)-O(6) | -13.5(6) | C(3)-C(2)-C(18)-O(18) | 14.1(7) |
| C(5)-O(5)-C(13)-C(15) | 107.0(5) | C(16)-C(2)-C(18)-O(18) | -106.8(5) |
| C(5)-O(5)-C(13)-C(14) | -129.3(5) | C(1)-C(2)-C(18)-C(17) | -115.1(5) |
| C(1)-C(2)-C(16)-C(17) | 112.0(5) | C(3)-C(2)-C(18)-C(17) | 123.0(5) |
| C(3)-C(2)-C(16)-C(17) | -120.7(5) | C(16)-C(2)-C(18)-C(17) | 2.1(4) |
| C(18)-C(2)-C(16)-C(17) | -2.2(4) | C(17)-C(18)-O(18)-C(19) | -17.9(6) |
| C(2)-C(16)-C(17)-C(20) | 107.7(6) | C(2)-C(18)-O(18)-C(19) | 80.2(5) |
| C(2)-C(16)-C(17)-C(18) | 2.2(4) | C(18)-O(18)-C(19)-C(20) | 32.5(5) |
| C(20)-C(17)-C(18)-O(18) | -3.8(6) | C(16)-C(17)-C(20)-C(19) | -74.4(7) |
| C(16)-C(17)-C(18)-O(18) | 113.7(4) | C(18)-C(17)-C(20)-C(19) | 22.3(6) |
| C(20)-C(17)-C(18)-C(2) | -119.6(4) | O(18)-C(19)-C(20)-C(17) | -33.4(6) |

**Tabelle 6: Hydrogen bonds for *[45]a* [Å and °].#**

| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(12)-H(12C)...O(6)[a] | 0.98 | 2.49 | 3.423(7) | 159.2 |
| C(8')-H(8'3)...O(1A)[b] | 0.99 | 2.46 | 3.169(7) | 128.0 |

Symmetry transformations used to generate equivalent atoms:

[a] -x,y+1/2,-z+3/2 [b] x+1/2,-y+3/2,-z+1

**10.** **(*1S,4R,7R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-1-(dimethylethoxy)-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro[3.4]oct]-5-yliden}chrom(0) *[47]b***

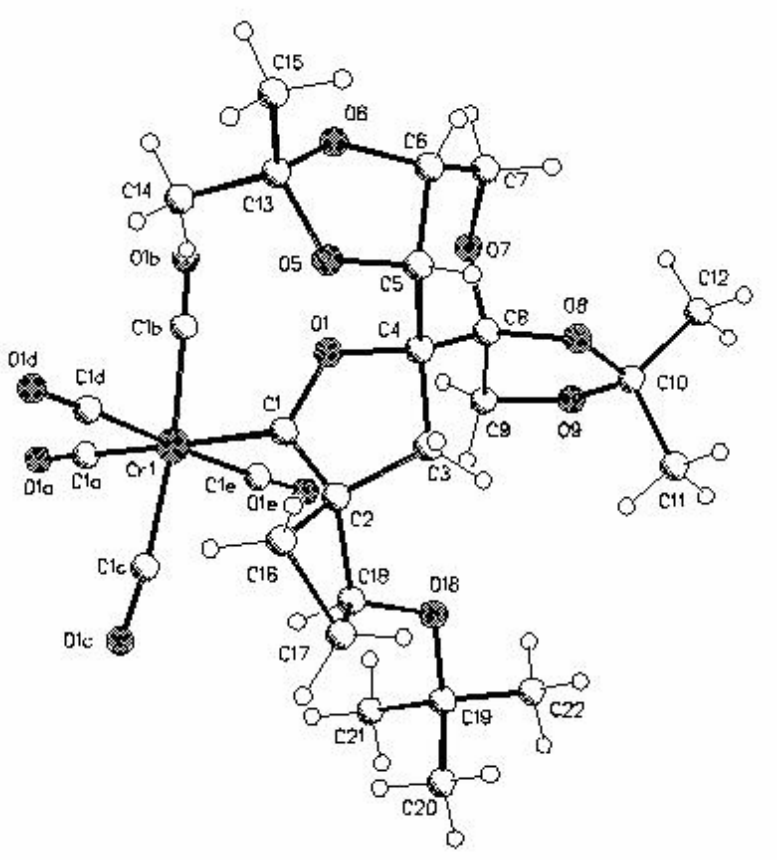


**Table 1: Crystal data and structure refinements for *[47]b***

| | |
|---|---|
| Identification code | ***[47]b*** |
| Empirical formula | $C_{27}H_{34}CrO_{12}$ |
| Formula weight | 602.54 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 7.69220(10) Å α = 90° |
| | b = 16.95310(10) Å β = 90° |
| | c = 22.4811(2) Å γ = 90° |
| Volume | 2931.68(5) $Å^3$ |
| Z | 4 |
| Calculated density | 1.365 $mg/m^3$ |
| Absorption coefficient | 0.450 $mm^{-1}$ |
| F(000) | 1264 |
| Crystal size | 0.60 x 0.40 x 0.30 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.91 to 25.00 ° |
| Limiting indices | $-9 \le h \le 9$, $-20 \le k \le 20$, $-26 \le l \le 26$ |
| Reflections collected / unique | 55377 / 5157 [R(int) = 0.0370] |
| Completeness to Θ = 25.00 | 99.7 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 5157 / 0 / 361 |
| Goodness-of-fit on $F^2$ | 1.060 |
| Final R indices [I>2σ(I)] | R1 = 0.0300, wR2 = 0.0746 |
| R indices (all data) | R1 = 0.0316, wR2 = 0.0755 |
| Absolute structure parameter | 0.008(16) |
| Largest diff. peak and hole | 0.234 and -0.410 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[47]b*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 6859(1) | 6680(1) | 6110(1) | 30(1) |
| C(1A) | 7870(5) | 7655(2) | 6274(1) | 61(1) |
| O(1A) | 8593(5) | 8233(1) | 6388(1) | 105(1) |
| C(1B) | 7300(3) | 6443(1) | 6926(1) | 34(1) |
| O(1B) | 7618(3) | 6357(1) | 7417(1) | 54(1) |
| C(1C) | 6526(4) | 7082(1) | 5331(1) | 46(1) |
| O(1C) | 6376(4) | 7400(1) | 4886(1) | 65(1) |
| C(1D) | 4567(4) | 7045(2) | 6292(1) | 42(1) |
| O(1D) | 3206(3) | 7278(1) | 6391(1) | 56(1) |
| C(1E) | 9079(3) | 6258(1) | 5927(1) | 34(1) |
| O(1E) | 10420(2) | 6000(1) | 5844(1) | 49(1) |
| O(1) | 6090(2) | 5054(1) | 6397(1) | 21(1) |
| C(1) | 6006(3) | 5565(1) | 5956(1) | 22(1) |
| C(2) | 5349(3) | 5150(1) | 5409(1) | 25(1) |
| C(3) | 5169(3) | 4279(1) | 5573(1) | 25(1) |
| C(4) | 5643(3) | 4229(1) | 6230(1) | 24(1) |
| C(5) | 4090(3) | 3960(2) | 6617(1) | 37(1) |
| O(5) | 2850(2) | 4568(1) | 6672(1) | 38(1) |
| C(6) | 4541(4) | 3740(2) | 7259(1) | 44(1) |
| O(6) | 4310(2) | 4473(1) | 7557(1) | 40(1) |
| C(7) | 6326(4) | 3417(2) | 7344(1) | 49(1) |
| O(7) | 7632(2) | 3803(1) | 6991(1) | 39(1) |
| C(8) | 7310(3) | 3774(1) | 6373(1) | 31(1) |
| O(8) | 7089(2) | 2994(1) | 6164(1) | 39(1) |
| C(9) | 8955(3) | 4050(1) | 6057(1) | 33(1) |
| O(9) | 9908(2) | 3341(1) | 5991(1) | 57(1) |
| C(10) | 8728(3) | 2706(1) | 5942(2) | 53(1) |
| C(11) | 8466(4) | 2489(2) | 5289(2) | 74(1) |
| C(12) | 9368(5) | 2032(2) | 6324(3) | 109(2) |
| C(13) | 2820(3) | 4839(2) | 7289(1) | 42(1) |
| C(14) | 3018(4) | 5710(2) | 7313(1) | 45(1) |
| C(15) | 1134(4) | 4559(2) | 7573(1) | 64(1) |
| C(16) | 3650(3) | 5476(2) | 5113(1) | 42(1) |
| C(17) | 4384(4) | 5223(2) | 4505(1) | 48(1) |
| C(18) | 6220(3) | 5291(1) | 4791(1) | 31(1) |
| O(18) | 7530(2) | 4745(1) | 4651(1) | 28(1) |
| C(19) | 8599(3) | 4919(1) | 4127(1) | 31(1) |
| C(20) | 7461(3) | 4954(2) | 3574(1) | 43(1) |
| C(21) | 9586(3) | 5691(2) | 4203(1) | 42(1) |
| C(22) | 9841(4) | 4232(2) | 4103(1) | 59(1) |

**Tabelle 3: Bond lengths [Å] for *[47]b*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1A) | 1.864(3) | O(5)-C(13) | 1.462(3) |
| Cr(1)-C(1E) | 1.896(3) | C(6)-O(6) | 1.422(3) |
| Cr(1)-C(1C) | 1.897(2) | C(6)-C(7) | 1.491(4) |
| Cr(1)-C(1B) | 1.907(2) | O(6)-C(13) | 1.436(3) |
| Cr(1)-C(1D) | 1.913(3) | C(7)-O(7) | 1.438(3) |
| Cr(1)-C(1) | 2.031(2) | O(7)-C(8) | 1.412(3) |
| C(1A)-O(1A) | 1.156(4) | C(8)-O(8) | 1.414(3) |
| C(1B)-O(1B) | 1.140(3) | C(8)-C(9) | 1.525(3) |
| C(1C)-O(1C) | 1.142(3) | O(8)-C(10) | 1.441(3) |
| C(1D)-O(1D) | 1.141(3) | C(9)-O(9) | 1.416(3) |
| C(1E)-O(1E) | 1.136(3) | O(9)-C(10) | 1.412(3) |
| O(1)-C(1) | 1.316(2) | C(10)-C(12) | 1.513(4) |
| O(1)-C(4) | 1.489(2) | C(10)-C(11) | 1.527(5) |
| C(1)-C(2) | 1.506(3) | C(13)-C(14) | 1.486(4) |
| C(2)-C(3) | 1.528(3) | C(13)-C(15) | 1.520(3) |
| C(2)-C(18) | 1.560(3) | C(16)-C(17) | 1.541(3) |
| C(2)-C(16) | 1.567(3) | C(17)-C(18) | 1.555(4) |
| C(3)-C(4) | 1.524(3) | C(18)-O(18) | 1.404(3) |
| C(4)-C(8) | 1.531(3) | O(18)-C(19) | 1.467(2) |
| C(4)-C(5) | 1.546(3) | C(19)-C(22) | 1.509(4) |
| C(5)-O(5) | 1.409(3) | C(19)-C(20) | 1.521(3) |
| C(5)-C(6) | 1.529(3) | C(19)-C(21) | 1.523(3) |

**Tabelle 4: Bond angles [°] for *[47]b*.**

| | | | |
|---|---|---|---|
| C(1A)-Cr(1)-C(1E) | 90.08(14) | C(5)-O(5)-C(13) | 108.83(17) |
| C(1A)-Cr(1)-C(1C) | 85.41(12) | O(6)-C(6)-C(7) | 112.1(2) |
| C(1E)-Cr(1)-C(1C) | 93.26(11) | O(6)-C(6)-C(5) | 101.7(2) |
| C(1A)-Cr(1)-C(1B) | 85.54(11) | C(7)-C(6)-C(5) | 114.8(2) |
| C(1E)-Cr(1)-C(1B) | 88.23(10) | C(6)-O(6)-C(13) | 106.24(18) |
| C(1C)-Cr(1)-C(1B) | 170.83(10) | O(7)-C(7)-C(6) | 113.91(19) |
| C(1A)-Cr(1)-C(1D) | 93.18(15) | C(8)-O(7)-C(7) | 113.86(17) |
| C(1E)-Cr(1)-C(1D) | 176.71(11) | O(7)-C(8)-O(8) | 112.40(17) |
| C(1C)-Cr(1)-C(1D) | 87.51(11) | O(7)-C(8)-C(9) | 107.57(18) |
| C(1B)-Cr(1)-C(1D) | 91.52(10) | O(8)-C(8)-C(9) | 103.40(19) |
| C(1A)-Cr(1)-C(1) | 173.84(13) | O(7)-C(8)-C(4) | 109.68(19) |
| C(1E)-Cr(1)-C(1) | 84.46(9) | O(8)-C(8)-C(4) | 107.45(17) |
| C(1C)-Cr(1)-C(1) | 97.71(10) | C(9)-C(8)-C(4) | 116.30(17) |
| C(1B)-Cr(1)-C(1) | 91.44(9) | C(8)-O(8)-C(10) | 109.05(17) |
| C(1D)-Cr(1)-C(1) | 92.27(10) | O(9)-C(9)-C(8) | 102.60(17) |
| O(1A)-C(1A)-Cr(1) | 175.5(3) | C(10)-O(9)-C(9) | 108.82(17) |
| O(1B)-C(1B)-Cr(1) | 174.7(2) | O(9)-C(10)-O(8) | 106.13(19) |
| O(1C)-C(1C)-Cr(1) | 172.8(2) | O(9)-C(10)-C(12) | 108.8(3) |
| O(1D)-C(1D)-Cr(1) | 178.2(2) | O(8)-C(10)-C(12) | 110.1(3) |
| O(1E)-C(1E)-Cr(1) | 177.0(2) | O(9)-C(10)-C(11) | 110.1(3) |
| C(1)-O(1)-C(4) | 114.64(15) | O(8)-C(10)-C(11) | 107.4(2) |
| O(1)-C(1)-C(2) | 108.97(17) | C(12)-C(10)-C(11) | 114.0(3) |
| O(1)-C(1)-Cr(1) | 117.89(14) | O(6)-C(13)-O(5) | 104.44(19) |
| C(2)-C(1)-Cr(1) | 133.03(14) | O(6)-C(13)-C(14) | 109.4(2) |
| C(1)-C(2)-C(3) | 106.49(16) | O(5)-C(13)-C(14) | 110.1(2) |
| C(1)-C(2)-C(18) | 120.87(19) | O(6)-C(13)-C(15) | 111.7(2) |
| C(3)-C(2)-C(18) | 113.70(17) | O(5)-C(13)-C(15) | 108.3(2) |
| C(1)-C(2)-C(16) | 117.46(17) | C(14)-C(13)-C(15) | 112.5(3) |
| C(3)-C(2)-C(16) | 111.6(2) | C(17)-C(16)-C(2) | 88.37(16) |
| C(18)-C(2)-C(16) | 85.82(16) | C(16)-C(17)-C(18) | 86.86(17) |
| C(4)-C(3)-C(2) | 105.48(16) | O(18)-C(18)-C(17) | 120.71(19) |
| O(1)-C(4)-C(3) | 104.28(15) | O(18)-C(18)-C(2) | 113.96(16) |
| O(1)-C(4)-C(8) | 103.09(15) | C(17)-C(18)-C(2) | 88.12(17) |
| C(3)-C(4)-C(8) | 115.63(18) | C(18)-O(18)-C(19) | 116.66(16) |
| O(1)-C(4)-C(5) | 108.24(17) | O(18)-C(19)-C(22) | 103.16(18) |
| C(3)-C(4)-C(5) | 112.19(17) | O(18)-C(19)-C(20) | 109.97(18) |
| C(8)-C(4)-C(5) | 112.38(18) | C(22)-C(19)-C(20) | 111.4(2) |
| O(5)-C(5)-C(6) | 104.5(2) | O(18)-C(19)-C(21) | 111.30(17) |
| O(5)-C(5)-C(4) | 110.90(18) | C(22)-C(19)-C(21) | 110.6(2) |
| C(6)-C(5)-C(4) | 115.32(19) | C(20)-C(19)-C(21) | 110.2(2) |

**Tabelle 5: Torsion angles [°] for *[47]b.***

| | | | |
|---|---|---|---|
| C(1E)-Cr(1)-C(1A)-O(1A) | -30(4) | C(1)-O(1)-C(4)-C(3) | -1.7(2) |
| C(1C)-Cr(1)-C(1A)-O(1A) | -124(4) | C(1)-O(1)-C(4)-C(8) | 119.51(17) |
| C(1B)-Cr(1)-C(1A)-O(1A) | 58(4) | C(1)-O(1)-C(4)-C(5) | -121.27(18) |
| C(1D)-Cr(1)-C(1A)-O(1A) | 149(4) | C(2)-C(3)-C(4)-O(1) | -1.0(2) |
| C(1)-Cr(1)-C(1A)-O(1A) | -3(5) | C(2)-C(3)-C(4)-C(8) | -113.45(19) |
| C(1A)-Cr(1)-C(1B)-O(1B) | 0(2) | C(2)-C(3)-C(4)-C(5) | 115.88(19) |
| C(1E)-Cr(1)-C(1B)-O(1B) | 90(2) | O(1)-C(4)-C(5)-O(5) | 41.5(2) |
| C(1C)-Cr(1)-C(1B)-O(1B) | -9(3) | C(3)-C(4)-C(5)-O(5) | -73.0(2) |
| C(1D)-Cr(1)-C(1B)-O(1B) | -93(2) | C(8)-C(4)-C(5)-O(5) | 154.73(17) |
| C(1)-Cr(1)-C(1B)-O(1B) | 175(2) | O(1)-C(4)-C(5)-C(6) | -76.9(2) |
| C(1A)-Cr(1)-C(1C)-O(1C) | -10(2) | C(3)-C(4)-C(5)-C(6) | 168.6(2) |
| C(1E)-Cr(1)-C(1C)-O(1C) | -100(2) | C(8)-C(4)-C(5)-C(6) | 36.3(3) |
| C(1B)-Cr(1)-C(1C)-O(1C) | -1(3) | C(6)-C(5)-O(5)-C(13) | 12.2(2) |
| C(1D)-Cr(1)-C(1C)-O(1C) | 83(2) | C(4)-C(5)-O(5)-C(13) | -112.6(2) |
| C(1)-Cr(1)-C(1C)-O(1C) | 175(2) | O(5)-C(5)-C(6)-O(6) | -30.8(2) |
| C(1A)-Cr(1)-C(1D)-O(1D) | 53(8) | C(4)-C(5)-C(6)-O(6) | 91.2(2) |
| C(1E)-Cr(1)-C(1D)-O(1D) | -136(7) | O(5)-C(5)-C(6)-C(7) | -152.0(2) |
| C(1C)-Cr(1)-C(1D)-O(1D) | -33(8) | C(4)-C(5)-C(6)-C(7) | -30.1(3) |
| C(1B)-Cr(1)-C(1D)-O(1D) | 138(8) | C(7)-C(6)-O(6)-C(13) | 161.38(19) |
| C(1)-Cr(1)-C(1D)-O(1D) | -130(8) | C(5)-C(6)-O(6)-C(13) | 38.2(2) |
| C(1A)-Cr(1)-C(1E)-O(1E) | 84(4) | O(6)-C(6)-C(7)-O(7) | -76.2(3) |
| C(1C)-Cr(1)-C(1E)-O(1E) | 170(4) | C(5)-C(6)-C(7)-O(7) | 39.2(3) |
| C(1B)-Cr(1)-C(1E)-O(1E) | -1(4) | C(6)-C(7)-O(7)-C(8) | -58.3(3) |
| C(1D)-Cr(1)-C(1E)-O(1E) | -87(4) | C(7)-O(7)-C(8)-O(8) | -55.4(3) |
| C(1)-Cr(1)-C(1E)-O(1E) | -93(4) | C(7)-O(7)-C(8)-C(9) | -168.59(18) |
| C(4)-O(1)-C(1)-C(2) | 3.7(2) | C(7)-O(7)-C(8)-C(4) | 64.1(2) |
| C(4)-O(1)-C(1)-Cr(1) | -172.99(12) | O(1)-C(4)-C(8)-O(7) | 64.35(19) |
| C(1A)-Cr(1)-C(1)-O(1) | 57.0(11) | C(3)-C(4)-C(8)-O(7) | 177.45(17) |
| C(1E)-Cr(1)-C(1)-O(1) | 84.55(16) | C(5)-C(4)-C(8)-O(7) | -52.0(2) |
| C(1C)-Cr(1)-C(1)-O(1) | 177.10(16) | O(1)-C(4)-C(8)-O(8) | -173.20(16) |
| C(1B)-Cr(1)-C(1)-O(1) | -3.53(16) | C(3)-C(4)-C(8)-O(8) | -60.1(2) |
| C(1D)-Cr(1)-C(1)-O(1) | -95.11(16) | C(5)-C(4)-C(8)-O(8) | 70.5(2) |
| C(1A)-Cr(1)-C(1)-C(2) | -118.6(10) | O(1)-C(4)-C(8)-C(9) | -58.0(2) |
| C(1E)-Cr(1)-C(1)-C(2) | -91.1(2) | C(3)-C(4)-C(8)-C(9) | 55.2(3) |
| C(1C)-Cr(1)-C(1)-C(2) | 1.4(2) | C(5)-C(4)-C(8)-C(9) | -174.26(18) |
| C(1B)-Cr(1)-C(1)-C(2) | -179.2(2) | O(7)-C(8)-O(8)-C(10) | -94.9(2) |
| C(1D)-Cr(1)-C(1)-C(2) | 89.22(19) | C(9)-C(8)-O(8)-C(10) | 20.8(2) |
| O(1)-C(1)-C(2)-C(3) | -4.1(2) | C(4)-C(8)-O(8)-C(10) | 144.4(2) |
| Cr(1)-C(1)-C(2)-C(3) | 171.84(15) | O(7)-C(8)-C(9)-O(9) | 88.6(2) |
| O(1)-C(1)-C(2)-C(18) | -135.82(18) | O(8)-C(8)-C(9)-O(9) | -30.5(2) |
| Cr(1)-C(1)-C(2)-C(18) | 40.1(3) | C(4)-C(8)-C(9)-O(9) | -148.04(19) |
| O(1)-C(1)-C(2)-C(16) | 121.8(2) | C(8)-C(9)-O(9)-C(10) | 29.8(3) |
| Cr(1)-C(1)-C(2)-C(16) | -62.3(3) | C(9)-O(9)-C(10)-O(8) | -17.7(3) |
| C(1)-C(2)-C(3)-C(4) | 3.0(2) | C(9)-O(9)-C(10)-C(12) | -136.2(3) |
| C(18)-C(2)-C(3)-C(4) | 138.58(18) | C(9)-O(9)-C(10)-C(11) | 98.2(3) |
| C(16)-C(2)-C(3)-C(4) | -126.37(17) | C(8)-O(8)-C(10)-O(9) | -3.2(3) |

| | | | |
|---|---|---|---|
| C(8)-O(8)-C(10)-C(12) | 114.4(3) | C(16)-C(17)-C(18)-C(2) | 24.69(19) |
| C(8)-O(8)-C(10)-C(11) | -121.0(2) | C(1)-C(2)-C(18)-O(18) | 93.0(2) |
| C(6)-O(6)-C(13)-O(5) | -31.5(2) | C(3)-C(2)-C(18)-O(18) | -35.5(3) |
| C(6)-O(6)-C(13)-C(14) | -149.35(19) | C(16)-C(2)-C(18)-O(18) | -147.3(2) |
| C(6)-O(6)-C(13)-C(15) | 85.4(2) | C(1)-C(2)-C(18)-C(17) | -144.0(2) |
| C(5)-O(5)-C(13)-O(6) | 10.8(2) | C(3)-C(2)-C(18)-C(17) | 87.5(2) |
| C(5)-O(5)-C(13)-C(14) | 128.2(2) | C(16)-C(2)-C(18)-C(17) | -24.30(19) |
| C(5)-O(5)-C(13)-C(15) | -108.5(2) | C(17)-C(18)-O(18)-C(19) | 87.2(3) |
| C(1)-C(2)-C(16)-C(17) | 147.3(2) | C(2)-C(18)-O(18)-C(19) | -169.96(18) |
| C(3)-C(2)-C(16)-C(17) | -89.3(2) | C(18)-O(18)-C(19)-C(22) | -179.8(2) |
| C(18)-C(2)-C(16)-C(17) | 24.5(2) | C(18)-O(18)-C(19)-C(20) | -60.8(2) |
| C(2)-C(16)-C(17)-C(18) | -24.6(2) | C(18)-O(18)-C(19)-C(21) | 61.5(2) |
| C(16)-C(17)-C(18)-O(18) | 141.6(2) | | |

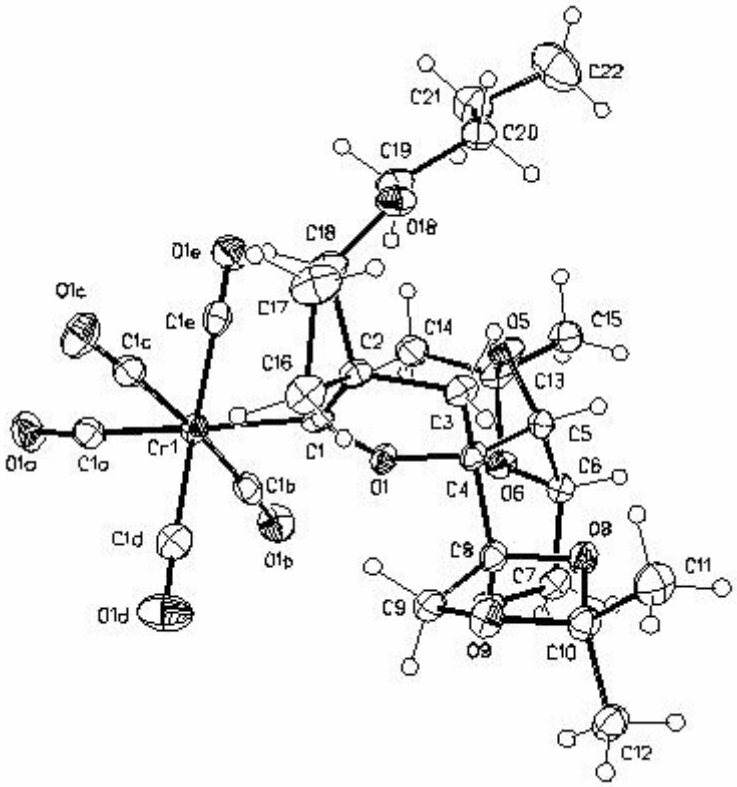
**Tabelle 6: Hydrogen bonds for *[47]b* [Å and °].**

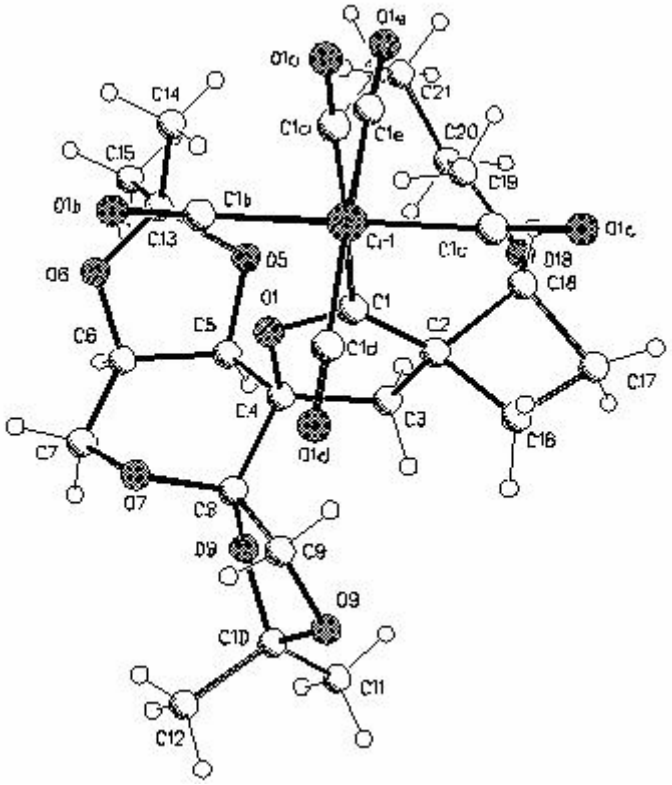
| **D-H...A** | **d(D-H)** | **d(H...A)** | **d(D...A)** | **<(DHA)** |
|---|---|---|---|---|
| C(17)-H(17A)...O(1A)[a] | 0.99 | 2.39 | 3.354(4) | 164.8 |
| C(16)-H(16A)...O(1E)[b] | 0.99 | 2.56 | 3.108(3) | 114.9 |

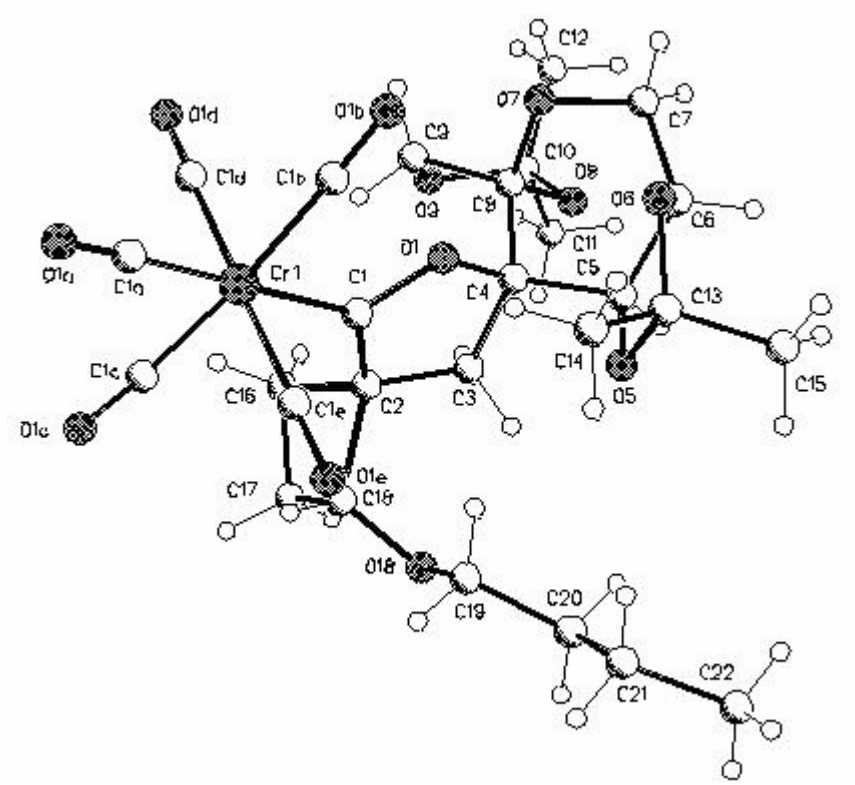
Symmetry transformations used to generate equivalent atoms:

[a] 1 x-1/2,-y+3/2,-z+1 [b] x-1,y,z

**11. (*1R,4S,7R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-1-*n*-butoxy-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxa-cyclopentan-5´,7-6-oxaspiro[3.4]oct]-5-yliden}chrom(0) *[48]a***




C12
O7
C7
O1d
O1b
C9
C10
O8
C1d
C1b
O3
C8
O6
C6
C11
O1
C5
O1a
C1a
Cr1
C1
C4
C13
C14
C1c
C16
C1e
C2
C3
O5
C15
O1c
O1e
C17
C1f
O18
C19
C20
C21
C22

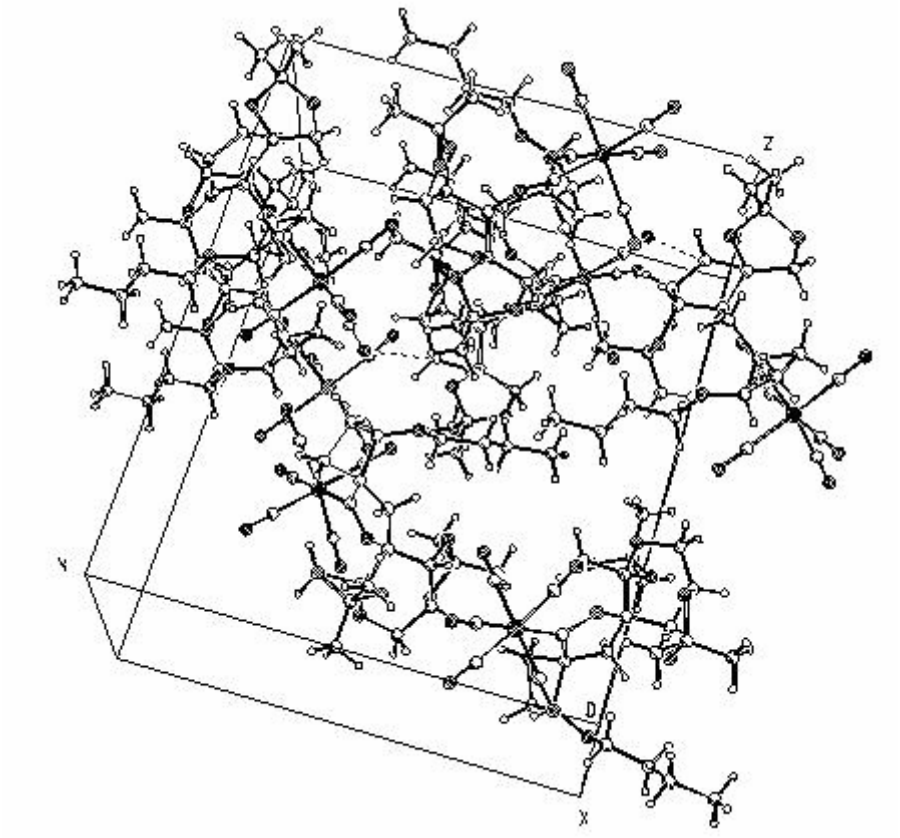

Z
Y
X

**Table 1: Crystal data and structure refinements for *[48]a***

| | |
|---|---|
| Identification code | ***[48]a*** |
| Empirical formula | $C_{27}H_{34}CrO_{12}$ |
| Formula weight | 602.54 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 8.8865(1) Å α = 90° |
| | b = 16.2665(2) Å β = 90° |
| | c = 20.0159(3) Å γ = 90° |
| Volume | 2893.34(6) $Å^3$ |
| Z | 4 |
| Calculated density | 1.383 $mg/m^3$ |
| Absorption coefficient | 0.456 $mm^{-1}$ |
| F(000) | 1264 |
| Crystal size | 0.3 x 0.15 x 0.05 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.61 to 25.00° |
| Limiting indices | $-10 \le h \le 10$, $-19 \le k \le 19$, $-23 \le l \le 23$ |
| Reflections collected / unique | 28530 / 5083 [R(int) = 0.0440] |
| Completeness to Θ = 25.00 | 99.7 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 5083 / 0 / 361 |
| Goodness-of-fit on $F^2$ | 0.962 |
| Final R indices [I>2σ(I)] | R1 = 0.0259, wR2 = 0.0502 |
| R indices (all data) | R1 = 0.0335, wR2 = 0.0517 |
| Absolute structure parameter | -0.007(12) |
| Largest diff. peak and hole | 0.175 and -0.249 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[48]a*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 1978(1) | 2023(1) | 1372(1) | 20(1) |
| C(1A) | 462(2) | 2736(1) | 1059(1) | 25(1) |
| O(1A) | -472(2) | 3144(1) | 842(1) | 36(1) |
| C(1B) | 499(2) | 1502(1) | 1927(1) | 23(1) |
| O(1B) | -400(2) | 1215(1) | 2258(1) | 32(1) |
| C(1C) | 3356(2) | 2616(1) | 843(1) | 25(1) |
| O(1C) | 4126(2) | 3019(1) | 511(1) | 37(1) |
| C(1D) | 2495(2) | 2710(1) | 2107(1) | 27(1) |
| O(1D) | 2829(2) | 3101(1) | 2557(1) | 43(1) |
| C(1E) | 1573(2) | 1312(1) | 640(1) | 23(1) |
| O(1E) | 1362(2) | 888(1) | 193(1) | 33(1) |
| O(1) | 3039(1) | 638(1) | 2147(1) | 19(1) |
| C(1) | 3492(2) | 1176(1) | 1696(1) | 18(1) |
| C(2) | 5117(2) | 1008(1) | 1526(1) | 19(1) |
| C(3) | 5570(2) | 256(1) | 1938(1) | 21(1) |
| C(4) | 4244(2) | 88(1) | 2398(1) | 18(1) |
| C(5) | 3672(2) | -802(1) | 2373(1) | 20(1) |
| O(5) | 2956(1) | -956(1) | 1746(1) | 23(1) |
| C(6) | 2487(2) | -1021(1) | 2889(1) | 21(1) |
| O(6) | 1112(1) | -879(1) | 2537(1) | 22(1) |
| C(7) | 2565(2) | -549(1) | 3540(1) | 25(1) |
| O(7) | 3095(1) | 277(1) | 3474(1) | 22(1) |
| C(8) | 4463(2) | 347(1) | 3130(1) | 20(1) |
| O(8) | 5605(1) | -174(1) | 3383(1) | 22(1) |
| C(9) | 5057(2) | 1221(1) | 3254(1) | 23(1) |
| O(9) | 6569(1) | 1112(1) | 3464(1) | 25(1) |
| C(10) | 6643(2) | 322(1) | 3773(1) | 23(1) |
| C(11) | 8210(2) | -22(1) | 3681(1) | 32(1) |
| C(12) | 6128(2) | 344(1) | 4498(1) | 28(1) |
| C(13) | 1375(2) | -1151(1) | 1871(1) | 22(1) |
| C(14) | 396(2) | -658(1) | 1409(1) | 29(1) |
| C(15) | 1168(2) | -2070(1) | 1795(1) | 28(1) |
| C(16) | 6259(2) | 1741(1) | 1556(1) | 27(1) |
| C(17) | 7003(2) | 1425(1) | 910(1) | 35(1) |
| C(18) | 5572(2) | 942(1) | 761(1) | 25(1) |
| O(18) | 5808(1) | 156(1) | 505(1) | 26(1) |
| C(19) | 4425(2) | -236(1) | 306(1) | 24(1) |
| C(20) | 4704(2) | -1139(1) | 199(1) | 28(1) |
| C(21) | 3344(2) | -1567(1) | -102(1) | 36(1) |
| C(22) | 3547(3) | -2492(2) | -149(1) | 56(1) |

**Tabelle 3: Bond lengths [Å] for *[48]a*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1C) | 1.884(2) | O(5)-C(13) | 1.462(2) |
| Cr(1)-C(1A) | 1.884(2) | C(6)-O(6) | 1.428(2) |
| Cr(1)-C(1E) | 1.901(2) | C(6)-C(7) | 1.514(3) |
| Cr(1)-C(1D) | 1.904(2) | O(6)-C(13) | 1.424(2) |
| Cr(1)-C(1B) | 1.918(2) | C(7)-O(7) | 1.430(2) |
| Cr(1)-C(1) | 2.032(2) | O(7)-C(8) | 1.403(2) |
| C(1A)-O(1A) | 1.148(2) | C(8)-O(8) | 1.416(2) |
| C(1B)-O(1B) | 1.139(2) | C(8)-C(9) | 1.538(3) |
| C(1C)-O(1C) | 1.157(2) | O(8)-C(10) | 1.453(2) |
| C(1D)-O(1D) | 1.141(2) | C(9)-O(9) | 1.419(2) |
| C(1E)-O(1E) | 1.145(2) | O(9)-C(10) | 1.427(2) |
| O(1)-C(1) | 1.321(2) | C(10)-C(11) | 1.512(2) |
| O(1)-C(4) | 1.483(2) | C(10)-C(12) | 1.522(3) |
| C(1)-C(2) | 1.508(2) | C(13)-C(14) | 1.501(3) |
| C(2)-C(3) | 1.529(3) | C(13)-C(15) | 1.513(3) |
| C(2)-C(16) | 1.567(3) | C(16)-C(17) | 1.541(3) |
| C(2)-C(18) | 1.589(3) | C(17)-C(18) | 1.524(3) |
| C(3)-C(4) | 1.521(2) | C(18)-O(18) | 1.394(2) |
| C(4)-C(5) | 1.535(3) | O(18)-C(19) | 1.441(2) |
| C(4)-C(8) | 1.536(3) | C(19)-C(20) | 1.505(3) |
| C(5)-O(5) | 1.429(2) | C(20)-C(21) | 1.519(3) |
| C(5)-C(6) | 1.518(2) | C(21)-C(22) | 1.519(3) |

**Tabelle 4: Bond angles [°] for *[48]a*.**

| | | | |
|---|---|---|---|
| C(1C)-Cr(1)-C(1A) | 87.87(8) | C(6)-C(5)-C(4) | 115.38(16) |
| C(1C)-Cr(1)-C(1E) | 90.08(8) | C(5)-O(5)-C(13) | 108.37(13) |
| C(1A)-Cr(1)-C(1E) | 89.01(8) | O(6)-C(6)-C(7) | 112.36(15) |
| C(1C)-Cr(1)-C(1D) | 88.66(9) | O(6)-C(6)-C(5) | 102.73(13) |
| C(1A)-Cr(1)-C(1D) | 93.90(9) | C(7)-C(6)-C(5) | 115.75(16) |
| C(1E)-Cr(1)-C(1D) | 176.79(8) | C(13)-O(6)-C(6) | 105.67(13) |
| C(1C)-Cr(1)-C(1B) | 175.37(9) | O(7)-C(7)-C(6) | 114.42(15) |
| C(1A)-Cr(1)-C(1B) | 88.54(8) | C(8)-O(7)-C(7) | 113.98(14) |
| C(1E)-Cr(1)-C(1B) | 92.77(8) | O(7)-C(8)-O(8) | 113.38(15) |
| C(1D)-Cr(1)-C(1B) | 88.67(8) | O(7)-C(8)-C(4) | 109.67(14) |
| C(1C)-Cr(1)-C(1) | 95.51(8) | O(8)-C(8)-C(4) | 105.59(14) |
| C(1A)-Cr(1)-C(1) | 175.23(8) | O(7)-C(8)-C(9) | 106.99(15) |
| C(1E)-Cr(1)-C(1) | 87.63(8) | O(8)-C(8)-C(9) | 104.47(14) |
| C(1D)-Cr(1)-C(1) | 89.55(8) | C(4)-C(8)-C(9) | 116.83(16) |
| C(1B)-Cr(1)-C(1) | 88.25(8) | C(8)-O(8)-C(10) | 108.36(14) |
| O(1A)-C(1A)-Cr(1) | 176.61(18) | O(9)-C(9)-C(8) | 104.87(15) |
| O(1B)-C(1B)-Cr(1) | 177.92(18) | C(9)-O(9)-C(10) | 106.58(14) |
| O(1C)-C(1C)-Cr(1) | 175.36(18) | O(9)-C(10)-O(8) | 103.74(13) |
| O(1D)-C(1D)-Cr(1) | 177.8(2) | O(9)-C(10)-C(11) | 108.86(15) |
| O(1E)-C(1E)-Cr(1) | 178.45(17) | O(8)-C(10)-C(11) | 108.26(16) |
| C(1)-O(1)-C(4) | 114.30(14) | O(9)-C(10)-C(12) | 112.27(16) |
| O(1)-C(1)-C(2) | 109.03(16) | O(8)-C(10)-C(12) | 109.50(15) |
| O(1)-C(1)-Cr(1) | 117.75(12) | C(11)-C(10)-C(12) | 113.68(17) |
| C(2)-C(1)-Cr(1) | 133.22(14) | O(6)-C(13)-O(5) | 104.56(14) |
| C(1)-C(2)-C(3) | 106.02(15) | O(6)-C(13)-C(14) | 108.40(16) |
| C(1)-C(2)-C(16) | 118.33(17) | O(5)-C(13)-C(14) | 109.59(16) |
| C(3)-C(2)-C(16) | 114.70(15) | O(6)-C(13)-C(15) | 112.42(16) |
| C(1)-C(2)-C(18) | 118.20(15) | O(5)-C(13)-C(15) | 108.27(15) |
| C(3)-C(2)-C(18) | 113.46(16) | C(14)-C(13)-C(15) | 113.22(17) |
| C(16)-C(2)-C(18) | 85.61(14) | C(17)-C(16)-C(2) | 89.54(15) |
| C(4)-C(3)-C(2) | 105.41(15) | C(18)-C(17)-C(16) | 88.79(15) |
| O(1)-C(4)-C(3) | 104.23(14) | O(18)-C(18)-C(17) | 114.79(17) |
| O(1)-C(4)-C(5) | 108.61(14) | O(18)-C(18)-C(2) | 117.06(16) |
| C(3)-C(4)-C(5) | 113.93(16) | C(17)-C(18)-C(2) | 89.31(14) |
| O(1)-C(4)-C(8) | 104.43(14) | C(18)-O(18)-C(19) | 112.24(14) |
| C(3)-C(4)-C(8) | 115.46(15) | O(18)-C(19)-C(20) | 109.28(16) |
| C(5)-C(4)-C(8) | 109.41(15) | C(19)-C(20)-C(21) | 111.86(17) |
| O(5)-C(5)-C(6) | 104.30(14) | C(22)-C(21)-C(20) | 112.55(19) |
| O(5)-C(5)-C(4) | 110.02(15) | | |

**Tabelle 5: Torsion angles [°] for *[48]a.***

| | | | |
|---|---|---|---|
| C(1C)-Cr(1)-C(1A)-O(1A) | 89(3) | C(1)-O(1)-C(4)-C(3) | -9.6(2) |
| C(1E)-Cr(1)-C(1A)-O(1A) | -1(3) | C(1)-O(1)-C(4)-C(5) | -131.37(15) |
| C(1D)-Cr(1)-C(1A)-O(1A) | 177(100) | C(1)-O(1)-C(4)-C(8) | 111.97(16) |
| C(1B)-Cr(1)-C(1A)-O(1A) | -94(3) | C(2)-C(3)-C(4)-O(1) | 9.74(19) |
| C(1)-Cr(1)-C(1A)-O(1A) | -47(4) | C(2)-C(3)-C(4)-C(5) | 127.95(16) |
| C(1C)-Cr(1)-C(1B)-O(1B) | 6(5) | C(2)-C(3)-C(4)-C(8) | -104.18(18) |
| C(1A)-Cr(1)-C(1B)-O(1B) | -33(5) | O(1)-C(4)-C(5)-O(5) | 45.70(18) |
| C(1E)-Cr(1)-C(1B)-O(1B) | -122(5) | C(3)-C(4)-C(5)-O(5) | -69.97(18) |
| C(1D)-Cr(1)-C(1B)-O(1B) | 61(5) | C(8)-C(4)-C(5)-O(5) | 159.11(14) |
| C(1)-Cr(1)-C(1B)-O(1B) | 151(5) | O(1)-C(4)-C(5)-C(6) | -71.94(18) |
| C(1A)-Cr(1)-C(1C)-O(1C) | 9(2) | C(3)-C(4)-C(5)-C(6) | 172.39(15) |
| C(1E)-Cr(1)-C(1C)-O(1C) | 98(2) | C(8)-C(4)-C(5)-C(6) | 41.5(2) |
| C(1D)-Cr(1)-C(1C)-O(1C) | -85(2) | C(6)-C(5)-O(5)-C(13) | 7.03(19) |
| C(1B)-Cr(1)-C(1C)-O(1C) | -30(3) | C(4)-C(5)-O(5)-C(13) | -117.28(16) |
| C(1)-Cr(1)-C(1C)-O(1C) | -174(2) | O(5)-C(5)-C(6)-O(6) | -27.42(18) |
| C(1C)-Cr(1)-C(1D)-O(1D) | -107(5) | C(4)-C(5)-C(6)-O(6) | 93.38(17) |
| C(1A)-Cr(1)-C(1D)-O(1D) | 165(5) | O(5)-C(5)-C(6)-C(7) | -150.23(16) |
| C(1E)-Cr(1)-C(1D)-O(1D) | -40(6) | C(4)-C(5)-C(6)-C(7) | -29.4(2) |
| C(1B)-Cr(1)-C(1D)-O(1D) | 77(5) | C(7)-C(6)-O(6)-C(13) | 163.32(16) |
| C(1)-Cr(1)-C(1D)-O(1D) | -11(5) | C(5)-C(6)-O(6)-C(13) | 38.26(18) |
| C(1C)-Cr(1)-C(1E)-O(1E) | 28(7) | O(6)-C(6)-C(7)-O(7) | -85.04(19) |
| C(1A)-Cr(1)-C(1E)-O(1E) | 115(7) | C(5)-C(6)-C(7)-O(7) | 32.5(2) |
| C(1D)-Cr(1)-C(1E)-O(1E) | -39(8) | C(6)-C(7)-O(7)-C(8) | -52.1(2) |
| C(1B)-Cr(1)-C(1E)-O(1E) | -156(7) | C(7)-O(7)-C(8)-O(8) | -51.7(2) |
| C(1)-Cr(1)-C(1E)-O(1E) | -68(7) | C(7)-O(7)-C(8)-C(4) | 66.06(18) |
| C(4)-O(1)-C(1)-C(2) | 5.0(2) | C(7)-O(7)-C(8)-C(9) | -166.31(14) |
| C(4)-O(1)-C(1)-Cr(1) | -175.10(11) | O(1)-C(4)-C(8)-O(7) | 57.17(18) |
| C(1C)-Cr(1)-C(1)-O(1) | 174.49(14) | C(3)-C(4)-C(8)-O(7) | 170.97(16) |
| C(1A)-Cr(1)-C(1)-O(1) | -50.5(11) | C(5)-C(4)-C(8)-O(7) | -58.94(19) |
| C(1E)-Cr(1)-C(1)-O(1) | -95.65(14) | O(1)-C(4)-C(8)-O(8) | 179.67(13) |
| C(1D)-Cr(1)-C(1)-O(1) | 85.88(14) | C(3)-C(4)-C(8)-O(8) | -66.52(19) |
| C(1B)-Cr(1)-C(1)-O(1) | -2.80(15) | C(5)-C(4)-C(8)-O(8) | 63.57(18) |
| C(1C)-Cr(1)-C(1)-C(2) | -5.64(19) | O(1)-C(4)-C(8)-C(9) | -64.75(19) |
| C(1A)-Cr(1)-C(1)-C(2) | 129.4(10) | C(3)-C(4)-C(8)-C(9) | 49.1(2) |
| C(1E)-Cr(1)-C(1)-C(2) | 84.22(18) | C(5)-C(4)-C(8)-C(9) | 179.15(15) |
| C(1D)-Cr(1)-C(1)-C(2) | -94.25(18) | O(7)-C(8)-O(8)-C(10) | -103.65(17) |
| C(1B)-Cr(1)-C(1)-C(2) | 177.07(18) | C(4)-C(8)-O(8)-C(10) | 136.25(14) |
| O(1)-C(1)-C(2)-C(3) | 1.7(2) | C(9)-C(8)-O(8)-C(10) | 12.48(18) |
| Cr(1)-C(1)-C(2)-C(3) | -178.14(14) | O(7)-C(8)-C(9)-O(9) | 129.67(14) |
| O(1)-C(1)-C(2)-C(16) | -128.70(17) | O(8)-C(8)-C(9)-O(9) | 9.18(18) |
| Cr(1)-C(1)-C(2)-C(16) | 51.4(2) | C(4)-C(8)-C(9)-O(9) | -107.03(17) |
| O(1)-C(1)-C(2)-C(18) | 130.34(17) | C(8)-C(9)-O(9)-C(10) | -27.76(18) |
| Cr(1)-C(1)-C(2)-C(18) | -49.5(3) | C(9)-O(9)-C(10)-O(8) | 35.49(17) |
| C(1)-C(2)-C(3)-C(4) | -7.31(19) | C(9)-O(9)-C(10)-C(11) | 150.60(15) |
| C(16)-C(2)-C(3)-C(4) | 125.18(17) | C(9)-O(9)-C(10)-C(12) | -82.64(18) |
| C(18)-C(2)-C(3)-C(4) | -138.64(16) | C(8)-O(8)-C(10)-O(9) | -29.58(17) |

| | | | |
|---|---|---|---|
| C(8)-O(8)-C(10)-C(11) | -145.11(16) | C(16)-C(17)-C(18)-C(2) | -19.60(15) |
| C(8)-O(8)-C(10)-C(12) | 90.46(17) | C(1)-C(2)-C(18)-O(18) | -103.1(2) |
| C(6)-O(6)-C(13)-O(5) | -34.29(18) | C(3)-C(2)-C(18)-O(18) | 22.0(2) |
| C(6)-O(6)-C(13)-C(14) | -151.14(16) | C(16)-C(2)-C(18)-O(18) | 137.01(17) |
| C(6)-O(6)-C(13)-C(15) | 82.94(18) | C(1)-C(2)-C(18)-C(17) | 139.24(19) |
| C(5)-O(5)-C(13)-O(6) | 16.08(19) | C(3)-C(2)-C(18)-C(17) | -95.74(18) |
| C(5)-O(5)-C(13)-C(14) | 132.10(16) | C(16)-C(2)-C(18)-C(17) | 19.31(15) |
| C(5)-O(5)-C(13)-C(15) | -103.97(17) | C(17)-C(18)-O(18)-C(19) | -173.73(16) |
| C(1)-C(2)-C(16)-C(17) | -138.90(17) | C(2)-C(18)-O(18)-C(19) | 83.49(19) |
| C(3)-C(2)-C(16)-C(17) | 94.74(17) | C(18)-O(18)-C(19)-C(20) | -166.05(15) |
| C(18)-C(2)-C(16)-C(17) | -19.10(15) | O(18)-C(19)-C(20)-C(21) | -172.21(16) |
| C(2)-C(16)-C(17)-C(18) | 19.89(15) | C(19)-C(20)-C(21)-C(22) | -174.21(19) |
| C(16)-C(17)-C(18)-O(18) | -139.31(17) | | |

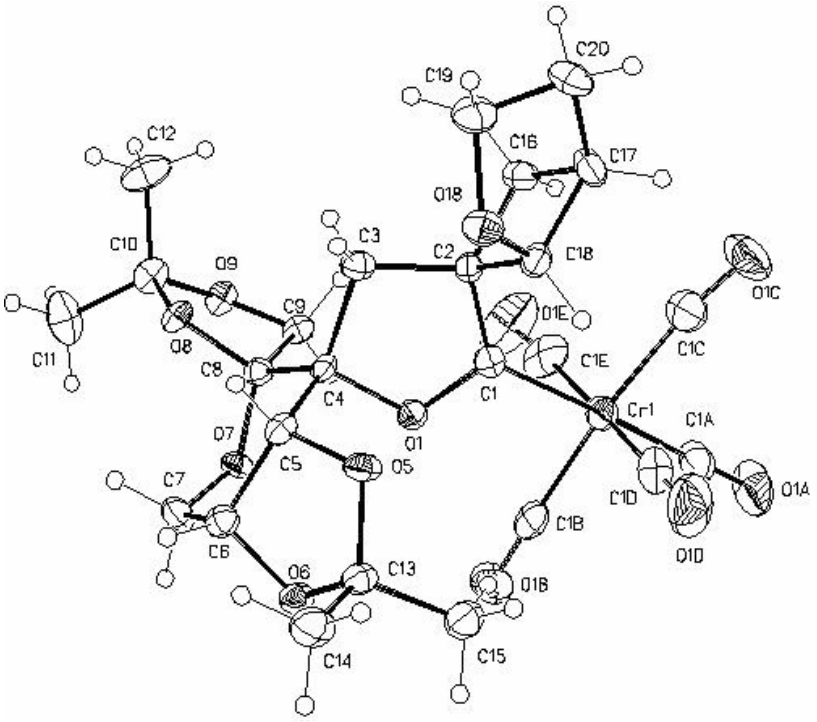
**Tabelle 6: Hydrogen bonds for *[48]a* [Å and °].**

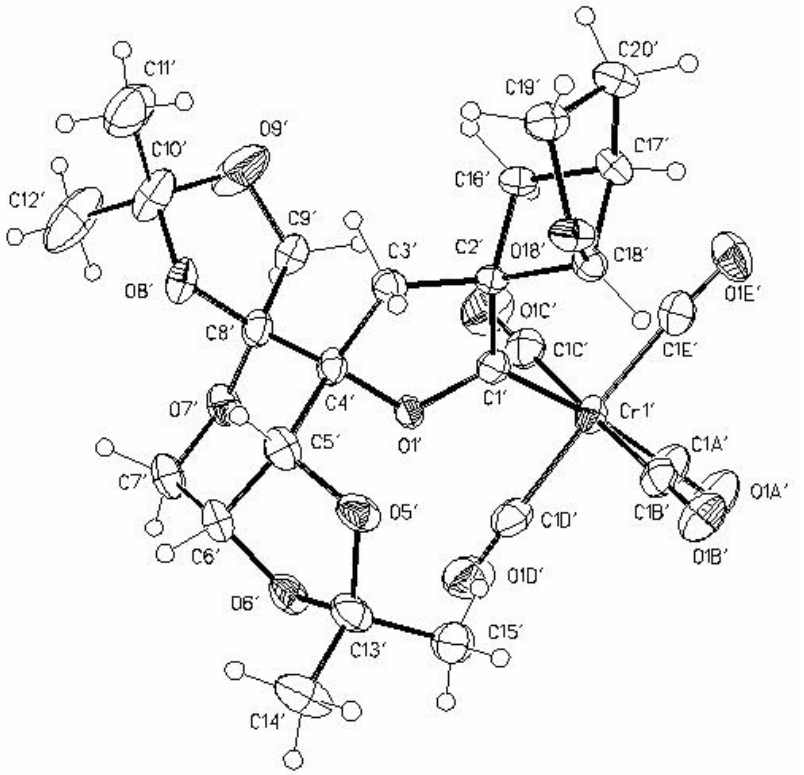
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(7)-H(7A)...O(1A)[a] | 0.99 | 2.49 | 3.084(2) | 118.0 |
| C(16)-H(16B)...O(1B)[b] | 0.99 | 2.59 | 3.394(2) | 138.6 |
| C(5)-H(5)...O(1D)[c] | 1.00 | 2.61 | 3.587(2) | 167.0 |

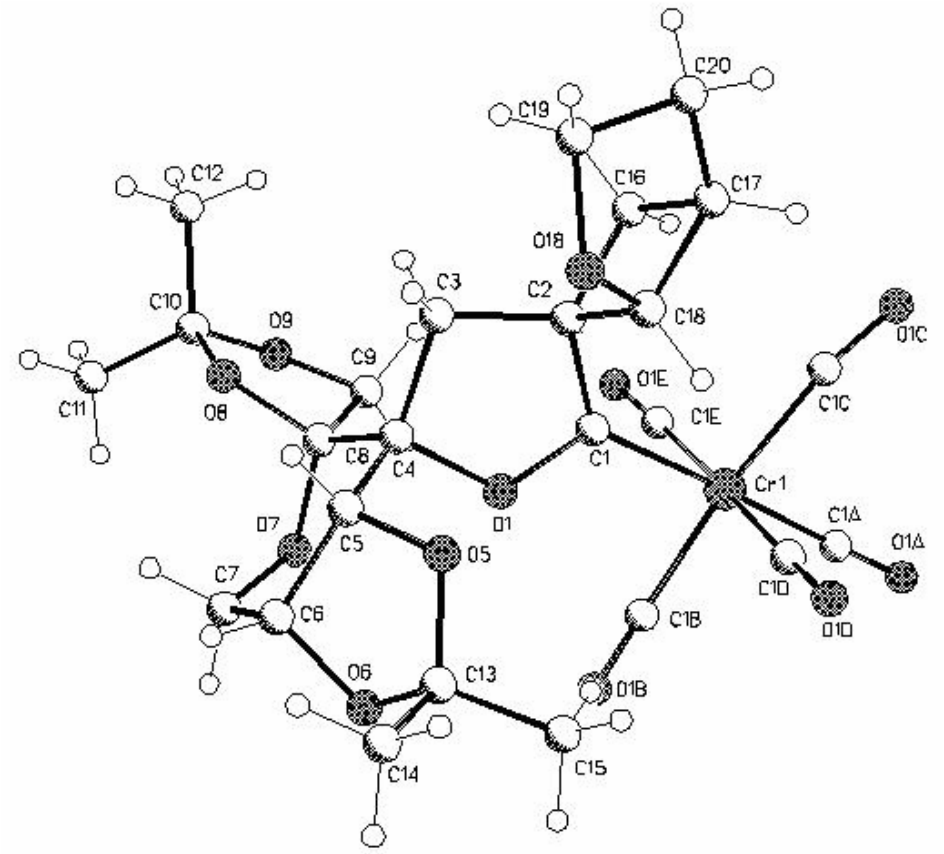
Symmetry transformations used to generate equivalent atoms:

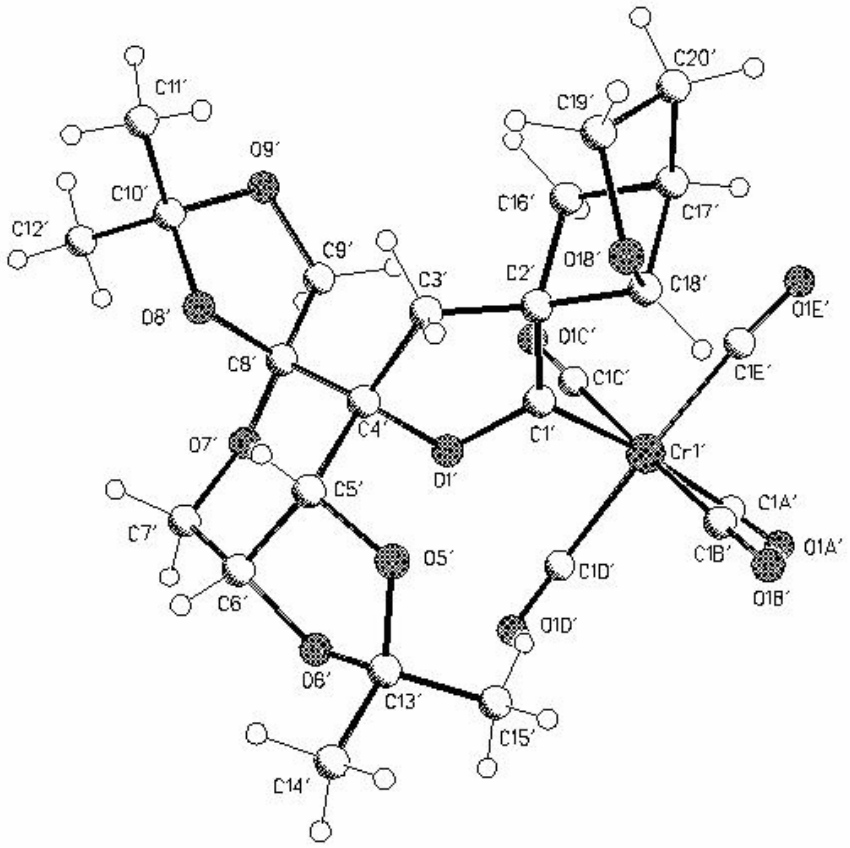
[a] -x,y-1/2,-z+1/2 [b] x+1,y,z [c] -x+1,y-1/2,-z+1/2

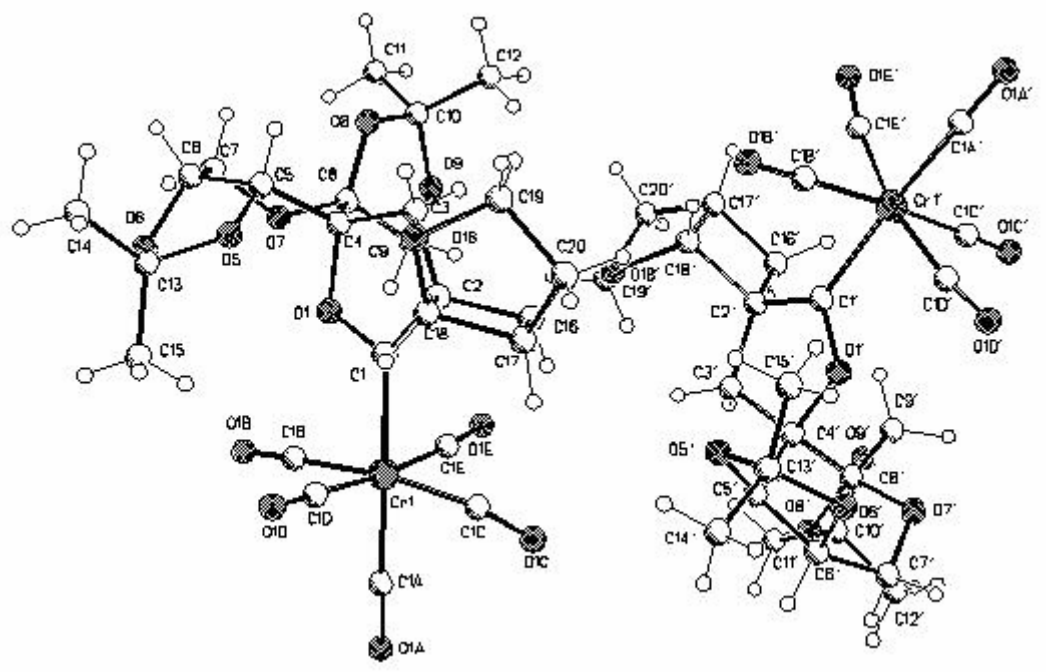
**12.** **(*3R,5S,1´R,5´S,1´´R,4´´S,6´´R*)-Pentacarbonyl{8´´,8´´,2´´´,2´´´-tetramethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´´,4´´´-1,3-dioxacyclopentan-5´´,3]-spiro[2-oxabicyclo[3.2.0]heptan-7´,5]-2-oxacyclopent-1-yliden}chrom(0)** ***[50]a***




C20
C19
C12
C16
C17
O18
C3
C2
C10
O9
C18
O1C
C9
O1E
C11
O8
C1E
C1C
C8
C4
C1
Cr1
O7
O1
C1A
O1A
C5
O5
C7
C1D
O1D
C6
C1B
O6
C13
O1B
C14
C15


C11′
C20′
C19′
O9′
C10′
C16′
C17′
C12′
C9′
O18′
C2′
C3′
C18′
O1E′
O8′
O1C′
C8′
C1C′
C1E′
C4′
C1′
O7′
Cr1′
O1′
C5′
C1A′
C7′
O5′
C1B′
O1A′
C1D′
C6′
O1B′
O1D′
O6′
C13′
C15′
C14′


C11
C12
O8
C10
C8
C7
C5
O6
O9
C19
C14
O5
O7
C4
C3
O16
C20
C13
C9
C2
O1
C18
C17
C15
C1
O1B
C1B
O1E
C1E
Cr1
O1D
C1D
C1C
O1C
C1A
O1A
O1E'
O1A'
C1E'
C1A'
O1B'
C1B'
C20'
C17'
Cr1'
C1C'
O1C'
C16'
C18'
O1C'
C19'
C2'
C1'
O1D'
C3'
C15'
O1'
O9'
C4'
O8'
O5'
C13'
C8'
C5'
O6'
O7'
C14'
C10'
C11'
C7'
C12'

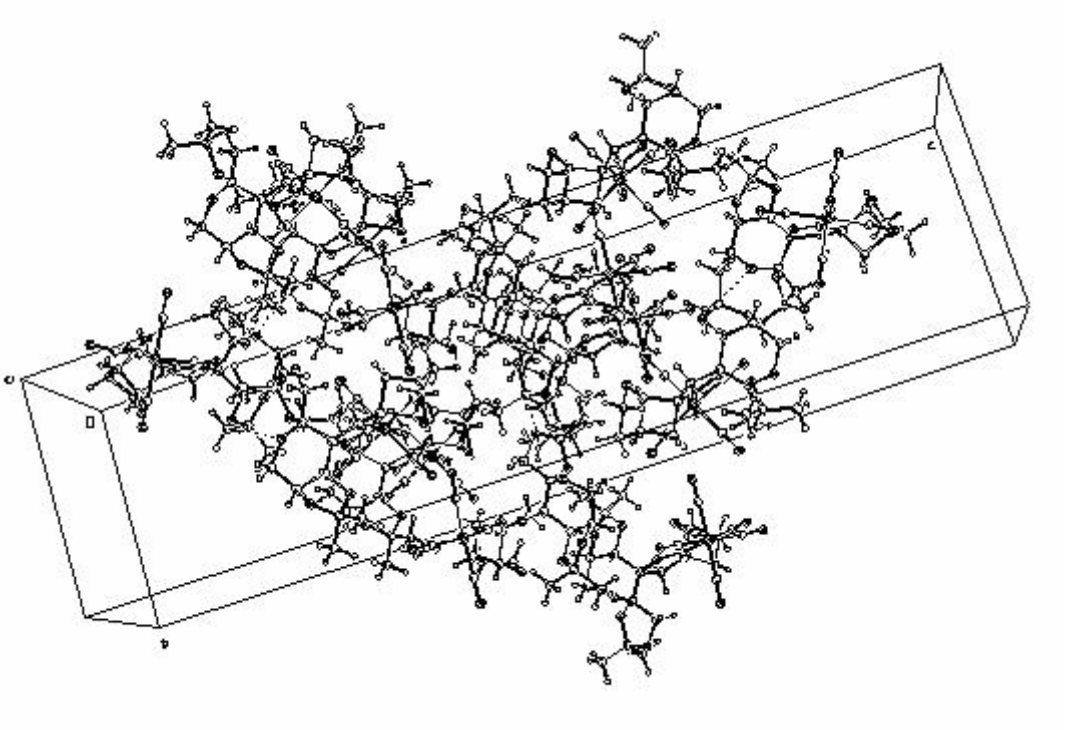

c
o
0
b

**Table 1: Crystal data and structure refinements for *[50]a***

| | |
|---|---|
| Identification code | ***[50]a*** |
| Empirical formula | $C_{25}H_{28}CrO_{12}$ |
| Formula weight | 572.47 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 10.7919(2) Å α = 90° |
| | b = 11.3057(2) Å β = 90° |
| | c = 43.5135(8) Å γ = 90° |
| Volume | 5309.08(17) $Å^3$ |
| Z | 8 |
| Calculated density | 1.432 $mg/m^3$ |
| Absorption coefficient | 0.493 $mm^{-1}$ |
| F(000) | 2384 |
| Crystal size | 0.25 x 0.10 x 0.05 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.95 to 27.48° |
| Limiting indices | $-13 \le h \le 14$, $-14 \le k \le 13$, $-56 \le l \le 56$ |
| Reflections collected / unique | 24907 / 10489 [R(int) = 0.0352] |
| Completeness to Θ = 25.00 | 94.1 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 10489 / 0 / 685 |
| Goodness-of-fit on $F^2$ | 0.907 |
| Final R indices [I>2σ(I)] | R1 = 0.0367, wR2 = 0.0616 |
| R indices (all data) | R1 = 0.0618, wR2 = 0.0662 |
| Absolute structure parameter | -0.019(12) |
| Largest diff. peak and hole | 0.673 and -0.394 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[50]a*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 4387(1) | 6167(1) | 1193(1) | 27(1) |
| C(1A) | 3096(3) | 6169(3) | 1483(1) | 38(1) |
| O(1A) | 2332(2) | 6170(2) | 1664(1) | 62(1) |
| C(1B) | 3175(2) | 6253(3) | 877(1) | 33(1) |
| O(1B) | 2411(2) | 6270(2) | 694(1) | 52(1) |
| C(1C) | 5399(3) | 6046(3) | 1549(1) | 40(1) |
| O(1C) | 5848(2) | 5994(2) | 1786(1) | 62(1) |
| C(1D) | 4521(3) | 7832(3) | 1212(1) | 40(1) |
| O(1D) | 4643(2) | 8846(2) | 1225(1) | 62(1) |
| C(1E) | 4372(3) | 4496(3) | 1150(1) | 42(1) |
| O(1E) | 4397(2) | 3499(2) | 1105(1) | 72(1) |
| O(1) | 5353(1) | 6135(1) | 576(1) | 19(1) |
| C(1) | 5724(2) | 6116(2) | 866(1) | 19(1) |
| C(2) | 7119(2) | 6038(2) | 871(1) | 18(1) |
| C(3) | 7500(2) | 5751(2) | 542(1) | 21(1) |
| C(4) | 6347(2) | 5938(2) | 347(1) | 15(1) |
| C(5) | 6432(2) | 7027(2) | 134(1) | 18(1) |
| O(5) | 6257(2) | 8086(1) | 309(1) | 21(1) |
| C(6) | 5471(2) | 7099(2) | -122(1) | 20(1) |
| O(6) | 4525(2) | 7819(1) | 15(1) | 21(1) |
| C(7) | 4964(2) | 5938(2) | -233(1) | 24(1) |
| O(7) | 4785(2) | 5097(1) | 11(1) | 19(1) |
| C(8) | 5894(2) | 4837(2) | 172(1) | 18(1) |
| O(8) | 6856(1) | 4454(1) | -24(1) | 19(1) |
| C(9) | 5623(2) | 3766(2) | 369(1) | 20(1) |
| O(9) | 5899(2) | 2799(1) | 171(1) | 23(1) |
| C(10) | 6816(2) | 3173(2) | -44(1) | 22(1) |
| C(11) | 6414(3) | 2812(2) | -362(1) | 40(1) |
| C(12) | 8071(2) | 2723(2) | 48(1) | 36(1) |
| C(13) | 5171(2) | 8682(2) | 192(1) | 21(1) |
| C(14) | 5575(3) | 9722(2) | -7(1) | 30(1) |
| C(15) | 4336(2) | 9052(2) | 454(1) | 29(1) |
| C(16) | 7747(2) | 5367(2) | 1138(1) | 23(1) |
| C(17) | 8225(2) | 6518(2) | 1284(1) | 27(1) |
| C(18) | 7671(2) | 7232(2) | 1012(1) | 21(1) |
| O(18) | 8655(2) | 7740(1) | 845(1) | 24(1) |
| C(19) | 9772(2) | 7125(2) | 928(1) | 28(1) |
| C(20) | 9613(2) | 6754(2) | 1259(1) | 29(1) |
| Cr(1') | 11501(1) | 1400(1) | 1530(1) | 26(1) |
| C(1A') | 13236(3) | 1376(3) | 1496(1) | 38(1) |
| O(1A') | 14302(2) | 1384(2) | 1479(1) | 59(1) |
| C(1B') | 11422(3) | 2886(2) | 1328(1) | 28(1) |

| | | | | |
|---|---|---|---|---|
| O(1B') | 11352(2) | 3781(2) | 1203(1) | 42(1) |
| C(1C') | 11437(3) | -110(3) | 1720(1) | 35(1) |
| O(1C') | 11373(2) | -1025(2) | 1831(1) | 56(1) |
| C(1D') | 11781(3) | 2067(3) | 1925(1) | 35(1) |
| O(1D') | 12042(2) | 2405(2) | 2165(1) | 57(1) |
| C(1E') | 11514(3) | 571(2) | 1153(1) | 32(1) |
| O(1E') | 11671(2) | 36(2) | 933(1) | 43(1) |
| O(1') | 9259(2) | 1857(1) | 1878(1) | 20(1) |
| C(1') | 9638(2) | 1537(2) | 1602(1) | 22(1) |
| C(2') | 8520(2) | 1292(2) | 1404(1) | 18(1) |
| C(3') | 7398(2) | 1632(2) | 1600(1) | 23(1) |
| C(4') | 7899(2) | 1831(2) | 1923(1) | 22(1) |
| C(5') | 7470(3) | 3016(2) | 2064(1) | 28(1) |
| O(5') | 8026(2) | 3986(2) | 1909(1) | 30(1) |
| C(6') | 7829(3) | 3192(2) | 2397(1) | 31(1) |
| O(6') | 9020(2) | 3733(2) | 2370(1) | 30(1) |
| C(7') | 7871(3) | 2086(2) | 2591(1) | 35(1) |
| O(7') | 8306(2) | 1058(2) | 2430(1) | 29(1) |
| C(8') | 7664(2) | 826(2) | 2153(1) | 25(1) |
| O(8') | 6369(2) | 762(2) | 2192(1) | 29(1) |
| C(9') | 8046(2) | -433(2) | 2058(1) | 29(1) |
| O(9') | 6911(2) | -1045(2) | 2027(1) | 65(1) |
| C(10') | 6006(3) | -465(3) | 2209(1) | 42(1) |
| C(11') | 4795(3) | -616(3) | 2044(1) | 59(1) |
| C(12') | 5949(3) | -852(3) | 2539(1) | 68(1) |
| C(13') | 8905(3) | 4547(2) | 2121(1) | 31(1) |
| C(14') | 8370(3) | 5720(2) | 2232(1) | 45(1) |
| C(15') | 10128(3) | 4688(2) | 1968(1) | 39(1) |
| C(16') | 8538(3) | 32(2) | 1252(1) | 22(1) |
| C(17') | 8625(3) | 576(2) | 928(1) | 24(1) |
| C(18') | 8564(3) | 1835(2) | 1069(1) | 22(1) |
| O(18') | 7442(2) | 2364(1) | 969(1) | 26(1) |
| C(19') | 6623(2) | 1419(2) | 876(1) | 27(1) |
| C(20') | 7465(3) | 504(2) | 731(1) | 28(1) |

**Tabelle 3: Bond lengths [Å] for *[50]a*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1A) | 1.880(3) | O(5)-C(13) | 1.444(3) |
| Cr(1)-C(1D) | 1.891(3) | C(6)-O(6) | 1.436(3) |
| Cr(1)-C(1E) | 1.898(3) | C(6)-C(7) | 1.501(3) |
| Cr(1)-C(1C) | 1.898(3) | O(6)-C(13) | 1.426(3) |
| Cr(1)-C(1B) | 1.901(3) | C(7)-O(7) | 1.439(3) |
| Cr(1)-C(1) | 2.028(2) | O(7)-C(8) | 1.417(3) |
| C(1A)-O(1A) | 1.139(3) | C(8)-O(8) | 1.409(3) |
| C(1B)-O(1B) | 1.145(3) | C(8)-C(9) | 1.512(3) |
| C(1C)-O(1C) | 1.144(3) | O(8)-C(10) | 1.451(2) |
| C(1D)-O(1D) | 1.154(3) | C(9)-O(9) | 1.423(3) |
| C(1E)-O(1E) | 1.144(3) | O(9)-C(10) | 1.426(3) |
| O(1)-C(1) | 1.325(3) | C(10)-C(12) | 1.501(3) |
| O(1)-C(4) | 1.480(3) | C(10)-C(11) | 1.506(3) |
| C(1)-C(2) | 1.508(3) | C(13)-C(15) | 1.512(3) |
| C(2)-C(3) | 1.525(3) | C(13)-C(14) | 1.524(3) |
| C(2)-C(16) | 1.545(3) | C(16)-C(17) | 1.537(3) |
| C(2)-C(18) | 1.598(3) | C(17)-C(20) | 1.525(4) |
| C(3)-C(4) | 1.520(3) | C(17)-C(18) | 1.550(3) |
| C(4)-C(8) | 1.539(3) | C(18)-O(18) | 1.409(3) |
| C(4)-C(5) | 1.544(3) | O(18)-C(19) | 1.437(3) |
| C(5)-O(5) | 1.431(3) | C(19)-C(20) | 1.513(3) |
| C(5)-C(6) | 1.525(3) | | |
| Cr(1')-C(1A') | 1.879(3) | O(5')-C(13') | 1.468(3) |
| Cr(1')-C(1E') | 1.892(3) | C(6')-O(6') | 1.428(3) |
| Cr(1')-C(1C') | 1.896(3) | C(6')-C(7') | 1.509(4) |
| Cr(1')-C(1B') | 1.899(3) | O(6')-C(13') | 1.425(3) |
| Cr(1')-C(1D') | 1.900(3) | C(7')-O(7') | 1.436(3) |
| Cr(1')-C(1') | 2.040(3) | O(7')-C(8') | 1.417(3) |
| C(1A')-O(1A') | 1.152(3) | C(8')-O(8') | 1.410(3) |
| C(1B')-O(1B') | 1.151(3) | C(8')-C(9') | 1.538(3) |
| C(1C')-O(1C') | 1.144(3) | O(8')-C(10') | 1.444(3) |
| C(1D')-O(1D') | 1.146(3) | C(9')-O(9') | 1.413(3) |
| C(1E')-O(1E') | 1.142(3) | O(9')-C(10') | 1.419(3) |
| O(1')-C(1') | 1.319(3) | C(10')-C(12') | 1.501(4) |
| O(1')-C(4') | 1.481(3) | C(10')-C(11') | 1.501(4) |
| C(1')-C(2') | 1.509(3) | C(13')-C(15') | 1.487(4) |
| C(2')-C(3') | 1.529(3) | C(13')-C(14') | 1.524(4) |
| C(2')-C(16') | 1.571(3) | C(16')-C(17') | 1.539(3) |
| C(2')-C(18') | 1.585(3) | C(17')-C(20') | 1.521(4) |
| C(3')-C(4') | 1.523(3) | C(17')-C(18') | 1.550(3) |
| C(4')-C(8') | 1.535(3) | C(18')-O(18') | 1.419(3) |
| C(4')-C(5') | 1.545(3) | O(18')-C(19') | 1.445(3) |
| C(5')-O(5') | 1.420(3) | C(19')-C(20') | 1.514(3) |
| C(5')-C(6') | 1.513(3) | | |

**Tabelle 4: Bond angles [°] for *[50]a*.**

| | | | |
|---|---|---|---|
| C(1A)-Cr(1)-C(1D) | 91.46(14) | C(6)-C(5)-C(4) | 116.2(2) |
| C(1A)-Cr(1)-C(1E) | 93.56(14) | C(5)-O(5)-C(13) | 108.10(18) |
| C(1D)-Cr(1)-C(1E) | 174.97(13) | O(6)-C(6)-C(7) | 111.7(2) |
| C(1A)-Cr(1)-C(1C) | 83.08(12) | O(6)-C(6)-C(5) | 102.14(18) |
| C(1D)-Cr(1)-C(1C) | 89.52(13) | C(7)-C(6)-C(5) | 115.8(2) |
| C(1E)-Cr(1)-C(1C) | 90.83(14) | C(13)-O(6)-C(6) | 105.38(17) |
| C(1A)-Cr(1)-C(1B) | 88.62(11) | O(7)-C(7)-C(6) | 112.97(19) |
| C(1D)-Cr(1)-C(1B) | 91.90(14) | C(8)-O(7)-C(7) | 112.82(18) |
| C(1E)-Cr(1)-C(1B) | 88.48(14) | O(8)-C(8)-O(7) | 112.85(19) |
| C(1C)-Cr(1)-C(1B) | 171.61(11) | O(8)-C(8)-C(9) | 103.75(19) |
| C(1A)-Cr(1)-C(1) | 177.10(11) | O(7)-C(8)-C(9) | 106.39(19) |
| C(1D)-Cr(1)-C(1) | 90.28(11) | O(8)-C(8)-C(4) | 108.29(18) |
| C(1E)-Cr(1)-C(1) | 84.71(11) | O(7)-C(8)-C(4) | 110.16(19) |
| C(1C)-Cr(1)-C(1) | 99.26(11) | C(9)-C(8)-C(4) | 115.4(2) |
| C(1B)-Cr(1)-C(1) | 89.00(10) | C(8)-O(8)-C(10) | 108.82(18) |
| O(1A)-C(1A)-Cr(1) | 178.6(2) | O(9)-C(9)-C(8) | 103.43(18) |
| O(1B)-C(1B)-Cr(1) | 176.8(3) | C(9)-O(9)-C(10) | 108.32(17) |
| O(1C)-C(1C)-Cr(1) | 169.8(2) | O(9)-C(10)-O(8) | 105.98(19) |
| O(1D)-C(1D)-Cr(1) | 177.8(3) | O(9)-C(10)-C(12) | 110.5(2) |
| O(1E)-C(1E)-Cr(1) | 175.7(3) | O(8)-C(10)-C(12) | 107.2(2) |
| C(1)-O(1)-C(4) | 114.78(18) | O(9)-C(10)-C(11) | 108.8(2) |
| O(1)-C(1)-C(2) | 108.4(2) | O(8)-C(10)-C(11) | 109.6(2) |
| O(1)-C(1)-Cr(1) | 117.00(17) | C(12)-C(10)-C(11) | 114.4(2) |
| C(2)-C(1)-Cr(1) | 134.56(18) | O(6)-C(13)-O(5) | 105.55(18) |
| C(1)-C(2)-C(3) | 105.6(2) | O(6)-C(13)-C(15) | 107.85(19) |
| C(1)-C(2)-C(16) | 118.5(2) | O(5)-C(13)-C(15) | 110.3(2) |
| C(3)-C(2)-C(16) | 118.9(2) | O(6)-C(13)-C(14) | 111.1(2) |
| C(1)-C(2)-C(18) | 109.2(2) | O(5)-C(13)-C(14) | 109.09(19) |
| C(3)-C(2)-C(18) | 116.1(2) | C(15)-C(13)-C(14) | 112.7(2) |
| C(16)-C(2)-C(18) | 87.82(18) | C(17)-C(16)-C(2) | 92.37(18) |
| C(4)-C(3)-C(2) | 105.8(2) | C(20)-C(17)-C(16) | 116.7(2) |
| O(1)-C(4)-C(3) | 103.88(17) | C(20)-C(17)-C(18) | 103.6(2) |
| O(1)-C(4)-C(8) | 102.99(17) | C(16)-C(17)-C(18) | 89.83(18) |
| C(3)-C(4)-C(8) | 115.06(19) | O(18)-C(18)-C(17) | 108.3(2) |
| O(1)-C(4)-C(5) | 109.10(18) | O(18)-C(18)-C(2) | 115.27(19) |
| C(3)-C(4)-C(5) | 113.4(2) | C(17)-C(18)-C(2) | 89.87(18) |
| C(8)-C(4)-C(5) | 111.45(18) | C(18)-O(18)-C(19) | 107.81(17) |
| O(5)-C(5)-C(6) | 104.73(18) | O(18)-C(19)-C(20) | 106.1(2) |
| O(5)-C(5)-C(4) | 109.86(17) | C(19)-C(20)-C(17) | 103.1(2) |

| | | | |
|---|---|---|---|
| C(1A')-Cr(1')-C(1E') | 85.18(13) | C(6')-C(5')-C(4') | 114.8(2) |
| C(1A')-Cr(1')-C(1C') | 93.35(13) | C(5')-O(5')-C(13') | 107.97(18) |
| C(1E')-Cr(1')-C(1C') | 86.10(12) | O(6')-C(6')-C(7') | 112.0(2) |
| C(1A')-Cr(1')-C(1B') | 91.16(13) | O(6')-C(6')-C(5') | 101.9(2) |
| C(1E')-Cr(1')-C(1B') | 92.05(11) | C(7')-C(6')-C(5') | 115.7(2) |
| C(1C')-Cr(1')-C(1B') | 174.96(13) | C(13')-O(6')-C(6') | 105.22(19) |
| C(1A')-Cr(1')-C(1D') | 85.39(13) | O(7')-C(7')-C(6') | 114.1(2) |
| C(1E')-Cr(1')-C(1D') | 168.76(13) | C(8')-O(7')-C(7') | 113.97(19) |
| C(1C')-Cr(1')-C(1D') | 88.35(12) | O(8')-C(8')-O(7') | 113.0(2) |
| C(1B')-Cr(1')-C(1D') | 94.26(12) | O(8')-C(8')-C(4') | 106.3(2) |
| C(1A')-Cr(1')-C(1') | 174.46(12) | O(7')-C(8')-C(4') | 109.7(2) |
| C(1E')-Cr(1')-C(1') | 100.27(11) | O(8')-C(8')-C(9') | 104.5(2) |
| C(1C')-Cr(1')-C(1') | 88.04(12) | O(7')-C(8')-C(9') | 105.6(2) |
| C(1B')-Cr(1')-C(1') | 87.69(11) | C(4')-C(8')-C(9') | 117.8(2) |
| C(1D')-Cr(1')-C(1') | 89.29(11) | C(8')-O(8')-C(10') | 108.9(2) |
| O(1A')-C(1A')-Cr(1') | 178.4(3) | O(9')-C(9')-C(8') | 104.2(2) |
| O(1B')-C(1B')-Cr(1') | 178.6(3) | C(9')-O(9')-C(10') | 108.5(2) |
| O(1C')-C(1C')-Cr(1') | 178.5(3) | O(9')-C(10')-O(8') | 103.2(2) |
| O(1D')-C(1D')-Cr(1') | 173.8(3) | O(9')-C(10')-C(12') | 115.4(3) |
| O(1E')-C(1E')-Cr(1') | 171.4(3) | O(8')-C(10')-C(12') | 109.9(3) |
| C(1')-O(1')-C(4') | 114.98(19) | O(9')-C(10')-C(11') | 106.2(3) |
| O(1')-C(1')-C(2') | 108.8(2) | O(8')-C(10')-C(11') | 108.6(2) |
| O(1')-C(1')-Cr(1') | 117.81(17) | C(12')-C(10')-C(11') | 112.9(3) |
| C(2')-C(1')-Cr(1') | 133.36(18) | O(6')-C(13')-O(5') | 104.6(2) |
| C(1')-C(2')-C(3') | 105.65(18) | O(6')-C(13')-C(15') | 109.4(2) |
| C(1')-C(2')-C(16') | 113.4(2) | O(5')-C(13')-C(15') | 109.7(2) |
| C(3')-C(2')-C(16') | 118.1(2) | O(6')-C(13')-C(14') | 110.8(2) |
| C(1')-C(2')-C(18') | 115.6(2) | O(5')-C(13')-C(14') | 109.3(2) |
| C(3')-C(2')-C(18') | 116.0(2) | C(15')-C(13')-C(14') | 112.7(2) |
| C(16')-C(2')-C(18') | 87.84(17) | C(17')-C(16')-C(2') | 91.39(17) |
| C(4')-C(3')-C(2') | 105.7(2) | C(20')-C(17')-C(16') | 116.4(2) |
| O(1')-C(4')-C(3') | 103.47(19) | C(20')-C(17')-C(18') | 103.7(2) |
| O(1')-C(4')-C(8') | 105.3(2) | C(16')-C(17')-C(18') | 90.23(17) |
| C(3')-C(4')-C(8') | 115.7(2) | O(18')-C(18')-C(17') | 107.6(2) |
| O(1')-C(4')-C(5') | 109.4(2) | O(18')-C(18')-C(2') | 114.8(2) |
| C(3')-C(4')-C(5') | 113.0(2) | C(17')-C(18')-C(2') | 90.48(18) |
| C(8')-C(4')-C(5') | 109.5(2) | C(18')-O(18')-C(19') | 107.23(17) |
| O(5')-C(5')-C(6') | 104.2(2) | O(18')-C(19')-C(20') | 104.8(2) |
| O(5')-C(5')-C(4') | 110.7(2) | C(19')-C(20')-C(17') | 102.8(2) |

**Tabelle 5: Torsion angles [°] for *[50]a.***

| | | | |
|---|---|---|---|
| C(1D)-Cr(1)-C(1A)-O(1A) | -86(13) | C(1)-O(1)-C(4)-C(3) | 1.1(2) |
| C(1E)-Cr(1)-C(1A)-O(1A) | 93(13) | C(1)-O(1)-C(4)-C(8) | 121.4(2) |
| C(1C)-Cr(1)-C(1A)-O(1A) | 3(13) | C(1)-O(1)-C(4)-C(5) | -120.1(2) |
| C(1B)-Cr(1)-C(1A)-O(1A) | -178(100) | C(2)-C(3)-C(4)-O(1) | -8.2(2) |
| C(1)-Cr(1)-C(1A)-O(1A) | 147(11) | C(2)-C(3)-C(4)-C(8) | -120.0(2) |
| C(1A)-Cr(1)-C(1B)-O(1B) | -38(5) | C(2)-C(3)-C(4)-C(5) | 110.0(2) |
| C(1D)-Cr(1)-C(1B)-O(1B) | -130(5) | O(1)-C(4)-C(5)-O(5) | 39.1(3) |
| C(1E)-Cr(1)-C(1B)-O(1B) | 55(5) | C(3)-C(4)-C(5)-O(5) | -76.2(2) |
| C(1C)-Cr(1)-C(1B)-O(1B) | -30(6) | C(8)-C(4)-C(5)-O(5) | 152.12(19) |
| C(1)-Cr(1)-C(1B)-O(1B) | 140(5) | O(1)-C(4)-C(5)-C(6) | -79.6(2) |
| C(1A)-Cr(1)-C(1C)-O(1C) | -5.7(17) | C(3)-C(4)-C(5)-C(6) | 165.2(2) |
| C(1D)-Cr(1)-C(1C)-O(1C) | 85.8(17) | C(8)-C(4)-C(5)-C(6) | 33.5(3) |
| C(1E)-Cr(1)-C(1C)-O(1C) | -99.2(17) | C(6)-C(5)-O(5)-C(13) | 6.8(2) |
| C(1B)-Cr(1)-C(1C)-O(1C) | -14(2) | C(4)-C(5)-O(5)-C(13) | -118.7(2) |
| C(1)-Cr(1)-C(1C)-O(1C) | 176.1(17) | O(5)-C(5)-C(6)-O(6) | -27.0(2) |
| C(1A)-Cr(1)-C(1D)-O(1D) | 132(7) | C(4)-C(5)-C(6)-O(6) | 94.4(2) |
| C(1E)-Cr(1)-C(1D)-O(1D) | -45(8) | O(5)-C(5)-C(6)-C(7) | -148.6(2) |
| C(1C)-Cr(1)-C(1D)-O(1D) | 49(7) | C(4)-C(5)-C(6)-C(7) | -27.2(3) |
| C(1B)-Cr(1)-C(1D)-O(1D) | -139(7) | C(7)-C(6)-O(6)-C(13) | 161.95(19) |
| C(1)-Cr(1)-C(1D)-O(1D) | -50(7) | C(5)-C(6)-O(6)-C(13) | 37.5(2) |
| C(1A)-Cr(1)-C(1E)-O(1E) | 156(4) | O(6)-C(6)-C(7)-O(7) | -78.5(2) |
| C(1D)-Cr(1)-C(1E)-O(1E) | -27(6) | C(5)-C(6)-C(7)-O(7) | 37.9(3) |
| C(1C)-Cr(1)-C(1E)-O(1E) | -121(4) | C(6)-C(7)-O(7)-C(8) | -59.1(3) |
| C(1B)-Cr(1)-C(1E)-O(1E) | 68(5) | C(7)-O(7)-C(8)-O(8) | -54.7(2) |
| C(1)-Cr(1)-C(1E)-O(1E) | -21(4) | C(7)-O(7)-C(8)-C(9) | -167.80(18) |
| C(4)-O(1)-C(1)-C(2) | 6.8(3) | C(7)-O(7)-C(8)-C(4) | 66.5(2) |
| C(4)-O(1)-C(1)-Cr(1) | -172.32(14) | O(1)-C(4)-C(8)-O(8) | -171.35(17) |
| C(1A)-Cr(1)-C(1)-O(1) | 34(3) | C(3)-C(4)-C(8)-O(8) | -59.0(2) |
| C(1D)-Cr(1)-C(1)-O(1) | -93.0(2) | C(5)-C(4)-C(8)-O(8) | 71.8(2) |
| C(1E)-Cr(1)-C(1)-O(1) | 87.4(2) | O(1)-C(4)-C(8)-O(7) | 64.8(2) |
| C(1C)-Cr(1)-C(1)-O(1) | 177.4(2) | C(3)-C(4)-C(8)-O(7) | 177.12(19) |
| C(1B)-Cr(1)-C(1)-O(1) | -1.12(19) | C(5)-C(4)-C(8)-O(7) | -52.0(3) |
| C(1A)-Cr(1)-C(1)-C(2) | -145(2) | O(1)-C(4)-C(8)-C(9) | -55.7(2) |
| C(1D)-Cr(1)-C(1)-C(2) | 88.2(3) | C(3)-C(4)-C(8)-C(9) | 56.7(3) |
| C(1E)-Cr(1)-C(1)-C(2) | -91.3(3) | C(5)-C(4)-C(8)-C(9) | -172.5(2) |
| C(1C)-Cr(1)-C(1)-C(2) | -1.4(3) | O(7)-C(8)-O(8)-C(10) | -91.7(2) |
| C(1B)-Cr(1)-C(1)-C(2) | -179.9(3) | C(9)-C(8)-O(8)-C(10) | 23.0(2) |
| O(1)-C(1)-C(2)-C(3) | -11.7(3) | C(4)-C(8)-O(8)-C(10) | 146.1(2) |
| Cr(1)-C(1)-C(2)-C(3) | 167.17(18) | O(8)-C(8)-C(9)-O(9) | -31.0(2) |
| O(1)-C(1)-C(2)-C(16) | -148.0(2) | O(7)-C(8)-C(9)-O(9) | 88.3(2) |
| Cr(1)-C(1)-C(2)-C(16) | 30.9(4) | C(4)-C(8)-C(9)-O(9) | -149.2(2) |
| O(1)-C(1)-C(2)-C(18) | 113.8(2) | C(8)-C(9)-O(9)-C(10) | 27.8(2) |
| Cr(1)-C(1)-C(2)-C(18) | -67.3(3) | C(9)-O(9)-C(10)-O(8) | -14.2(2) |
| C(1)-C(2)-C(3)-C(4) | 12.0(3) | C(9)-O(9)-C(10)-C(12) | 101.6(2) |
| C(16)-C(2)-C(3)-C(4) | 148.0(2) | C(9)-O(9)-C(10)-C(11) | -132.1(2) |
| C(18)-C(2)-C(3)-C(4) | -109.1(2) | C(8)-O(8)-C(10)-O(9) | -6.5(3) |

| | | | |
|---|---|---|---|
| C(8)-O(8)-C(10)-C(12) | -124.5(2) | C(20)-C(17)-C(18)-C(2) | 120.1(2) |
| C(8)-O(8)-C(10)-C(11) | 110.8(2) | C(16)-C(17)-C(18)-C(2) | 2.63(19) |
| C(6)-O(6)-C(13)-O(5) | -34.3(2) | C(1)-C(2)-C(18)-O(18) | -132.8(2) |
| C(6)-O(6)-C(13)-C(15) | -152.27(19) | C(3)-C(2)-C(18)-O(18) | -13.7(3) |
| C(6)-O(6)-C(13)-C(14) | 83.8(2) | C(16)-C(2)-C(18)-O(18) | 107.7(2) |
| C(5)-O(5)-C(13)-O(6) | 16.3(2) | C(1)-C(2)-C(18)-C(17) | 116.9(2) |
| C(5)-O(5)-C(13)-C(15) | 132.6(2) | C(3)-C(2)-C(18)-C(17) | -124.0(2) |
| C(5)-O(5)-C(13)-C(14) | -103.1(2) | C(16)-C(2)-C(18)-C(17) | -2.62(19) |
| C(1)-C(2)-C(16)-C(17) | -108.1(2) | C(17)-C(18)-O(18)-C(19) | 17.5(2) |
| C(3)-C(2)-C(16)-C(17) | 121.5(2) | C(2)-C(18)-O(18)-C(19) | -81.3(2) |
| C(18)-C(2)-C(16)-C(17) | 2.65(19) | C(18)-O(18)-C(19)-C(20) | -32.0(2) |
| C(2)-C(16)-C(17)-C(20) | -107.9(2) | O(18)-C(19)-C(20)-C(17) | 33.0(3) |
| C(2)-C(16)-C(17)-C(18) | -2.73(19) | C(16)-C(17)-C(20)-C(19) | 75.2(3) |
| C(20)-C(17)-C(18)-O(18) | 3.5(3) | C(18)-C(17)-C(20)-C(19) | -21.6(3) |
| C(16)-C(17)-C(18)-O(18) | -114.0(2) | | |
| C(1E')-Cr(1')-C(1A')-O(1A') | 159(12) | C(1C')-Cr(1')-C(1')-C(2') | -96.1(2) |
| C(1C')-Cr(1')-C(1A')-O(1A') | -115(12) | C(1B')-Cr(1')-C(1')-C(2') | 81.2(2) |
| C(1B')-Cr(1')-C(1A')-O(1A') | 67(12) | C(1D')-Cr(1')-C(1')-C(2') | 175.5(2) |
| C(1D')-Cr(1')-C(1A')-O(1A') | -27(12) | O(1')-C(1')-C(2')-C(3') | 5.1(3) |
| C(1')-Cr(1')-C(1A')-O(1A') | -11(13) | Cr(1')-C(1')-C(2')-C(3') | -177.88(19) |
| C(1A')-Cr(1')-C(1B')-O(1B') | 149(81) | O(1')-C(1')-C(2')-C(16') | -125.8(2) |
| C(1E')-Cr(1')-C(1B')-O(1B') | 64(11) | Cr(1')-C(1')-C(2')-C(16') | 51.2(3) |
| C(1C')-Cr(1')-C(1B')-O(1B') | -4(12) | O(1')-C(1')-C(2')-C(18') | 134.9(2) |
| C(1D')-Cr(1')-C(1B')-O(1B') | -125(11) | Cr(1')-C(1')-C(2')-C(18') | -48.1(3) |
| C(1')-Cr(1')-C(1B')-O(1B') | -36(11) | C(1')-C(2')-C(3')-C(4') | -10.7(3) |
| C(1A')-Cr(1')-C(1C')-O(1C') | -152(88) | C(16')-C(2')-C(3')-C(4') | 117.5(2) |
| C(1E')-Cr(1')-C(1C')-O(1C') | -67(11) | C(18')-C(2')-C(3')-C(4') | -140.1(2) |
| C(1B')-Cr(1')-C(1C')-O(1C') | 1(12) | C(1')-O(1')-C(4')-C(3') | -9.7(3) |
| C(1D')-Cr(1')-C(1C')-O(1C') | 123(11) | C(1')-O(1')-C(4')-C(8') | 112.1(2) |
| C(1')-Cr(1')-C(1C')-O(1C') | 33(11) | C(1')-O(1')-C(4')-C(5') | -130.4(2) |
| C(1A')-Cr(1')-C(1D')-O(1D') | -35(2) | C(2')-C(3')-C(4')-O(1') | 11.9(2) |
| C(1E')-Cr(1')-C(1D')-O(1D') | -2(3) | C(2')-C(3')-C(4')-C(8') | -102.6(2) |
| C(1C')-Cr(1')-C(1D')-O(1D') | 58(2) | C(2')-C(3')-C(4')-C(5') | 130.1(2) |
| C(1B')-Cr(1')-C(1D')-O(1D') | -126(2) | O(1')-C(4')-C(5')-O(5') | 45.9(3) |
| C(1')-Cr(1')-C(1D')-O(1D') | 146(2) | C(3')-C(4')-C(5')-O(5') | -68.7(3) |
| C(1A')-Cr(1')-C(1E')-O(1E') | 20.2(16) | C(8')-C(4')-C(5')-O(5') | 160.8(2) |
| C(1C')-Cr(1')-C(1E')-O(1E') | -73.5(16) | O(1')-C(4')-C(5')-C(6') | -71.6(3) |
| C(1B')-Cr(1')-C(1E')-O(1E') | 111.2(16) | C(3')-C(4')-C(5')-C(6') | 173.7(2) |
| C(1D')-Cr(1')-C(1E')-O(1E') | -13(2) | C(8')-C(4')-C(5')-C(6') | 43.2(3) |
| C(1')-Cr(1')-C(1E')-O(1E') | -160.8(16) | C(6')-C(5')-O(5')-C(13') | 12.6(3) |
| C(4')-O(1')-C(1')-C(2') | 3.0(3) | C(4')-C(5')-O(5')-C(13') | -111.3(2) |
| C(4')-O(1')-C(1')-Cr(1') | -174.57(15) | O(5')-C(5')-C(6')-O(6') | -32.4(2) |
| C(1A')-Cr(1')-C(1')-O(1') | -23.9(13) | C(4')-C(5')-C(6')-O(6') | 88.8(3) |
| C(1E')-Cr(1')-C(1')-O(1') | 166.37(18) | O(5')-C(5')-C(6')-C(7') | -154.2(2) |
| C(1C')-Cr(1')-C(1')-O(1') | 80.69(19) | C(4')-C(5')-C(6')-C(7') | -33.0(4) |
| C(1B')-Cr(1')-C(1')-O(1') | -101.97(19) | C(7')-C(6')-O(6')-C(13') | 164.8(2) |
| C(1D')-Cr(1')-C(1')-O(1') | -7.7(2) | C(5')-C(6')-O(6')-C(13') | 40.6(2) |
| C(1A')-Cr(1')-C(1')-C(2') | 159.3(11) | O(6')-C(6')-C(7')-O(7') | -80.3(3) |
| C(1E')-Cr(1')-C(1')-C(2') | -10.5(3) | C(5')-C(6')-C(7')-O(7') | 35.9(4) |

| | | | |
|---|---|---|---|
| C(6')-C(7')-O(7')-C(8') | -52.9(3) | C(6')-O(6')-C(13')-C(15') | -150.9(2) |
| C(7')-O(7')-C(8')-O(8') | -53.5(3) | C(6')-O(6')-C(13')-C(14') | 84.3(3) |
| C(7')-O(7')-C(8')-C(4') | 64.9(3) | C(5')-O(5')-C(13')-O(6') | 11.9(3) |
| C(7')-O(7')-C(8')-C(9') | -167.2(2) | C(5')-O(5')-C(13')-C(15') | 129.2(2) |
| O(1')-C(4')-C(8')-O(8') | -178.33(19) | C(5')-O(5')-C(13')-C(14') | -106.8(2) |
| C(3')-C(4')-C(8')-O(8') | -64.8(3) | C(1')-C(2')-C(16')-C(17') | -115.2(2) |
| C(5')-C(4')-C(8')-O(8') | 64.2(3) | C(3')-C(2')-C(16')-C(17') | 120.4(2) |
| O(1')-C(4')-C(8')-O(7') | 59.2(2) | C(18')-C(2')-C(16')-C(17') | 1.8(2) |
| C(3')-C(4')-C(8')-O(7') | 172.8(2) | C(2')-C(16')-C(17')-C(20') | -107.3(2) |
| C(5')-C(4')-C(8')-O(7') | -58.3(3) | C(2')-C(16')-C(17')-C(18') | -1.8(2) |
| O(1')-C(4')-C(8')-C(9') | -61.6(3) | C(20')-C(17')-C(18')-O(18') | 2.9(3) |
| C(3')-C(4')-C(8')-C(9') | 51.9(3) | C(16')-C(17')-C(18')-O(18') | -114.5(2) |
| C(5')-C(4')-C(8')-C(9') | -179.1(2) | C(20')-C(17')-C(18')-C(2') | 119.2(2) |
| O(7')-C(8')-O(8')-C(10') | -99.2(3) | C(16')-C(17')-C(18')-C(2') | 1.8(2) |
| C(4')-C(8')-O(8')-C(10') | 140.4(2) | C(1')-C(2')-C(18')-O(18') | -137.1(2) |
| C(9')-C(8')-O(8')-C(10') | 15.1(3) | C(3')-C(2')-C(18')-O(18') | -12.5(3) |
| O(8')-C(8')-C(9')-O(9') | 5.1(3) | C(16')-C(2')-C(18')-O(18') | 107.9(2) |
| O(7')-C(8')-C(9')-O(9') | 124.5(2) | C(1')-C(2')-C(18')-C(17') | 113.2(2) |
| C(4')-C(8')-C(9')-O(9') | -112.6(3) | C(3')-C(2')-C(18')-C(17') | -122.3(2) |
| C(8')-C(9')-O(9')-C(10') | -23.9(3) | C(16')-C(2')-C(18')-C(17') | -1.8(2) |
| C(9')-O(9')-C(10')-O(8') | 33.2(3) | C(17')-C(18')-O(18')-C(19') | 20.3(2) |
| C(9')-O(9')-C(10')-C(12') | -86.8(3) | C(2')-C(18')-O(18')-C(19') | -78.7(2) |
| C(9')-O(9')-C(10')-C(11') | 147.4(2) | C(18')-O(18')-C(19')-C(20') | -35.9(2) |
| C(8')-O(8')-C(10')-O(9') | -29.6(3) | O(18')-C(19')-C(20')-C(17') | 36.4(2) |
| C(8')-O(8')-C(10')-C(12') | 93.9(3) | C(16')-C(17')-C(20')-C(19') | 73.8(3) |
| C(8')-O(8')-C(10')-C(11') | -142.1(2) | C(18')-C(17')-C(20')-C(19') | -23.4(3) |
| C(6')-O(6')-C(13')-O(5') | -33.3(2) | | |

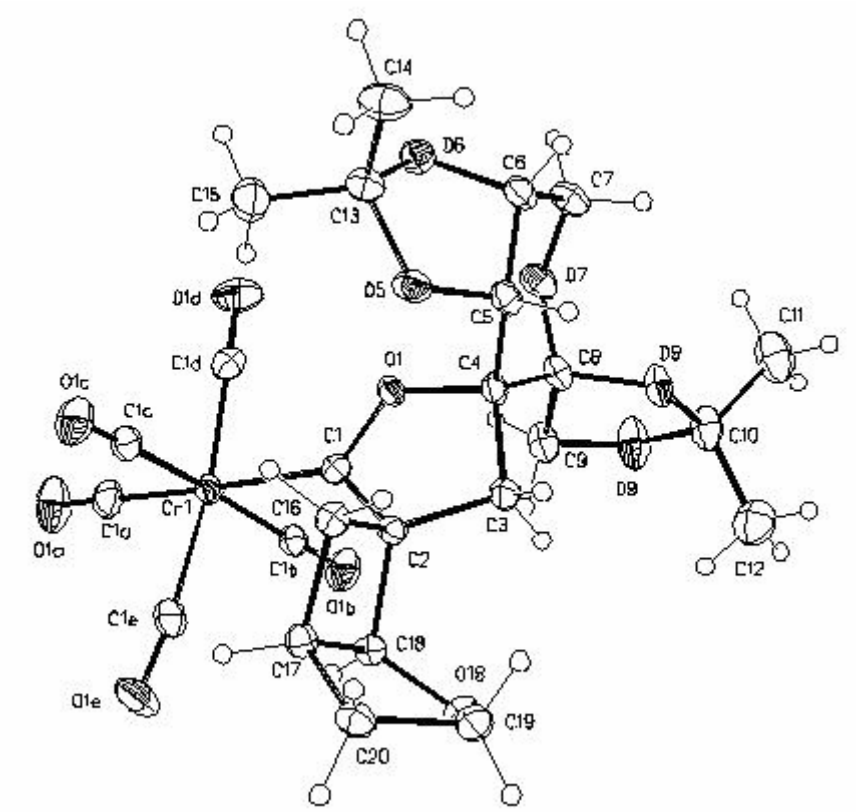
**Tabelle 6: Hydrogen bonds for *[50]a* [Å and °].**

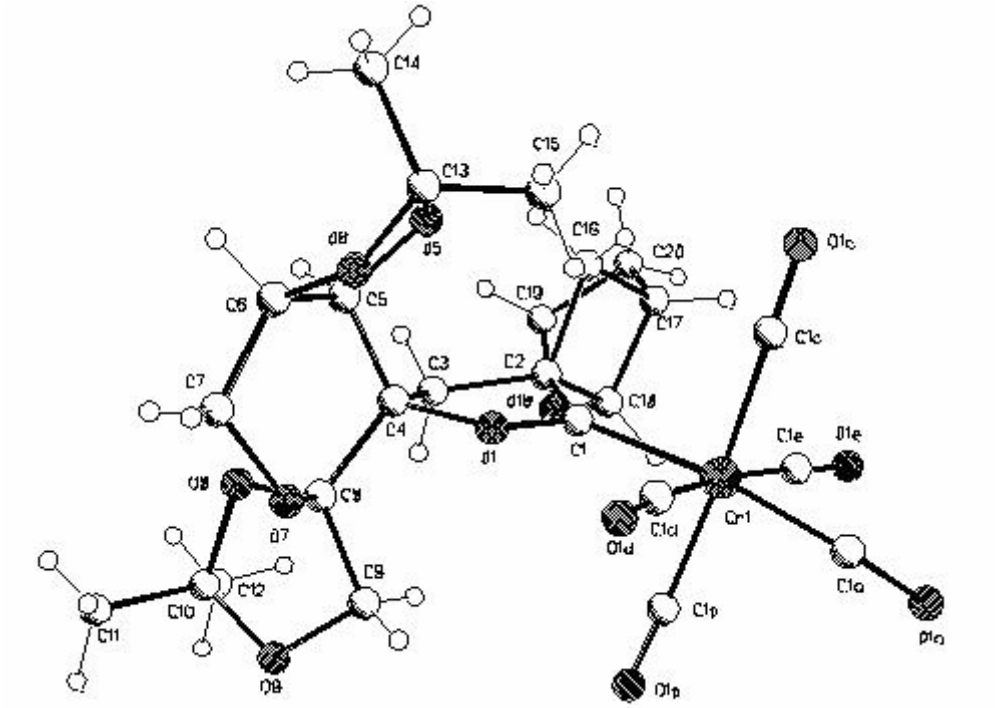
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(5)-H(5)...O(6)[a] | 1.00 | 2.44 | 3.404(3) | 161.2 |
| C(12)-H(12A)...O(9)[b] | 0.98 | 2.56 | 3.251(3) | 127.3 |
| C(11')-H(11F)...O(1A')[c] | 0.98 | 2.50 | 3.382(4) | 150.1 |
| C(15')-H(15F)...O(7')[d] | 0.98 | 2.54 | 3.480(3) | 161.6 |

Symmetry transformations used to generate equivalent atoms:

[a] x+1/2,-y+3/2,-z [b] x+1/2,-y+1/2,-z [c] x-1,y,z [d] -x+2,y+1/2,-z+1/2

**13.** **(*3R,5R,1´S,5´R,1´´R,4´´S,6´´R*)-Pentacarbonyl{8´´,8´´,2´´´,2´´´-tetramethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´´,4´´´-1,3-dioxacyclopentan-5´´,3]-spiro[2-oxabicyclo[3.2.0]heptan-7´,5]-2-oxacyclopent-1-yliden}chrom(0)** ***[50]b***



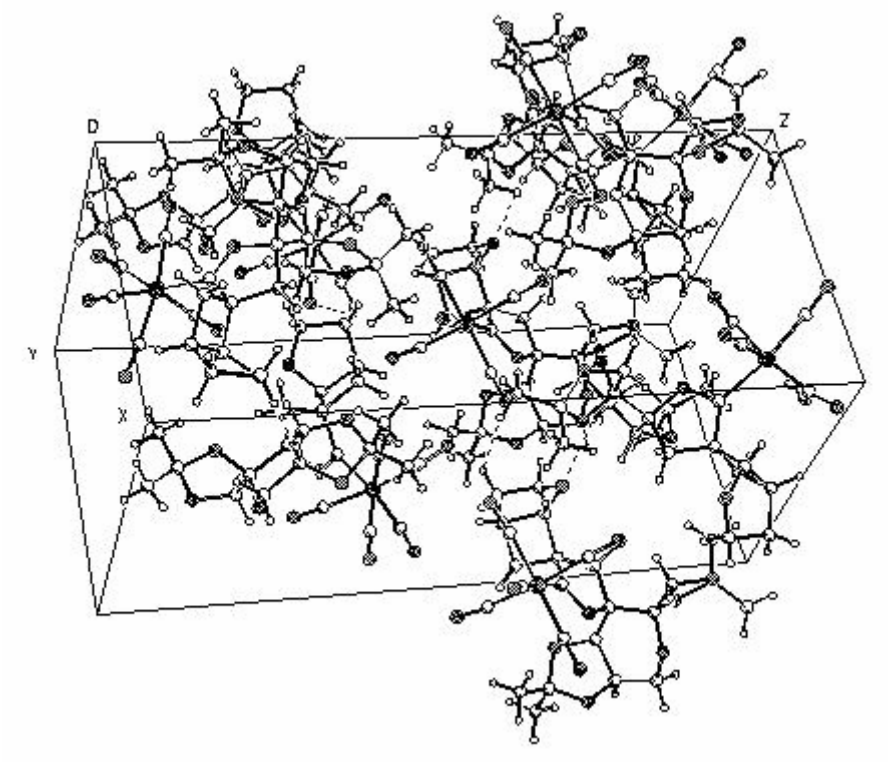



**Table 1: Crystal data and structure refinements for *[50]b***

| | |
|---|---|
| Identification code | ***[50]b*** |
| Empirical formula | $C_{25}H_{28}CrO_{12}$ |
| Formula weight | 572.47 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoK$\alpha$) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 10.2664(1) Å $\alpha$ = 90° |
| | b = 11.2195(1) Å $\beta$ = 90° |
| | c = 22.8418(2) Å $\gamma$ = 90° |
| Volume | 2631.01(4) $Å^3$ |
| Z | 4 |
| Calculated density | 1.445 $mg/m^3$ |
| Absorption coefficient | 0.498 $mm^{-1}$ |
| F(000) | 1192 |
| Crystal size | 0.40 x 0.35 x 0.30 mm |

| | |
|---|---|
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.67 to 25.00 ° |
| Limiting indices | $-12 \leq h \leq 12$, $-13 \leq k \leq 13$, $-27 \leq l \leq 27$ |
| Reflections collected / unique | 46483 / 4624 [R(int) = 0.0374] |
| Completeness to $\Theta = 25.00$ | 99.8 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 4624 / 0 / 343 |
| Goodness-of-fit on $F^2$ | 1.058 |
| Final R indices [$I>2\sigma(I)$] | R1 = 0.0212, wR2 = 0.0553 |
| R indices (all data) | R1 = 0.0229, wR2 = 0.0559 |
| Absolute structure parameter | -0.011(11) |
| Largest diff. peak and hole | 0.205 and -0.234 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[50]b*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 2805(1) | 3406(1) | 876(1) | 16(1) |
| C(1A) | 2069(2) | 4601(2) | 397(1) | 25(1) |
| O(1A) | 1599(2) | 5357(1) | 130(1) | 40(1) |
| C(1B) | 2983(2) | 4512(2) | 1494(1) | 20(1) |
| O(1B) | 3101(1) | 5163(1) | 1874(1) | 32(1) |
| C(1C) | 2866(2) | 2189(2) | 289(1) | 22(1) |
| O(1C) | 3008(1) | 1468(1) | -60(1) | 31(1) |
| C(1D) | 1054(2) | 3065(2) | 1110(1) | 21(1) |
| O(1D) | -6(1) | 2951(1) | 1240(1) | 35(1) |
| C(1E) | 4422(2) | 3904(2) | 561(1) | 23(1) |
| O(1E) | 5320(1) | 4268(1) | 327(1) | 35(1) |
| O(1) | 2558(1) | 1789(1) | 1861(1) | 17(1) |
| C(1) | 3427(2) | 2205(1) | 1487(1) | 15(1) |
| C(2) | 4731(2) | 1680(2) | 1641(1) | 16(1) |
| C(3) | 4589(2) | 1263(2) | 2276(1) | 18(1) |
| C(4) | 3124(2) | 1076(2) | 2348(1) | 17(1) |
| C(5) | 2727(2) | -245(1) | 2288(1) | 18(1) |
| O(5) | 2771(1) | -592(1) | 1689(1) | 22(1) |
| C(6) | 1348(2) | -572(2) | 2481(1) | 21(1) |

| | | | | |
|---|---|---|---|---|
| O(6) | 628(1) | -463(1) | 1947(1) | 22(1) |
| C(7) | 802(2) | 207(2) | 2959(1) | 25(1) |
| O(7) | 1149(1) | 1435(1) | 2896(1) | 23(1) |
| C(8) | 2506(2) | 1642(2) | 2895(1) | 20(1) |
| O(8) | 3146(1) | 1155(1) | 3386(1) | 23(1) |
| C(9) | 2670(2) | 2979(2) | 2972(1) | 25(1) |
| O(9) | 2675(2) | 3111(1) | 3591(1) | 35(1) |
| C(10) | 3218(2) | 2060(2) | 3844(1) | 27(1) |
| C(11) | 2370(2) | 1701(2) | 4351(1) | 38(1) |
| C(12) | 4637(2) | 2238(2) | 3999(1) | 37(1) |
| C(13) | 1472(2) | -925(2) | 1507(1) | 22(1) |
| C(14) | 1402(2) | -2277(2) | 1471(1) | 29(1) |
| C(15) | 1141(2) | -322(2) | 935(1) | 30(1) |
| C(16) | 5131(2) | 738(2) | 1161(1) | 18(1) |
| C(17) | 6271(2) | 1548(2) | 966(1) | 18(1) |
| C(18) | 5981(2) | 2412(2) | 1479(1) | 17(1) |
| O(18) | 7011(1) | 2294(1) | 1889(1) | 20(1) |
| C(19) | 7736(2) | 1230(2) | 1755(1) | 21(1) |
| C(20) | 7632(2) | 1073(2) | 1092(1) | 22(1) |

**Tabelle 3: Bond lengths [Å] for *[50]b*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1B) | 1.8880(17) | O(5)-C(13) | 1.447(2) |
| Cr(1)-C(1A) | 1.8887(18) | C(6)-O(6) | 1.431(2) |
| Cr(1)-C(1E) | 1.8938(19) | C(6)-C(7) | 1.506(2) |
| Cr(1)-C(1D) | 1.9135(19) | O(6)-C(13) | 1.426(2) |
| Cr(1)-C(1C) | 1.9144(18) | C(7)-O(7) | 1.430(2) |
| Cr(1)-C(1) | 2.0430(17) | O(7)-C(8) | 1.412(2) |
| C(1A)-O(1A) | 1.150(2) | C(8)-O(8) | 1.411(2) |
| C(1B)-O(1B) | 1.142(2) | C(8)-C(9) | 1.519(2) |
| C(1C)-O(1C) | 1.144(2) | O(8)-C(10) | 1.460(2) |
| C(1D)-O(1D) | 1.136(2) | C(9)-O(9) | 1.421(2) |
| C(1E)-O(1E) | 1.141(2) | O(9)-C(10) | 1.427(2) |
| O(1)-C(1) | 1.320(2) | C(10)-C(11) | 1.504(3) |
| O(1)-C(4) | 1.4878(18) | C(10)-C(12) | 1.512(3) |
| C(1)-C(2) | 1.504(2) | C(13)-C(15) | 1.508(3) |
| C(2)-C(3) | 1.531(2) | C(13)-C(14) | 1.520(2) |
| C(2)-C(18) | 1.567(2) | C(16)-C(17) | 1.548(2) |
| C(2)-C(16) | 1.577(2) | C(17)-C(20) | 1.523(2) |
| C(3)-C(4) | 1.527(2) | C(17)-C(18) | 1.550(2) |
| C(4)-C(8) | 1.540(2) | C(18)-O(18) | 1.419(2) |
| C(4)-C(5) | 1.542(2) | O(18)-C(19) | 1.440(2) |
| C(5)-O(5) | 1.4246(19) | C(19)-C(20) | 1.528(2) |
| C(5)-C(6) | 1.528(2) | | |

**Tabelle 4: Bond angles [°] for *[50]b*.**

| | | | |
|---|---|---|---|
| C(1B)-Cr(1)-C(1A) | 90.30(7) | C(6)-C(5)-C(4) | 116.80(14) |
| C(1B)-Cr(1)-C(1E) | 90.27(8) | C(5)-O(5)-C(13) | 108.52(13) |
| C(1A)-Cr(1)-C(1E) | 85.50(8) | O(6)-C(6)-C(7) | 112.05(14) |
| C(1B)-Cr(1)-C(1D) | 90.77(7) | O(6)-C(6)-C(5) | 102.22(13) |
| C(1A)-Cr(1)-C(1D) | 85.86(8) | C(7)-C(6)-C(5) | 114.45(15) |
| C(1E)-Cr(1)-C(1D) | 171.30(7) | C(13)-O(6)-C(6) | 104.91(12) |
| C(1B)-Cr(1)-C(1C) | 171.45(8) | O(7)-C(7)-C(6) | 113.22(14) |
| C(1A)-Cr(1)-C(1C) | 96.55(7) | C(8)-O(7)-C(7) | 113.86(13) |
| C(1E)-Cr(1)-C(1C) | 85.19(8) | O(8)-C(8)-O(7) | 113.22(13) |
| C(1D)-Cr(1)-C(1C) | 94.81(8) | O(8)-C(8)-C(9) | 103.82(13) |
| C(1B)-Cr(1)-C(1) | 83.81(6) | O(7)-C(8)-C(9) | 105.76(14) |
| C(1A)-Cr(1)-C(1) | 171.68(7) | O(8)-C(8)-C(4) | 107.10(13) |
| C(1E)-Cr(1)-C(1) | 100.36(7) | O(7)-C(8)-C(4) | 109.89(13) |
| C(1D)-Cr(1)-C(1) | 88.33(7) | C(9)-C(8)-C(4) | 117.12(14) |
| C(1C)-Cr(1)-C(1) | 89.87(7) | C(8)-O(8)-C(10) | 108.90(12) |
| O(1A)-C(1A)-Cr(1) | 176.51(16) | O(9)-C(9)-C(8) | 102.63(13) |
| O(1B)-C(1B)-Cr(1) | 178.66(15) | C(9)-O(9)-C(10) | 108.58(13) |
| O(1C)-C(1C)-Cr(1) | 174.52(17) | O(9)-C(10)-O(8) | 105.33(13) |
| O(1D)-C(1D)-Cr(1) | 174.80(15) | O(9)-C(10)-C(11) | 107.93(16) |
| O(1E)-C(1E)-Cr(1) | 172.57(15) | O(8)-C(10)-C(11) | 109.61(15) |
| C(1)-O(1)-C(4) | 114.20(12) | O(9)-C(10)-C(12) | 111.29(17) |
| O(1)-C(1)-C(2) | 108.18(13) | O(8)-C(10)-C(12) | 107.96(16) |
| O(1)-C(1)-Cr(1) | 117.67(11) | C(11)-C(10)-C(12) | 114.34(17) |
| C(2)-C(1)-Cr(1) | 134.10(12) | O(6)-C(13)-O(5) | 105.23(13) |
| C(1)-C(2)-C(3) | 104.82(13) | O(6)-C(13)-C(15) | 108.12(15) |
| C(1)-C(2)-C(18) | 117.94(13) | O(5)-C(13)-C(15) | 109.88(15) |
| C(3)-C(2)-C(18) | 117.53(13) | O(6)-C(13)-C(14) | 111.87(15) |
| C(1)-C(2)-C(16) | 109.40(13) | O(5)-C(13)-C(14) | 108.45(15) |
| C(3)-C(2)-C(16) | 118.55(14) | C(15)-C(13)-C(14) | 113.00(16) |
| C(18)-C(2)-C(16) | 88.49(12) | C(17)-C(16)-C(2) | 90.24(12) |
| C(4)-C(3)-C(2) | 103.75(13) | C(20)-C(17)-C(16) | 115.71(14) |
| O(1)-C(4)-C(3) | 103.31(12) | C(20)-C(17)-C(18) | 104.60(13) |
| O(1)-C(4)-C(8) | 102.95(12) | C(16)-C(17)-C(18) | 90.18(12) |
| C(3)-C(4)-C(8) | 115.86(14) | O(18)-C(18)-C(17) | 107.32(13) |
| O(1)-C(4)-C(5) | 110.34(13) | O(18)-C(18)-C(2) | 113.90(13) |
| C(3)-C(4)-C(5) | 112.53(14) | C(17)-C(18)-C(2) | 90.50(12) |
| C(8)-C(4)-C(5) | 111.02(13) | C(18)-O(18)-C(19) | 108.78(12) |
| O(5)-C(5)-C(6) | 103.96(14) | O(18)-C(19)-C(20) | 105.68(13) |
| O(5)-C(5)-C(4) | 109.81(12) | C(17)-C(20)-C(19) | 102.21(13) |

**Tabelle 5: Torsion angles [°] for *[50]b.***

| | | | |
|---|---|---|---|
| C(1B)-Cr(1)-C(1A)-O(1A) | -27(3) | C(1)-O(1)-C(4)-C(3) | 8.59(17) |
| C(1E)-Cr(1)-C(1A)-O(1A) | -117(3) | C(1)-O(1)-C(4)-C(8) | 129.54(13) |
| C(1D)-Cr(1)-C(1A)-O(1A) | 64(3) | C(1)-O(1)-C(4)-C(5) | -111.92(15) |
| C(1C)-Cr(1)-C(1A)-O(1A) | 158(3) | C(2)-C(3)-C(4)-O(1) | -19.72(16) |
| C(1)-Cr(1)-C(1A)-O(1A) | 18(3) | C(2)-C(3)-C(4)-C(8) | -131.47(14) |
| C(1A)-Cr(1)-C(1B)-O(1B) | 179(100) | C(2)-C(3)-C(4)-C(5) | 99.27(15) |
| C(1E)-Cr(1)-C(1B)-O(1B) | -95(7) | O(1)-C(4)-C(5)-O(5) | 39.94(18) |
| C(1D)-Cr(1)-C(1B)-O(1B) | 93(7) | C(3)-C(4)-C(5)-O(5) | -74.87(17) |
| C(1C)-Cr(1)-C(1B)-O(1B) | -37(8) | C(8)-C(4)-C(5)-O(5) | 153.42(13) |
| C(1)-Cr(1)-C(1B)-O(1B) | 5(7) | O(1)-C(4)-C(5)-C(6) | -78.04(16) |
| C(1B)-Cr(1)-C(1C)-O(1C) | -30.6(18) | C(3)-C(4)-C(5)-C(6) | 167.15(13) |
| C(1A)-Cr(1)-C(1C)-O(1C) | 112.4(15) | C(8)-C(4)-C(5)-C(6) | 35.44(19) |
| C(1E)-Cr(1)-C(1C)-O(1C) | 27.5(15) | C(6)-C(5)-O(5)-C(13) | 8.36(16) |
| C(1D)-Cr(1)-C(1C)-O(1C) | -161.2(15) | C(4)-C(5)-O(5)-C(13) | -117.33(15) |
| C(1)-Cr(1)-C(1C)-O(1C) | -72.9(15) | O(5)-C(5)-C(6)-O(6) | -28.93(16) |
| C(1B)-Cr(1)-C(1D)-O(1D) | 62.0(18) | C(4)-C(5)-C(6)-O(6) | 92.19(16) |
| C(1A)-Cr(1)-C(1D)-O(1D) | -28.3(18) | O(5)-C(5)-C(6)-C(7) | -150.29(14) |
| C(1E)-Cr(1)-C(1D)-O(1D) | -35(2) | C(4)-C(5)-C(6)-C(7) | -29.2(2) |
| C(1C)-Cr(1)-C(1D)-O(1D) | -124.5(18) | C(7)-C(6)-O(6)-C(13) | 162.06(14) |
| C(1)-Cr(1)-C(1D)-O(1D) | 145.8(18) | C(5)-C(6)-O(6)-C(13) | 39.06(16) |
| C(1B)-Cr(1)-C(1E)-O(1E) | -104.7(13) | O(6)-C(6)-C(7)-O(7) | -77.28(19) |
| C(1A)-Cr(1)-C(1E)-O(1E) | -14.4(13) | C(5)-C(6)-C(7)-O(7) | 38.5(2) |
| C(1D)-Cr(1)-C(1E)-O(1E) | -7.8(17) | C(6)-C(7)-O(7)-C(8) | -59.07(19) |
| C(1C)-Cr(1)-C(1E)-O(1E) | 82.5(13) | C(7)-O(7)-C(8)-O(8) | -53.80(18) |
| C(1)-Cr(1)-C(1E)-O(1E) | 171.5(13) | C(7)-O(7)-C(8)-C(9) | -166.83(13) |
| C(4)-O(1)-C(1)-C(2) | 6.96(17) | C(7)-O(7)-C(8)-C(4) | 65.89(16) |
| C(4)-O(1)-C(1)-Cr(1) | -170.68(10) | O(1)-C(4)-C(8)-O(8) | -170.55(12) |
| C(1B)-Cr(1)-C(1)-O(1) | 81.15(12) | C(3)-C(4)-C(8)-O(8) | -58.59(17) |
| C(1A)-Cr(1)-C(1)-O(1) | 36.0(6) | C(5)-C(4)-C(8)-O(8) | 71.39(16) |
| C(1E)-Cr(1)-C(1)-O(1) | 170.29(12) | O(1)-C(4)-C(8)-O(7) | 66.10(15) |
| C(1D)-Cr(1)-C(1)-O(1) | -9.80(12) | C(3)-C(4)-C(8)-O(7) | 178.05(13) |
| C(1C)-Cr(1)-C(1)-O(1) | -104.62(13) | C(5)-C(4)-C(8)-O(7) | -51.97(17) |
| C(1B)-Cr(1)-C(1)-C(2) | -95.73(16) | O(1)-C(4)-C(8)-C(9) | -54.55(18) |
| C(1A)-Cr(1)-C(1)-C(2) | -140.9(5) | C(3)-C(4)-C(8)-C(9) | 57.4(2) |
| C(1E)-Cr(1)-C(1)-C(2) | -6.59(17) | C(5)-C(4)-C(8)-C(9) | -172.61(14) |
| C(1D)-Cr(1)-C(1)-C(2) | 173.31(16) | O(7)-C(8)-O(8)-C(10) | -92.36(17) |
| C(1C)-Cr(1)-C(1)-C(2) | 78.49(16) | C(9)-C(8)-O(8)-C(10) | 21.84(17) |
| O(1)-C(1)-C(2)-C(3) | -19.61(17) | C(4)-C(8)-O(8)-C(10) | 146.36(14) |
| Cr(1)-C(1)-C(2)-C(3) | 157.49(13) | O(8)-C(8)-C(9)-O(9) | -31.88(17) |
| O(1)-C(1)-C(2)-C(18) | -152.57(13) | O(7)-C(8)-C(9)-O(9) | 87.54(16) |
| Cr(1)-C(1)-C(2)-C(18) | 24.5(2) | C(4)-C(8)-C(9)-O(9) | -149.67(15) |
| O(1)-C(1)-C(2)-C(16) | 108.50(14) | C(8)-C(9)-O(9)-C(10) | 30.75(19) |
| Cr(1)-C(1)-C(2)-C(16) | -74.40(18) | C(9)-O(9)-C(10)-O(8) | -18.0(2) |
| C(1)-C(2)-C(3)-C(4) | 23.91(16) | C(9)-O(9)-C(10)-C(11) | -134.99(17) |
| C(18)-C(2)-C(3)-C(4) | 157.11(14) | C(9)-O(9)-C(10)-C(12) | 98.78(18) |
| C(16)-C(2)-C(3)-C(4) | -98.43(16) | C(8)-O(8)-C(10)-O(9) | -3.57(18) |

| | | | |
|---|---|---|---|
| C(8)-O(8)-C(10)-C(11) | 112.32(16) | C(20)-C(17)-C(18)-C(2) | -122.56(13) |
| C(8)-O(8)-C(10)-C(12) | -122.56(16) | C(16)-C(17)-C(18)-C(2) | -5.88(12) |
| C(6)-O(6)-C(13)-O(5) | -34.70(16) | C(1)-C(2)-C(18)-O(18) | 145.30(14) |
| C(6)-O(6)-C(13)-C(15) | -152.09(14) | C(3)-C(2)-C(18)-O(18) | 18.2(2) |
| C(6)-O(6)-C(13)-C(14) | 82.86(18) | C(16)-C(2)-C(18)-O(18) | -103.47(14) |
| C(5)-O(5)-C(13)-O(6) | 15.52(17) | C(1)-C(2)-C(18)-C(17) | -105.46(15) |
| C(5)-O(5)-C(13)-C(15) | 131.70(15) | C(3)-C(2)-C(18)-C(17) | 127.46(15) |
| C(5)-O(5)-C(13)-C(14) | -104.34(16) | C(16)-C(2)-C(18)-C(17) | 5.77(12) |
| C(1)-C(2)-C(16)-C(17) | 113.41(14) | C(17)-C(18)-O(18)-C(19) | -14.57(17) |
| C(3)-C(2)-C(16)-C(17) | -126.57(14) | C(2)-C(18)-O(18)-C(19) | 83.96(15) |
| C(18)-C(2)-C(16)-C(17) | -5.78(12) | C(18)-O(18)-C(19)-C(20) | 30.85(17) |
| C(2)-C(16)-C(17)-C(20) | 112.17(14) | C(16)-C(17)-C(20)-C(19) | -72.99(16) |
| C(2)-C(16)-C(17)-C(18) | 5.84(12) | C(18)-C(17)-C(20)-C(19) | 24.42(16) |
| C(20)-C(17)-C(18)-O(18) | -7.27(17) | O(18)-C(19)-C(20)-C(17) | -33.88(16) |
| C(16)-C(17)-C(18)-O(18) | 109.42(13) | | |

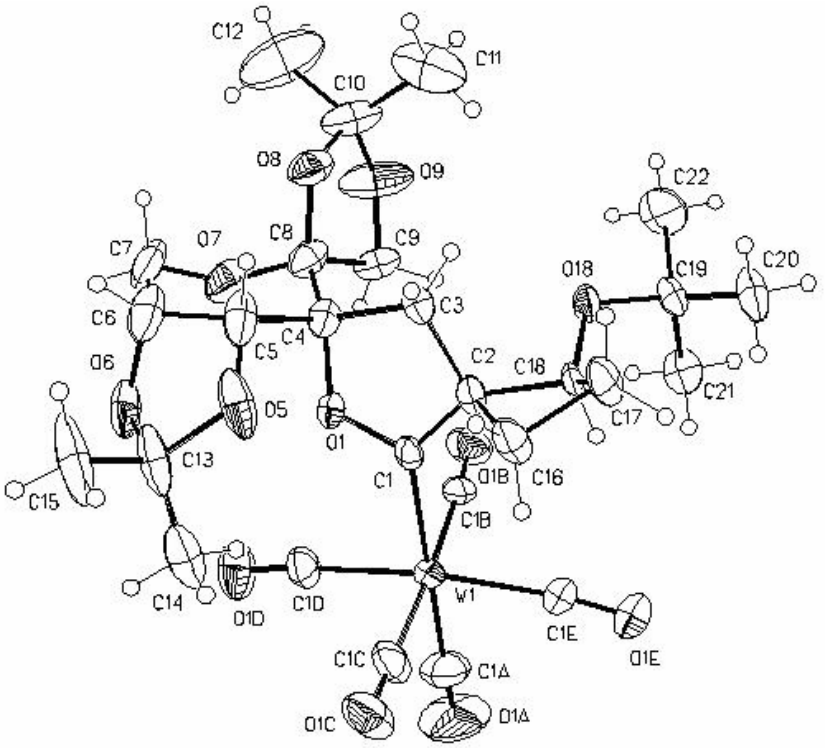
**Tabelle 6: Hydrogen bonds for *[50]b* [Å and °].**

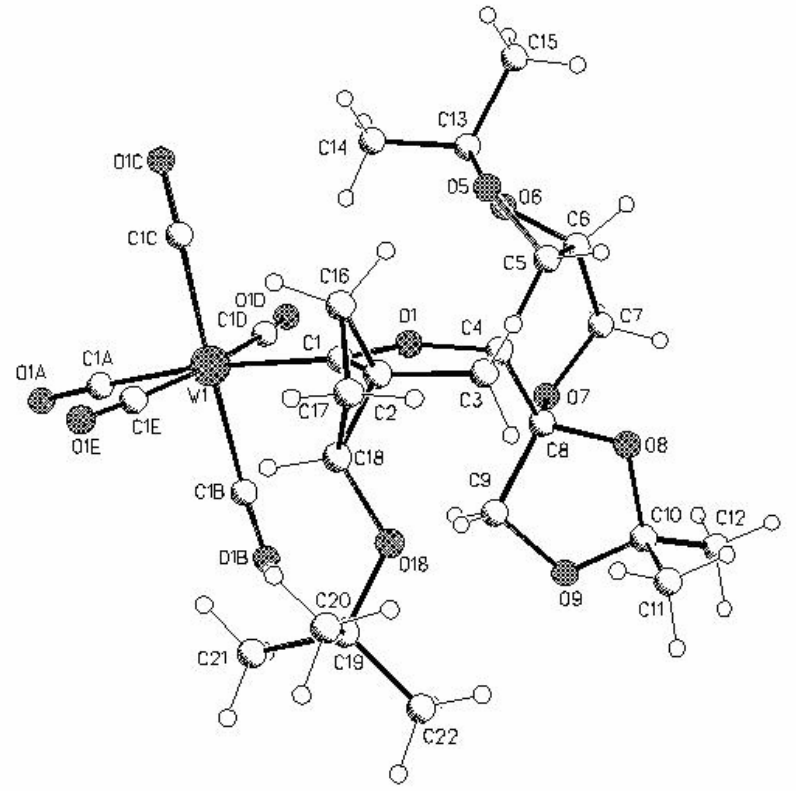
| **D-H...A** | **d(D-H)** | **d(H...A)** | **d(D...A)** | **<(DHA)** |
|---|---|---|---|---|
| C(20)-H(20A)...O(1D)[a] | 0.99 | 2.48 | 3.230(2) | 132.1 |
| C(6)-H(6)...O(18)[b] | 1.00 | 2.50 | 3.262(2) | 132.5 |

Symmetry transformations used to generate equivalent atoms:

[a] x+1,y,z [b] -x+1,y-1/2,-z+1/2

**14. (*1S,4R,7R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-1-(dimethylethoxy)-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro[3.4]oct]-5-yliden}wolfram(0) *[51]b***



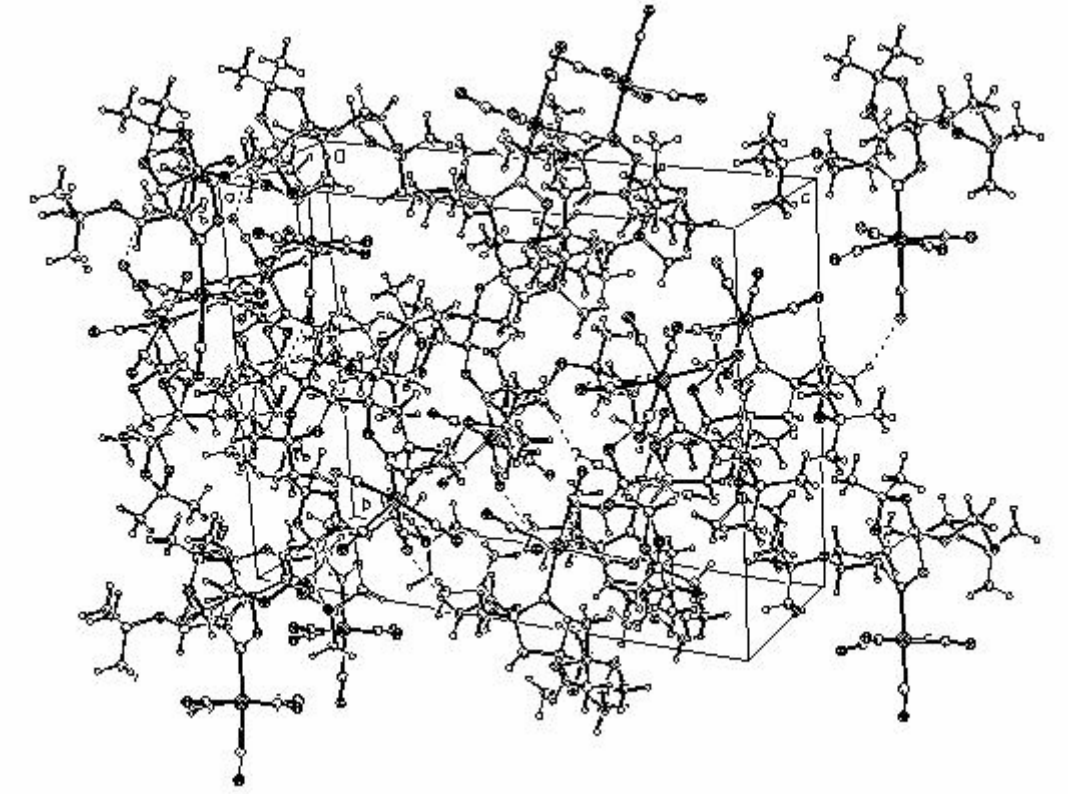


**Table 1: Crystal data and structure refinements for *[51]b***

| | |
|---|---|
| Identification code | ***[51]b*** |
| Empirical formula | $C_{27}H_{34}O_{12}W$ |
| Formula weight | 734.39 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 7.6884(1) Å α = 90° |
| | b = 17.1796(2) Å β = 90° |
| | c = 22.6085(3) Å γ = 90° |
| Volume | 2986.21(7) $Å^3$ |
| Z | 4 |
| Calculated density | 1.633 $mg/m^3$ |
| Absorption coefficient | 3.928 $mm^{-1}$ |
| F(000) | 1464 |
| Crystal size | 0.30 x 0.20 x 0.10 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.95 to 27.48° |
| Limiting indices | $-8 \le h \le 9$, $-22 \le k \le 22$, $-29 \le l \le 29$ |
| Reflections collected / unique | 28872 / 6767 [R(int) = 0.0565] |
| Completeness to Θ = 27.48 | 99.2 % |
| Absorption correction | Empirical from multiple refl |
| Max. and min. transmission | 0.45015 and 0.40226 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 6767 / 0 / 361 |
| Goodness-of-fit on $F^2$ | 0.990 |
| Final R indices [I>2σ(I)] | R1 = 0.0297, wR2 = 0.0516 |
| R indices (all data) | R1 = 0.0396, wR2 = 0.0536 |
| Absolute structure parameter | -0.015(6) |
| Largest diff. peak and hole | 1.104 and -1.113 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[51]b*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| W(1) | 3174(1) | 6706(1) | 3893(1) | 30(1) |
| C(1A) | 2043(9) | 7745(3) | 3740(2) | 63(2) |
| O(1A) | 1337(7) | 8316(2) | 3658(2) | 108(2) |
| C(1B) | 743(7) | 6275(2) | 4057(2) | 37(1) |
| O(1B) | -606(5) | 6013(2) | 4110(2) | 49(1) |
| C(1C) | 5611(7) | 7113(3) | 3698(2) | 42(1) |
| O(1C) | 6960(5) | 7349(2) | 3581(1) | 57(1) |
| C(1D) | 2733(5) | 6469(3) | 3017(2) | 37(1) |
| O(1D) | 2451(4) | 6400(2) | 2524(2) | 60(1) |
| C(1E) | 3542(7) | 7097(3) | 4741(2) | 45(1) |
| O(1E) | 3692(5) | 7380(2) | 5188(2) | 67(1) |
| O(1) | 3923(3) | 5027(2) | 3594(1) | 24(1) |
| C(1) | 4042(5) | 5527(2) | 4036(2) | 24(1) |
| C(2) | 4704(4) | 5091(2) | 4578(2) | 25(1) |
| C(3) | 4822(5) | 4234(2) | 4401(2) | 27(1) |
| C(4) | 4346(5) | 4207(2) | 3745(2) | 27(1) |
| C(5) | 5904(7) | 3946(3) | 3354(2) | 44(1) |
| O(5) | 7139(4) | 4546(2) | 3310(1) | 51(1) |
| C(6) | 5412(7) | 3761(3) | 2716(2) | 50(1) |
| O(6) | 5647(4) | 4495(2) | 2436(1) | 45(1) |
| C(7) | 3619(7) | 3451(3) | 2624(2) | 61(2) |
| O(7) | 2331(4) | 3820(2) | 2981(1) | 44(1) |
| C(8) | 2690(6) | 3770(3) | 3601(2) | 35(1) |
| O(8) | 2849(4) | 2997(2) | 3789(2) | 42(1) |
| C(9) | 1035(5) | 4047(2) | 3922(3) | 35(1) |
| O(9) | 75(4) | 3352(2) | 3990(2) | 73(1) |
| C(10) | 1219(6) | 2722(3) | 4016(3) | 55(2) |
| C(11) | 1463(7) | 2446(4) | 4633(3) | 81(2) |
| C(12) | 581(10) | 2082(4) | 3601(4) | 119(3) |
| C(13) | 7159(6) | 4848(4) | 2703(2) | 52(2) |
| C(14) | 6954(7) | 5708(3) | 2699(2) | 55(1) |
| C(15) | 8842(6) | 4567(4) | 2406(2) | 82(2) |
| C(16) | 6418(5) | 5387(3) | 4870(2) | 38(1) |
| C(17) | 5664(5) | 5133(3) | 5480(2) | 40(1) |
| C(18) | 3847(5) | 5233(3) | 5191(2) | 26(1) |
| O(18) | 2498(3) | 4710(2) | 5331(1) | 28(1) |
| C(19) | 1436(5) | 4896(3) | 5849(2) | 31(1) |
| C(20) | 2570(6) | 4940(3) | 6403(2) | 44(1) |
| C(21) | 448(6) | 5656(3) | 5761(2) | 41(1) |
| C(22) | 182(6) | 4216(3) | 5876(2) | 58(2) |

**Tabelle 3: Bond lengths [Å] for *[51]b*.**

| | | | |
|---|---|---|---|
| W(1)-C(1A) | 2.015(6) | O(5)-C(13) | 1.468(5) |
| W(1)-C(1B) | 2.044(5) | C(6)-O(6) | 1.423(6) |
| W(1)-C(1C) | 2.048(5) | C(6)-C(7) | 1.492(7) |
| W(1)-C(1D) | 2.049(4) | O(6)-C(13) | 1.444(6) |
| W(1)-C(1E) | 2.051(5) | C(7)-O(7) | 1.425(5) |
| W(1)-C(1) | 2.157(4) | O(7)-C(8) | 1.432(5) |
| C(1A)-O(1A) | 1.137(6) | C(8)-O(8) | 1.399(5) |
| C(1B)-O(1B) | 1.137(5) | C(8)-C(9) | 1.540(6) |
| C(1C)-O(1C) | 1.145(5) | O(8)-C(10) | 1.435(6) |
| C(1D)-O(1D) | 1.142(5) | C(9)-O(9) | 1.412(5) |
| C(1E)-O(1E) | 1.128(5) | O(9)-C(10) | 1.396(6) |
| O(1)-C(1) | 1.322(4) | C(10)-C(11) | 1.484(8) |
| O(1)-C(4) | 1.486(5) | C(10)-C(12) | 1.526(8) |
| C(1)-C(2) | 1.523(5) | C(13)-C(14) | 1.485(8) |
| C(2)-C(3) | 1.527(6) | C(13)-C(15) | 1.534(6) |
| C(2)-C(18) | 1.553(5) | C(16)-C(17) | 1.558(6) |
| C(2)-C(16) | 1.559(5) | C(17)-C(18) | 1.551(6) |
| C(3)-C(4) | 1.528(5) | C(18)-O(18) | 1.409(4) |
| C(4)-C(8) | 1.513(6) | O(18)-C(19) | 1.464(4) |
| C(4)-C(5) | 1.554(6) | C(19)-C(22) | 1.517(6) |
| C(5)-O(5) | 1.405(6) | C(19)-C(21) | 1.523(6) |
| C(5)-C(6) | 1.525(6) | C(19)-C(20) | 1.527(6) |

**Tabelle 4: Bond angles [°] for *[51]b*.**

| | | | |
|---|---|---|---|
| C(1A)-W(1)-C(1B) | 87.6(2) | C(5)-O(5)-C(13) | 109.5(4) |
| C(1A)-W(1)-C(1C) | 93.2(2) | O(6)-C(6)-C(7) | 111.7(4) |
| C(1B)-W(1)-C(1C) | 177.76(18) | O(6)-C(6)-C(5) | 101.9(4) |
| C(1A)-W(1)-C(1D) | 86.5(2) | C(7)-C(6)-C(5) | 115.8(5) |
| C(1B)-W(1)-C(1D) | 87.28(17) | C(6)-O(6)-C(13) | 106.7(4) |
| C(1C)-W(1)-C(1D) | 90.66(17) | O(7)-C(7)-C(6) | 113.9(4) |
| C(1A)-W(1)-C(1E) | 86.0(2) | C(7)-O(7)-C(8) | 113.2(3) |
| C(1B)-W(1)-C(1E) | 94.28(18) | O(8)-C(8)-O(7) | 111.8(4) |
| C(1C)-W(1)-C(1E) | 87.88(19) | O(8)-C(8)-C(4) | 109.4(3) |
| C(1D)-W(1)-C(1E) | 172.25(18) | O(7)-C(8)-C(4) | 110.0(4) |
| C(1A)-W(1)-C(1) | 172.3(2) | O(8)-C(8)-C(9) | 102.9(4) |
| C(1B)-W(1)-C(1) | 85.13(15) | O(7)-C(8)-C(9) | 106.4(4) |
| C(1C)-W(1)-C(1) | 94.01(17) | C(4)-C(8)-C(9) | 116.1(3) |
| C(1D)-W(1)-C(1) | 90.57(16) | C(8)-O(8)-C(10) | 110.2(3) |
| C(1E)-W(1)-C(1) | 97.12(17) | O(9)-C(9)-C(8) | 102.8(3) |
| O(1A)-C(1A)-W(1) | 177.0(6) | C(10)-O(9)-C(9) | 109.4(3) |
| O(1B)-C(1B)-W(1) | 175.2(4) | O(9)-C(10)-O(8) | 106.2(4) |
| O(1C)-C(1C)-W(1) | 178.7(4) | O(9)-C(10)-C(11) | 111.6(5) |
| O(1D)-C(1D)-W(1) | 174.4(4) | O(8)-C(10)-C(11) | 109.4(4) |
| O(1E)-C(1E)-W(1) | 173.3(4) | O(9)-C(10)-C(12) | 109.2(5) |
| C(1)-O(1)-C(4) | 115.3(3) | O(8)-C(10)-C(12) | 107.3(5) |
| O(1)-C(1)-C(2) | 108.2(3) | C(11)-C(10)-C(12) | 112.8(5) |
| O(1)-C(1)-W(1) | 118.3(3) | O(6)-C(13)-O(5) | 103.5(4) |
| C(2)-C(1)-W(1) | 133.3(3) | O(6)-C(13)-C(14) | 109.3(4) |
| C(1)-C(2)-C(3) | 106.4(3) | O(5)-C(13)-C(14) | 110.9(4) |
| C(1)-C(2)-C(18) | 119.8(3) | O(6)-C(13)-C(15) | 111.3(4) |
| C(3)-C(2)-C(18) | 114.2(3) | O(5)-C(13)-C(15) | 107.8(4) |
| C(1)-C(2)-C(16) | 117.5(3) | C(14)-C(13)-C(15) | 113.6(5) |
| C(3)-C(2)-C(16) | 112.0(3) | C(17)-C(16)-C(2) | 88.2(3) |
| C(18)-C(2)-C(16) | 85.9(3) | C(18)-C(17)-C(16) | 86.0(3) |
| C(2)-C(3)-C(4) | 105.7(3) | O(18)-C(18)-C(17) | 119.9(3) |
| O(1)-C(4)-C(8) | 103.7(3) | O(18)-C(18)-C(2) | 114.4(3) |
| O(1)-C(4)-C(3) | 104.2(3) | C(17)-C(18)-C(2) | 88.7(3) |
| C(8)-C(4)-C(3) | 115.1(4) | C(18)-O(18)-C(19) | 116.8(3) |
| O(1)-C(4)-C(5) | 108.2(3) | O(18)-C(19)-C(22) | 102.6(3) |
| C(8)-C(4)-C(5) | 112.5(3) | O(18)-C(19)-C(21) | 111.2(3) |
| C(3)-C(4)-C(5) | 112.1(3) | C(22)-C(19)-C(21) | 110.4(3) |
| O(5)-C(5)-C(6) | 104.7(4) | O(18)-C(19)-C(20) | 110.4(3) |
| O(5)-C(5)-C(4) | 110.5(4) | C(22)-C(19)-C(20) | 111.5(4) |
| C(6)-C(5)-C(4) | 114.0(4) | C(21)-C(19)-C(20) | 110.5(4) |

**Tabelle 5: Torsion angles [°] for *[51]b.***

| | | | |
|---|---|---|---|
| C(1B)-W(1)-C(1A)-O(1A) | -2(10) | C(1)-O(1)-C(4)-C(8) | 120.2(3) |
| C(1C)-W(1)-C(1A)-O(1A) | 176(100) | C(1)-O(1)-C(4)-C(3) | -0.7(4) |
| C(1D)-W(1)-C(1A)-O(1A) | 85(10) | C(1)-O(1)-C(4)-C(5) | -120.1(3) |
| C(1E)-W(1)-C(1A)-O(1A) | -96(10) | C(2)-C(3)-C(4)-O(1) | -2.2(4) |
| C(1)-W(1)-C(1A)-O(1A) | 18(11) | C(2)-C(3)-C(4)-C(8) | -115.1(4) |
| C(1A)-W(1)-C(1B)-O(1B) | 101(5) | C(2)-C(3)-C(4)-C(5) | 114.6(4) |
| C(1C)-W(1)-C(1B)-O(1B) | -9(9) | O(1)-C(4)-C(5)-O(5) | 40.9(4) |
| C(1D)-W(1)-C(1B)-O(1B) | 15(5) | C(8)-C(4)-C(5)-O(5) | 154.9(4) |
| C(1E)-W(1)-C(1B)-O(1B) | -173(5) | C(3)-C(4)-C(5)-O(5) | -73.5(4) |
| C(1)-W(1)-C(1B)-O(1B) | -76(5) | O(1)-C(4)-C(5)-C(6) | -76.6(5) |
| C(1A)-W(1)-C(1C)-O(1C) | -38(23) | C(8)-C(4)-C(5)-C(6) | 37.4(6) |
| C(1B)-W(1)-C(1C)-O(1C) | 72(25) | C(3)-C(4)-C(5)-C(6) | 169.0(4) |
| C(1D)-W(1)-C(1C)-O(1C) | 49(23) | C(6)-C(5)-O(5)-C(13) | 11.8(5) |
| C(1E)-W(1)-C(1C)-O(1C) | -123(23) | C(4)-C(5)-O(5)-C(13) | -111.3(4) |
| C(1)-W(1)-C(1C)-O(1C) | 140(23) | O(5)-C(5)-C(6)-O(6) | -30.0(4) |
| C(1A)-W(1)-C(1D)-O(1D) | 10(4) | C(4)-C(5)-C(6)-O(6) | 90.7(5) |
| C(1B)-W(1)-C(1D)-O(1D) | 98(4) | O(5)-C(5)-C(6)-C(7) | -151.5(4) |
| C(1C)-W(1)-C(1D)-O(1D) | -83(4) | C(4)-C(5)-C(6)-C(7) | -30.7(6) |
| C(1E)-W(1)-C(1D)-O(1D) | -4(5) | C(7)-C(6)-O(6)-C(13) | 161.9(4) |
| C(1)-W(1)-C(1D)-O(1D) | -177(4) | C(5)-C(6)-O(6)-C(13) | 37.6(4) |
| C(1A)-W(1)-C(1E)-O(1E) | -9(4) | O(6)-C(6)-C(7)-O(7) | -76.2(5) |
| C(1B)-W(1)-C(1E)-O(1E) | -96(4) | C(5)-C(6)-C(7)-O(7) | 39.8(6) |
| C(1C)-W(1)-C(1E)-O(1E) | 84(4) | C(6)-C(7)-O(7)-C(8) | -57.4(5) |
| C(1D)-W(1)-C(1E)-O(1E) | 5(5) | C(7)-O(7)-C(8)-O(8) | -57.7(5) |
| C(1)-W(1)-C(1E)-O(1E) | 178(4) | C(7)-O(7)-C(8)-C(4) | 64.1(5) |
| C(4)-O(1)-C(1)-C(2) | 3.3(4) | C(7)-O(7)-C(8)-C(9) | -169.3(4) |
| C(4)-O(1)-C(1)-W(1) | -173.0(2) | O(1)-C(4)-C(8)-O(8) | -173.2(3) |
| C(1A)-W(1)-C(1)-O(1) | 62.5(15) | C(3)-C(4)-C(8)-O(8) | -60.0(5) |
| C(1B)-W(1)-C(1)-O(1) | 82.1(3) | C(5)-C(4)-C(8)-O(8) | 70.1(5) |
| C(1C)-W(1)-C(1)-O(1) | -95.8(3) | O(1)-C(4)-C(8)-O(7) | 63.6(4) |
| C(1D)-W(1)-C(1)-O(1) | -5.1(3) | C(3)-C(4)-C(8)-O(7) | 176.8(3) |
| C(1E)-W(1)-C(1)-O(1) | 175.8(3) | C(5)-C(4)-C(8)-O(7) | -53.1(5) |
| C(1A)-W(1)-C(1)-C(2) | -112.8(14) | O(1)-C(4)-C(8)-C(9) | -57.3(5) |
| C(1B)-W(1)-C(1)-C(2) | -93.1(4) | C(3)-C(4)-C(8)-C(9) | 55.9(5) |
| C(1C)-W(1)-C(1)-C(2) | 89.0(3) | C(5)-C(4)-C(8)-C(9) | -174.0(4) |
| C(1D)-W(1)-C(1)-C(2) | 179.7(3) | O(7)-C(8)-O(8)-C(10) | -94.9(5) |
| C(1E)-W(1)-C(1)-C(2) | 0.6(4) | C(4)-C(8)-O(8)-C(10) | 143.0(4) |
| O(1)-C(1)-C(2)-C(3) | -4.5(4) | C(9)-C(8)-O(8)-C(10) | 18.9(5) |
| W(1)-C(1)-C(2)-C(3) | 171.1(3) | O(8)-C(8)-C(9)-O(9) | -28.0(5) |
| O(1)-C(1)-C(2)-C(18) | -136.0(3) | O(7)-C(8)-C(9)-O(9) | 89.7(5) |
| W(1)-C(1)-C(2)-C(18) | 39.5(5) | C(4)-C(8)-C(9)-O(9) | -147.5(4) |
| O(1)-C(1)-C(2)-C(16) | 122.0(4) | C(8)-C(9)-O(9)-C(10) | 27.8(5) |
| W(1)-C(1)-C(2)-C(16) | -62.4(5) | C(9)-O(9)-C(10)-O(8) | -16.9(6) |
| C(1)-C(2)-C(3)-C(4) | 4.0(4) | C(9)-O(9)-C(10)-C(11) | 102.2(5) |
| C(18)-C(2)-C(3)-C(4) | 138.5(3) | C(9)-O(9)-C(10)-C(12) | -132.4(6) |
| C(16)-C(2)-C(3)-C(4) | -125.8(3) | C(8)-O(8)-C(10)-O(9) | -2.7(6) |

| | | | |
|---|---|---|---|
| C(8)-O(8)-C(10)-C(11) | -123.2(5) | C(16)-C(17)-C(18)-C(2) | 24.9(3) |
| C(8)-O(8)-C(10)-C(12) | 114.1(5) | C(1)-C(2)-C(18)-O(18) | 93.1(4) |
| C(6)-O(6)-C(13)-O(5) | -30.9(5) | C(3)-C(2)-C(18)-O(18) | -34.9(5) |
| C(6)-O(6)-C(13)-C(14) | -149.0(4) | C(16)-C(2)-C(18)-O(18) | -147.3(4) |
| C(6)-O(6)-C(13)-C(15) | 84.7(5) | C(1)-C(2)-C(18)-C(17) | -144.5(4) |
| C(5)-O(5)-C(13)-O(6) | 10.7(5) | C(3)-C(2)-C(18)-C(17) | 87.5(4) |
| C(5)-O(5)-C(13)-C(14) | 127.7(4) | C(16)-C(2)-C(18)-C(17) | -24.9(3) |
| C(5)-O(5)-C(13)-C(15) | -107.4(5) | C(17)-C(18)-O(18)-C(19) | 87.3(4) |
| C(1)-C(2)-C(16)-C(17) | 146.5(4) | C(2)-C(18)-O(18)-C(19) | -169.3(3) |
| C(3)-C(2)-C(16)-C(17) | -89.8(4) | C(18)-O(18)-C(19)-C(22) | -178.5(3) |
| C(18)-C(2)-C(16)-C(17) | 24.8(3) | C(18)-O(18)-C(19)-C(21) | 63.5(4) |
| C(2)-C(16)-C(17)-C(18) | -24.8(3) | C(18)-O(18)-C(19)-C(20) | -59.5(5) |
| C(16)-C(17)-C(18)-O(18) | 142.4(4) | | |

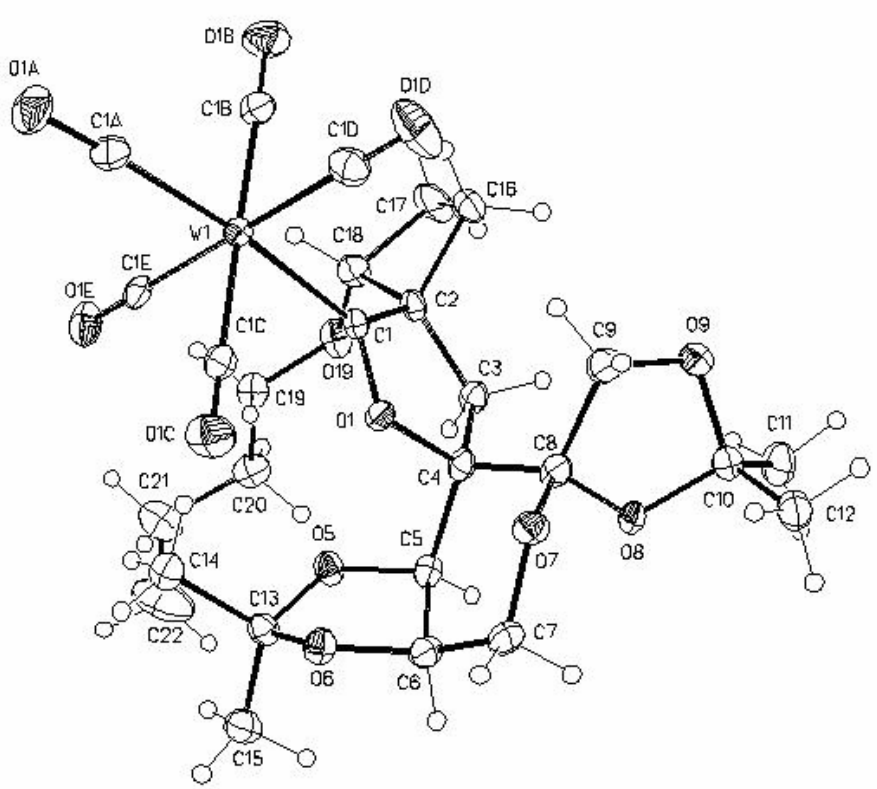
**Tabelle 6: Hydrogen bonds for *[51]b* [Å and °].**

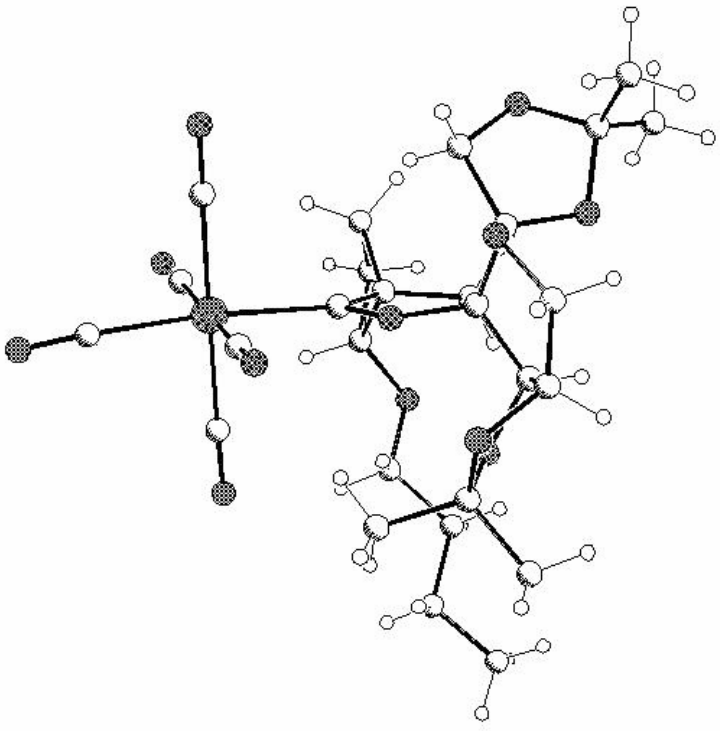
| **D-H...A** | **d(D-H)** | **d(H...A)** | **d(D...A)** | **<(DHA)** |
|---|---|---|---|---|
| C(17)-H(17B)...O(1A)[a] | 0.99 | 2.39 | 3.342(7) | 161.5 |
| C(16)-H(16A)...O(1B)[b] | 0.99 | 2.60 | 3.056(5) | 108.0 |
| C(20)-H(20C)...O(6)[c] | 0.98 | 2.60 | 3.537(5) | 160.5 |

Symmetry transformations used to generate equivalent atoms:

[a] x+1/2,-y+3/2,-z+1 [b] x+1,y,z [c] -x+1/2,-y+1,z+1/2

**15. (*1R,4S,7R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-1-(*n*-butoxy)-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxa-cyclopentan-5´,7-6-oxaspiro[3.4]oct]-5-yliden}wolfram(0) *[52]a***



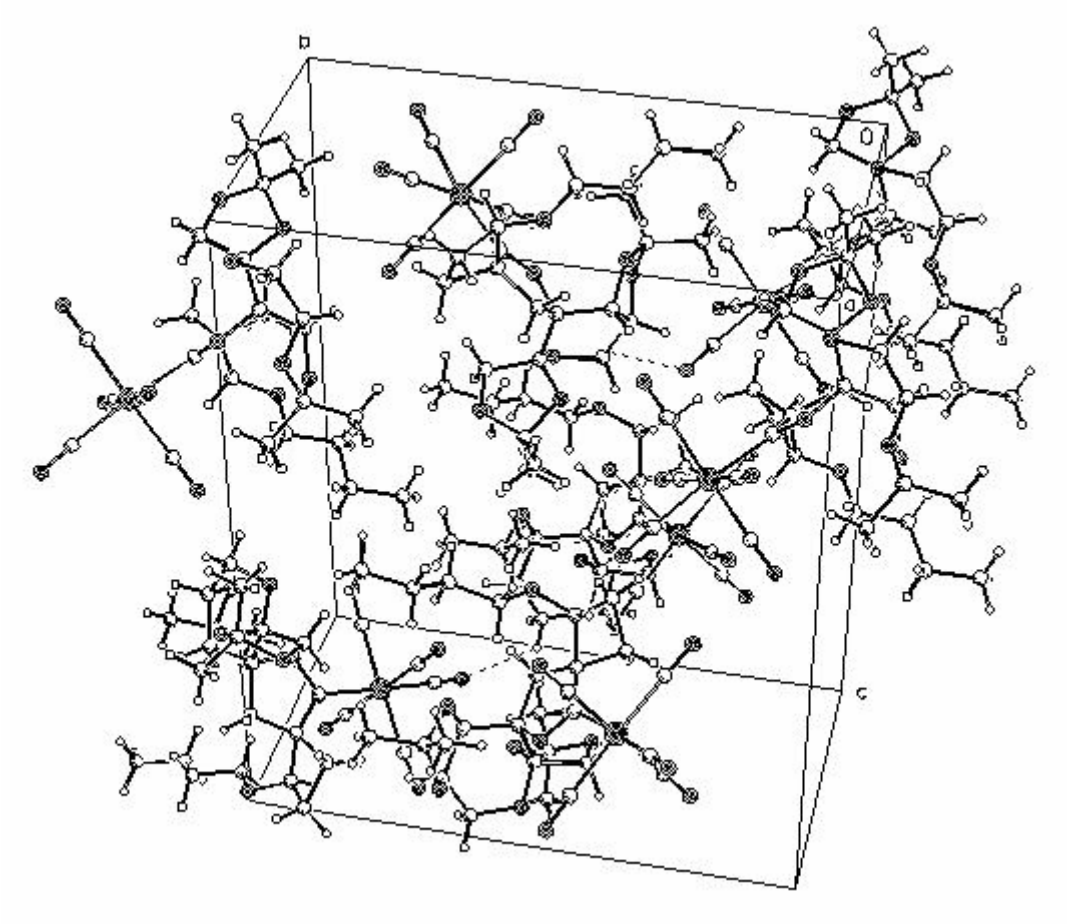

b
c

**Table 1: Crystal data and structure refinements for *[52]a***

| | |
|---|---|
| Identification code | ***[52]a*** |
| Empirical formula | $C_{27}H_{34}O_{12}W$ |
| Formula weight | 734.39 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 8.9508(1) Å α = 90° |
| | b = 16.3343(1) Å β = 90° |
| | c = 20.1719(2) Å γ = 90° |
| Volume | 2949.23(5) $Å^3$ |
| Z | 4 |
| Calculated density | 1.654 $mg/m^3$ |
| Absorption coefficient | 3.977 $mm^{-1}$ |
| F(000) | 1464 |
| Crystal size | 0.40 x 0.25 x 0.15 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 3.04 to 27.46° |
| Limiting indices | $-11 \le h \le 11$, $-21 \le k \le 21$, $-26 \le l \le 26$ |
| Reflections collected / unique | 25549 / 6649 [R(int) = 0.0526] |
| Completeness to Θ = 25.00 | 99.6 % |
| Absorption correction | Empirical from multiple refl |
| Max. and min. transmission | 0.44504 and 0.29060 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 6649 / 0 / 361 |
| Goodness-of-fit on $F^2$ | 1.042 |
| Final R indices [I>2σ(I)] | R1 = 0.0185, wR2 = 0.0402 |
| R indices (all data) | R1 = 0.0194, wR2 = 0.0404 |
| Absolute structure parameter | -0.010(4) |
| Largest diff. peak and hole | 0.565 and -0.686 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[52]a*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| W(1) | 1960(1) | 7041(1) | 1368(1) | 15(1) |
| C(1A) | 319(3) | 7783(2) | 1008(1) | 23(1) |
| O(1A) | -622(2) | 8167(1) | 787(1) | 33(1) |
| C(1B) | 3460(3) | 7667(2) | 805(1) | 22(1) |
| O(1B) | 4264(2) | 8048(1) | 491(1) | 35(1) |
| C(1C) | 432(3) | 6459(2) | 1968(1) | 19(1) |
| O(1C) | -416(2) | 6161(1) | 2309(1) | 30(1) |
| C(1D) | 2410(3) | 7814(2) | 2138(2) | 25(1) |
| O(1D) | 2681(2) | 8241(2) | 2574(1) | 41(1) |
| C(1E) | 1557(3) | 6267(2) | 599(1) | 20(1) |
| O(1E) | 1366(2) | 5821(1) | 162(1) | 30(1) |
| O(1) | 3121(2) | 5621(1) | 2172(1) | 16(1) |
| C(1) | 3570(3) | 6149(2) | 1725(1) | 14(1) |
| C(2) | 5178(3) | 5980(2) | 1552(1) | 16(1) |
| C(3) | 5635(3) | 5237(2) | 1963(1) | 18(1) |
| C(4) | 4308(3) | 5075(2) | 2422(1) | 17(1) |
| C(5) | 3745(3) | 4187(2) | 2402(1) | 18(1) |
| O(5) | 3047(2) | 4026(1) | 1779(1) | 20(1) |
| C(6) | 2542(3) | 3981(2) | 2915(1) | 20(1) |
| O(6) | 1198(2) | 4129(1) | 2555(1) | 20(1) |
| C(7) | 2628(3) | 4455(2) | 3554(1) | 23(1) |
| O(7) | 3169(2) | 5276(1) | 3490(1) | 20(1) |
| C(8) | 4545(3) | 5335(2) | 3146(1) | 18(1) |
| O(8) | 5667(2) | 4822(1) | 3403(1) | 19(1) |
| C(9) | 5131(3) | 6210(2) | 3261(1) | 22(1) |
| O(9) | 6635(2) | 6099(1) | 3473(1) | 22(1) |
| C(10) | 6688(3) | 5316(2) | 3791(1) | 21(1) |
| C(11) | 8252(3) | 4967(2) | 3707(2) | 29(1) |
| C(12) | 6175(3) | 5354(2) | 4506(1) | 26(1) |
| C(13) | 1479(3) | 3852(2) | 1900(1) | 18(1) |
| C(14) | 518(3) | 4342(2) | 1427(2) | 26(1) |
| C(15) | 1247(3) | 2930(2) | 1826(1) | 26(1) |
| C(16) | 6303(3) | 6711(2) | 1559(1) | 24(1) |
| C(17) | 7028(4) | 6382(2) | 922(2) | 33(1) |
| C(18) | 5598(3) | 5904(2) | 787(1) | 21(1) |
| O(19) | 5816(2) | 5114(1) | 535(1) | 23(1) |
| C(19) | 4442(3) | 4745(2) | 327(1) | 22(1) |
| C(20) | 4697(3) | 3841(2) | 203(2) | 27(1) |
| C(21) | 3338(4) | 3435(2) | -104(2) | 37(1) |
| C(22) | 3552(5) | 2520(2) | -190(2) | 62(1) |

**Tabelle 3: Bond lengths [Å] for *[52]a*.**

| | | | |
|---|---|---|---|
| W(1)-C(1B) | 2.033(3) | O(5)-C(13) | 1.453(3) |
| W(1)-C(1E) | 2.034(3) | C(6)-O(6) | 1.425(3) |
| W(1)-C(1A) | 2.038(3) | C(6)-C(7) | 1.507(4) |
| W(1)-C(1D) | 2.042(3) | O(6)-C(13) | 1.420(3) |
| W(1)-C(1C) | 2.060(3) | C(7)-O(7) | 1.432(3) |
| W(1)-C(1) | 2.172(2) | O(7)-C(8) | 1.417(3) |
| C(1A)-O(1A) | 1.141(3) | C(8)-O(8) | 1.407(3) |
| C(1B)-O(1B) | 1.143(3) | C(8)-C(9) | 1.540(4) |
| C(1C)-O(1C) | 1.134(3) | O(8)-C(10) | 1.448(3) |
| C(1D)-O(1D) | 1.148(3) | C(9)-O(9) | 1.424(3) |
| C(1E)-O(1E) | 1.155(3) | O(9)-C(10) | 1.431(3) |
| O(1)-C(1) | 1.312(3) | C(10)-C(12) | 1.514(4) |
| O(1)-C(4) | 1.476(3) | C(10)-C(11) | 1.521(4) |
| C(1)-C(2) | 1.507(3) | C(13)-C(14) | 1.513(4) |
| C(2)-C(3) | 1.525(4) | C(13)-C(15) | 1.527(4) |
| C(2)-C(16) | 1.563(3) | C(16)-C(17) | 1.537(4) |
| C(2)-C(18) | 1.593(4) | C(17)-C(18) | 1.523(4) |
| C(3)-C(4) | 1.528(3) | C(18)-O(19) | 1.401(3) |
| C(4)-C(5) | 1.535(3) | O(19)-C(19) | 1.432(3) |
| C(4)-C(8) | 1.536(4) | C(19)-C(20) | 1.514(4) |
| C(5)-O(5) | 1.428(3) | C(20)-C(21) | 1.517(4) |
| C(5)-C(6) | 1.531(3) | C(21)-C(22) | 1.517(5) |

**Tabelle 4: Bond angles [°] for *[52]a*.**

| | | | |
|---|---|---|---|
| C(1B)-W(1)-C(1E) | 90.25(11) | C(6)-C(5)-C(4) | 114.9(2) |
| C(1B)-W(1)-C(1A) | 88.76(10) | C(5)-O(5)-C(13) | 108.10(19) |
| C(1E)-W(1)-C(1A) | 88.28(10) | O(6)-C(6)-C(7) | 113.0(2) |
| C(1B)-W(1)-C(1D) | 89.05(11) | O(6)-C(6)-C(5) | 102.27(19) |
| C(1E)-W(1)-C(1D) | 178.85(11) | C(7)-C(6)-C(5) | 115.4(2) |
| C(1A)-W(1)-C(1D) | 92.62(11) | C(13)-O(6)-C(6) | 105.62(19) |
| C(1B)-W(1)-C(1C) | 177.10(11) | O(7)-C(7)-C(6) | 114.9(2) |
| C(1E)-W(1)-C(1C) | 92.46(11) | C(8)-O(7)-C(7) | 113.7(2) |
| C(1A)-W(1)-C(1C) | 90.31(11) | O(8)-C(8)-O(7) | 113.5(2) |
| C(1D)-W(1)-C(1C) | 88.25(11) | O(8)-C(8)-C(4) | 106.5(2) |
| C(1B)-W(1)-C(1) | 94.85(10) | O(7)-C(8)-C(4) | 109.1(2) |
| C(1E)-W(1)-C(1) | 87.30(9) | O(8)-C(8)-C(9) | 104.7(2) |
| C(1A)-W(1)-C(1) | 174.30(10) | O(7)-C(8)-C(9) | 106.6(2) |
| C(1D)-W(1)-C(1) | 91.84(10) | C(4)-C(8)-C(9) | 116.6(2) |
| C(1C)-W(1)-C(1) | 86.29(9) | C(8)-O(8)-C(10) | 108.5(2) |
| O(1A)-C(1A)-W(1) | 176.7(2) | O(9)-C(9)-C(8) | 104.4(2) |
| O(1B)-C(1B)-W(1) | 177.0(2) | C(9)-O(9)-C(10) | 106.23(19) |
| O(1C)-C(1C)-W(1) | 177.8(2) | O(9)-C(10)-O(8) | 103.55(19) |
| O(1D)-C(1D)-W(1) | 178.9(3) | O(9)-C(10)-C(12) | 112.3(2) |
| O(1E)-C(1E)-W(1) | 178.3(2) | O(8)-C(10)-C(12) | 110.3(2) |
| C(1)-O(1)-C(4) | 114.29(19) | O(9)-C(10)-C(11) | 108.3(2) |
| O(1)-C(1)-C(2) | 109.4(2) | O(8)-C(10)-C(11) | 108.1(2) |
| O(1)-C(1)-W(1) | 117.80(16) | C(12)-C(10)-C(11) | 113.6(2) |
| C(2)-C(1)-W(1) | 132.82(19) | O(6)-C(13)-O(5) | 105.40(19) |
| C(1)-C(2)-C(3) | 106.0(2) | O(6)-C(13)-C(14) | 108.5(2) |
| C(1)-C(2)-C(16) | 118.2(2) | O(5)-C(13)-C(14) | 109.9(2) |
| C(3)-C(2)-C(16) | 115.5(2) | O(6)-C(13)-C(15) | 112.4(2) |
| C(1)-C(2)-C(18) | 117.7(2) | O(5)-C(13)-C(15) | 107.9(2) |
| C(3)-C(2)-C(18) | 113.7(2) | C(14)-C(13)-C(15) | 112.5(2) |
| C(16)-C(2)-C(18) | 85.23(18) | C(17)-C(16)-C(2) | 89.8(2) |
| C(2)-C(3)-C(4) | 105.0(2) | C(18)-C(17)-C(16) | 88.5(2) |
| O(1)-C(4)-C(3) | 104.36(19) | O(19)-C(18)-C(17) | 114.9(2) |
| O(1)-C(4)-C(5) | 108.99(19) | O(19)-C(18)-C(2) | 117.1(2) |
| C(3)-C(4)-C(5) | 113.8(2) | C(17)-C(18)-C(2) | 89.16(19) |
| O(1)-C(4)-C(8) | 104.80(19) | C(18)-O(19)-C(19) | 112.02(19) |
| C(3)-C(4)-C(8) | 114.9(2) | O(19)-C(19)-C(20) | 109.2(2) |
| C(5)-C(4)-C(8) | 109.4(2) | C(19)-C(20)-C(21) | 111.9(2) |
| O(5)-C(5)-C(6) | 104.3(2) | C(22)-C(21)-C(20) | 112.1(3) |
| O(5)-C(5)-C(4) | 109.9(2) | | |

**Tabelle 5: Torsion angles [°] for *[52]a.***

| | | | |
|---|---|---|---|
| C(1B)-W(1)-C(1A)-O(1A) | 104(5) | C(1)-O(1)-C(4)-C(3) | -9.5(3) |
| C(1E)-W(1)-C(1A)-O(1A) | 14(5) | C(1)-O(1)-C(4)-C(5) | -131.4(2) |
| C(1D)-W(1)-C(1A)-O(1A) | -167(5) | C(1)-O(1)-C(4)-C(8) | 111.6(2) |
| C(1C)-W(1)-C(1A)-O(1A) | -79(5) | C(2)-C(3)-C(4)-O(1) | 9.3(2) |
| C(1)-W(1)-C(1A)-O(1A) | -25(5) | C(2)-C(3)-C(4)-C(5) | 128.0(2) |
| C(1E)-W(1)-C(1B)-O(1B) | 117(5) | C(2)-C(3)-C(4)-C(8) | -104.9(2) |
| C(1A)-W(1)-C(1B)-O(1B) | 29(5) | O(1)-C(4)-C(5)-O(5) | 46.3(3) |
| C(1D)-W(1)-C(1B)-O(1B) | -64(5) | C(3)-C(4)-C(5)-O(5) | -69.7(3) |
| C(1C)-W(1)-C(1B)-O(1B) | -43(6) | C(8)-C(4)-C(5)-O(5) | 160.34(19) |
| C(1)-W(1)-C(1B)-O(1B) | -156(5) | O(1)-C(4)-C(5)-C(6) | -70.9(3) |
| C(1B)-W(1)-C(1C)-O(1C) | 8(8) | C(3)-C(4)-C(5)-C(6) | 173.1(2) |
| C(1E)-W(1)-C(1C)-O(1C) | -151(6) | C(8)-C(4)-C(5)-C(6) | 43.1(3) |
| C(1A)-W(1)-C(1C)-O(1C) | -63(6) | C(6)-C(5)-O(5)-C(13) | 7.7(2) |
| C(1D)-W(1)-C(1C)-O(1C) | 30(6) | C(4)-C(5)-O(5)-C(13) | -116.0(2) |
| C(1)-W(1)-C(1C)-O(1C) | 122(6) | O(5)-C(5)-C(6)-O(6) | -27.8(2) |
| C(1B)-W(1)-C(1D)-O(1D) | -91(14) | C(4)-C(5)-C(6)-O(6) | 92.6(2) |
| C(1E)-W(1)-C(1D)-O(1D) | -38(18) | O(5)-C(5)-C(6)-C(7) | -150.9(2) |
| C(1A)-W(1)-C(1D)-O(1D) | -179(100) | C(4)-C(5)-C(6)-C(7) | -30.5(3) |
| C(1C)-W(1)-C(1D)-O(1D) | 90(14) | C(7)-C(6)-O(6)-C(13) | 162.8(2) |
| C(1)-W(1)-C(1D)-O(1D) | 4(14) | C(5)-C(6)-O(6)-C(13) | 38.1(2) |
| C(1B)-W(1)-C(1E)-O(1E) | 58(8) | O(6)-C(6)-C(7)-O(7) | -84.2(3) |
| C(1A)-W(1)-C(1E)-O(1E) | 146(8) | C(5)-C(6)-C(7)-O(7) | 33.1(3) |
| C(1D)-W(1)-C(1E)-O(1E) | 5(12) | C(6)-C(7)-O(7)-C(8) | -52.1(3) |
| C(1C)-W(1)-C(1E)-O(1E) | -123(8) | C(7)-O(7)-C(8)-O(8) | -52.6(3) |
| C(1)-W(1)-C(1E)-O(1E) | -37(8) | C(7)-O(7)-C(8)-C(4) | 66.0(3) |
| C(4)-O(1)-C(1)-C(2) | 5.3(3) | C(7)-O(7)-C(8)-C(9) | -167.3(2) |
| C(4)-O(1)-C(1)-W(1) | -175.96(15) | O(1)-C(4)-C(8)-O(8) | 179.61(18) |
| C(1B)-W(1)-C(1)-O(1) | 174.58(19) | C(3)-C(4)-C(8)-O(8) | -66.5(3) |
| C(1E)-W(1)-C(1)-O(1) | -95.4(2) | C(5)-C(4)-C(8)-O(8) | 62.9(2) |
| C(1A)-W(1)-C(1)-O(1) | -56.2(12) | O(1)-C(4)-C(8)-O(7) | 56.7(2) |
| C(1D)-W(1)-C(1)-O(1) | 85.4(2) | C(3)-C(4)-C(8)-O(7) | 170.6(2) |
| C(1C)-W(1)-C(1)-O(1) | -2.7(2) | C(5)-C(4)-C(8)-O(7) | -60.0(3) |
| C(1B)-W(1)-C(1)-C(2) | -7.0(3) | O(1)-C(4)-C(8)-C(9) | -64.0(3) |
| C(1E)-W(1)-C(1)-C(2) | 83.0(2) | C(3)-C(4)-C(8)-C(9) | 49.9(3) |
| C(1A)-W(1)-C(1)-C(2) | 122.2(10) | C(5)-C(4)-C(8)-C(9) | 179.3(2) |
| C(1D)-W(1)-C(1)-C(2) | -96.2(2) | O(7)-C(8)-O(8)-C(10) | -102.9(2) |
| C(1C)-W(1)-C(1)-C(2) | 175.7(2) | C(4)-C(8)-O(8)-C(10) | 137.0(2) |
| O(1)-C(1)-C(2)-C(3) | 1.2(3) | C(9)-C(8)-O(8)-C(10) | 12.9(3) |
| W(1)-C(1)-C(2)-C(3) | -177.28(19) | O(8)-C(8)-C(9)-O(9) | 9.3(3) |
| O(1)-C(1)-C(2)-C(16) | -130.2(2) | O(7)-C(8)-C(9)-O(9) | 129.9(2) |
| W(1)-C(1)-C(2)-C(16) | 51.3(3) | C(4)-C(8)-C(9)-O(9) | -108.0(2) |
| O(1)-C(1)-C(2)-C(18) | 129.7(2) | C(8)-C(9)-O(9)-C(10) | -28.2(3) |
| W(1)-C(1)-C(2)-C(18) | -48.8(3) | C(9)-O(9)-C(10)-O(8) | 36.3(2) |
| C(1)-C(2)-C(3)-C(4) | -6.7(3) | C(9)-O(9)-C(10)-C(12) | -82.7(3) |
| C(16)-C(2)-C(3)-C(4) | 126.3(2) | C(9)-O(9)-C(10)-C(11) | 150.9(2) |
| C(18)-C(2)-C(3)-C(4) | -137.5(2) | C(8)-O(8)-C(10)-O(9) | -30.4(2) |

| | | | |
|---|---|---|---|
| C(8)-O(8)-C(10)-C(12) | 90.0(2) | C(16)-C(17)-C(18)-C(2) | -20.3(2) |
| C(8)-O(8)-C(10)-C(11) | -145.2(2) | C(1)-C(2)-C(18)-O(19) | -102.8(3) |
| C(6)-O(6)-C(13)-O(5) | -34.2(2) | C(3)-C(2)-C(18)-O(19) | 22.0(3) |
| C(6)-O(6)-C(13)-C(14) | -151.8(2) | C(16)-C(2)-C(18)-O(19) | 137.8(2) |
| C(6)-O(6)-C(13)-C(15) | 83.2(2) | C(1)-C(2)-C(18)-C(17) | 139.5(3) |
| C(5)-O(5)-C(13)-O(6) | 15.5(3) | C(3)-C(2)-C(18)-C(17) | -95.7(2) |
| C(5)-O(5)-C(13)-C(14) | 132.2(2) | C(16)-C(2)-C(18)-C(17) | 20.1(2) |
| C(5)-O(5)-C(13)-C(15) | -104.8(2) | C(17)-C(18)-O(19)-C(19) | -171.3(2) |
| C(1)-C(2)-C(16)-C(17) | -138.8(2) | C(2)-C(18)-O(19)-C(19) | 86.0(3) |
| C(3)-C(2)-C(16)-C(17) | 94.1(3) | C(18)-O(19)-C(19)-C(20) | -167.8(2) |
| C(18)-C(2)-C(16)-C(17) | -19.9(2) | O(19)-C(19)-C(20)-C(21) | -172.6(2) |
| C(2)-C(16)-C(17)-C(18) | 20.7(2) | C(19)-C(20)-C(21)-C(22) | -176.5(3) |
| C(16)-C(17)-C(18)-O(19) | -140.0(2) | | |

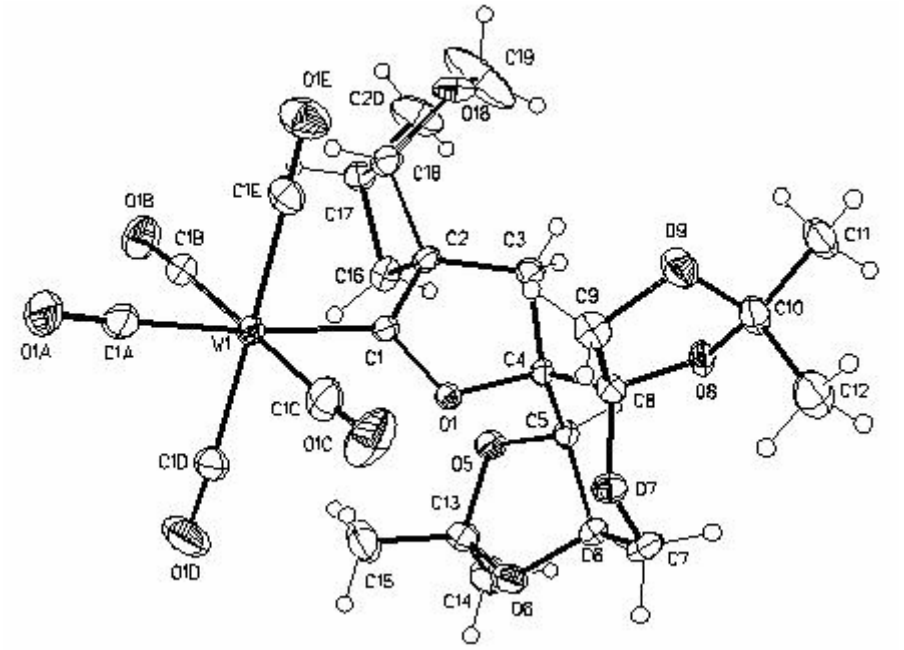
**Tabelle 6: Hydrogen bonds for *[52]a* [Å and °].**

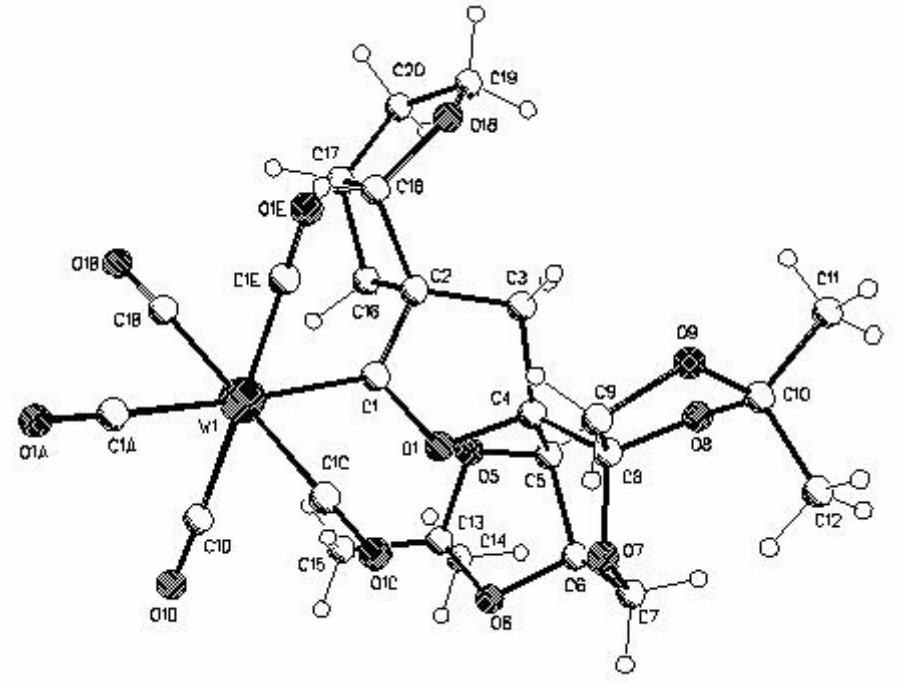
| **D-H...A** | **d(D-H)** | **d(H...A)** | **d(D...A)** | **<(DHA)** |
|---|---|---|---|---|
| C C(7)-H(7A)...O(1A)[a] | 0.99 | 2.49 | 3.069(3) | 117.0 |

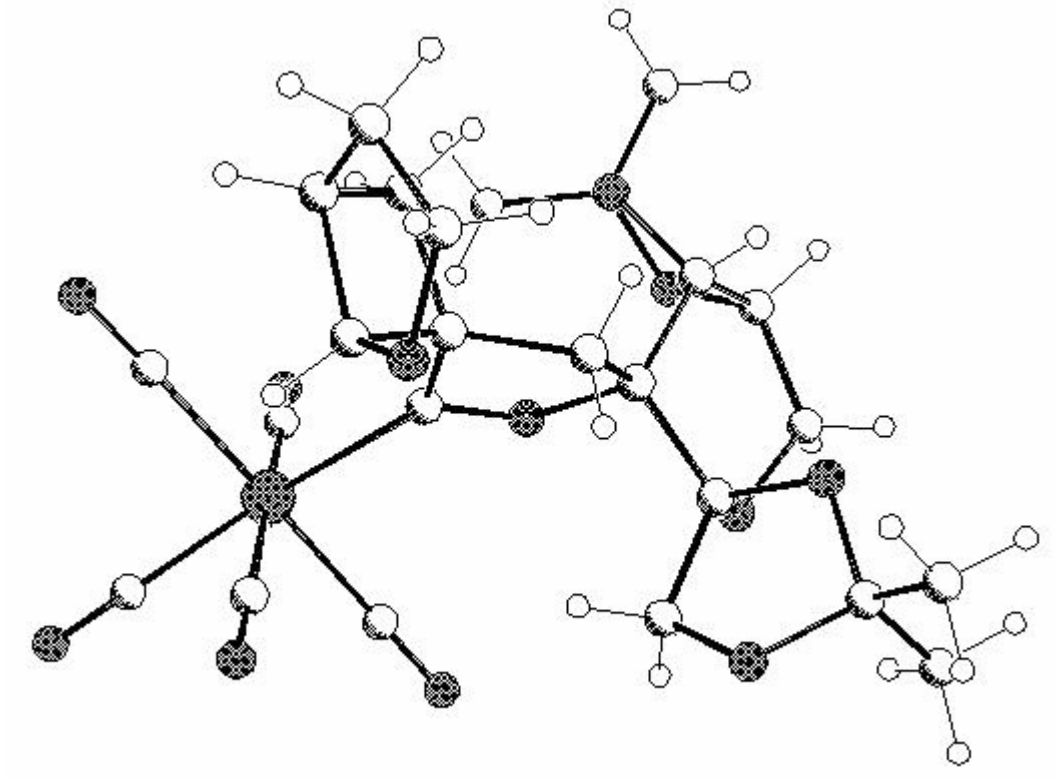
Symmetry transformations used to generate equivalent atoms:

[a] -x,y-1/2,-z+1/2

**16.** **(*3R,5R,1´S,5´R,1´´R,4´´S,6´´R*)-Pentacarbonyl{8´´,8´´,2´´´,2´´´-tetramethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´´,4´´´-1,3-dioxacyclopentan-5´´,3]-spiro[2-oxabicyclo[3.2.0]heptan-7´,5]-2-oxacyclo-pent-1-yliden}wolfram(0) *[53]a/b***





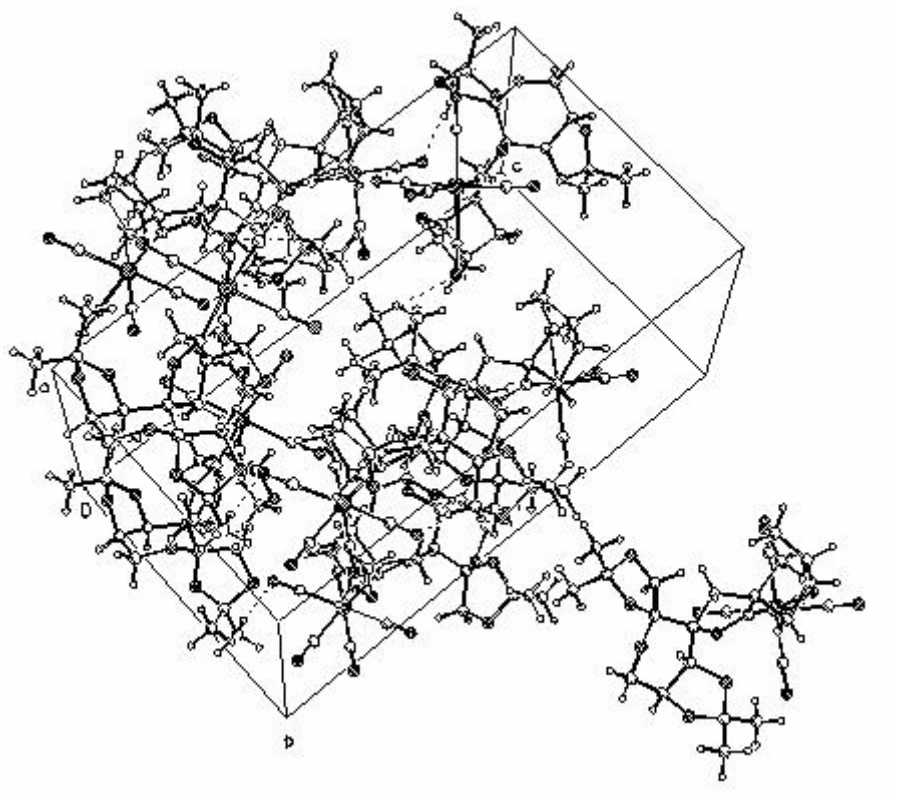


**Table 1: Crystal data and structure refinements for *[53]b***

| | |
|---|---|
| Identification code | ***[53]b*** |
| Empirical formula | $C_{25}H_{28}O_{12}W$ |
| Formula weight | 704.32 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 10.6837(2) Å α = 90° |
| | b = 11.8647(2) Å β = 90° |
| | c = 21.0204(3) Å γ = 90° |
| Volume | 2224.49(4) $Å^3$ |
| Z | 4 |
| Calculated density | 1.756 $mg/m^3$ |
| Absorption coefficient | 4.398 $mm^{-1}$ |
| F(000) | 1392 |
| Crystal size | 0.45 x 0.30 x 0.15 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 3.22 to 27.48° |
| Limiting indices | $-13 \le h \le 9$, $-13 \le k \le 154$, $-27 \le l \le 18$ |
| Reflections collected / unique | 13079 / 5907 [R(int) = 0.0449] |
| Completeness to Θ = 25.00 | 99.7 % |
| Absorption correction | empirical, multiscan |
| Max. and min. transmission | 0.42931 and 0.26645 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 5907 / 0 / 343 |
| Goodness-of-fit on $F^2$ | 1.059 |
| Final R indices [I>2σ(I)] | R1 = 0.0250, wR2 = 0.0615 |
| R indices (all data) | R1 = 0.0265, wR2 = 0.0622 |
| Absolute structure parameter | 0.003(6) |
| Largest diff. peak and hole | 2.241 (near W) and -1.365 (near W) $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[53]b*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| W(1) | 1548(1) | 7454(1) | 7546(1) | 18(1) |
| C(1A) | 229(3) | 7508(4) | 8239(1) | 27(1) |
| O(1A) | -494(2) | 7526(3) | 8640(1) | 42(1) |
| C(1B) | 2592(3) | 8570(3) | 8063(2) | 24(1) |
| O(1B) | 3131(3) | 9196(2) | 8374(1) | 36(1) |
| C(1C) | 481(4) | 6358(4) | 7027(2) | 31(1) |
| O(1C) | -94(3) | 5726(3) | 6743(2) | 49(1) |
| C(1D) | 670(4) | 8743(4) | 7082(2) | 30(1) |
| O(1D) | 116(3) | 9450(3) | 6847(2) | 56(1) |
| C(1E) | 2276(3) | 6114(3) | 8026(2) | 24(1) |
| O(1E) | 2637(3) | 5358(3) | 8305(1) | 39(1) |
| O(1) | 2692(2) | 7018(2) | 6242(1) | 17(1) |
| C(1) | 3012(3) | 7233(3) | 6837(1) | 15(1) |
| C(2) | 4414(3) | 7157(3) | 6888(1) | 17(1) |
| C(3) | 4782(3) | 6380(3) | 6338(1) | 18(1) |
| C(4) | 3725(3) | 6543(3) | 5852(1) | 16(1) |
| C(5) | 4084(3) | 7337(3) | 5307(1) | 17(1) |
| O(5) | 4134(2) | 8473(2) | 5525(1) | 22(1) |
| C(6) | 3154(3) | 7388(3) | 4753(1) | 23(1) |
| O(6) | 2356(2) | 8287(2) | 4933(1) | 27(1) |
| C(7) | 2451(4) | 6308(3) | 4633(2) | 29(1) |
| O(7) | 2118(2) | 5719(2) | 5201(1) | 23(1) |
| C(8) | 3149(3) | 5446(3) | 5592(2) | 19(1) |
| O(8) | 4109(2) | 4877(2) | 5258(1) | 20(1) |
| C(9) | 2676(3) | 4603(3) | 6082(2) | 23(1) |
| O(9) | 3437(2) | 3627(2) | 5989(1) | 28(1) |
| C(10) | 3916(3) | 3689(3) | 5360(2) | 24(1) |
| C(11) | 5166(3) | 3108(3) | 5342(2) | 32(1) |
| C(12) | 3005(4) | 3212(4) | 4876(2) | 37(1) |
| C(13) | 3141(3) | 9106(3) | 5225(2) | 26(1) |
| C(14) | 3726(4) | 9894(3) | 4737(2) | 36(1) |
| C(15) | 2405(4) | 9733(4) | 5726(2) | 41(1) |
| C(16) | 5021(3) | 8381(3) | 6903(2) | 22(1) |
| C(17) | 5729(3) | 8082(3) | 7520(2) | 28(1) |
| C(18) | 5025(3) | 6958(3) | 7551(2) | 27(1) |
| O(18) | 5944(2) | 6070(2) | 7564(1) | 28(1) |
| C(19) | 7125(4) | 6559(4) | 7411(4) | 79(2) |
| C(20) | 7100(4) | 7755(4) | 7481(3) | 45(1) |

**Tabelle 3: Bond lengths [Å] for *[53]b*.**

| | | | |
|---|---|---|---|
| W(1)-C(1A) | 2.029(3) | O(5)-C(13) | 1.446(4) |
| W(1)-C(1E) | 2.038(4) | C(6)-O(6) | 1.416(4) |
| W(1)-C(1D) | 2.042(4) | C(6)-C(7) | 1.506(5) |
| W(1)-C(1C) | 2.044(4) | O(6)-C(13) | 1.423(4) |
| W(1)-C(1B) | 2.044(4) | C(7)-O(7) | 1.428(4) |
| W(1)-C(1) | 2.176(3) | O(7)-C(8) | 1.411(4) |
| C(1A)-O(1A) | 1.143(4) | C(8)-O(8) | 1.415(4) |
| C(1B)-O(1B) | 1.144(4) | C(8)-C(9) | 1.522(5) |
| C(1C)-O(1C) | 1.139(5) | O(8)-C(10) | 1.440(4) |
| C(1D)-O(1D) | 1.139(5) | C(9)-O(9) | 1.429(4) |
| C(1E)-O(1E) | 1.139(4) | O(9)-C(10) | 1.421(4) |
| O(1)-C(1) | 1.322(4) | C(10)-C(11) | 1.504(5) |
| O(1)-C(4) | 1.485(3) | C(10)-C(12) | 1.517(5) |
| C(1)-C(2) | 1.505(4) | C(13)-C(15) | 1.509(5) |
| C(2)-C(3) | 1.529(4) | C(13)-C(14) | 1.523(5) |
| C(2)-C(18) | 1.558(4) | C(16)-C(17) | 1.542(5) |
| C(2)-C(16) | 1.591(5) | C(17)-C(20) | 1.518(5) |
| C(3)-C(4) | 1.536(4) | C(17)-C(18) | 1.533(6) |
| C(4)-C(5) | 1.532(4) | C(18)-O(18) | 1.441(4) |
| C(4)-C(8) | 1.541(4) | O(18)-C(19) | 1.425(5) |
| C(5)-O(5) | 1.424(4) | C(19)-C(20) | 1.427(6) |
| C(5)-C(6) | 1.532(4) | | |

**Tabelle 4: Bond angles [°] for *[53]b*.**

| | | | |
|---|---|---|---|
| C(1A)-W(1)-C(1E) | 86.18(15) | C(4)-C(5)-C(6) | 115.5(3) |
| C(1A)-W(1)-C(1D) | 90.03(15) | C(5)-O(5)-C(13) | 108.9(2) |
| C(1E)-W(1)-C(1D) | 175.06(15) | O(6)-C(6)-C(7) | 112.7(3) |
| C(1A)-W(1)-C(1C) | 90.88(15) | O(6)-C(6)-C(5) | 102.5(2) |
| C(1E)-W(1)-C(1C) | 88.90(17) | C(7)-C(6)-C(5) | 114.6(3) |
| C(1D)-W(1)-C(1C) | 88.00(17) | C(6)-O(6)-C(13) | 106.0(3) |
| C(1A)-W(1)-C(1B) | 88.64(15) | O(7)-C(7)-C(6) | 113.6(3) |
| C(1E)-W(1)-C(1B) | 91.91(15) | C(8)-O(7)-C(7) | 113.9(3) |
| C(1D)-W(1)-C(1B) | 91.16(17) | O(7)-C(8)-O(8) | 112.8(3) |
| C(1C)-W(1)-C(1B) | 179.03(15) | O(7)-C(8)-C(9) | 106.6(3) |
| C(1A)-W(1)-C(1) | 174.36(15) | O(8)-C(8)-C(9) | 105.3(3) |
| C(1E)-W(1)-C(1) | 88.35(13) | O(7)-C(8)-C(4) | 108.9(3) |
| C(1D)-W(1)-C(1) | 95.37(13) | O(8)-C(8)-C(4) | 106.8(3) |
| C(1C)-W(1)-C(1) | 87.65(13) | C(9)-C(8)-C(4) | 116.6(3) |
| C(1B)-W(1)-C(1) | 92.91(13) | C(8)-O(8)-C(10) | 106.8(2) |
| O(1A)-C(1A)-W(1) | 178.3(3) | O(9)-C(9)-C(8) | 104.6(3) |
| O(1B)-C(1B)-W(1) | 176.8(3) | C(10)-O(9)-C(9) | 106.8(3) |
| O(1C)-C(1C)-W(1) | 178.3(4) | O(9)-C(10)-O(8) | 103.9(3) |
| O(1D)-C(1D)-W(1) | 175.7(3) | O(9)-C(10)-C(11) | 108.6(3) |
| O(1E)-C(1E)-W(1) | 177.3(3) | O(8)-C(10)-C(11) | 108.5(3) |
| C(1)-O(1)-C(4) | 113.8(2) | O(9)-C(10)-C(12) | 112.0(3) |
| O(1)-C(1)-C(2) | 108.3(3) | O(8)-C(10)-C(12) | 111.0(3) |
| O(1)-C(1)-W(1) | 119.0(2) | C(11)-C(10)-C(12) | 112.5(3) |
| C(2)-C(1)-W(1) | 132.4(2) | O(6)-C(13)-O(5) | 105.4(3) |
| C(1)-C(2)-C(3) | 103.8(2) | O(6)-C(13)-C(15) | 109.3(3) |
| C(1)-C(2)-C(18) | 119.3(3) | O(5)-C(13)-C(15) | 109.5(3) |
| C(3)-C(2)-C(18) | 118.5(3) | O(6)-C(13)-C(14) | 111.8(3) |
| C(1)-C(2)-C(16) | 110.7(3) | O(5)-C(13)-C(14) | 108.2(3) |
| C(3)-C(2)-C(16) | 117.4(3) | C(15)-C(13)-C(14) | 112.5(3) |
| C(18)-C(2)-C(16) | 87.1(2) | C(17)-C(16)-C(2) | 90.4(3) |
| C(2)-C(3)-C(4) | 103.8(2) | C(20)-C(17)-C(18) | 104.7(3) |
| O(1)-C(4)-C(5) | 111.5(2) | C(20)-C(17)-C(16) | 119.1(4) |
| O(1)-C(4)-C(3) | 103.1(2) | C(18)-C(17)-C(16) | 89.8(3) |
| C(5)-C(4)-C(3) | 113.0(3) | O(18)-C(18)-C(17) | 107.6(3) |
| O(1)-C(4)-C(8) | 102.6(2) | O(18)-C(18)-C(2) | 114.4(3) |
| C(5)-C(4)-C(8) | 110.8(3) | C(17)-C(18)-C(2) | 92.0(3) |
| C(3)-C(4)-C(8) | 115.1(3) | C(19)-O(18)-C(18) | 107.6(3) |
| O(5)-C(5)-C(4) | 110.5(2) | O(18)-C(19)-C(20) | 111.5(4) |
| O(5)-C(5)-C(6) | 103.4(2) | C(19)-C(20)-C(17) | 106.1(3) |

**Tabelle 5: Torsion angles [°] for *[53]b.***

| | | | |
|---|---|---|---|
| C(1E)-W(1)-C(1A)-O(1A) | 28(14) | C(1)-O(1)-C(4)-C(5) | -116.3(3) |
| C(1D)-W(1)-C(1A)-O(1A) | -156(14) | C(1)-O(1)-C(4)-C(3) | 5.3(3) |
| C(1C)-W(1)-C(1A)-O(1A) | 116(14) | C(1)-O(1)-C(4)-C(8) | 125.2(3) |
| C(1B)-W(1)-C(1A)-O(1A) | -64(14) | C(2)-C(3)-C(4)-O(1) | -19.8(3) |
| C(1)-W(1)-C(1A)-O(1A) | 42(14) | C(2)-C(3)-C(4)-C(5) | 100.7(3) |
| C(1A)-W(1)-C(1B)-O(1B) | -2(6) | C(2)-C(3)-C(4)-C(8) | -130.7(3) |
| C(1E)-W(1)-C(1B)-O(1B) | -88(6) | O(1)-C(4)-C(5)-O(5) | 43.2(3) |
| C(1D)-W(1)-C(1B)-O(1B) | 88(6) | C(3)-C(4)-C(5)-O(5) | -72.5(3) |
| C(1C)-W(1)-C(1B)-O(1B) | 58(12) | C(8)-C(4)-C(5)-O(5) | 156.8(2) |
| C(1)-W(1)-C(1B)-O(1B) | -177(100) | O(1)-C(4)-C(5)-C(6) | -73.8(3) |
| C(1A)-W(1)-C(1C)-O(1C) | -105(12) | C(3)-C(4)-C(5)-C(6) | 170.6(3) |
| C(1E)-W(1)-C(1C)-O(1C) | -19(12) | C(8)-C(4)-C(5)-C(6) | 39.9(4) |
| C(1D)-W(1)-C(1C)-O(1C) | 165(12) | C(4)-C(5)-O(5)-C(13) | -113.2(3) |
| C(1B)-W(1)-C(1C)-O(1C) | -166(10) | C(6)-C(5)-O(5)-C(13) | 10.9(3) |
| C(1)-W(1)-C(1C)-O(1C) | 69(12) | O(5)-C(5)-C(6)-O(6) | -29.6(3) |
| C(1A)-W(1)-C(1D)-O(1D) | -14(6) | C(4)-C(5)-C(6)-O(6) | 91.2(3) |
| C(1E)-W(1)-C(1D)-O(1D) | 26(7) | O(5)-C(5)-C(6)-C(7) | -152.0(3) |
| C(1C)-W(1)-C(1D)-O(1D) | 77(6) | C(4)-C(5)-C(6)-C(7) | -31.2(4) |
| C(1B)-W(1)-C(1D)-O(1D) | -103(6) | C(7)-C(6)-O(6)-C(13) | 161.4(3) |
| C(1)-W(1)-C(1D)-O(1D) | 164(6) | C(5)-C(6)-O(6)-C(13) | 37.7(3) |
| C(1A)-W(1)-C(1E)-O(1E) | 25(8) | O(6)-C(6)-C(7)-O(7) | -79.1(4) |
| C(1D)-W(1)-C(1E)-O(1E) | -15(9) | C(5)-C(6)-C(7)-O(7) | 37.6(4) |
| C(1C)-W(1)-C(1E)-O(1E) | -66(8) | C(6)-C(7)-O(7)-C(8) | -57.3(4) |
| C(1B)-W(1)-C(1E)-O(1E) | 113(8) | C(7)-O(7)-C(8)-O(8) | -52.1(4) |
| C(1)-W(1)-C(1E)-O(1E) | -154(8) | C(7)-O(7)-C(8)-C(9) | -167.1(3) |
| C(4)-O(1)-C(1)-C(2) | 12.2(4) | C(7)-O(7)-C(8)-C(4) | 66.3(3) |
| C(4)-O(1)-C(1)-W(1) | -162.2(2) | O(1)-C(4)-C(8)-O(7) | 63.2(3) |
| C(1A)-W(1)-C(1)-O(1) | 105.6(12) | C(5)-C(4)-C(8)-O(7) | -55.9(3) |
| C(1E)-W(1)-C(1)-O(1) | 119.6(3) | C(3)-C(4)-C(8)-O(7) | 174.4(2) |
| C(1D)-W(1)-C(1)-O(1) | -57.2(3) | O(1)-C(4)-C(8)-O(8) | -174.8(2) |
| C(1C)-W(1)-C(1)-O(1) | 30.6(3) | C(5)-C(4)-C(8)-O(8) | 66.2(3) |
| C(1B)-W(1)-C(1)-O(1) | -148.6(2) | C(3)-C(4)-C(8)-O(8) | -63.6(3) |
| C(1A)-W(1)-C(1)-C(2) | -67.2(13) | O(1)-C(4)-C(8)-C(9) | -57.5(3) |
| C(1E)-W(1)-C(1)-C(2) | -53.2(3) | C(5)-C(4)-C(8)-C(9) | -176.5(3) |
| C(1D)-W(1)-C(1)-C(2) | 130.1(3) | C(3)-C(4)-C(8)-C(9) | 53.7(4) |
| C(1C)-W(1)-C(1)-C(2) | -142.2(3) | O(7)-C(8)-O(8)-C(10) | -95.7(3) |
| C(1B)-W(1)-C(1)-C(2) | 38.6(3) | C(9)-C(8)-O(8)-C(10) | 20.2(3) |
| O(1)-C(1)-C(2)-C(3) | -24.5(3) | C(4)-C(8)-O(8)-C(10) | 144.7(3) |
| W(1)-C(1)-C(2)-C(3) | 148.9(2) | O(7)-C(8)-C(9)-O(9) | 121.1(3) |
| O(1)-C(1)-C(2)-C(18) | -159.0(3) | O(8)-C(8)-C(9)-O(9) | 1.1(3) |
| W(1)-C(1)-C(2)-C(18) | 14.3(5) | C(4)-C(8)-C(9)-O(9) | -117.1(3) |
| O(1)-C(1)-C(2)-C(16) | 102.4(3) | C(8)-C(9)-O(9)-C(10) | -22.2(3) |
| W(1)-C(1)-C(2)-C(16) | -84.3(3) | C(9)-O(9)-C(10)-O(8) | 34.9(3) |
| C(1)-C(2)-C(3)-C(4) | 26.6(3) | C(9)-O(9)-C(10)-C(11) | 150.3(3) |
| C(18)-C(2)-C(3)-C(4) | 161.5(3) | C(9)-O(9)-C(10)-C(12) | -84.9(3) |
| C(16)-C(2)-C(3)-C(4) | -95.9(3) | C(8)-O(8)-C(10)-O(9) | -34.3(3) |

| | | | |
|---|---|---|---|
| C(8)-O(8)-C(10)-C(11) | -149.7(3) | C(20)-C(17)-C(18)-C(2) | -113.5(3) |
| C(8)-O(8)-C(10)-C(12) | 86.3(3) | C(16)-C(17)-C(18)-C(2) | 6.7(3) |
| C(6)-O(6)-C(13)-O(5) | -31.6(3) | C(1)-C(2)-C(18)-O(18) | 131.1(3) |
| C(6)-O(6)-C(13)-C(15) | -149.2(3) | C(3)-C(2)-C(18)-O(18) | 3.1(4) |
| C(6)-O(6)-C(13)-C(14) | 85.6(3) | C(16)-C(2)-C(18)-O(18) | -116.8(3) |
| C(5)-O(5)-C(13)-O(6) | 11.7(3) | C(1)-C(2)-C(18)-C(17) | -118.6(3) |
| C(5)-O(5)-C(13)-C(15) | 129.1(3) | C(3)-C(2)-C(18)-C(17) | 113.4(3) |
| C(5)-O(5)-C(13)-C(14) | -108.0(3) | C(16)-C(2)-C(18)-C(17) | -6.5(2) |
| C(1)-C(2)-C(16)-C(17) | 126.7(3) | C(17)-C(18)-O(18)-C(19) | -11.8(5) |
| C(3)-C(2)-C(16)-C(17) | -114.4(3) | C(2)-C(18)-O(18)-C(19) | 88.8(5) |
| C(18)-C(2)-C(16)-C(17) | 6.4(2) | C(18)-O(18)-C(19)-C(20) | 17.1(7) |
| C(2)-C(16)-C(17)-C(20) | 100.3(4) | O(18)-C(19)-C(20)-C(17) | -15.0(8) |
| C(2)-C(16)-C(17)-C(18) | -6.5(3) | C(18)-C(17)-C(20)-C(19) | 7.0(7) |
| C(20)-C(17)-C(18)-O(18) | 2.9(4) | C(16)-C(17)-C(20)-C(19) | -91.2(6) |
| C(16)-C(17)-C(18)-O(18) | 123.0(3) | | |

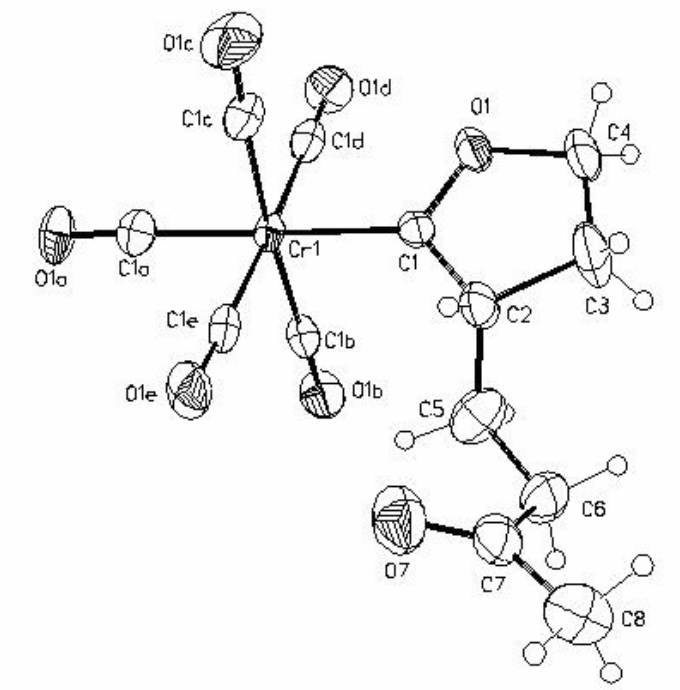
**Tabelle 6: Hydrogen bonds for *[53]b* [Å and °].**

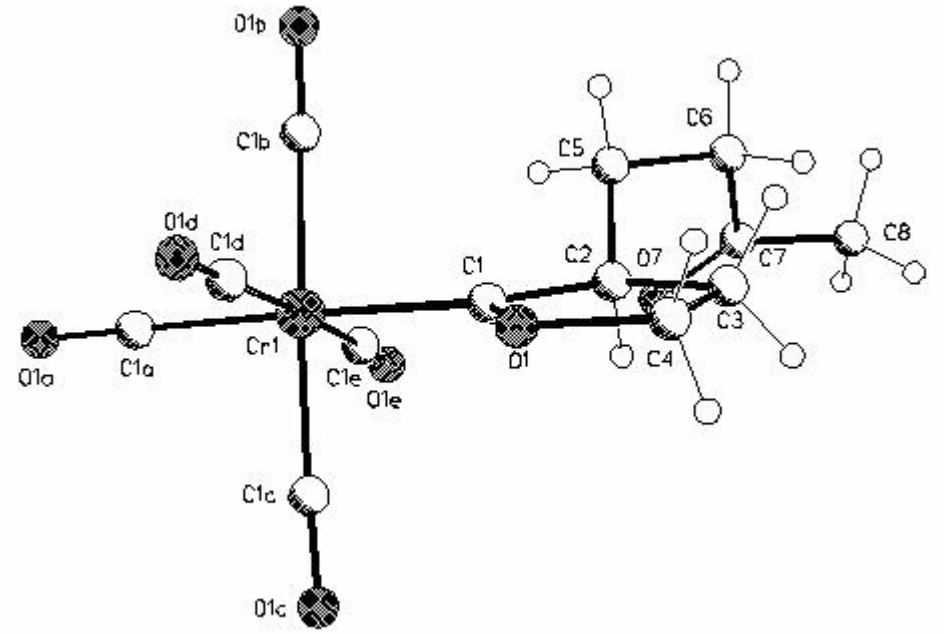
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(11)-H(11C)...O(1B)[a] | 0.98 | 2.55 | 3.502(5) | 164.4 |
| C(14)-H(14B)...O(1C)[b] | 0.98 | 2.57 | 3.435(5) | 147.2 |

Symmetry transformations used to generate equivalent atoms:

[a] -x+1,y-1/2,-z+3/2 [b] x+1/2,-y+3/2,-z+1

## 17. Pentacarbonyl[5-(3´-oxobutyl)-2-oxacyclopentyliden]-chrom(0) *[55]*


O1c
C1c
O1d
C1d
O1
C4
O1a
C1a
Cr1
C1
C3
C1e
C2
C1b
O1e
O1b
C5
C6
O7
C7
C8


O1b
C1b
C6
C5
O1d
C1d
C1
C2
O7
C7
C8
C3
O1a
C1a
Cr1
C1e
O1
C4
O1e
C1c
O1c

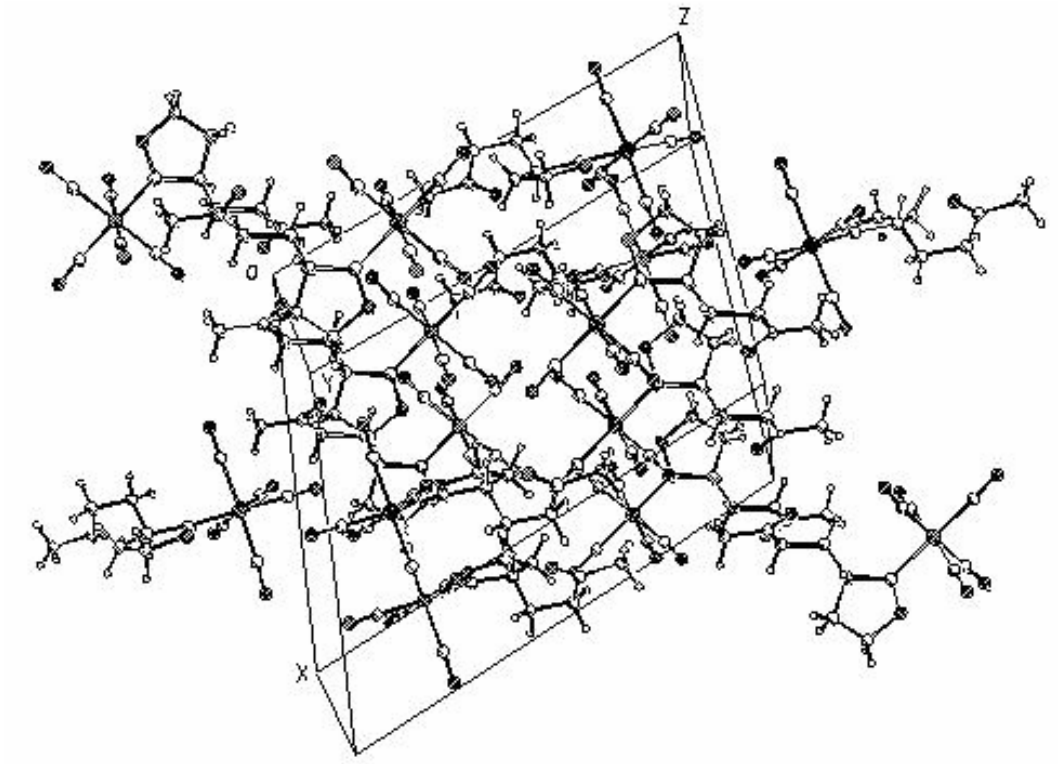



**Table 1: Crystal data and structure refinements for *[55]***

| | | |
|---|---|---|
| Identification code | ***[55]*** | |
| Empirical formula | $C_{13}H_{12}CrO_7$ | |
| Formula weight | 332.23 | |
| Temperature | 123(2) K | |
| Wavelength | 0.71073 Å (MoKα) | |
| Crystal system | Monoclinic | |
| Space group | P2/c (No.13) | |
| Unit cell dimensions | a = 15.1786(4) Å | α = 90° |
| | b = 6.1994(2) Å | β = 114.830(1)° |
| | c = 16.8868(5) Å | γ = 90° |
| Volume | 1442.12(7) $Å^3$ | |
| Z | 4 | |
| Calculated density | 1.530 $mg/m^3$ | |
| Absorption coefficient | 0.822 $mm^{-1}$ | |
| F(000) | 680 | |
| Crystal size | 0.50 x 0.25 x 0.15 mm | |
| Diffractometer | Nonius KappaCCD | |

| | |
|---|---|
| Theta range for data collection | 2.66 to 25.00° |
| Limiting indices | $-18 \leq h \leq 17$, $-7 \leq k \leq 7$, $-20 \leq l \leq 20$ |
| Reflections collected / unique | 12310 / 2545 [R(int) = 0.0528] |
| Completeness to $\Theta = 25.00$ | 99.8 % |
| Absorption correction | Empirical from multiple refl |
| Max. and min. transmission | 0.8356 and 0.6954 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 2545 / 0 / 191 |
| Goodness-of-fit on $F^2$ | 1.105 |
| Final R indices [$I>2\sigma(I)$] | R1 = 0.0433, wR2 = 0.1183 |
| R indices (all data) | R1 = 0.0529, wR2 = 0.1237 |
| Largest diff. peak and hole | 1.070 and -0.331 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[55]*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 3127(1) | 2449(1) | 3208(1) | 21(1) |
| C(1A) | 3148(2) | 1067(5) | 4211(2) | 31(1) |
| O(1A) | 3141(2) | 182(4) | 4805(1) | 41(1) |
| C(1B) | 1918(2) | 3781(5) | 2986(2) | 28(1) |
| O(1B) | 1195(2) | 4595(4) | 2863(2) | 43(1) |
| C(1C) | 4353(2) | 1299(5) | 3387(2) | 32(1) |
| O(1C) | 5095(2) | 675(4) | 3475(2) | 54(1) |
| C(1D) | 3780(2) | 4918(5) | 3869(2) | 29(1) |
| O(1D) | 4177(2) | 6382(4) | 4265(2) | 42(1) |
| C(1E) | 2499(2) | -57(5) | 2578(2) | 29(1) |
| O(1E) | 2129(2) | -1616(4) | 2246(2) | 45(1) |
| O(1) | 3789(2) | 5305(4) | 2213(1) | 43(1) |
| C(1) | 3142(2) | 3844(4) | 2129(2) | 24(1) |
| C(2) | 2552(2) | 3391(6) | 1171(2) | 37(1) |
| C(3) | 2943(3) | 4998(6) | 705(2) | 53(1) |
| C(4) | 3732(3) | 6138(8) | 1372(2) | 64(1) |
| C(5) | 1472(3) | 3472(6) | 882(2) | 48(1) |
| C(6) | 902(3) | 2977(6) | -92(2) | 48(1) |
| C(7) | 886(2) | 656(6) | -366(2) | 39(1) |
| O(7) | 1166(2) | -770(4) | 171(2) | 58(1) |
| C(8) | 468(3) | 274(7) | -1327(2) | 52(1) |

**Tabelle 3: Bond lengths [Å] for *[55]*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1A) | 1.887(3) | O(1)-C(1) | 1.299(3) |
| Cr(1)-C(1C) | 1.895(3) | O(1)-C(4) | 1.479(4) |
| Cr(1)-C(1E) | 1.898(3) | C(1)-C(2) | 1.512(4) |
| Cr(1)-C(1B) | 1.901(3) | C(2)-C(5) | 1.502(5) |
| Cr(1)-C(1D) | 1.909(3) | C(2)-C(3) | 1.534(4) |
| Cr(1)-C(1) | 2.025(3) | C(3)-C(4) | 1.438(5) |
| C(1A)-O(1A) | 1.147(4) | C(5)-C(6) | 1.534(5) |
| C(1B)-O(1B) | 1.145(3) | C(6)-C(7) | 1.509(5) |
| C(1C)-O(1C) | 1.141(4) | C(7)-O(7) | 1.209(4) |
| C(1D)-O(1D) | 1.138(4) | C(7)-C(8) | 1.493(5) |
| C(1E)-O(1E) | 1.140(4) | | |

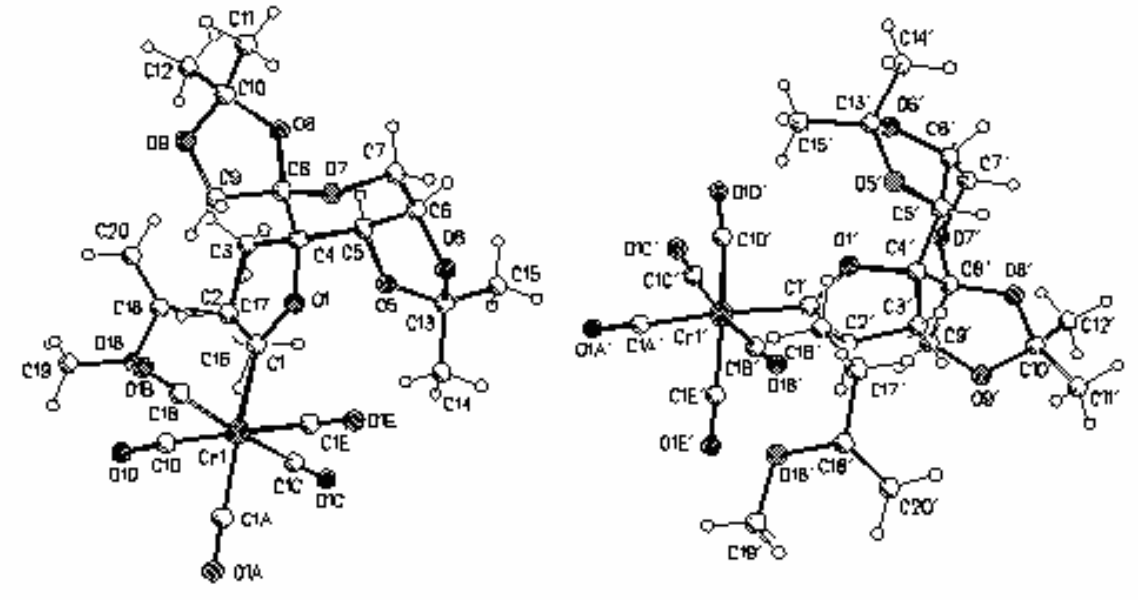
**Tabelle 4: Bond angles [°] for *[55]*.**

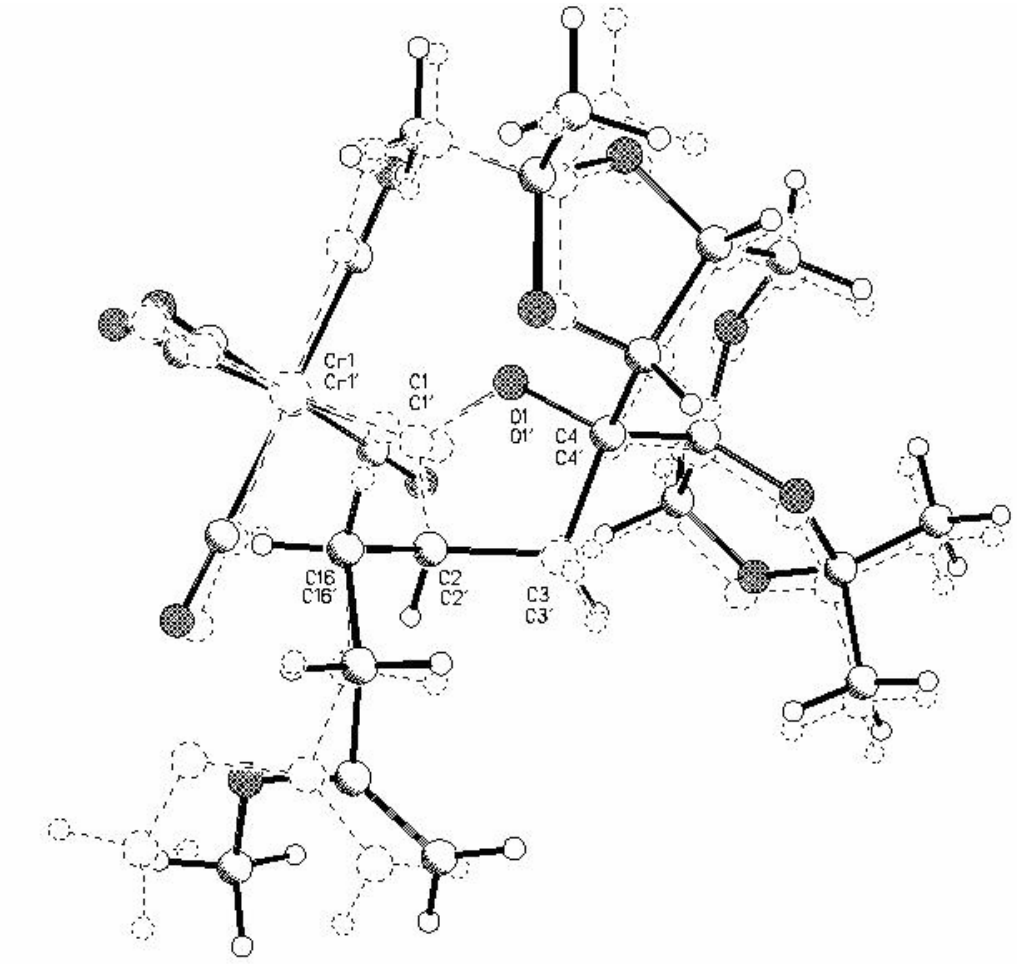
| | | | |
|---|---|---|---|
| C(1A)-Cr(1)-C(1C) | 92.31(13) | O(1C)-C(1C)-Cr(1) | 177.2(3) |
| C(1A)-Cr(1)-C(1E) | 86.95(13) | O(1D)-C(1D)-Cr(1) | 179.4(3) |
| C(1C)-Cr(1)-C(1E) | 90.56(13) | O(1E)-C(1E)-Cr(1) | 175.9(3) |
| C(1A)-Cr(1)-C(1B) | 91.44(12) | C(1)-O(1)-C(4) | 113.7(2) |
| C(1C)-Cr(1)-C(1B) | 175.73(12) | O(1)-C(1)-C(2) | 109.1(2) |
| C(1E)-Cr(1)-C(1B) | 91.69(12) | O(1)-C(1)-Cr(1) | 119.6(2) |
| C(1A)-Cr(1)-C(1D) | 91.71(13) | C(2)-C(1)-Cr(1) | 131.2(2) |
| C(1C)-Cr(1)-C(1D) | 88.53(13) | C(5)-C(2)-C(1) | 114.6(3) |
| C(1E)-Cr(1)-C(1D) | 178.35(12) | C(5)-C(2)-C(3) | 114.6(3) |
| C(1B)-Cr(1)-C(1D) | 89.31(12) | C(1)-C(2)-C(3) | 104.2(3) |
| C(1A)-Cr(1)-C(1) | 177.71(11) | C(4)-C(3)-C(2) | 107.0(3) |
| C(1C)-Cr(1)-C(1) | 85.57(12) | C(3)-C(4)-O(1) | 105.9(3) |
| C(1E)-Cr(1)-C(1) | 92.21(12) | C(2)-C(5)-C(6) | 112.9(3) |
| C(1B)-Cr(1)-C(1) | 90.71(11) | C(7)-C(6)-C(5) | 116.7(3) |
| C(1D)-Cr(1)-C(1) | 89.10(12) | O(7)-C(7)-C(8) | 123.7(4) |
| O(1A)-C(1A)-Cr(1) | 177.9(3) | O(7)-C(7)-C(6) | 120.8(3) |
| O(1B)-C(1B)-Cr(1) | 179.1(3) | C(8)-C(7)-C(6) | 115.5(3) |

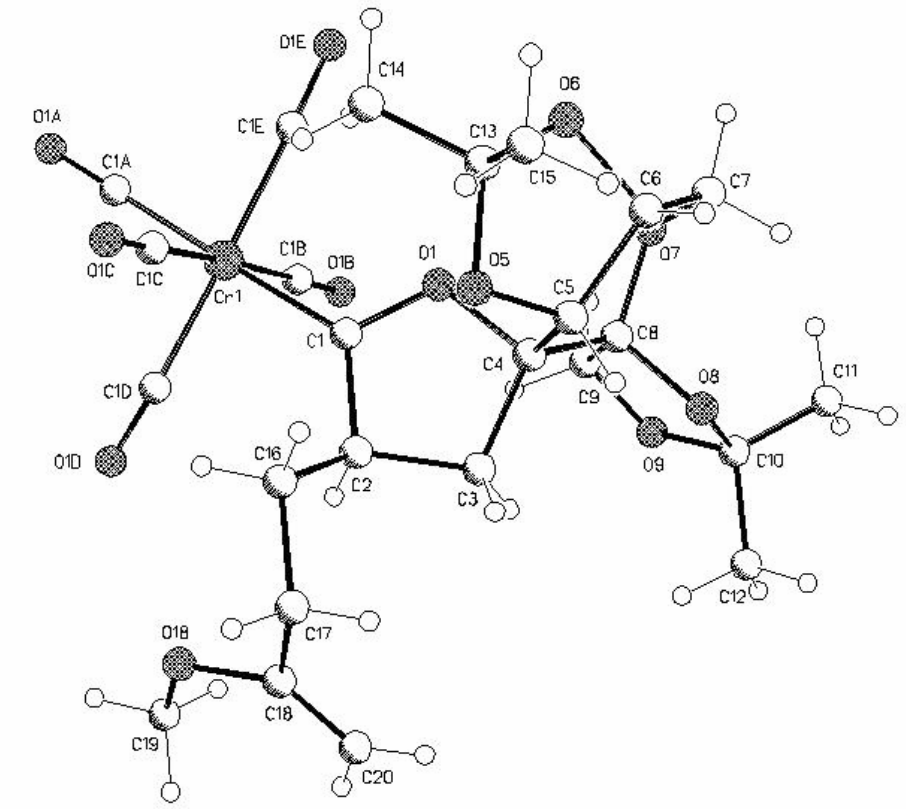
**Tabelle 5: Torsion angles [°] for *[45]a.***

| | | | |
|---|---|---|---|
| C(1C)-Cr(1)-C(1A)-O(1A) | -105(7) | C(4)-O(1)-C(1)-C(2) | 2.4(4) |
| C(1E)-Cr(1)-C(1A)-O(1A) | -14(7) | C(4)-O(1)-C(1)-Cr(1) | 178.4(3) |
| C(1B)-Cr(1)-C(1A)-O(1A) | 77(7) | C(1A)-Cr(1)-C(1)-O(1) | -87(3) |
| C(1D)-Cr(1)-C(1A)-O(1A) | 167(7) | C(1C)-Cr(1)-C(1)-O(1) | -65.2(2) |
| C(1)-Cr(1)-C(1A)-O(1A) | -83(8) | C(1E)-Cr(1)-C(1)-O(1) | -155.6(2) |
| C(1A)-Cr(1)-C(1B)-O(1B) | 55(17) | C(1B)-Cr(1)-C(1)-O(1) | 112.7(2) |
| C(1C)-Cr(1)-C(1B)-O(1B) | -97(18) | C(1D)-Cr(1)-C(1)-O(1) | 23.3(2) |
| C(1E)-Cr(1)-C(1B)-O(1B) | 142(17) | C(1A)-Cr(1)-C(1)-C(2) | 88(3) |
| C(1D)-Cr(1)-C(1B)-O(1B) | -37(17) | C(1C)-Cr(1)-C(1)-C(2) | 109.7(3) |
| C(1)-Cr(1)-C(1B)-O(1B) | -126(17) | C(1E)-Cr(1)-C(1)-C(2) | 19.3(3) |
| C(1A)-Cr(1)-C(1C)-O(1C) | -157(6) | C(1B)-Cr(1)-C(1)-C(2) | -72.4(3) |
| C(1E)-Cr(1)-C(1C)-O(1C) | 116(6) | C(1D)-Cr(1)-C(1)-C(2) | -161.7(3) |
| C(1B)-Cr(1)-C(1C)-O(1C) | -5(7) | O(1)-C(1)-C(2)-C(5) | -129.6(3) |
| C(1D)-Cr(1)-C(1C)-O(1C) | -65(6) | Cr(1)-C(1)-C(2)-C(5) | 55.1(4) |
| C(1)-Cr(1)-C(1C)-O(1C) | 24(6) | O(1)-C(1)-C(2)-C(3) | -3.5(4) |
| C(1A)-Cr(1)-C(1D)-O(1D) | 88(25) | Cr(1)-C(1)-C(2)-C(3) | -178.9(2) |
| C(1C)-Cr(1)-C(1D)-O(1D) | -4(25) | C(5)-C(2)-C(3)-C(4) | 129.4(4) |
| C(1E)-Cr(1)-C(1D)-O(1D) | 52(27) | C(1)-C(2)-C(3)-C(4) | 3.4(4) |
| C(1B)-Cr(1)-C(1D)-O(1D) | 179(100) | C(2)-C(3)-C(4)-O(1) | -2.1(5) |
| C(1)-Cr(1)-C(1D)-O(1D) | -90(25) | C(1)-O(1)-C(4)-C(3) | -0.1(5) |
| C(1A)-Cr(1)-C(1E)-O(1E) | -4(4) | C(1)-C(2)-C(5)-C(6) | -179.3(3) |
| C(1C)-Cr(1)-C(1E)-O(1E) | 88(4) | C(3)-C(2)-C(5)-C(6) | 60.3(4) |
| C(1B)-Cr(1)-C(1E)-O(1E) | -95(4) | C(2)-C(5)-C(6)-C(7) | 73.6(4) |
| C(1D)-Cr(1)-C(1E)-O(1E) | 32(7) | C(5)-C(6)-C(7)-O(7) | 12.1(5) |
| C(1)-Cr(1)-C(1E)-O(1E) | 174(4) | C(5)-C(6)-C(7)-C(8) | -170.3(3) |

**18.** **(*3R,5R,1´R,4´S,6´R*)-Pentacarbonyl{5-(3´´´-methoxybut-3-enyl)-8´,8´,2´´,2´´-tetramethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,3-2-oxacyclopent]-1-yliden}chrom(0)** ***[56]a***




O1E
C14
O6
O1A
C1E
C13
C15
C6
C7
C1A
O1C
C1C
C1B
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O1
O5
C5
O7
Cr1
C1
C8
C4
C11
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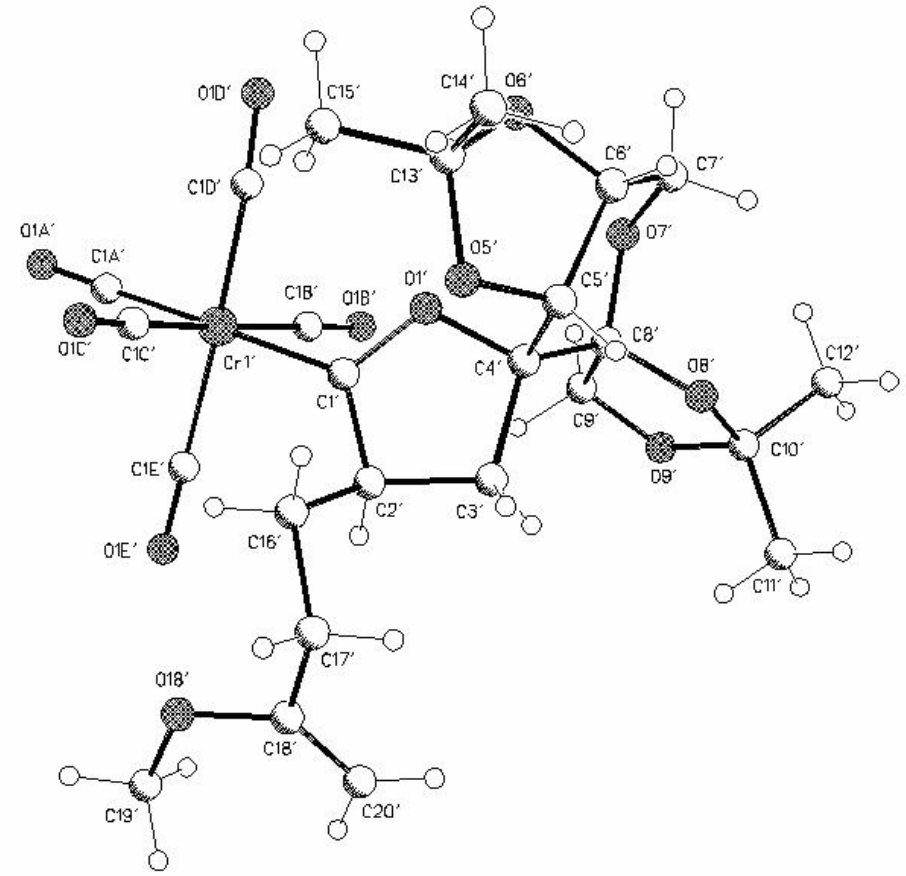
C16
C2
C3
O9
C10
C12
O18
C17
C18
C19
C20


O1D'
C15'
C14'
O6'
C6'
C7'
C13'
C1D'
O1A'
C1A'
O5'
O7'
O1'
C5'
C1B'
O1B'
O1C'
C1C'
Cr1'
C8'
C12'
C4'
O8'
C1'
C9'
C10'
C1E'
O9'
C2'
C3'
O1E'
C16'
C11'
C17'
O18'
C18'
C19'
C20'

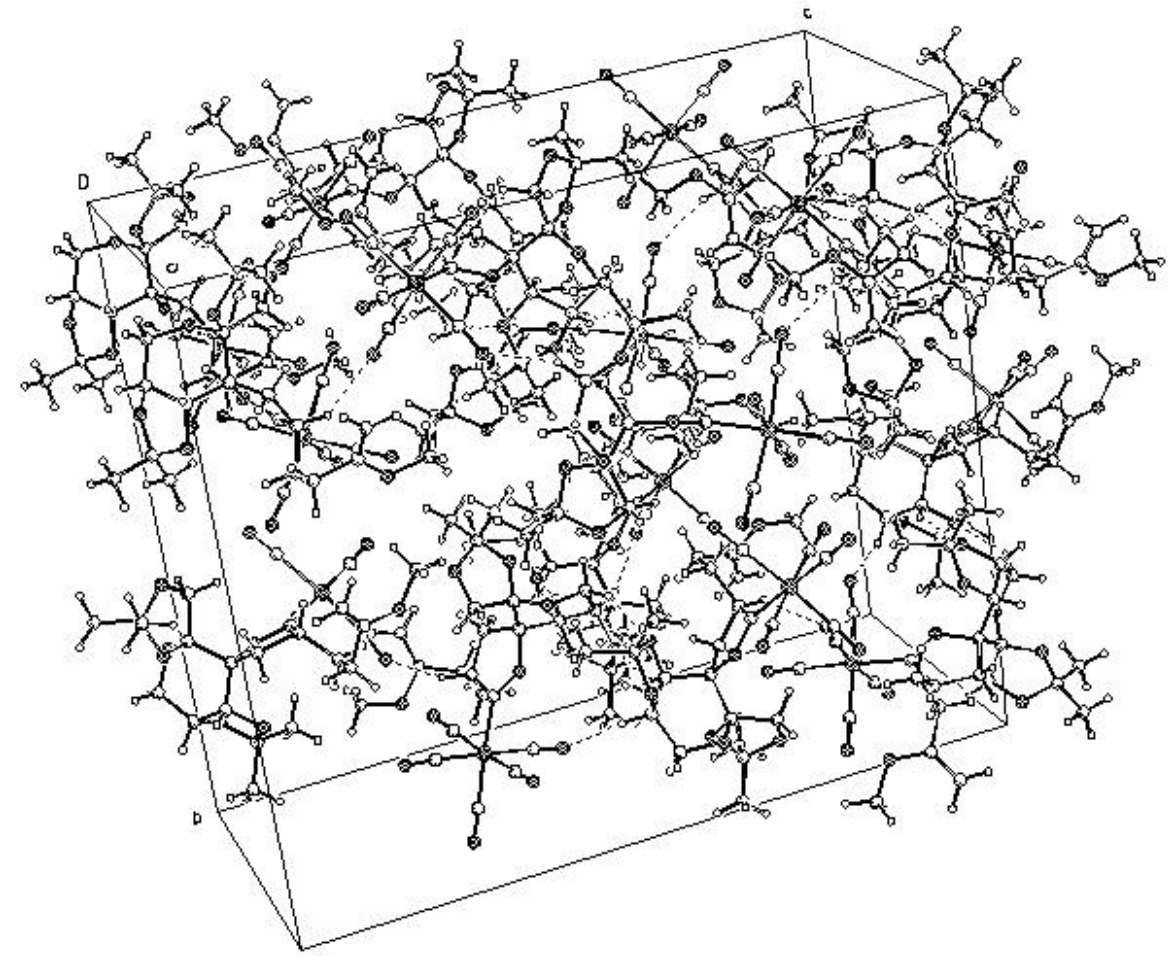

c
D
b

**Table 1: Crystal data and structure refinements for *[56]a***

| | |
|---|---|
| Identification code | ***[56]a*** |
| Empirical formula | $C_{25}H_{30}CrO_{12}$ |
| Formula weight | 574.49 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 10.3549(1) Å α = 90° |
| | b = 22.0427(3) Å β = 90° |
| | c = 24.9082(3) Å γ = 90° |
| Volume | 5685.30(12) $Å^3$ |
| Z | 8 |
| Calculated density | 1.342 $mg/m^3$ |
| Absorption coefficient | 0.461 $mm^{-1}$ |
| F(000) | 2400 |
| Crystal size | 0.50 x 0.30 x 0.10 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 3.07 to 25.03° |
| Limiting indices | $-12 \le h \le 12$, $-26 \le k \le 26$, $-29 \le l \le 29$ |
| Reflections collected / unique | 71492 / 10036 [R(int) = 0.0632] |
| Completeness to Θ = 25.03 | 99.7 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 10036 / 0 / 685 |
| Goodness-of-fit on $F^2$ | 1.047 |
| Final R indices [I>2σ(I)] | R1 = 0.0423, wR2 = 0.0958 |
| R indices (all data) | R1 = 0.0616, wR2 = 0.1024 |
| Absolute structure parameter | -0.018(16) |
| Largest diff. peak and hole | 0.938 and -0.860 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[56]a*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 6616(1) | 8699(1) | 3142(1) | 31(1) |
| C(1A) | 7761(4) | 9245(2) | 2802(1) | 39(1) |
| O(1A) | 8450(3) | 9582(1) | 2598(1) | 61(1) |
| C(1B) | 7193(3) | 8035(2) | 2716(2) | 42(1) |
| O(1B) | 7508(3) | 7630(2) | 2463(1) | 70(1) |
| C(1C) | 5932(3) | 9316(2) | 3592(1) | 32(1) |
| O(1C) | 5509(2) | 9682(1) | 3866(1) | 41(1) |
| C(1D) | 5377(3) | 8868(2) | 2605(2) | 38(1) |
| O(1D) | 4682(3) | 8980(1) | 2254(1) | 51(1) |
| C(1E) | 7941(3) | 8531(2) | 3650(2) | 37(1) |
| O(1E) | 8760(2) | 8447(1) | 3945(1) | 54(1) |
| O(1) | 5873(2) | 7734(1) | 3881(1) | 28(1) |
| C(1) | 5448(3) | 8117(2) | 3522(1) | 28(1) |
| C(2) | 3999(3) | 8011(2) | 3442(1) | 29(1) |
| C(3) | 3722(3) | 7414(2) | 3740(1) | 30(1) |
| C(4) | 4906(3) | 7292(2) | 4089(1) | 28(1) |
| C(5) | 4666(3) | 7397(2) | 4684(1) | 29(1) |
| O(5) | 4599(2) | 8031(1) | 4796(1) | 32(1) |
| C(6) | 5700(3) | 7161(2) | 5067(1) | 31(1) |
| O(6) | 6495(2) | 7682(1) | 5146(1) | 32(1) |
| C(7) | 6467(3) | 6626(2) | 4870(1) | 38(1) |
| O(7) | 6694(2) | 6623(1) | 4300(1) | 35(1) |
| C(8) | 5552(3) | 6670(2) | 3996(1) | 33(1) |
| O(8) | 4614(2) | 6231(1) | 4140(1) | 33(1) |
| C(9) | 5917(3) | 6514(2) | 3417(2) | 42(1) |
| O(9) | 5268(5) | 6006(2) | 3313(2) | 197(3) |
| C(10) | 4653(4) | 5750(2) | 3753(2) | 51(1) |
| C(11) | 5366(9) | 5233(3) | 3954(4) | 207(6) |
| C(12) | 3332(4) | 5595(2) | 3591(2) | 58(1) |
| C(13) | 5637(3) | 8188(2) | 5164(1) | 33(1) |
| C(14) | 6346(3) | 8737(2) | 4962(1) | 41(1) |
| C(15) | 5071(4) | 8273(2) | 5721(1) | 47(1) |
| C(16) | 3199(3) | 8552(2) | 3635(1) | 34(1) |
| C(17) | 1737(3) | 8442(2) | 3571(1) | 36(1) |
| C(18) | 1377(3) | 8244(2) | 3015(2) | 37(1) |
| O(18) | 1744(2) | 8679(1) | 2656(1) | 47(1) |
| C(19) | 1595(5) | 8527(2) | 2106(2) | 65(1) |
| C(20) | 810(3) | 7728(2) | 2900(2) | 49(1) |

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1') | 7874(1) | 8750(1) | 8245(1) | 32(1) |
| C(1A') | 6687(4) | 8877(2) | 7673(2) | 40(1) |
| O(1A') | 5963(3) | 8938(1) | 7331(1) | 55(1) |
| C(1B') | 7101(4) | 9380(2) | 8667(2) | 41(1) |
| O(1B') | 6667(2) | 9744(1) | 8931(1) | 55(1) |
| C(1C') | 8691(3) | 8093(2) | 7893(1) | 36(1) |
| O(1C') | 9191(3) | 7685(1) | 7696(1) | 50(1) |
| C(1D') | 6694(4) | 8201(2) | 8549(1) | 39(1) |
| O(1D') | 5905(3) | 7868(1) | 8698(1) | 57(1) |
| C(1E') | 9017(3) | 9317(2) | 7911(1) | 33(1) |
| O(1E') | 9660(2) | 9671(1) | 7707(1) | 43(1) |
| O(1') | 8774(2) | 8440(1) | 9326(1) | 30(1) |
| C(1') | 9151(3) | 8651(2) | 8851(1) | 28(1) |
| C(2') | 10564(3) | 8817(2) | 8880(1) | 28(1) |
| C(3') | 10942(3) | 8770(2) | 9476(1) | 33(1) |
| C(4') | 9783(3) | 8479(2) | 9753(1) | 30(1) |
| C(5') | 10072(3) | 7836(2) | 9966(1) | 33(1) |
| O(5') | 10188(2) | 7418(1) | 9534(1) | 36(1) |
| C(6') | 9021(3) | 7563(2) | 10323(1) | 36(1) |
| O(6') | 8264(2) | 7235(1) | 9940(1) | 34(1) |
| C(7') | 8268(3) | 8007(2) | 10648(1) | 41(1) |
| O(7') | 8031(2) | 8575(1) | 10384(1) | 41(1) |
| C(8') | 9160(3) | 8857(2) | 10193(1) | 34(1) |
| O(8') | 10105(2) | 8942(1) | 10595(1) | 36(1) |
| C(9') | 8759(4) | 9502(2) | 10038(2) | 48(1) |
| O(9') | 9365(7) | 9870(2) | 10391(2) | 179(3) |
| C(10') | 10032(5) | 9561(2) | 10796(2) | 64(1) |
| C(11') | 11393(6) | 9800(2) | 10855(2) | 110(2) |
| C(12') | 9311(6) | 9582(3) | 11305(2) | 97(2) |
| C(13') | 9175(3) | 6974(2) | 9578(2) | 39(1) |
| C(14') | 9728(4) | 6384(2) | 9797(2) | 56(1) |
| C(15') | 8529(4) | 6891(2) | 9042(1) | 46(1) |
| C(16') | 11368(3) | 8404(2) | 8508(1) | 32(1) |
| C(17') | 12818(3) | 8567(2) | 8507(2) | 39(1) |
| C(18') | 13061(3) | 9188(2) | 8276(2) | 39(1) |
| O(18') | 12716(2) | 9195(1) | 7747(1) | 49(1) |
| C(19') | 12881(4) | 9756(2) | 7469(2) | 68(1) |
| C(20') | 13546(4) | 9645(2) | 8544(2) | 66(1) |

**Tabelle 3: Bond lengths [Å] for *[56]a*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1A) | 1.890(4) | O(5)-C(13) | 1.455(4) |
| Cr(1)-C(1D) | 1.891(4) | C(6)-O(6) | 1.427(4) |
| Cr(1)-C(1C) | 1.898(4) | C(6)-C(7) | 1.504(5) |
| Cr(1)-C(1E) | 1.901(4) | O(6)-C(13) | 1.426(4) |
| Cr(1)-C(1B) | 1.905(4) | C(7)-O(7) | 1.438(4) |
| Cr(1)-C(1) | 2.001(3) | O(7)-C(8) | 1.408(4) |
| C(1A)-O(1A) | 1.147(4) | C(8)-O(8) | 1.417(4) |
| C(1B)-O(1B) | 1.141(4) | C(8)-C(9) | 1.529(5) |
| C(1C)-O(1C) | 1.144(4) | O(8)-C(10) | 1.433(4) |
| C(1D)-O(1D) | 1.159(4) | C(9)-O(9) | 1.331(5) |
| C(1E)-O(1E) | 1.138(4) | O(9)-C(10) | 1.389(6) |
| O(1)-C(1) | 1.307(4) | C(10)-C(11) | 1.445(7) |
| O(1)-C(4) | 1.490(4) | C(10)-C(12) | 1.466(5) |
| C(1)-C(2) | 1.531(4) | C(13)-C(14) | 1.502(5) |
| C(2)-C(16) | 1.531(4) | C(13)-C(15) | 1.516(5) |
| C(2)-C(3) | 1.537(5) | C(16)-C(17) | 1.542(5) |
| C(3)-C(4) | 1.527(4) | C(17)-C(18) | 1.500(5) |
| C(4)-C(5) | 1.521(5) | C(18)-C(20) | 1.312(5) |
| C(4)-C(8) | 1.545(5) | C(18)-O(18) | 1.364(4) |
| C(5)-O(5) | 1.427(4) | O(18)-C(19) | 1.418(4) |
| C(5)-C(6) | 1.525(5) | | |
| Cr(1')-C(1D') | 1.879(4) | O(5')-C(13') | 1.438(4) |
| Cr(1')-C(1C') | 1.892(4) | C(6')-O(6') | 1.432(4) |
| Cr(1')-C(1A') | 1.902(4) | C(6')-C(7') | 1.491(5) |
| Cr(1')-C(1E') | 1.912(4) | O(6')-C(13') | 1.426(4) |
| Cr(1')-C(1B') | 1.916(4) | C(7')-O(7') | 1.435(4) |
| Cr(1')-C(1') | 2.019(3) | O(7')-C(8') | 1.407(4) |
| C(1A')-O(1A') | 1.144(4) | C(8')-O(8') | 1.413(4) |
| C(1B')-O(1B') | 1.131(4) | C(8')-C(9') | 1.532(5) |
| C(1C')-O(1C') | 1.149(4) | O(8')-C(10') | 1.457(5) |
| C(1D')-O(1D') | 1.159(4) | C(9')-O(9') | 1.350(5) |
| C(1E')-O(1E') | 1.144(4) | O(9')-C(10') | 1.399(5) |
| O(1')-C(1') | 1.328(4) | C(10')-C(12') | 1.473(6) |
| O(1')-C(4') | 1.495(4) | C(10')-C(11') | 1.511(7) |
| C(1')-C(2') | 1.509(4) | C(13')-C(15') | 1.505(5) |
| C(2')-C(3') | 1.540(4) | C(13')-C(14') | 1.522(5) |
| C(2')-C(16') | 1.543(4) | C(16')-C(17') | 1.544(5) |
| C(3')-C(4') | 1.525(4) | C(17')-C(18') | 1.506(5) |
| C(4')-C(8') | 1.519(5) | C(18')-C(20') | 1.308(5) |
| C(4')-C(5') | 1.543(5) | C(18')-O(18') | 1.365(4) |
| C(5')-O(5') | 1.421(4) | O(18')-C(19') | 1.428(5) |
| C(5')-C(6') | 1.528(5) | | |

**Tabelle 4: Bond angles [°] for *[56]a*.**

| | | | |
|---|---|---|---|
| C(1A)-Cr(1)-C(1D) | 89.05(15) | C(4)-C(5)-C(6) | 116.3(3) |
| C(1A)-Cr(1)-C(1C) | 92.42(15) | C(5)-O(5)-C(13) | 108.6(2) |
| C(1D)-Cr(1)-C(1C) | 91.34(15) | O(6)-C(6)-C(7) | 111.8(3) |
| C(1A)-Cr(1)-C(1E) | 88.26(15) | O(6)-C(6)-C(5) | 102.6(3) |
| C(1D)-Cr(1)-C(1E) | 176.51(15) | C(7)-C(6)-C(5) | 115.7(3) |
| C(1C)-Cr(1)-C(1E) | 90.99(15) | C(13)-O(6)-C(6) | 105.9(2) |
| C(1A)-Cr(1)-C(1B) | 92.44(16) | O(7)-C(7)-C(6) | 114.3(3) |
| C(1D)-Cr(1)-C(1B) | 88.19(16) | C(8)-O(7)-C(7) | 113.2(2) |
| C(1C)-Cr(1)-C(1B) | 175.11(15) | O(7)-C(8)-O(8) | 112.9(3) |
| C(1E)-Cr(1)-C(1B) | 89.70(16) | O(7)-C(8)-C(9) | 106.5(3) |
| C(1A)-Cr(1)-C(1) | 178.09(15) | O(8)-C(8)-C(9) | 104.8(3) |
| C(1D)-Cr(1)-C(1) | 92.86(14) | O(7)-C(8)-C(4) | 110.3(3) |
| C(1C)-Cr(1)-C(1) | 87.46(13) | O(8)-C(8)-C(4) | 105.8(2) |
| C(1E)-Cr(1)-C(1) | 89.83(14) | C(9)-C(8)-C(4) | 116.6(3) |
| C(1B)-Cr(1)-C(1) | 87.70(15) | C(8)-O(8)-C(10) | 108.4(3) |
| O(1A)-C(1A)-Cr(1) | 179.3(4) | O(9)-C(9)-C(8) | 104.4(3) |
| O(1B)-C(1B)-Cr(1) | 178.2(3) | C(9)-O(9)-C(10) | 114.8(4) |
| O(1C)-C(1C)-Cr(1) | 179.0(3) | O(9)-C(10)-O(8) | 104.0(3) |
| O(1D)-C(1D)-Cr(1) | 175.6(3) | O(9)-C(10)-C(11) | 111.1(6) |
| O(1E)-C(1E)-Cr(1) | 177.4(3) | O(8)-C(10)-C(11) | 111.4(4) |
| C(1)-O(1)-C(4) | 115.7(2) | O(9)-C(10)-C(12) | 107.7(4) |
| O(1)-C(1)-C(2) | 108.7(3) | O(8)-C(10)-C(12) | 109.3(3) |
| O(1)-C(1)-Cr(1) | 122.3(2) | C(11)-C(10)-C(12) | 112.9(5) |
| C(2)-C(1)-Cr(1) | 129.0(2) | O(6)-C(13)-O(5) | 104.7(3) |
| C(1)-C(2)-C(16) | 111.8(3) | O(6)-C(13)-C(14) | 108.3(3) |
| C(1)-C(2)-C(3) | 104.6(3) | O(5)-C(13)-C(14) | 109.9(3) |
| C(16)-C(2)-C(3) | 114.5(3) | O(6)-C(13)-C(15) | 111.5(3) |
| C(4)-C(3)-C(2) | 105.9(3) | O(5)-C(13)-C(15) | 108.8(3) |
| O(1)-C(4)-C(5) | 110.4(3) | C(14)-C(13)-C(15) | 113.3(3) |
| O(1)-C(4)-C(3) | 103.1(2) | C(2)-C(16)-C(17) | 112.1(3) |
| C(5)-C(4)-C(3) | 113.4(3) | C(18)-C(17)-C(16) | 112.7(3) |
| O(1)-C(4)-C(8) | 103.7(2) | C(20)-C(18)-O(18) | 126.3(4) |
| C(5)-C(4)-C(8) | 110.6(3) | C(20)-C(18)-C(17) | 124.3(4) |
| C(3)-C(4)-C(8) | 114.7(3) | O(18)-C(18)-C(17) | 109.4(3) |
| O(5)-C(5)-C(4) | 110.2(3) | C(18)-O(18)-C(19) | 115.9(3) |
| O(5)-C(5)-C(6) | 104.3(3) | | |
| C(1D')-Cr(1')-C(1C') | 89.08(15) | C(6')-C(5')-C(4') | 115.0(3) |
| C(1D')-Cr(1')-C(1A') | 88.62(16) | C(5')-O(5')-C(13') | 108.7(2) |
| C(1C')-Cr(1')-C(1A') | 93.10(15) | O(6')-C(6')-C(7') | 114.0(3) |
| C(1D')-Cr(1')-C(1E') | 177.33(15) | O(6')-C(6')-C(5') | 101.6(3) |
| C(1C')-Cr(1')-C(1E') | 91.21(14) | C(7')-C(6')-C(5') | 115.5(3) |
| C(1A')-Cr(1')-C(1E') | 88.71(15) | C(13')-O(6')-C(6') | 105.2(3) |
| C(1D')-Cr(1')-C(1B') | 88.55(16) | O(7')-C(7')-C(6') | 114.4(3) |
| C(1C')-Cr(1')-C(1B') | 174.35(16) | C(8')-O(7')-C(7') | 113.4(3) |
| C(1A')-Cr(1')-C(1B') | 91.97(15) | O(7')-C(8')-O(8') | 113.3(2) |
| C(1E')-Cr(1')-C(1B') | 91.39(16) | O(7')-C(8')-C(4') | 110.8(3) |

| | | | |
|---|---|---|---|
| C(1D')-Cr(1')-C(1') | 93.17(14) | O(8')-C(8')-C(4') | 106.8(3) |
| C(1C')-Cr(1')-C(1') | 88.32(14) | O(7')-C(8')-C(9') | 105.6(3) |
| C(1A')-Cr(1')-C(1') | 177.73(16) | O(8')-C(8')-C(9') | 104.1(3) |
| C(1E')-Cr(1')-C(1') | 89.49(14) | C(4')-C(8')-C(9') | 116.3(3) |
| C(1B')-Cr(1')-C(1') | 86.69(14) | C(8')-O(8')-C(10') | 109.4(3) |
| O(1A')-C(1A')-Cr(1') | 178.2(3) | O(9')-C(9')-C(8') | 105.5(3) |
| O(1B')-C(1B')-Cr(1') | 177.5(3) | C(9')-O(9')-C(10') | 114.0(3) |
| O(1C')-C(1C')-Cr(1') | 177.7(3) | O(9')-C(10')-O(8') | 103.5(3) |
| O(1D')-C(1D')-Cr(1') | 174.3(3) | O(9')-C(10')-C(12') | 110.9(5) |
| O(1E')-C(1E')-Cr(1') | 177.3(3) | O(8')-C(10')-C(12') | 110.6(4) |
| C(1')-O(1')-C(4') | 114.1(2) | O(9')-C(10')-C(11') | 111.2(5) |
| O(1')-C(1')-C(2') | 109.1(3) | O(8')-C(10')-C(11') | 108.1(4) |
| O(1')-C(1')-Cr(1') | 120.7(2) | C(12')-C(10')-C(11') | 112.2(4) |
| C(2')-C(1')-Cr(1') | 130.1(2) | O(6')-C(13')-O(5') | 104.9(3) |
| C(1')-C(2')-C(3') | 106.0(2) | O(6')-C(13')-C(15') | 108.4(3) |
| C(1')-C(2')-C(16') | 110.6(3) | O(5')-C(13')-C(15') | 109.8(3) |
| C(3')-C(2')-C(16') | 113.7(3) | O(6')-C(13')-C(14') | 111.6(3) |
| C(4')-C(3')-C(2') | 105.4(3) | O(5')-C(13')-C(14') | 109.5(3) |
| O(1')-C(4')-C(8') | 104.4(2) | C(15')-C(13')-C(14') | 112.4(3) |
| O(1')-C(4')-C(3') | 104.6(2) | C(2')-C(16')-C(17') | 112.9(3) |
| C(8')-C(4')-C(3') | 115.5(3) | C(18')-C(17')-C(16') | 112.1(3) |
| O(1')-C(4')-C(5') | 109.1(3) | C(20')-C(18')-O(18') | 125.7(4) |
| C(8')-C(4')-C(5') | 109.8(3) | C(20')-C(18')-C(17') | 124.7(4) |
| C(3')-C(4')-C(5') | 112.9(3) | O(18')-C(18')-C(17') | 109.6(3) |
| O(5')-C(5')-C(6') | 104.3(3) | C(18')-O(18')-C(19') | 116.5(3) |
| O(5')-C(5')-C(4') | 110.6(3) | | |

**Tabelle 5: Torsion angles [°] for *[56]a.***

| | | | |
|---|---|---|---|
| C(1D)-Cr(1)-C(1A)-O(1A) | 79(30) | C(1C)-Cr(1)-C(1D)-O(1D) | 119(4) |
| C(1C)-Cr(1)-C(1A)-O(1A) | -12(30) | C(1E)-Cr(1)-C(1D)-O(1D) | -13(6) |
| C(1E)-Cr(1)-C(1A)-O(1A) | -103(30) | C(1B)-Cr(1)-C(1D)-O(1D) | -66(4) |
| C(1B)-Cr(1)-C(1A)-O(1A) | 167(100) | C(1)-Cr(1)-C(1D)-O(1D) | -154(4) |
| C(1)-Cr(1)-C(1A)-O(1A) | -99(30) | C(1A)-Cr(1)-C(1E)-O(1E) | 7(8) |
| C(1A)-Cr(1)-C(1B)-O(1B) | -159(12) | C(1D)-Cr(1)-C(1E)-O(1E) | 46(9) |
| C(1D)-Cr(1)-C(1B)-O(1B) | -70(12) | C(1C)-Cr(1)-C(1E)-O(1E) | -86(8) |
| C(1C)-Cr(1)-C(1B)-O(1B) | 15(14) | C(1B)-Cr(1)-C(1E)-O(1E) | 99(8) |
| C(1E)-Cr(1)-C(1B)-O(1B) | 113(12) | C(1)-Cr(1)-C(1E)-O(1E) | -173(100) |
| C(1)-Cr(1)-C(1B)-O(1B) | 23(12) | C(4)-O(1)-C(1)-C(2) | 3.1(4) |
| C(1A)-Cr(1)-C(1C)-O(1C) | -176(100) | C(4)-O(1)-C(1)-Cr(1) | -174.5(2) |
| C(1D)-Cr(1)-C(1C)-O(1C) | 95(19) | C(1A)-Cr(1)-C(1)-O(1) | -18(5) |
| C(1E)-Cr(1)-C(1C)-O(1C) | -88(19) | C(1D)-Cr(1)-C(1)-O(1) | 164.6(3) |
| C(1B)-Cr(1)-C(1C)-O(1C) | 10(21) | C(1C)-Cr(1)-C(1)-O(1) | -104.2(3) |
| C(1)-Cr(1)-C(1C)-O(1C) | 2(19) | C(1E)-Cr(1)-C(1)-O(1) | -13.2(3) |
| C(1A)-Cr(1)-C(1D)-O(1D) | 26(4) | C(1B)-Cr(1)-C(1)-O(1) | 76.5(3) |

| | | | |
|---|---|---|---|
| C(1A)-Cr(1)-C(1)-C(2) | 165(4) | C(7)-O(7)-C(8)-C(4) | 65.3(3) |
| C(1D)-Cr(1)-C(1)-C(2) | -12.5(3) | O(1)-C(4)-C(8)-O(7) | 63.1(3) |
| C(1C)-Cr(1)-C(1)-C(2) | 78.7(3) | C(5)-C(4)-C(8)-O(7) | -55.3(3) |
| C(1E)-Cr(1)-C(1)-C(2) | 169.7(3) | C(3)-C(4)-C(8)-O(7) | 174.8(3) |
| C(1B)-Cr(1)-C(1)-C(2) | -100.6(3) | O(1)-C(4)-C(8)-O(8) | -174.5(2) |
| O(1)-C(1)-C(2)-C(16) | 113.6(3) | C(5)-C(4)-C(8)-O(8) | 67.1(3) |
| Cr(1)-C(1)-C(2)-C(16) | -69.1(4) | C(3)-C(4)-C(8)-O(8) | -62.8(3) |
| O(1)-C(1)-C(2)-C(3) | -10.9(3) | O(1)-C(4)-C(8)-C(9) | -58.5(3) |
| Cr(1)-C(1)-C(2)-C(3) | 166.5(2) | C(5)-C(4)-C(8)-C(9) | -176.9(3) |
| C(1)-C(2)-C(3)-C(4) | 14.1(3) | C(3)-C(4)-C(8)-C(9) | 53.2(4) |
| C(16)-C(2)-C(3)-C(4) | -108.6(3) | O(7)-C(8)-O(8)-C(10) | -100.7(3) |
| C(1)-O(1)-C(4)-C(5) | -115.6(3) | C(9)-C(8)-O(8)-C(10) | 14.8(3) |
| C(1)-O(1)-C(4)-C(3) | 6.0(3) | C(4)-C(8)-O(8)-C(10) | 138.6(3) |
| C(1)-O(1)-C(4)-C(8) | 125.9(3) | O(7)-C(8)-C(9)-O(9) | 115.4(4) |
| C(2)-C(3)-C(4)-O(1) | -12.2(3) | O(8)-C(8)-C(9)-O(9) | -4.5(4) |
| C(2)-C(3)-C(4)-C(5) | 107.2(3) | C(4)-C(8)-C(9)-O(9) | -121.0(4) |
| C(2)-C(3)-C(4)-C(8) | -124.3(3) | C(8)-C(9)-O(9)-C(10) | -8.1(6) |
| O(1)-C(4)-C(5)-O(5) | 41.2(3) | C(9)-O(9)-C(10)-O(8) | 17.3(6) |
| C(3)-C(4)-C(5)-O(5) | -74.0(3) | C(9)-O(9)-C(10)-C(11) | -102.7(6) |
| C(8)-C(4)-C(5)-O(5) | 155.5(2) | C(9)-O(9)-C(10)-C(12) | 133.2(5) |
| O(1)-C(4)-C(5)-C(6) | -77.1(3) | C(8)-O(8)-C(10)-O(9) | -19.3(5) |
| C(3)-C(4)-C(5)-C(6) | 167.7(3) | C(8)-O(8)-C(10)-C(11) | 100.4(6) |
| C(8)-C(4)-C(5)-C(6) | 37.1(4) | C(8)-O(8)-C(10)-C(12) | -134.1(3) |
| C(4)-C(5)-O(5)-C(13) | -118.2(3) | C(6)-O(6)-C(13)-O(5) | -33.6(3) |
| C(6)-C(5)-O(5)-C(13) | 7.3(3) | C(6)-O(6)-C(13)-C(14) | -150.9(3) |
| O(5)-C(5)-C(6)-O(6) | -27.2(3) | C(6)-O(6)-C(13)-C(15) | 83.8(3) |
| C(4)-C(5)-C(6)-O(6) | 94.4(3) | C(5)-O(5)-C(13)-O(6) | 15.4(3) |
| O(5)-C(5)-C(6)-C(7) | -149.3(3) | C(5)-O(5)-C(13)-C(14) | 131.6(3) |
| C(4)-C(5)-C(6)-C(7) | -27.7(4) | C(5)-O(5)-C(13)-C(15) | -103.9(3) |
| C(7)-C(6)-O(6)-C(13) | 162.3(3) | C(1)-C(2)-C(16)-C(17) | -178.5(3) |
| C(5)-C(6)-O(6)-C(13) | 37.6(3) | C(3)-C(2)-C(16)-C(17) | -59.8(4) |
| O(6)-C(6)-C(7)-O(7) | -82.8(3) | C(2)-C(16)-C(17)-C(18) | -51.9(4) |
| C(5)-C(6)-C(7)-O(7) | 34.2(4) | C(16)-C(17)-C(18)-C(20) | 120.0(4) |
| C(6)-C(7)-O(7)-C(8) | -54.5(4) | C(16)-C(17)-C(18)-O(18) | -59.3(4) |
| C(7)-O(7)-C(8)-O(8) | -52.8(4) | C(20)-C(18)-O(18)-C(19) | -5.9(5) |
| C(7)-O(7)-C(8)-C(9) | -167.2(3) | C(17)-C(18)-O(18)-C(19) | 173.4(3) |
| C(1D')-Cr(1')-C(1A')-O(1A') | -31(11) | C(1B')-Cr(1')-C(1C')-O(1C') | 12(9) |
| C(1C')-Cr(1')-C(1A')-O(1A') | 58(11) | C(1')-Cr(1')-C(1C')-O(1C') | 40(8) |
| C(1E')-Cr(1')-C(1A')-O(1A') | 149(11) | C(1C')-Cr(1')-C(1D')-O(1D') | -88(3) |
| C(1B')-Cr(1')-C(1A')-O(1A') | -120(11) | C(1A')-Cr(1')-C(1D')-O(1D') | 5(3) |
| C(1')-Cr(1')-C(1A')-O(1A') | -174(100) | C(1E')-Cr(1')-C(1D')-O(1D') | 8(6) |
| C(1D')-Cr(1')-C(1B')-O(1B') | 76(8) | C(1B')-Cr(1')-C(1D')-O(1D') | 97(3) |
| C(1C')-Cr(1')-C(1B')-O(1B') | 11(10) | C(1')-Cr(1')-C(1D')-O(1D') | -176(3) |
| C(1A')-Cr(1')-C(1B')-O(1B') | 165(8) | C(1D')-Cr(1')-C(1E')-O(1E') | 43(9) |
| C(1E')-Cr(1')-C(1B')-O(1B') | -106(8) | C(1C')-Cr(1')-C(1E')-O(1E') | 139(7) |
| C(1')-Cr(1')-C(1B')-O(1B') | -17(8) | C(1A')-Cr(1')-C(1E')-O(1E') | 46(7) |
| C(1D')-Cr(1')-C(1C')-O(1C') | -53(8) | C(1B')-Cr(1')-C(1E')-O(1E') | -46(7) |
| C(1A')-Cr(1')-C(1C')-O(1C') | -142(8) | C(1')-Cr(1')-C(1E')-O(1E') | -133(7) |
| C(1E')-Cr(1')-C(1C')-O(1C') | 129(8) | C(4')-O(1')-C(1')-C(2') | 5.3(4) |

| | | | |
|---|---|---|---|
| C(4')-O(1')-C(1')-Cr(1') | -171.4(2) | C(6')-C(7')-O(7')-C(8') | -53.8(4) |
| C(1D')-Cr(1')-C(1')-O(1') | -20.8(3) | C(7')-O(7')-C(8')-O(8') | -54.8(4) |
| C(1C')-Cr(1')-C(1')-O(1') | -109.8(3) | C(7')-O(7')-C(8')-C(4') | 65.1(3) |
| C(1A')-Cr(1')-C(1')-O(1') | 121(3) | C(7')-O(7')-C(8')-C(9') | -168.1(3) |
| C(1E')-Cr(1')-C(1')-O(1') | 159.0(3) | O(1')-C(4')-C(8')-O(7') | 60.0(3) |
| C(1B')-Cr(1')-C(1')-O(1') | 67.6(3) | C(3')-C(4')-C(8')-O(7') | 174.1(3) |
| C(1D')-Cr(1')-C(1')-C(2') | 163.3(3) | C(5')-C(4')-C(8')-O(7') | -56.8(3) |
| C(1C')-Cr(1')-C(1')-C(2') | 74.3(3) | O(1')-C(4')-C(8')-O(8') | -176.3(2) |
| C(1A')-Cr(1')-C(1')-C(2') | -54(4) | C(3')-C(4')-C(8')-O(8') | -62.1(3) |
| C(1E')-Cr(1')-C(1')-C(2') | -16.9(3) | C(5')-C(4')-C(8')-O(8') | 66.9(3) |
| C(1B')-Cr(1')-C(1')-C(2') | -108.3(3) | O(1')-C(4')-C(8')-C(9') | -60.7(4) |
| O(1')-C(1')-C(2')-C(3') | -8.9(4) | C(3')-C(4')-C(8')-C(9') | 53.5(4) |
| Cr(1')-C(1')-C(2')-C(3') | 167.4(3) | C(5')-C(4')-C(8')-C(9') | -177.5(3) |
| O(1')-C(1')-C(2')-C(16') | 114.8(3) | O(7')-C(8')-O(8')-C(10') | -98.2(4) |
| Cr(1')-C(1')-C(2')-C(16') | -68.9(4) | C(4')-C(8')-O(8')-C(10') | 139.6(3) |
| C(1')-C(2')-C(3')-C(4') | 8.9(4) | C(9')-C(8')-O(8')-C(10') | 16.1(4) |
| C(16')-C(2')-C(3')-C(4') | -112.8(3) | O(7')-C(8')-C(9')-O(9') | 112.8(4) |
| C(1')-O(1')-C(4')-C(8') | 122.3(3) | O(8')-C(8')-C(9')-O(9') | -6.7(5) |
| C(1')-O(1')-C(4')-C(3') | 0.6(4) | C(4')-C(8')-C(9')-O(9') | -123.8(4) |
| C(1')-O(1')-C(4')-C(5') | -120.4(3) | C(8')-C(9')-O(9')-C(10') | -5.6(7) |
| C(2')-C(3')-C(4')-O(1') | -6.0(3) | C(9')-O(9')-C(10')-O(8') | 15.2(7) |
| C(2')-C(3')-C(4')-C(8') | -120.1(3) | C(9')-O(9')-C(10')-C(12') | -103.4(6) |
| C(2')-C(3')-C(4')-C(5') | 112.5(3) | C(9')-O(9')-C(10')-C(11') | 131.1(5) |
| O(1')-C(4')-C(5')-O(5') | 44.4(3) | C(8')-O(8')-C(10')-O(9') | -19.3(5) |
| C(8')-C(4')-C(5')-O(5') | 158.1(3) | C(8')-O(8')-C(10')-C(12') | 99.6(4) |
| C(3')-C(4')-C(5')-O(5') | -71.5(3) | C(8')-O(8')-C(10')-C(11') | -137.3(4) |
| O(1')-C(4')-C(5')-C(6') | -73.4(3) | C(6')-O(6')-C(13')-O(5') | -35.5(3) |
| C(8')-C(4')-C(5')-C(6') | 40.3(4) | C(6')-O(6')-C(13')-C(15') | -152.7(3) |
| C(3')-C(4')-C(5')-C(6') | 170.7(3) | C(6')-O(6')-C(13')-C(14') | 83.0(3) |
| C(6')-C(5')-O(5')-C(13') | 7.6(3) | C(5')-O(5')-C(13')-O(6') | 16.4(3) |
| C(4')-C(5')-O(5')-C(13') | -116.6(3) | C(5')-O(5')-C(13')-C(15') | 132.7(3) |
| O(5')-C(5')-C(6')-O(6') | -28.5(3) | C(5')-O(5')-C(13')-C(14') | -103.5(3) |
| C(4')-C(5')-C(6')-O(6') | 92.8(3) | C(1')-C(2')-C(16')-C(17') | 178.1(3) |
| O(5')-C(5')-C(6')-C(7') | -152.4(3) | C(3')-C(2')-C(16')-C(17') | -62.8(4) |
| C(4')-C(5')-C(6')-C(7') | -31.1(4) | C(2')-C(16')-C(17')-C(18') | -64.9(4) |
| C(7')-C(6')-O(6')-C(13') | 164.1(3) | C(16')-C(17')-C(18')-C(20') | 116.3(4) |
| C(5')-C(6')-O(6')-C(13') | 39.2(3) | C(16')-C(17')-C(18')-O(18') | -64.5(4) |
| O(6')-C(6')-C(7')-O(7') | -81.2(4) | C(20')-C(18')-O(18')-C(19') | -1.2(5) |
| C(5')-C(6')-C(7')-O(7') | 35.9(4) | C(17')-C(18')-O(18')-C(19') | 179.6(3) |

**Tabelle 6: Hydrogen bonds for *[56]a* [Å and °].**

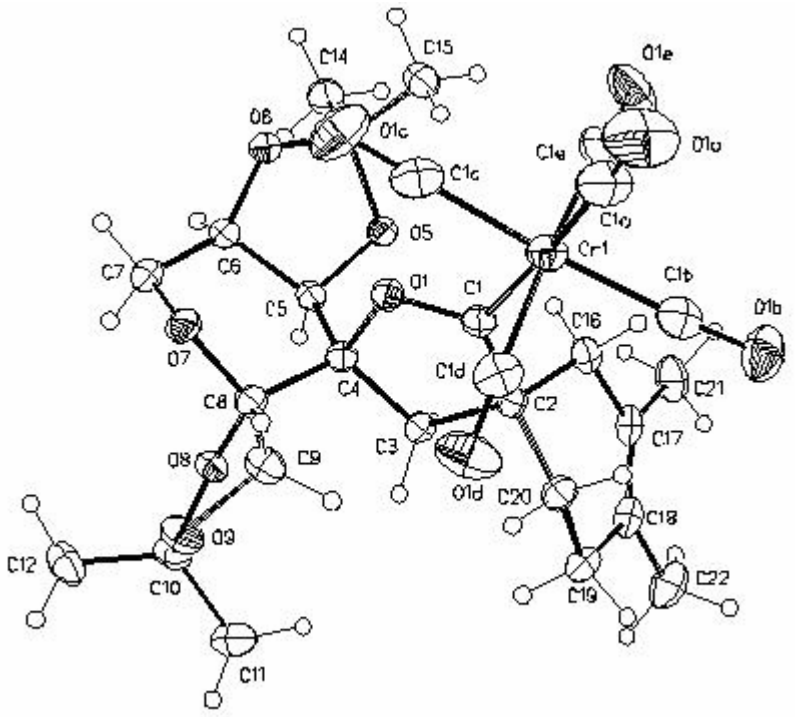
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(9')-H(9'1)...O(1C)[a] | 0.99 | 2.59 | 3.512(5) | 154.0 |
| C(6)-H(6)...O(1E)[b] | 1.00 | 2.50 | 3.448(4) | 159.0 |
| C(7)-H(7A)...O(5)[c] | 0.99 | 2.52 | 3.432(4) | 153.7 |
| C(5)-H(5)...O(6)[b] | 1.00 | 2.43 | 3.315(4) | 147.6 |
| C(2)-H(2)...O(1C')[b] | 1.00 | 2.35 | 3.229(4) | 145.5 |
| C(6')-H(6')...O(1D')[d] | 1.00 | 2.39 | 3.264(4) | 145.7 |
| C(7')-H(7'1)...O(5')[e] | 0.99 | 2.48 | 3.354(4) | 147.4 |
| C(5')-H(5')...O(6')[d] | 1.00 | 2.47 | 3.317(4) | 142.7 |
| C(3')-H(3'1)...O(6')[d] | 0.99 | 2.66 | 3.578(4) | 153.9 |

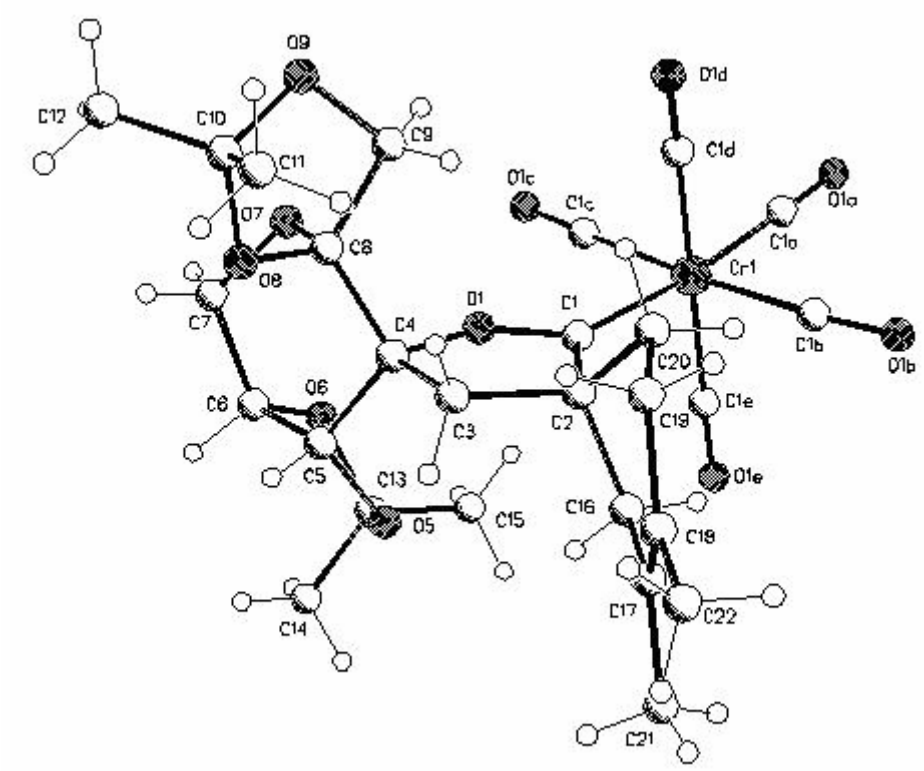
Symmetry transformations used to generate equivalent atoms:

[a] -x+3/2,-y+2,z+1/2 [b] x-1/2,-y+3/2,-z+1 [c] x+1/2,-y+3/2,-z+1 [d] x+1/2,-y+3/2,-z+2

[e] x-1/2,-y+3/2,-z+2

**19. (*3R,5R, 1´R,4´S,6´R*)-Pentacarbonyl{7,8,8´,8´,2´´,2´´-hexamethyl-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,3-2-oxa-spiro[4.5]dec-7-en]-1-yliden}chrom(0) *[64]a***




O9
C12
C10
C9
C11
O7
C8
O8
C7
C4
O1
C1
O1d
C1d
O1c
C1c
O1a
C1a
Cr1
C1b
O1b
C20
C1e
C19
O1e
C6
O6
C3
C2
C5
C13
C16
C18
O5
C15
C14
C17
C22
C21

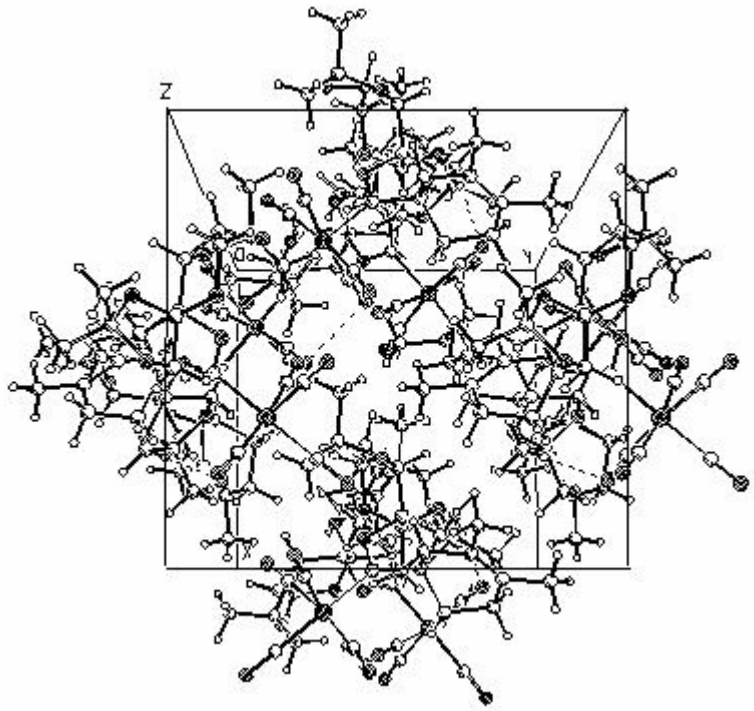

Z

**Table 1: Crystal data and structure refinements for *[64]a***

| | |
|---|---|
| Identification code | ***[64]a*** |
| Empirical formula | $C_{27}H_{32}CrO_{11}$ |
| Formula weight | 584.53 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Tetragonal |
| Space group | P4(3)2(1)2 (No.96) |
| Unit cell dimensions | a = 11.4112(1) Å α = 90° |
| | b = 11.4112(1) Å β = 90° |
| | c = 42.5860(3) Å γ = 90° |
| Volume | 5545.36(8) $Å^3$ |
| Z | 8 |
| Calculated density | 1.400 $mg/m^3$ |
| Absorption coefficient | 0.471 $mm^{-1}$ |
| F(000) | 2448 |
| Crystal size | 0.50 x 0.35 x 0.25 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.90 to 24.99° |
| Limiting indices | $-12 \le h \le 13$, $-13 \le k \le 13$, $-50 \le l \le 50$ |
| Reflections collected / unique | 40374 / 4875 [R(int) = 0.0364] |
| Completeness to Θ = 24.99 | 99.4 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 4875 / 0 / 354 |
| Goodness-of-fit on $F^2$ | 1.030 |
| Final R indices [I>2σ(I)] | R1 = 0.0230, wR2 = 0.0596 |
| R indices (all data) | R1 = 0.0257, wR2 = 0.0607 |
| Absolute structure parameter | -0.017(12) |
| Largest diff. peak and hole | 0.197 and -0.205 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[64]a*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 3185(1) | 1197(1) | 4380(1) | 24(1) |
| C(1A) | 1910(2) | 2241(2) | 4322(1) | 38(1) |
| O(1A) | 1137(1) | 2871(1) | 4289(1) | 59(1) |
| C(1B) | 3995(2) | 2112(2) | 4082(1) | 30(1) |
| O(1B) | 4415(1) | 2726(1) | 3899(1) | 43(1) |
| C(1C) | 2224(2) | 359(2) | 4675(1) | 33(1) |
| O(1C) | 1627(1) | -108(1) | 4851(1) | 51(1) |
| C(1D) | 3854(2) | 2124(2) | 4701(1) | 30(1) |
| O(1D) | 4325(1) | 2662(1) | 4890(1) | 44(1) |
| C(1E) | 2709(2) | 227(2) | 4036(1) | 34(1) |
| O(1E) | 2458(2) | -329(1) | 3825(1) | 55(1) |
| O(1) | 4281(1) | -767(1) | 4689(1) | 20(1) |
| C(1) | 4508(1) | 80(1) | 4487(1) | 20(1) |
| C(2) | 5800(1) | 26(1) | 4395(1) | 19(1) |
| C(3) | 6343(1) | -873(1) | 4624(1) | 20(1) |
| C(4) | 5325(1) | -1403(1) | 4806(1) | 19(1) |
| C(5) | 5189(1) | -2727(1) | 4746(1) | 19(1) |
| O(5) | 4768(1) | -2935(1) | 4436(1) | 22(1) |
| C(6) | 4302(1) | -3350(2) | 4954(1) | 22(1) |
| O(6) | 3247(1) | -3217(1) | 4779(1) | 23(1) |
| C(7) | 4227(2) | -2876(2) | 5285(1) | 26(1) |
| O(7) | 4292(1) | -1626(1) | 5300(1) | 24(1) |
| C(8) | 5314(1) | -1157(1) | 5162(1) | 21(1) |
| O(8) | 6355(1) | -1632(1) | 5288(1) | 20(1) |
| C(9) | 5344(2) | 121(2) | 5266(1) | 25(1) |
| O(9) | 5920(1) | 41(1) | 5562(1) | 27(1) |
| C(10) | 6788(2) | -848(2) | 5533(1) | 24(1) |
| C(11) | 7953(2) | -340(2) | 5425(1) | 33(1) |
| C(12) | 6866(2) | -1506(2) | 5839(1) | 33(1) |
| C(13) | 3583(1) | -3413(1) | 4460(1) | 22(1) |
| C(14) | 3641(2) | -4716(2) | 4380(1) | 30(1) |
| C(15) | 2777(2) | -2741(2) | 4249(1) | 30(1) |
| C(16) | 5861(2) | -408(2) | 4052(1) | 23(1) |
| C(17) | 7088(2) | -360(2) | 3916(1) | 24(1) |
| C(18) | 7934(2) | 296(2) | 4041(1) | 26(1) |
| C(19) | 7734(2) | 1043(2) | 4329(1) | 25(1) |
| C(20) | 6442(1) | 1204(1) | 4413(1) | 23(1) |
| C(21) | 7215(2) | -1109(2) | 3626(1) | 35(1) |
| C(22) | 9174(2) | 362(2) | 3916(1) | 38(1) |

**Tabelle 3: Bond lengths [Å] for *[64]a*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1B) | 1.884(2) | O(5)-C(13) | 1.4617(19) |
| Cr(1)-C(1D) | 1.891(2) | C(6)-O(6) | 1.424(2) |
| Cr(1)-C(1A) | 1.8957(19) | C(6)-C(7) | 1.512(2) |
| Cr(1)-C(1E) | 1.911(2) | O(6)-C(13) | 1.428(2) |
| Cr(1)-C(1C) | 1.923(2) | C(7)-O(7) | 1.430(2) |
| Cr(1)-C(1) | 2.0284(17) | O(7)-C(8) | 1.411(2) |
| C(1A)-O(1A) | 1.147(2) | C(8)-O(8) | 1.4131(19) |
| C(1B)-O(1B) | 1.152(2) | C(8)-C(9) | 1.524(2) |
| C(1C)-O(1C) | 1.145(2) | O(8)-C(10) | 1.459(2) |
| C(1D)-O(1D) | 1.146(2) | C(9)-O(9) | 1.426(2) |
| C(1E)-O(1E) | 1.137(2) | O(9)-C(10) | 1.423(2) |
| O(1)-C(1) | 1.319(2) | C(10)-C(12) | 1.507(3) |
| O(1)-C(4) | 1.4808(18) | C(10)-C(11) | 1.520(2) |
| C(1)-C(2) | 1.527(2) | C(13)-C(15) | 1.499(2) |
| C(2)-C(20) | 1.533(2) | C(13)-C(14) | 1.527(2) |
| C(2)-C(16) | 1.543(2) | C(16)-C(17) | 1.516(2) |
| C(2)-C(3) | 1.546(2) | C(17)-C(18) | 1.332(3) |
| C(3)-C(4) | 1.520(2) | C(17)-C(21) | 1.509(2) |
| C(4)-C(5) | 1.540(2) | C(18)-C(19) | 1.511(2) |
| C(4)-C(8) | 1.541(2) | C(18)-C(22) | 1.515(2) |
| C(5)-O(5) | 1.4270(18) | C(19)-C(20) | 1.528(2) |
| C(5)-C(6) | 1.522(2) | | |

**Tabelle 4: Bond angles [°] for *[64]a*.**

| | | | |
|---|---|---|---|
| C(1B)-Cr(1)-C(1D) | 88.78(8) | C(6)-C(5)-C(4) | 115.44(14) |
| C(1B)-Cr(1)-C(1A) | 86.62(8) | C(5)-O(5)-C(13) | 107.88(12) |
| C(1D)-Cr(1)-C(1A) | 93.01(8) | O(6)-C(6)-C(7) | 113.75(14) |
| C(1B)-Cr(1)-C(1E) | 86.93(8) | O(6)-C(6)-C(5) | 101.96(13) |
| C(1D)-Cr(1)-C(1E) | 172.68(9) | C(7)-C(6)-C(5) | 114.38(14) |
| C(1A)-Cr(1)-C(1E) | 92.65(8) | C(6)-O(6)-C(13) | 104.75(12) |
| C(1B)-Cr(1)-C(1C) | 174.26(8) | O(7)-C(7)-C(6) | 113.22(14) |
| C(1D)-Cr(1)-C(1C) | 92.04(8) | C(8)-O(7)-C(7) | 113.76(12) |
| C(1A)-Cr(1)-C(1C) | 87.67(9) | O(7)-C(8)-O(8) | 112.94(13) |
| C(1E)-Cr(1)-C(1C) | 92.82(8) | O(7)-C(8)-C(9) | 105.07(13) |
| C(1B)-Cr(1)-C(1) | 97.77(7) | O(8)-C(8)-C(9) | 103.71(13) |
| C(1D)-Cr(1)-C(1) | 83.53(7) | O(7)-C(8)-C(4) | 110.36(13) |
| C(1A)-Cr(1)-C(1) | 174.33(8) | O(8)-C(8)-C(4) | 107.35(12) |
| C(1E)-Cr(1)-C(1) | 91.18(7) | C(9)-C(8)-C(4) | 117.41(14) |
| C(1C)-Cr(1)-C(1) | 87.97(7) | C(8)-O(8)-C(10) | 108.79(12) |
| O(1A)-C(1A)-Cr(1) | 179.5(2) | O(9)-C(9)-C(8) | 101.92(13) |
| O(1B)-C(1B)-Cr(1) | 174.73(16) | C(10)-O(9)-C(9) | 106.79(12) |
| O(1C)-C(1C)-Cr(1) | 177.70(17) | O(9)-C(10)-O(8) | 105.27(13) |
| O(1D)-C(1D)-Cr(1) | 175.85(16) | O(9)-C(10)-C(12) | 108.66(13) |
| O(1E)-C(1E)-Cr(1) | 177.33(19) | O(8)-C(10)-C(12) | 109.35(14) |
| C(1)-O(1)-C(4) | 114.82(12) | O(9)-C(10)-C(11) | 111.31(15) |
| O(1)-C(1)-C(2) | 109.10(13) | O(8)-C(10)-C(11) | 108.43(13) |
| O(1)-C(1)-Cr(1) | 117.49(11) | C(12)-C(10)-C(11) | 113.49(16) |
| C(2)-C(1)-Cr(1) | 133.33(11) | O(6)-C(13)-O(5) | 104.97(12) |
| C(1)-C(2)-C(20) | 114.40(13) | O(6)-C(13)-C(15) | 109.12(14) |
| C(1)-C(2)-C(16) | 107.41(13) | O(5)-C(13)-C(15) | 109.45(13) |
| C(20)-C(2)-C(16) | 107.90(13) | O(6)-C(13)-C(14) | 112.07(14) |
| C(1)-C(2)-C(3) | 104.58(13) | O(5)-C(13)-C(14) | 107.88(13) |
| C(20)-C(2)-C(3) | 111.04(13) | C(15)-C(13)-C(14) | 113.01(14) |
| C(16)-C(2)-C(3) | 111.49(13) | C(17)-C(16)-C(2) | 113.07(14) |
| C(4)-C(3)-C(2) | 106.17(13) | C(18)-C(17)-C(21) | 125.16(17) |
| O(1)-C(4)-C(3) | 104.44(12) | C(18)-C(17)-C(16) | 122.43(16) |
| O(1)-C(4)-C(5) | 110.15(13) | C(21)-C(17)-C(16) | 112.40(16) |
| C(3)-C(4)-C(5) | 112.55(13) | C(17)-C(18)-C(19) | 122.13(15) |
| O(1)-C(4)-C(8) | 103.54(12) | C(17)-C(18)-C(22) | 124.34(17) |
| C(3)-C(4)-C(8) | 115.69(14) | C(19)-C(18)-C(22) | 113.53(16) |
| C(5)-C(4)-C(8) | 109.85(13) | C(18)-C(19)-C(20) | 113.81(14) |
| O(5)-C(5)-C(6) | 103.76(13) | C(19)-C(20)-C(2) | 110.08(13) |
| O(5)-C(5)-C(4) | 110.43(13) | | |

**Tabelle 5: Torsion angles [°] for *[64]a.***

| | | | |
|---|---|---|---|
| C(1B)-Cr(1)-C(1A)-O(1A) | -149(72) | C(1)-O(1)-C(4)-C(3) | -3.79(17) |
| C(1D)-Cr(1)-C(1A)-O(1A) | -60(54) | C(1)-O(1)-C(4)-C(5) | -124.88(14) |
| C(1E)-Cr(1)-C(1A)-O(1A) | 124(40) | C(1)-O(1)-C(4)-C(8) | 117.70(14) |
| C(1C)-Cr(1)-C(1A)-O(1A) | 32(40) | C(2)-C(3)-C(4)-O(1) | -2.64(16) |
| C(1)-Cr(1)-C(1A)-O(1A) | -8(41) | C(2)-C(3)-C(4)-C(5) | 116.84(14) |
| C(1D)-Cr(1)-C(1B)-O(1B) | -92.1(18) | C(2)-C(3)-C(4)-C(8) | -115.73(15) |
| C(1A)-Cr(1)-C(1B)-O(1B) | 1.0(18) | O(1)-C(4)-C(5)-O(5) | 45.06(17) |
| C(1E)-Cr(1)-C(1B)-O(1B) | 93.9(18) | C(3)-C(4)-C(5)-O(5) | -71.04(17) |
| C(1C)-Cr(1)-C(1B)-O(1B) | 6(2) | C(8)-C(4)-C(5)-O(5) | 158.49(12) |
| C(1)-Cr(1)-C(1B)-O(1B) | -175(68) | O(1)-C(4)-C(5)-C(6) | -72.23(17) |
| C(1B)-Cr(1)-C(1C)-O(1C) | -23(5) | C(3)-C(4)-C(5)-C(6) | 171.67(13) |
| C(1D)-Cr(1)-C(1C)-O(1C) | 75(4) | C(8)-C(4)-C(5)-C(6) | 41.21(19) |
| C(1A)-Cr(1)-C(1C)-O(1C) | -18(4) | C(6)-C(5)-O(5)-C(13) | 11.85(16) |
| C(1E)-Cr(1)-C(1C)-O(1C) | -110(4) | C(4)-C(5)-O(5)-C(13) | -112.43(14) |
| C(1)-Cr(1)-C(1C)-O(1C) | 159(4) | O(5)-C(5)-C(6)-O(6) | -32.43(15) |
| C(1B)-Cr(1)-C(1D)-O(1D) | -62(2) | C(4)-C(5)-C(6)-O(6) | 88.54(16) |
| C(1A)-Cr(1)-C(1D)-O(1D) | -148(2) | O(5)-C(5)-C(6)-C(7) | -155.66(14) |
| C(1E)-Cr(1)-C(1D)-O(1D) | -8(3) | C(4)-C(5)-C(6)-C(7) | -34.7(2) |
| C(1C)-Cr(1)-C(1D)-O(1D) | 124(2) | C(7)-C(6)-O(6)-C(13) | 164.86(13) |
| C(1)-Cr(1)-C(1D)-O(1D) | 36(2) | C(5)-C(6)-O(6)-C(13) | 41.20(15) |
| C(1B)-Cr(1)-C(1E)-O(1E) | 14(4) | O(6)-C(6)-C(7)-O(7) | -76.29(18) |
| C(1D)-Cr(1)-C(1E)-O(1E) | -40(4) | C(5)-C(6)-C(7)-O(7) | 40.3(2) |
| C(1A)-Cr(1)-C(1E)-O(1E) | 101(4) | C(6)-C(7)-O(7)-C(8) | -57.00(19) |
| C(1C)-Cr(1)-C(1E)-O(1E) | -172(4) | C(7)-O(7)-C(8)-O(8) | -55.25(18) |
| C(1)-Cr(1)-C(1E)-O(1E) | -84(4) | C(7)-O(7)-C(8)-C(9) | -167.61(13) |
| C(4)-O(1)-C(1)-C(2) | 8.67(17) | C(7)-O(7)-C(8)-C(4) | 64.92(17) |
| C(4)-O(1)-C(1)-Cr(1) | -168.43(10) | O(1)-C(4)-C(8)-O(7) | 62.54(15) |
| C(1B)-Cr(1)-C(1)-O(1) | -177.58(12) | C(3)-C(4)-C(8)-O(7) | 176.15(13) |
| C(1D)-Cr(1)-C(1)-O(1) | 94.54(12) | C(5)-C(4)-C(8)-O(7) | -55.08(17) |
| C(1A)-Cr(1)-C(1)-O(1) | 42.0(8) | O(1)-C(4)-C(8)-O(8) | -173.99(11) |
| C(1E)-Cr(1)-C(1)-O(1) | -90.53(13) | C(3)-C(4)-C(8)-O(8) | -60.38(17) |
| C(1C)-Cr(1)-C(1)-O(1) | 2.26(12) | C(5)-C(4)-C(8)-O(8) | 68.39(16) |
| C(1B)-Cr(1)-C(1)-C(2) | 6.18(16) | O(1)-C(4)-C(8)-C(9) | -57.78(18) |
| C(1D)-Cr(1)-C(1)-C(2) | -81.69(16) | C(3)-C(4)-C(8)-C(9) | 55.83(19) |
| C(1A)-Cr(1)-C(1)-C(2) | -134.3(7) | C(5)-C(4)-C(8)-C(9) | -175.40(14) |
| C(1E)-Cr(1)-C(1)-C(2) | 93.24(16) | O(7)-C(8)-O(8)-C(10) | -95.90(15) |
| C(1C)-Cr(1)-C(1)-C(2) | -173.98(16) | C(9)-C(8)-O(8)-C(10) | 17.30(16) |
| O(1)-C(1)-C(2)-C(20) | -131.36(14) | C(4)-C(8)-O(8)-C(10) | 142.23(13) |
| Cr(1)-C(1)-C(2)-C(20) | 45.1(2) | O(7)-C(8)-C(9)-O(9) | 85.98(15) |
| O(1)-C(1)-C(2)-C(16) | 108.90(14) | O(8)-C(8)-C(9)-O(9) | -32.78(16) |
| Cr(1)-C(1)-C(2)-C(16) | -74.64(17) | C(4)-C(8)-C(9)-O(9) | -150.97(13) |
| O(1)-C(1)-C(2)-C(3) | -9.67(17) | C(8)-C(9)-O(9)-C(10) | 36.72(16) |
| Cr(1)-C(1)-C(2)-C(3) | 166.79(12) | C(9)-O(9)-C(10)-O(8) | -26.78(16) |
| C(1)-C(2)-C(3)-C(4) | 7.13(16) | C(9)-O(9)-C(10)-C(12) | -143.82(15) |
| C(20)-C(2)-C(3)-C(4) | 131.01(14) | C(9)-O(9)-C(10)-C(11) | 90.49(17) |
| C(16)-C(2)-C(3)-C(4) | -108.63(15) | C(8)-O(8)-C(10)-O(9) | 4.74(16) |

| | | | |
|---|---|---|---|
| C(8)-O(8)-C(10)-C(12) | 121.32(14) | C(2)-C(16)-C(17)-C(21) | 162.49(14) |
| C(8)-O(8)-C(10)-C(11) | -114.47(15) | C(21)-C(17)-C(18)-C(19) | 178.76(16) |
| C(6)-O(6)-C(13)-O(5) | -34.51(15) | C(16)-C(17)-C(18)-C(19) | 0.1(3) |
| C(6)-O(6)-C(13)-C(15) | -151.74(13) | C(21)-C(17)-C(18)-C(22) | -2.0(3) |
| C(6)-O(6)-C(13)-C(14) | 82.32(16) | C(16)-C(17)-C(18)-C(22) | 179.36(16) |
| C(5)-O(5)-C(13)-O(6) | 13.05(16) | C(17)-C(18)-C(19)-C(20) | -13.8(2) |
| C(5)-O(5)-C(13)-C(15) | 130.05(14) | C(22)-C(18)-C(19)-C(20) | 166.84(15) |
| C(5)-O(5)-C(13)-C(14) | -106.62(15) | C(18)-C(19)-C(20)-C(2) | 45.5(2) |
| C(1)-C(2)-C(16)-C(17) | 172.87(13) | C(1)-C(2)-C(20)-C(19) | 177.78(14) |
| C(20)-C(2)-C(16)-C(17) | 49.07(17) | C(16)-C(2)-C(20)-C(19) | -62.76(17) |
| C(3)-C(2)-C(16)-C(17) | -73.11(17) | C(3)-C(2)-C(20)-C(19) | 59.70(18) |
| C(2)-C(16)-C(17)-C(18) | -18.7(2) | | |

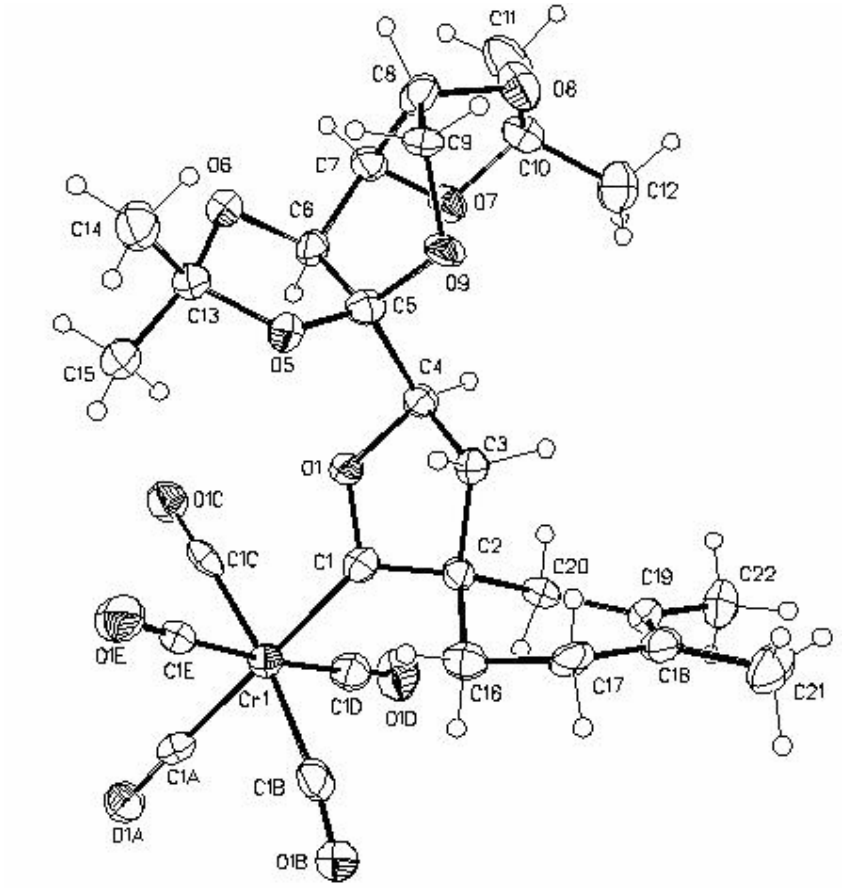
**Tabelle 6: Hydrogen bonds for *[64]a* [Å and °].**

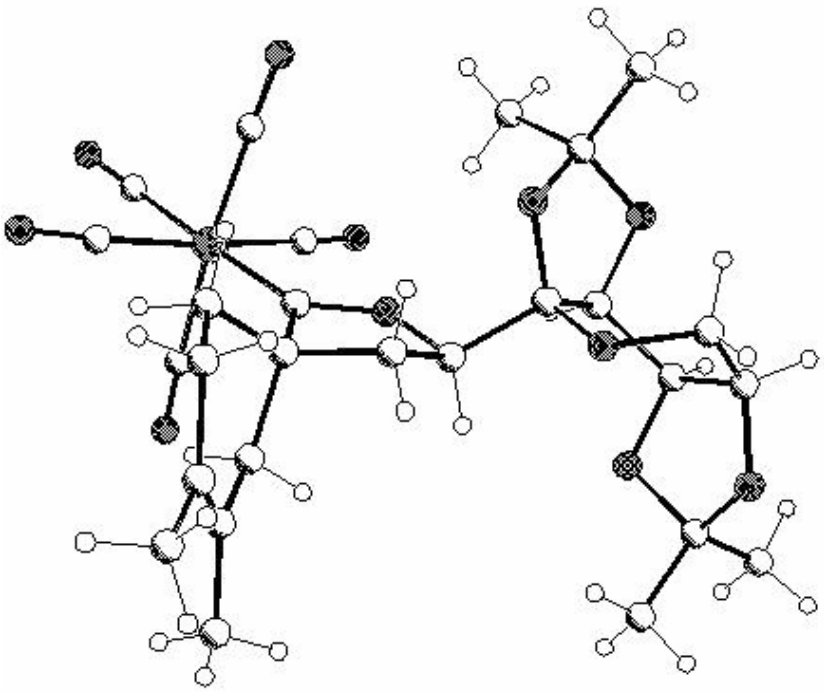
| **D-H...A** | **d(D-H)** | **d(H...A)** | **d(D...A)** | **<(DHA)** |
|---|---|---|---|---|
| C(12)-H(12C)...O(1B)[a] | 0.98 | 2.54 | 3.514(2) | 173.8 |
| C(14)-H(14A)...O(1E)[b] | 0.98 | 2.63 | 3.331(2) | 128.7 |

Symmetry transformations used to generate equivalent atoms:

[a] y+1/2,-x+1/2,z+1/4 [b] -x+1/2,y-1/2,-z+3/4

**20.** **(*3R,5S,1´R,2´S,6´S,9´R*)-Pentacarbonyl{7,8-dimethyl-3-(4´,4´,11´,11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´-yl)-2-oxaspiro[4.5]dec-7-en-1-yliden}chrom(0)** ***[65]a***



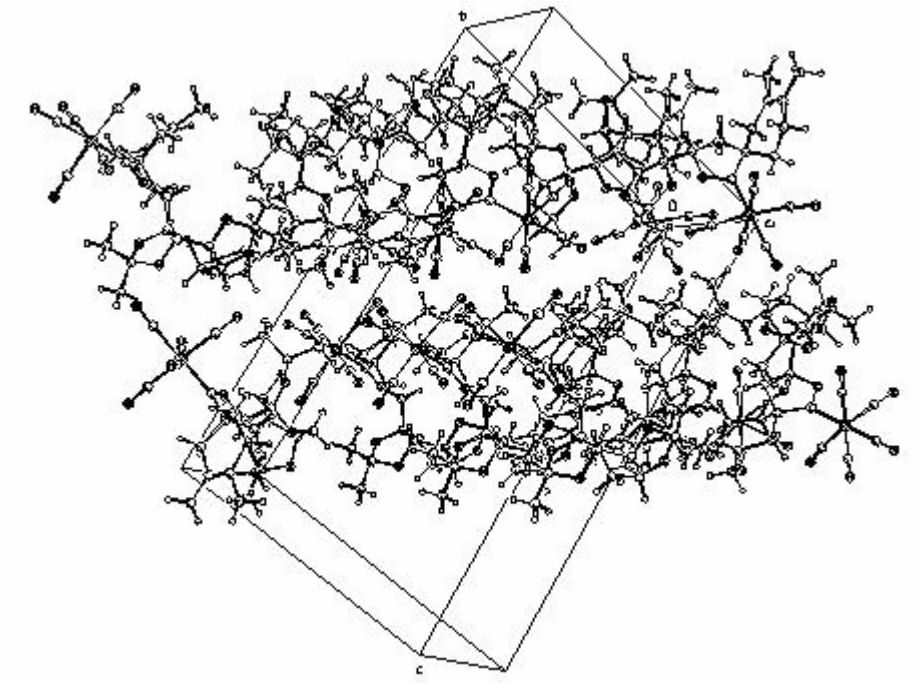


**Table 1: Crystal data and structure refinements for *[65]a***

| | |
|---|---|
| Identification code | ***[65]a*** |
| Empirical formula | $C_{27}H_{32}CrO_{11}$ |
| Formula weight | 584.53 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 7.0224(1) Å α = 90° |
| | b = 15.5740(4) Å β = 90° |
| | c = 25.9722(7) Å γ = 90° |
| Volume | 2840.50(11) $Å^3$ |
| Z | 4 |
| Calculated density | 1.367 $mg/m^3$ |
| Absorption coefficient | 0.460 $mm^{-1}$ |
| F(000) | 1224 |
| Crystal size | 0.20 x 0.08 x 0.04 mm |
| Diffractometer | Nonius KappaCCD |

| | |
|---|---|
| Theta range for data collection | 3.00 to 25.03° |
| Limiting indices | $-8 \leq h \leq 8$, $-18 \leq k \leq 18$, $-30 \leq l \leq 30$ |
| Reflections collected / unique | 22230 / 4825 [R(int) = 0.0880] |
| Completeness to $\Theta = 25.03$ | 97.5 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 4825 / 0 / 354 |
| Goodness-of-fit on $F^2$ | 0.827 |
| Final R indices [$I>2\sigma(I)$] | R1 = 0.0383, wR2 = 0.0500 |
| R indices (all data) | R1 = 0.0869, wR2 = 0.0563 |
| Absolute structure parameter | -0.005(19) |
| Largest diff. peak and hole | 0.261 and -0.248 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[65]a*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 7551(1) | 2669(1) | 1294(1) | 26(1) |
| C(1A) | 7212(5) | 1492(2) | 1463(1) | 32(1) |
| O(1A) | 7079(4) | 779(2) | 1559(1) | 39(1) |
| C(1B) | 9811(5) | 2242(2) | 979(1) | 34(1) |
| O(1B) | 11100(4) | 1879(2) | 811(1) | 46(1) |
| C(1C) | 5238(5) | 2935(2) | 1634(1) | 27(1) |
| O(1C) | 3829(3) | 3059(2) | 1846(1) | 35(1) |
| C(1D) | 6154(5) | 2555(2) | 667(1) | 32(1) |
| O(1D) | 5274(3) | 2507(2) | 298(1) | 45(1) |
| C(1E) | 8830(5) | 2870(2) | 1922(1) | 27(1) |
| O(1E) | 9564(3) | 3008(2) | 2310(1) | 43(1) |
| O(1) | 6792(3) | 4496(1) | 1336(1) | 23(1) |
| C(1) | 7975(4) | 3923(2) | 1136(1) | 23(1) |
| C(2) | 9399(4) | 4421(2) | 804(1) | 21(1) |
| C(3) | 9271(4) | 5342(2) | 1020(1) | 24(1) |
| C(4) | 7256(5) | 5402(2) | 1209(1) | 25(1) |
| C(5) | 6972(4) | 5949(2) | 1691(1) | 25(1) |
| O(5) | 8048(3) | 5634(1) | 2105(1) | 28(1) |
| C(6) | 4902(4) | 5971(2) | 1897(1) | 26(1) |
| O(6) | 5171(3) | 6032(1) | 2440(1) | 29(1) |
| C(7) | 3743(4) | 6735(2) | 1730(1) | 26(1) |

| | | | | |
|---|---|---|---|---|
| O(7) | 3368(3) | 6642(1) | 1195(1) | 29(1) |
| C(8) | 4825(4) | 7593(2) | 1769(1) | 32(1) |
| O(8) | 4544(3) | 7993(1) | 1281(1) | 40(1) |
| C(9) | 6944(4) | 7460(2) | 1838(1) | 29(1) |
| O(9) | 7626(3) | 6761(1) | 1533(1) | 26(1) |
| C(10) | 3238(4) | 7497(2) | 989(1) | 31(1) |
| C(11) | 1230(4) | 7848(2) | 1058(1) | 52(1) |
| C(12) | 3908(5) | 7475(3) | 434(1) | 57(1) |
| C(13) | 6866(5) | 5563(2) | 2561(1) | 29(1) |
| C(14) | 7872(5) | 5971(2) | 3010(1) | 39(1) |
| C(15) | 6405(5) | 4618(2) | 2661(1) | 36(1) |
| C(16) | 11447(4) | 4091(2) | 799(1) | 28(1) |
| C(17) | 12704(5) | 4676(2) | 476(1) | 30(1) |
| C(18) | 11834(4) | 4911(2) | -40(1) | 28(1) |
| C(19) | 10005(5) | 4785(2) | -151(1) | 25(1) |
| C(20) | 8640(4) | 4394(2) | 244(1) | 24(1) |
| C(21) | 13227(4) | 5310(2) | -413(1) | 41(1) |
| C(22) | 9050(4) | 4969(2) | -649(1) | 35(1) |

**Tabelle 3: Bond lengths [Å] for *[65]a*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1E) | 1.888(4) | O(5)-C(13) | 1.449(3) |
| Cr(1)-C(1C) | 1.894(4) | C(6)-O(6) | 1.427(3) |
| Cr(1)-C(1A) | 1.900(4) | C(6)-C(7) | 1.505(4) |
| Cr(1)-C(1B) | 1.905(4) | O(6)-C(13) | 1.432(3) |
| Cr(1)-C(1D) | 1.908(4) | C(7)-O(7) | 1.422(4) |
| Cr(1)-C(1) | 2.017(3) | C(7)-C(8) | 1.541(4) |
| C(1A)-O(1A) | 1.143(3) | O(7)-C(10) | 1.438(4) |
| C(1B)-O(1B) | 1.154(4) | C(8)-O(8) | 1.425(3) |
| C(1C)-O(1C) | 1.149(3) | C(8)-C(9) | 1.513(4) |
| C(1D)-O(1D) | 1.142(3) | O(8)-C(10) | 1.419(4) |
| C(1E)-O(1E) | 1.153(3) | C(9)-O(9) | 1.429(3) |
| O(1)-C(1) | 1.326(3) | C(10)-C(12) | 1.518(4) |
| O(1)-C(4) | 1.486(3) | C(10)-C(11) | 1.522(4) |
| C(1)-C(2) | 1.531(4) | C(13)-C(14) | 1.504(4) |
| C(2)-C(16) | 1.528(4) | C(13)-C(15) | 1.529(4) |
| C(2)-C(3) | 1.542(4) | C(16)-C(17) | 1.519(4) |
| C(2)-C(20) | 1.550(4) | C(17)-C(18) | 1.519(4) |
| C(3)-C(4) | 1.501(4) | C(18)-C(19) | 1.331(4) |
| C(4)-C(5) | 1.527(4) | C(18)-C(21) | 1.511(4) |
| C(5)-O(5) | 1.403(3) | C(19)-C(22) | 1.484(4) |
| C(5)-O(9) | 1.407(4) | C(19)-C(20) | 1.530(4) |
| C(5)-C(6) | 1.549(4) | | |

**Tabelle 4: Bond angles [°] for *[65]a*.**

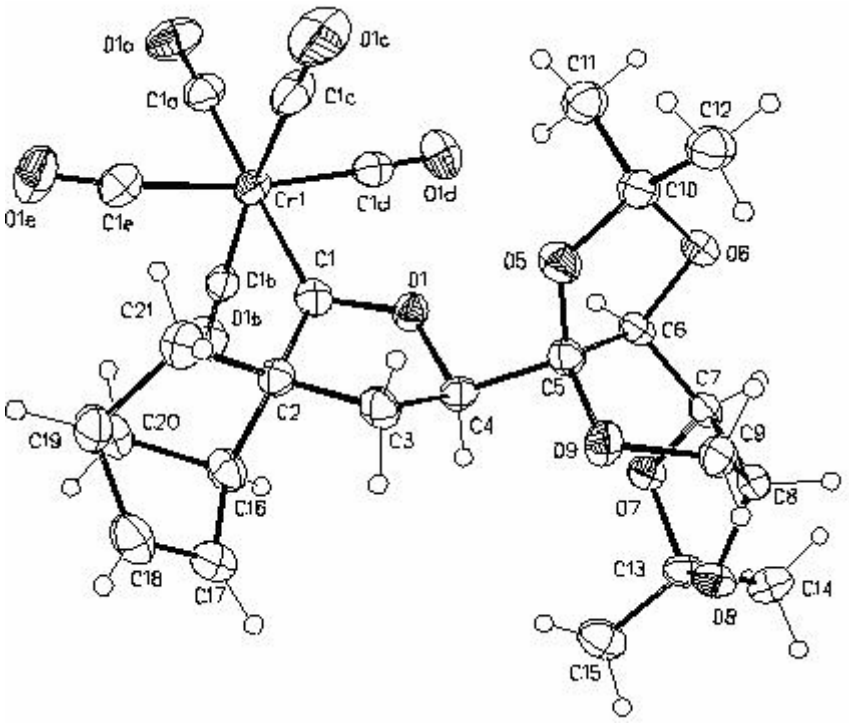
| | | | |
|---|---|---|---|
| C(1E)-Cr(1)-C(1C) | 88.22(15) | O(9)-C(5)-C(6) | 112.8(3) |
| C(1E)-Cr(1)-C(1A) | 91.11(14) | C(4)-C(5)-C(6) | 114.7(3) |
| C(1C)-Cr(1)-C(1A) | 89.74(14) | C(5)-O(5)-C(13) | 110.1(2) |
| C(1E)-Cr(1)-C(1B) | 91.81(14) | O(6)-C(6)-C(7) | 107.7(3) |
| C(1C)-Cr(1)-C(1B) | 172.08(15) | O(6)-C(6)-C(5) | 102.6(2) |
| C(1A)-Cr(1)-C(1B) | 82.34(15) | C(7)-C(6)-C(5) | 115.2(3) |
| C(1E)-Cr(1)-C(1D) | 175.27(15) | C(6)-O(6)-C(13) | 107.0(2) |
| C(1C)-Cr(1)-C(1D) | 88.72(14) | O(7)-C(7)-C(6) | 107.5(3) |
| C(1A)-Cr(1)-C(1D) | 92.48(14) | O(7)-C(7)-C(8) | 104.1(3) |
| C(1B)-Cr(1)-C(1D) | 91.72(14) | C(6)-C(7)-C(8) | 113.6(3) |
| C(1E)-Cr(1)-C(1) | 86.88(13) | C(7)-O(7)-C(10) | 106.3(2) |
| C(1C)-Cr(1)-C(1) | 90.59(13) | O(8)-C(8)-C(9) | 107.5(3) |
| C(1A)-Cr(1)-C(1) | 177.95(13) | O(8)-C(8)-C(7) | 104.6(2) |
| C(1B)-Cr(1)-C(1) | 97.32(14) | C(9)-C(8)-C(7) | 112.0(3) |
| C(1D)-Cr(1)-C(1) | 89.55(13) | C(10)-O(8)-C(8) | 109.1(3) |
| O(1A)-C(1A)-Cr(1) | 177.4(3) | O(9)-C(9)-C(8) | 111.6(3) |
| O(1B)-C(1B)-Cr(1) | 171.0(3) | C(5)-O(9)-C(9) | 114.4(2) |
| O(1C)-C(1C)-Cr(1) | 177.1(3) | O(8)-C(10)-O(7) | 105.3(2) |
| O(1D)-C(1D)-Cr(1) | 177.6(3) | O(8)-C(10)-C(12) | 108.6(3) |
| O(1E)-C(1E)-Cr(1) | 177.9(3) | O(7)-C(10)-C(12) | 108.2(3) |
| C(1)-O(1)-C(4) | 114.5(2) | O(8)-C(10)-C(11) | 109.9(3) |
| O(1)-C(1)-C(2) | 106.8(3) | O(7)-C(10)-C(11) | 110.4(3) |
| O(1)-C(1)-Cr(1) | 118.6(2) | C(12)-C(10)-C(11) | 114.0(3) |
| C(2)-C(1)-Cr(1) | 134.6(2) | O(6)-C(13)-O(5) | 105.0(2) |
| C(16)-C(2)-C(1) | 116.7(3) | O(6)-C(13)-C(14) | 110.1(3) |
| C(16)-C(2)-C(3) | 111.8(2) | O(5)-C(13)-C(14) | 109.3(3) |
| C(1)-C(2)-C(3) | 103.3(2) | O(6)-C(13)-C(15) | 110.7(3) |
| C(16)-C(2)-C(20) | 107.8(2) | O(5)-C(13)-C(15) | 109.5(3) |
| C(1)-C(2)-C(20) | 106.8(2) | C(14)-C(13)-C(15) | 112.0(3) |
| C(3)-C(2)-C(20) | 110.3(3) | C(17)-C(16)-C(2) | 110.5(3) |
| C(4)-C(3)-C(2) | 103.4(2) | C(16)-C(17)-C(18) | 113.4(3) |
| O(1)-C(4)-C(3) | 102.7(2) | C(19)-C(18)-C(21) | 123.1(3) |
| O(1)-C(4)-C(5) | 108.6(2) | C(19)-C(18)-C(17) | 122.9(3) |
| C(3)-C(4)-C(5) | 115.2(3) | C(21)-C(18)-C(17) | 113.9(3) |
| O(5)-C(5)-O(9) | 111.2(2) | C(18)-C(19)-C(22) | 126.6(3) |
| O(5)-C(5)-C(4) | 111.4(3) | C(18)-C(19)-C(20) | 121.2(3) |
| O(9)-C(5)-C(4) | 102.6(2) | C(22)-C(19)-C(20) | 112.2(3) |
| O(5)-C(5)-C(6) | 104.4(2) | C(19)-C(20)-C(2) | 113.8(3) |

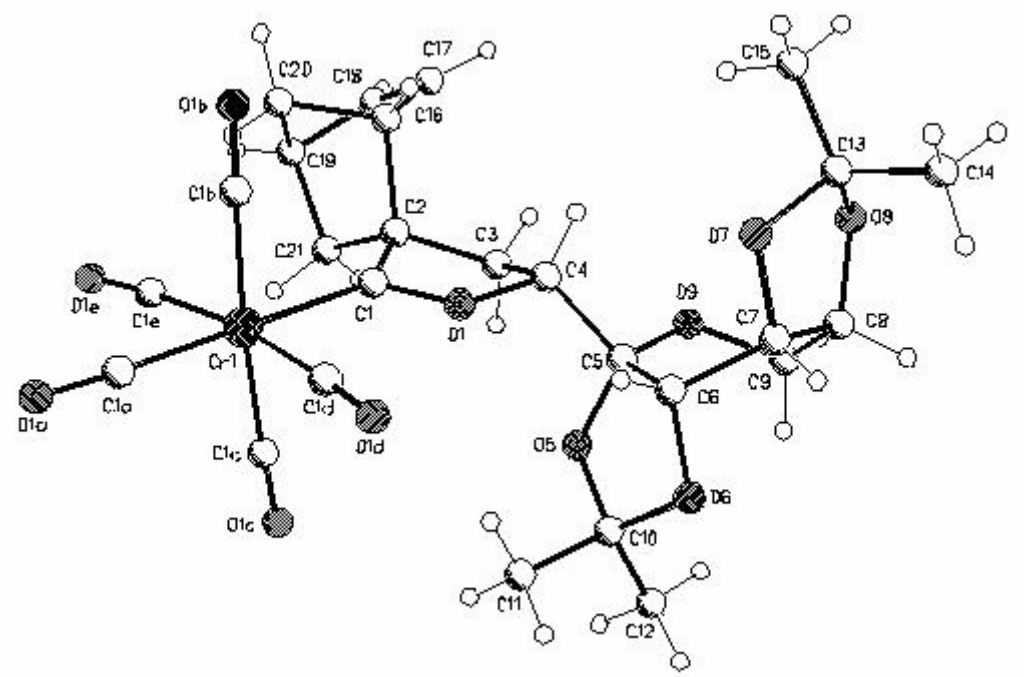
**Tabelle 5: Torsion angles [°] for *[65]a.***

| | | | |
|---|---|---|---|
| C(1E)-Cr(1)-C(1A)-O(1A) | -79(7) | C(1)-O(1)-C(4)-C(3) | -16.2(3) |
| C(1C)-Cr(1)-C(1A)-O(1A) | -168(7) | C(1)-O(1)-C(4)-C(5) | -138.6(2) |
| C(1B)-Cr(1)-C(1A)-O(1A) | 12(7) | C(2)-C(3)-C(4)-O(1) | 27.6(3) |
| C(1D)-Cr(1)-C(1A)-O(1A) | 104(7) | C(2)-C(3)-C(4)-C(5) | 145.5(3) |
| C(1)-Cr(1)-C(1A)-O(1A) | -68(8) | O(1)-C(4)-C(5)-O(5) | 55.5(3) |
| C(1E)-Cr(1)-C(1B)-O(1B) | 91.6(19) | C(3)-C(4)-C(5)-O(5) | -59.0(3) |
| C(1C)-Cr(1)-C(1B)-O(1B) | 2(3) | O(1)-C(4)-C(5)-O(9) | 174.6(2) |
| C(1A)-Cr(1)-C(1B)-O(1B) | 0.7(19) | C(3)-C(4)-C(5)-O(9) | 60.1(3) |
| C(1D)-Cr(1)-C(1B)-O(1B) | -92(2) | O(1)-C(4)-C(5)-C(6) | -62.7(3) |
| C(1)-Cr(1)-C(1B)-O(1B) | 179(100) | C(3)-C(4)-C(5)-C(6) | -177.2(3) |
| C(1E)-Cr(1)-C(1C)-O(1C) | -89(6) | O(9)-C(5)-O(5)-C(13) | 115.0(3) |
| C(1A)-Cr(1)-C(1C)-O(1C) | 2(6) | C(4)-C(5)-O(5)-C(13) | -131.2(3) |
| C(1B)-Cr(1)-C(1C)-O(1C) | 2(7) | C(6)-C(5)-O(5)-C(13) | -6.9(3) |
| C(1D)-Cr(1)-C(1C)-O(1C) | 95(6) | O(5)-C(5)-C(6)-O(6) | 24.4(3) |
| C(1)-Cr(1)-C(1C)-O(1C) | -175(100) | O(9)-C(5)-C(6)-O(6) | -96.4(3) |
| C(1E)-Cr(1)-C(1D)-O(1D) | -20(9) | C(4)-C(5)-C(6)-O(6) | 146.6(3) |
| C(1C)-Cr(1)-C(1D)-O(1D) | 29(8) | O(5)-C(5)-C(6)-C(7) | 141.1(3) |
| C(1A)-Cr(1)-C(1D)-O(1D) | 119(8) | O(9)-C(5)-C(6)-C(7) | 20.2(4) |
| C(1B)-Cr(1)-C(1D)-O(1D) | -159(8) | C(4)-C(5)-C(6)-C(7) | -96.8(4) |
| C(1)-Cr(1)-C(1D)-O(1D) | -61(8) | C(7)-C(6)-O(6)-C(13) | -155.2(3) |
| C(1C)-Cr(1)-C(1E)-O(1E) | -22(9) | C(5)-C(6)-O(6)-C(13) | -33.3(3) |
| C(1A)-Cr(1)-C(1E)-O(1E) | -112(9) | O(6)-C(6)-C(7)-O(7) | -176.1(2) |
| C(1B)-Cr(1)-C(1E)-O(1E) | 166(9) | C(5)-C(6)-C(7)-O(7) | 70.1(3) |
| C(1D)-Cr(1)-C(1E)-O(1E) | 28(10) | O(6)-C(6)-C(7)-C(8) | 69.2(3) |
| C(1)-Cr(1)-C(1E)-O(1E) | 69(9) | C(5)-C(6)-C(7)-C(8) | -44.5(4) |
| C(4)-O(1)-C(1)-C(2) | -3.2(3) | C(6)-C(7)-O(7)-C(10) | -149.6(2) |
| C(4)-O(1)-C(1)-Cr(1) | 178.36(18) | C(8)-C(7)-O(7)-C(10) | -28.7(3) |
| C(1E)-Cr(1)-C(1)-O(1) | -81.7(2) | O(7)-C(7)-C(8)-O(8) | 13.8(3) |
| C(1C)-Cr(1)-C(1)-O(1) | 6.5(2) | C(6)-C(7)-C(8)-O(8) | 130.4(3) |
| C(1A)-Cr(1)-C(1)-O(1) | -93(4) | O(7)-C(7)-C(8)-C(9) | -102.4(3) |
| C(1B)-Cr(1)-C(1)-O(1) | -173.1(2) | C(6)-C(7)-C(8)-C(9) | 14.3(4) |
| C(1D)-Cr(1)-C(1)-O(1) | 95.2(2) | C(9)-C(8)-O(8)-C(10) | 125.8(3) |
| C(1E)-Cr(1)-C(1)-C(2) | 100.4(3) | C(7)-C(8)-O(8)-C(10) | 6.6(3) |
| C(1C)-Cr(1)-C(1)-C(2) | -171.4(3) | O(8)-C(8)-C(9)-O(9) | -75.3(3) |
| C(1A)-Cr(1)-C(1)-C(2) | 89(4) | C(7)-C(8)-C(9)-O(9) | 39.1(3) |
| C(1B)-Cr(1)-C(1)-C(2) | 9.0(3) | O(5)-C(5)-O(9)-C(9) | -81.3(3) |
| C(1D)-Cr(1)-C(1)-C(2) | -82.7(3) | C(4)-C(5)-O(9)-C(9) | 159.6(2) |
| O(1)-C(1)-C(2)-C(16) | 143.8(3) | C(6)-C(5)-O(9)-C(9) | 35.6(3) |
| Cr(1)-C(1)-C(2)-C(16) | -38.1(4) | C(8)-C(9)-O(9)-C(5) | -68.1(3) |
| O(1)-C(1)-C(2)-C(3) | 20.8(3) | C(8)-O(8)-C(10)-O(7) | -24.4(3) |
| Cr(1)-C(1)-C(2)-C(3) | -161.2(2) | C(8)-O(8)-C(10)-C(12) | -140.2(3) |
| O(1)-C(1)-C(2)-C(20) | -95.6(3) | C(8)-O(8)-C(10)-C(11) | 94.5(3) |
| Cr(1)-C(1)-C(2)-C(20) | 82.5(3) | C(7)-O(7)-C(10)-O(8) | 33.5(3) |
| C(16)-C(2)-C(3)-C(4) | -156.1(2) | C(7)-O(7)-C(10)-C(12) | 149.6(2) |
| C(1)-C(2)-C(3)-C(4) | -29.9(3) | C(7)-O(7)-C(10)-C(11) | -85.1(3) |
| C(20)-C(2)-C(3)-C(4) | 84.0(3) | C(6)-O(6)-C(13)-O(5) | 29.8(3) |

| | | | |
|---|---|---|---|
| C(6)-O(6)-C(13)-C(14) | 147.4(3) | C(16)-C(17)-C(18)-C(21) | -166.3(3) |
| C(6)-O(6)-C(13)-C(15) | -88.3(3) | C(21)-C(18)-C(19)-C(22) | 2.8(6) |
| C(5)-O(5)-C(13)-O(6) | -13.3(3) | C(17)-C(18)-C(19)-C(22) | -178.0(3) |
| C(5)-O(5)-C(13)-C(14) | -131.4(3) | C(21)-C(18)-C(19)-C(20) | -178.8(3) |
| C(5)-O(5)-C(13)-C(15) | 105.6(3) | C(17)-C(18)-C(19)-C(20) | 0.4(5) |
| C(1)-C(2)-C(16)-C(17) | -177.1(3) | C(18)-C(19)-C(20)-C(2) | 17.2(4) |
| C(3)-C(2)-C(16)-C(17) | -58.6(3) | C(22)-C(19)-C(20)-C(2) | -164.2(3) |
| C(20)-C(2)-C(16)-C(17) | 62.8(3) | C(16)-C(2)-C(20)-C(19) | -47.8(3) |
| C(2)-C(16)-C(17)-C(18) | -46.6(3) | C(1)-C(2)-C(20)-C(19) | -174.0(3) |
| C(16)-C(17)-C(18)-C(19) | 14.4(5) | C(3)-C(2)-C(20)-C(19) | 74.5(3) |

**21.** **5.10.3 (*3R,5R,1´R,4´S,1´´R,2´´S,5´´S,9´´R*)-Pentacarbonyl{3-(4´´,4´´,-11´´,11´´-tetramethyl-3´´,5´´,7´´,10´´,12´´-pentaoxatricyclo-[7.3.0.0$^{2,6}$]-dodec-6´´-yl)-spiro[bicyclo[2.2.1]hept-2´-en-5´,5-2-oxacyclo-pent]-1-yliden}chrom(0) *[66]a***




C20
C18
C17
C16
C15
O1b
C19
C13
C14
C1b
C2
O9
O7
C21
C3
C4
O1e
C1e
C1
O1
O3
C7
C8
Cr1
C5
C6
C9
O1a
C1a
C1d
O1d
O5
C1c
O6
O1c
C10
C11
C12

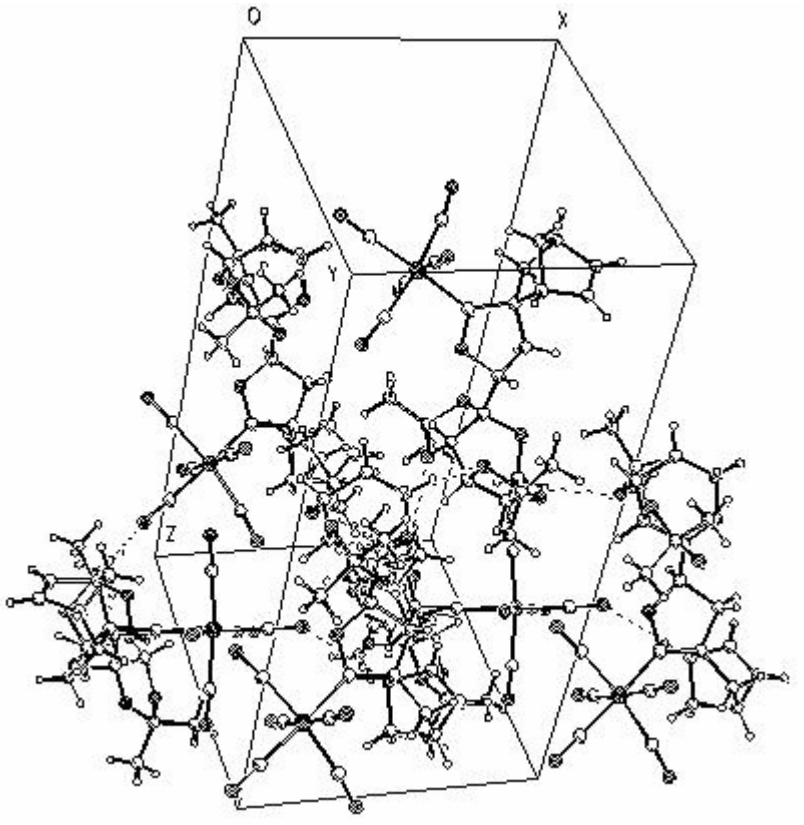

O
X
Y
Z

**Table 1: Crystal data and structure refinements for *[66]a***

| | |
|---|---|
| Identification code | ***[66]a*** |
| Empirical formula | $C_{26}H_{28}CrO_{11}$ |
| Formula weight | 568.48 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 10.4519(1) Å α = 90° |
| | b = 12.1090(2) Å β = 90° |
| | c = 21.3688(4) Å γ = 90° |
| Volume | 2704.48(7) $Å^3$ |
| Z | 4 |
| Calculated density | 1.396 $mg/m^3$ |
| Absorption coefficient | 0.481 $mm^{-1}$ |
| F(000) | 1184 |
| Crystal size | 0.25 x 0.20 x 0.15 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 3.20 to 25.00° |
| Limiting indices | $-12 \le h \le 11$, $-14 \le k \le 14$, $-25 \le l \le 25$ |
| Reflections collected / unique | 22708 / 4766 [R(int) = 0.0522] |
| Completeness to Θ = 25.00 | 99.7 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 4766 / 0 / 343 |
| Goodness-of-fit on $F^2$ | 0.972 |
| Final R indices [I>2σ(I)] | R1 = 0.0293, wR2 = 0.0573 |
| R indices (all data) | R1 = 0.0398, wR2 = 0.0599 |
| Absolute structure parameter | -0.008(14) |
| Largest diff. peak and hole | 0.169 and -0.260 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[66]a*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 5522(1) | 3070(1) | 2918(1) | 26(1) |
| C(1A) | 4094(2) | 2355(2) | 2580(1) | 33(1) |
| O(1A) | 3220(2) | 1936(2) | 2348(1) | 50(1) |
| C(1B) | 5447(2) | 4183(2) | 2293(1) | 27(1) |
| O(1B) | 5408(2) | 4860(1) | 1927(1) | 41(1) |
| C(1C) | 5728(2) | 2046(2) | 3589(1) | 38(1) |
| O(1C) | 5900(2) | 1454(2) | 3993(1) | 64(1) |
| C(1D) | 4365(2) | 3890(2) | 3422(1) | 29(1) |
| O(1D) | 3604(2) | 4345(2) | 3717(1) | 43(1) |
| C(1E) | 6444(2) | 2095(2) | 2394(1) | 34(1) |
| O(1E) | 6887(2) | 1443(2) | 2072(1) | 50(1) |
| O(1) | 6734(1) | 4607(1) | 3747(1) | 25(1) |
| C(1) | 7035(2) | 3904(2) | 3304(1) | 23(1) |
| C(2) | 8490(2) | 3939(2) | 3218(1) | 24(1) |
| C(3) | 8945(2) | 4409(2) | 3843(1) | 28(1) |
| C(4) | 7859(2) | 5154(2) | 4039(1) | 25(1) |
| C(5) | 7645(2) | 5211(2) | 4743(1) | 24(1) |
| O(5) | 7497(1) | 4129(1) | 4985(1) | 29(1) |
| C(6) | 6447(2) | 5817(2) | 4970(1) | 22(1) |
| O(6) | 6130(1) | 5225(1) | 5531(1) | 25(1) |
| C(7) | 6625(2) | 7018(2) | 5143(1) | 23(1) |
| O(7) | 6674(1) | 7639(1) | 4575(1) | 25(1) |
| C(8) | 7913(2) | 7277(2) | 5468(1) | 24(1) |
| O(8) | 8538(1) | 8033(1) | 5051(1) | 28(1) |
| C(9) | 8739(2) | 6265(2) | 5531(1) | 26(1) |
| O(9) | 8807(1) | 5686(1) | 4951(1) | 28(1) |
| C(10) | 6478(2) | 4102(2) | 5436(1) | 28(1) |
| C(11) | 5365(2) | 3450(2) | 5170(1) | 35(1) |
| C(12) | 6975(2) | 3632(2) | 6046(1) | 35(1) |
| C(13) | 7571(2) | 8517(2) | 4671(1) | 27(1) |
| C(14) | 6961(2) | 9480(2) | 5007(1) | 34(1) |
| C(15) | 8129(2) | 8816(2) | 4043(1) | 38(1) |
| C(16) | 8817(2) | 4724(2) | 2636(1) | 30(1) |
| C(17) | 10246(2) | 4930(2) | 2681(1) | 35(1) |
| C(18) | 10837(2) | 4000(2) | 2531(1) | 37(1) |
| C(19) | 9820(2) | 3146(2) | 2385(1) | 33(1) |
| C(20) | 8799(2) | 3894(2) | 2089(1) | 32(1) |
| C(21) | 9163(2) | 2854(2) | 3022(1) | 31(1) |

**Tabelle 3: Bond lengths [Å] for *[66]a*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1A) | 1.871(2) | O(5)-C(10) | 1.437(2) |
| Cr(1)-C(1E) | 1.891(3) | C(6)-O(6) | 1.436(2) |
| Cr(1)-C(1B) | 1.898(2) | C(6)-C(7) | 1.512(3) |
| Cr(1)-C(1D) | 1.899(3) | O(6)-C(10) | 1.422(3) |
| Cr(1)-C(1C) | 1.907(3) | C(7)-O(7) | 1.430(2) |
| Cr(1)-C(1) | 2.049(2) | C(7)-C(8) | 1.546(3) |
| C(1A)-O(1A) | 1.156(3) | O(7)-C(13) | 1.433(3) |
| C(1B)-O(1B) | 1.134(2) | C(8)-O(8) | 1.435(2) |
| C(1C)-O(1C) | 1.138(3) | C(8)-C(9) | 1.505(3) |
| C(1D)-O(1D) | 1.154(3) | O(8)-C(13) | 1.422(3) |
| C(1E)-O(1E) | 1.146(3) | C(9)-O(9) | 1.426(2) |
| O(1)-C(1) | 1.312(2) | C(10)-C(12) | 1.516(3) |
| O(1)-C(4) | 1.487(2) | C(10)-C(11) | 1.516(3) |
| C(1)-C(2) | 1.533(3) | C(13)-C(15) | 1.508(3) |
| C(2)-C(3) | 1.527(3) | C(13)-C(14) | 1.510(3) |
| C(2)-C(21) | 1.549(3) | C(16)-C(17) | 1.518(3) |
| C(2)-C(16) | 1.601(3) | C(16)-C(20) | 1.541(3) |
| C(3)-C(4) | 1.510(3) | C(17)-C(18) | 1.324(3) |
| C(4)-C(5) | 1.521(3) | C(18)-C(19) | 1.515(3) |
| C(5)-O(9) | 1.415(2) | C(19)-C(20) | 1.537(3) |
| C(5)-O(5) | 1.418(3) | C(19)-C(21) | 1.565(3) |
| C(5)-C(6) | 1.530(3) | | |

**Tabelle 4: Bond angles [°] for *[66]a*.**

| | | | |
|---|---|---|---|
| C(1A)-Cr(1)-C(1E) | 83.61(10) | C(4)-C(5)-C(6) | 117.09(18) |
| C(1A)-Cr(1)-C(1B) | 91.38(10) | C(5)-O(5)-C(10) | 110.23(17) |
| C(1E)-Cr(1)-C(1B) | 92.74(10) | O(6)-C(6)-C(7) | 107.65(17) |
| C(1A)-Cr(1)-C(1D) | 87.32(10) | O(6)-C(6)-C(5) | 102.38(16) |
| C(1E)-Cr(1)-C(1D) | 170.57(10) | C(7)-C(6)-C(5) | 115.98(17) |
| C(1B)-Cr(1)-C(1D) | 90.06(9) | C(10)-O(6)-C(6) | 107.37(16) |
| C(1A)-Cr(1)-C(1C) | 94.57(10) | O(7)-C(7)-C(6) | 107.53(16) |
| C(1E)-Cr(1)-C(1C) | 88.95(10) | O(7)-C(7)-C(8) | 104.09(16) |
| C(1B)-Cr(1)-C(1C) | 173.95(10) | C(6)-C(7)-C(8) | 114.38(17) |
| C(1D)-Cr(1)-C(1C) | 89.19(10) | C(7)-O(7)-C(13) | 106.92(15) |
| C(1A)-Cr(1)-C(1) | 177.55(10) | O(8)-C(8)-C(9) | 108.31(16) |
| C(1E)-Cr(1)-C(1) | 98.78(9) | O(8)-C(8)-C(7) | 104.31(16) |
| C(1B)-Cr(1)-C(1) | 87.99(9) | C(9)-C(8)-C(7) | 111.95(18) |
| C(1D)-Cr(1)-C(1) | 90.31(9) | C(13)-O(8)-C(8) | 107.07(14) |
| C(1C)-Cr(1)-C(1) | 86.01(9) | O(9)-C(9)-C(8) | 110.58(17) |
| O(1A)-C(1A)-Cr(1) | 177.3(2) | C(5)-O(9)-C(9) | 115.48(15) |
| O(1B)-C(1B)-Cr(1) | 178.91(19) | O(6)-C(10)-O(5) | 105.35(17) |
| O(1C)-C(1C)-Cr(1) | 177.2(2) | O(6)-C(10)-C(12) | 108.90(19) |
| O(1D)-C(1D)-Cr(1) | 175.9(2) | O(5)-C(10)-C(12) | 109.40(17) |
| O(1E)-C(1E)-Cr(1) | 172.9(2) | O(6)-C(10)-C(11) | 110.79(17) |
| C(1)-O(1)-C(4) | 113.77(15) | O(5)-C(10)-C(11) | 109.22(18) |
| O(1)-C(1)-C(2) | 107.83(17) | C(12)-C(10)-C(11) | 112.89(19) |
| O(1)-C(1)-Cr(1) | 115.19(14) | O(8)-C(13)-O(7) | 103.99(17) |
| C(2)-C(1)-Cr(1) | 136.98(16) | O(8)-C(13)-C(15) | 109.39(17) |
| C(3)-C(2)-C(1) | 102.37(17) | O(7)-C(13)-C(15) | 107.61(18) |
| C(3)-C(2)-C(21) | 114.25(18) | O(8)-C(13)-C(14) | 110.29(19) |
| C(1)-C(2)-C(21) | 117.34(19) | O(7)-C(13)-C(14) | 111.42(17) |
| C(3)-C(2)-C(16) | 113.05(18) | C(15)-C(13)-C(14) | 113.7(2) |
| C(1)-C(2)-C(16) | 108.79(17) | C(17)-C(16)-C(20) | 99.68(17) |
| C(21)-C(2)-C(16) | 101.36(16) | C(17)-C(16)-C(2) | 104.99(18) |
| C(4)-C(3)-C(2) | 103.42(16) | C(20)-C(16)-C(2) | 101.51(18) |
| O(1)-C(4)-C(3) | 102.18(16) | C(18)-C(17)-C(16) | 107.6(2) |
| O(1)-C(4)-C(5) | 108.59(16) | C(17)-C(18)-C(19) | 107.7(2) |
| C(3)-C(4)-C(5) | 114.40(19) | C(18)-C(19)-C(20) | 99.73(19) |
| O(9)-C(5)-O(5) | 110.75(17) | C(18)-C(19)-C(21) | 106.44(18) |
| O(9)-C(5)-C(4) | 101.69(16) | C(20)-C(19)-C(21) | 100.72(17) |
| O(5)-C(5)-C(4) | 109.52(19) | C(19)-C(20)-C(16) | 93.65(17) |
| O(9)-C(5)-C(6) | 114.08(18) | C(2)-C(21)-C(19) | 104.03(18) |
| O(5)-C(5)-C(6) | 103.82(16) | | |

**Tabelle 5: Torsion angles [°] for *[66]a.***

| | | | |
|---|---|---|---|
| C(1E)-Cr(1)-C(1A)-O(1A) | -74(5) | C(1)-O(1)-C(4)-C(3) | -17.2(2) |
| C(1B)-Cr(1)-C(1A)-O(1A) | 18(5) | C(1)-O(1)-C(4)-C(5) | -138.49(19) |
| C(1D)-Cr(1)-C(1A)-O(1A) | 108(5) | C(2)-C(3)-C(4)-O(1) | 29.4(2) |
| C(1C)-Cr(1)-C(1A)-O(1A) | -163(5) | C(2)-C(3)-C(4)-C(5) | 146.6(2) |
| C(1)-Cr(1)-C(1A)-O(1A) | 94(5) | O(1)-C(4)-C(5)-O(9) | 176.87(16) |
| C(1A)-Cr(1)-C(1B)-O(1B) | 140(11) | C(3)-C(4)-C(5)-O(9) | 63.5(2) |
| C(1E)-Cr(1)-C(1B)-O(1B) | -136(11) | O(1)-C(4)-C(5)-O(5) | 59.7(2) |
| C(1D)-Cr(1)-C(1B)-O(1B) | 53(11) | C(3)-C(4)-C(5)-O(5) | -53.7(2) |
| C(1C)-Cr(1)-C(1B)-O(1B) | -30(12) | O(1)-C(4)-C(5)-C(6) | -58.1(3) |
| C(1)-Cr(1)-C(1B)-O(1B) | -37(11) | C(3)-C(4)-C(5)-C(6) | -171.52(18) |
| C(1A)-Cr(1)-C(1C)-O(1C) | 167(5) | O(9)-C(5)-O(5)-C(10) | 110.82(18) |
| C(1E)-Cr(1)-C(1C)-O(1C) | 84(5) | C(4)-C(5)-O(5)-C(10) | -137.83(16) |
| C(1B)-Cr(1)-C(1C)-O(1C) | -23(5) | C(6)-C(5)-O(5)-C(10) | -12.0(2) |
| C(1D)-Cr(1)-C(1C)-O(1C) | -106(5) | O(9)-C(5)-C(6)-O(6) | -93.3(2) |
| C(1)-Cr(1)-C(1C)-O(1C) | -15(5) | O(5)-C(5)-C(6)-O(6) | 27.34(19) |
| C(1A)-Cr(1)-C(1D)-O(1D) | 11(3) | C(4)-C(5)-C(6)-O(6) | 148.16(19) |
| C(1E)-Cr(1)-C(1D)-O(1D) | -5(3) | O(9)-C(5)-C(6)-C(7) | 23.6(3) |
| C(1B)-Cr(1)-C(1D)-O(1D) | 103(3) | O(5)-C(5)-C(6)-C(7) | 144.23(17) |
| C(1C)-Cr(1)-C(1D)-O(1D) | -83(3) | C(4)-C(5)-C(6)-C(7) | -94.9(2) |
| C(1)-Cr(1)-C(1D)-O(1D) | -169(3) | C(7)-C(6)-O(6)-C(10) | -156.21(15) |
| C(1A)-Cr(1)-C(1E)-O(1E) | -15.9(17) | C(5)-C(6)-O(6)-C(10) | -33.49(19) |
| C(1B)-Cr(1)-C(1E)-O(1E) | -107.0(17) | O(6)-C(6)-C(7)-O(7) | -168.06(14) |
| C(1D)-Cr(1)-C(1E)-O(1E) | 0(2) | C(5)-C(6)-C(7)-O(7) | 78.0(2) |
| C(1C)-Cr(1)-C(1E)-O(1E) | 78.8(17) | O(6)-C(6)-C(7)-C(8) | 76.88(19) |
| C(1)-Cr(1)-C(1E)-O(1E) | 164.6(17) | C(5)-C(6)-C(7)-C(8) | -37.0(3) |
| C(4)-O(1)-C(1)-C(2) | -3.0(2) | C(6)-C(7)-O(7)-C(13) | -144.41(16) |
| C(4)-O(1)-C(1)-Cr(1) | 176.99(13) | C(8)-C(7)-O(7)-C(13) | -22.7(2) |
| C(1A)-Cr(1)-C(1)-O(1) | 18(2) | O(7)-C(7)-C(8)-O(8) | 1.0(2) |
| C(1E)-Cr(1)-C(1)-O(1) | -174.26(16) | C(6)-C(7)-C(8)-O(8) | 118.05(18) |
| C(1B)-Cr(1)-C(1)-O(1) | 93.27(16) | O(7)-C(7)-C(8)-C(9) | -115.90(19) |
| C(1D)-Cr(1)-C(1)-O(1) | 3.22(16) | C(6)-C(7)-C(8)-C(9) | 1.1(2) |
| C(1C)-Cr(1)-C(1)-O(1) | -85.95(16) | C(9)-C(8)-O(8)-C(13) | 140.64(18) |
| C(1A)-Cr(1)-C(1)-C(2) | -162(2) | C(7)-C(8)-O(8)-C(13) | 21.3(2) |
| C(1E)-Cr(1)-C(1)-C(2) | 5.7(2) | O(8)-C(8)-C(9)-O(9) | -66.2(2) |
| C(1B)-Cr(1)-C(1)-C(2) | -86.8(2) | C(7)-C(8)-C(9)-O(9) | 48.3(2) |
| C(1D)-Cr(1)-C(1)-C(2) | -176.8(2) | O(5)-C(5)-O(9)-C(9) | -88.7(2) |
| C(1C)-Cr(1)-C(1)-C(2) | 94.0(2) | C(4)-C(5)-O(9)-C(9) | 155.03(18) |
| O(1)-C(1)-C(2)-C(3) | 21.7(2) | C(6)-C(5)-O(9)-C(9) | 28.0(3) |
| Cr(1)-C(1)-C(2)-C(3) | -158.23(18) | C(8)-C(9)-O(9)-C(5) | -66.2(2) |
| O(1)-C(1)-C(2)-C(21) | 147.64(18) | C(6)-O(6)-C(10)-O(5) | 26.8(2) |
| Cr(1)-C(1)-C(2)-C(21) | -32.3(3) | C(6)-O(6)-C(10)-C(12) | 144.04(16) |
| O(1)-C(1)-C(2)-C(16) | -98.15(19) | C(6)-O(6)-C(10)-C(11) | -91.22(19) |
| Cr(1)-C(1)-C(2)-C(16) | 81.9(3) | C(5)-O(5)-C(10)-O(6) | -8.2(2) |
| C(1)-C(2)-C(3)-C(4) | -31.2(2) | C(5)-O(5)-C(10)-C(12) | -125.1(2) |
| C(21)-C(2)-C(3)-C(4) | -159.16(18) | C(5)-O(5)-C(10)-C(11) | 110.83(19) |
| C(16)-C(2)-C(3)-C(4) | 85.6(2) | C(8)-O(8)-C(13)-O(7) | -35.7(2) |

| | | | |
|---|---|---|---|
| C(8)-O(8)-C(13)-C(15) | -150.45(19) | C(16)-C(17)-C(18)-C(19) | -0.3(3) |
| C(8)-O(8)-C(13)-C(14) | 83.8(2) | C(17)-C(18)-C(19)-C(20) | 34.1(2) |
| C(7)-O(7)-C(13)-O(8) | 36.5(2) | C(17)-C(18)-C(19)-C(21) | -70.2(2) |
| C(7)-O(7)-C(13)-C(15) | 152.45(18) | C(18)-C(19)-C(20)-C(16) | -50.87(19) |
| C(7)-O(7)-C(13)-C(14) | -82.3(2) | C(21)-C(19)-C(20)-C(16) | 58.07(19) |
| C(3)-C(2)-C(16)-C(17) | 53.8(2) | C(17)-C(16)-C(20)-C(19) | 50.7(2) |
| C(1)-C(2)-C(16)-C(17) | 166.82(19) | C(2)-C(16)-C(20)-C(19) | -56.94(17) |
| C(21)-C(2)-C(16)-C(17) | -68.9(2) | C(3)-C(2)-C(21)-C(19) | -119.84(19) |
| C(3)-C(2)-C(16)-C(20) | 157.25(16) | C(1)-C(2)-C(21)-C(19) | 120.33(19) |
| C(1)-C(2)-C(16)-C(20) | -89.75(19) | C(16)-C(2)-C(21)-C(19) | 2.1(2) |
| C(21)-C(2)-C(16)-C(20) | 34.52(19) | C(18)-C(19)-C(21)-C(2) | 65.5(2) |
| C(20)-C(16)-C(17)-C(18) | -33.5(2) | C(20)-C(19)-C(21)-C(2) | -38.1(2) |
| C(2)-C(16)-C(17)-C(18) | 71.3(2) | | |

**Tabelle 6: Hydrogen bonds for *[66]a* [Å and °].**

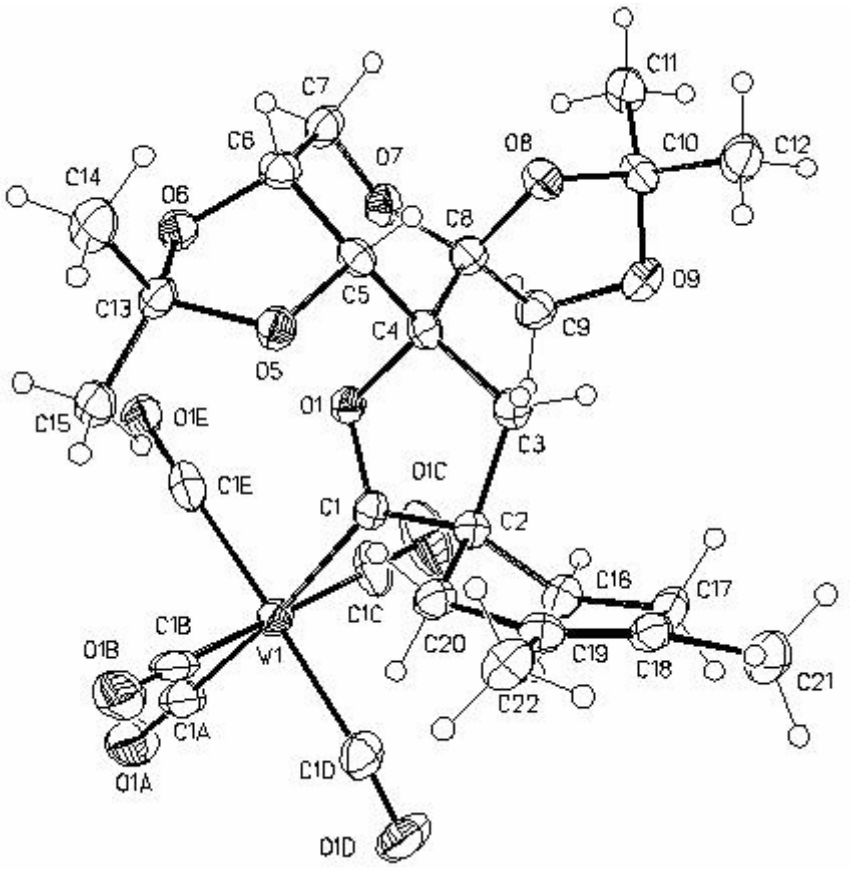
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(16)-H(16)...O(1A)[a] | 1.00 | 2.43 | 3.422(3) | 169.7 |
| C(14)-H(14A)...O(1C)[b] | 0.98 | 2.59 | 3.411(3) | 141.9 |
| C(20)-H(20B)...O(6)[c] | 0.99 | 2.57 | 3.497(3) | 156.1 |
| C(15)-H(15C)...O(6)[d] | 0.98 | 2.58 | 3.467(3) | 150.4 |
| C(9)-H(9A)...O(7)[d] | 0.99 | 2.46 | 3.350(3) | 149.9 |
| C(7)-H(7)...O(8)[e] | 1.00 | 2.68 | 3.254(2) | 116.7 |

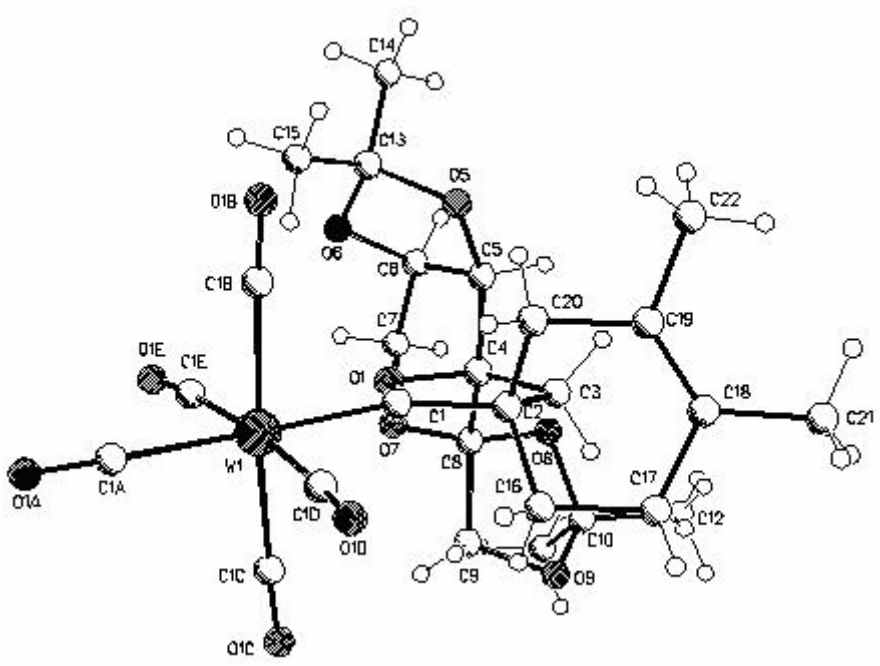
Symmetry transformations used to generate equivalent atoms:

[a] -x+1,y+1/2,-z+1/2 [b] x,y+1,z [c] -x+3/2,-y+1,z-1/2 [d] x+1/2,-y+3/2,-z+1

[e] x-1/2,-y+3/2,-z+1

**22. (*3R,5R,1´R,4´S,6´R*)-Pentacarbonyl{7,8,8´,8´,2´´,2´´-hexamethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,3-2-oxaspiro[4.5]dec-7-en-1-yliden}wolfram(0) *[70]a***



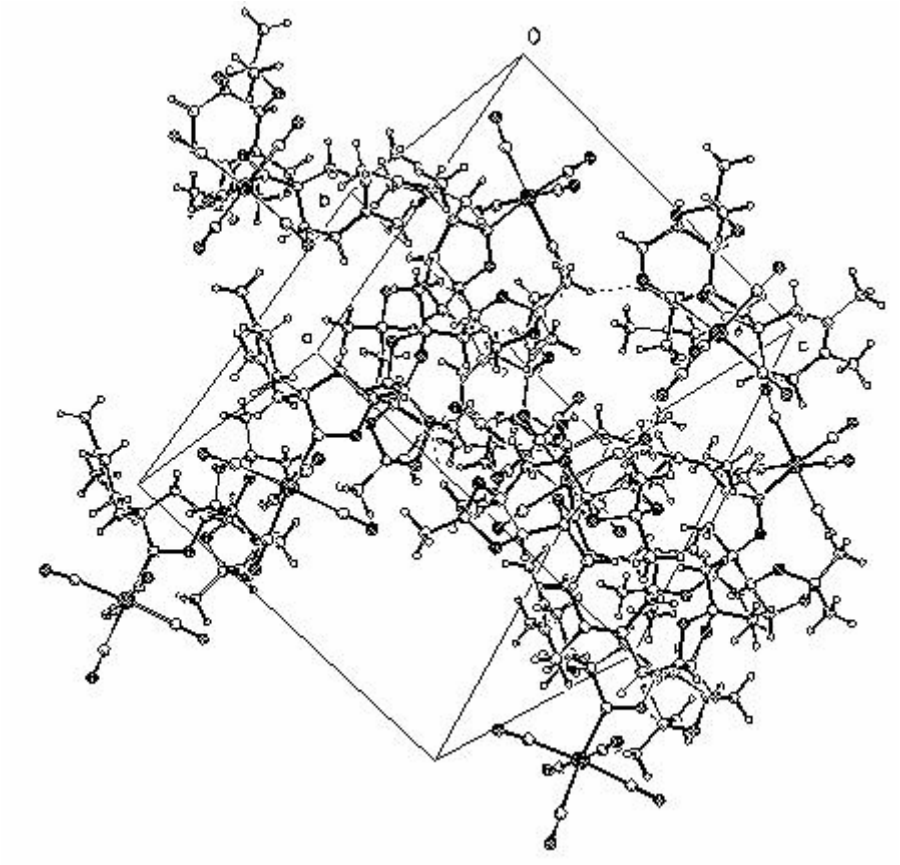

O
D
C

**Table 1: Crystal data and structure refinements for *[70]a***

| | |
|---|---|
| Identification code | ***[70]a*** |
| Empirical formula | $C_{27}H_{32}O_{11}W$ |
| Formula weight | 716.38 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | P2(1)2(1)2(1) (No.19) |
| Unit cell dimensions | a = 18.1394(4) Å α = 90° |
| | b = 10.7708(3) Å β = 90° |
| | c = 14.4879(3) Å γ = 90° |
| Volume | 2830.59(12) $Å^3$ |
| Z | 4 |
| Calculated density | 1.681 $mg/m^3$ |
| Absorption coefficient | 4.139 $mm^{-1}$ |
| F(000) | 1424 |
| Crystal size | 0.40 x 0.20 x 0.10 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.94 to 25.02° |
| Limiting indices | $-21 \le h \le 21$, $-12 \le k \le 12$, $-17 \le l \le 17$ |
| Reflections collected / unique | 20164 / 4990 [R(int) = 0.0783] |
| Completeness to Θ = 25.02 | 99.3 % |
| Absorption correction | Empirical from multiple refl |
| Max. and min. transmission | 0.41587 and 0.31681 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 4990 / 0 / 354 |
| Goodness-of-fit on $F^2$ | 1.065 |
| Final R indices [I>2σ(I)] | R1 = 0.0311, wR2 = 0.0670 |
| R indices (all data) | R1 = 0.0353, wR2 = 0.0684 |
| Absolute structure parameter | -0.022(9) |
| Largest diff. peak and hole | 2.212 and -1.131 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[70]a*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| W(1) | 1292(1) | 2449(1) | 2630(1) | 23(1) |
| C(1A) | 276(3) | 1815(6) | 3013(4) | 33(1) |
| O(1A) | -289(2) | 1460(4) | 3237(3) | 44(1) |
| C(1B) | 1729(3) | 673(6) | 2813(4) | 29(1) |
| O(1B) | 1972(2) | -282(4) | 2885(3) | 40(1) |
| C(1C) | 942(3) | 4221(6) | 2409(5) | 38(2) |
| O(1C) | 790(3) | 5233(4) | 2294(4) | 68(2) |
| C(1D) | 1040(3) | 1943(6) | 1307(4) | 34(1) |
| O(1D) | 848(2) | 1612(5) | 602(3) | 49(1) |
| C(1E) | 1444(3) | 3000(5) | 3979(4) | 32(1) |
| O(1E) | 1477(2) | 3345(4) | 4721(3) | 37(1) |
| O(1) | 2725(2) | 3760(3) | 3034(2) | 22(1) |
| C(1) | 2382(3) | 3197(5) | 2357(4) | 21(1) |
| C(2) | 2862(3) | 3232(5) | 1491(4) | 22(1) |
| C(3) | 3520(3) | 4066(5) | 1763(4) | 22(1) |
| C(4) | 3439(3) | 4342(5) | 2788(4) | 22(1) |
| C(5) | 4046(3) | 3779(5) | 3398(4) | 20(1) |
| O(5) | 3979(2) | 2450(4) | 3443(2) | 24(1) |
| C(6) | 4018(3) | 4174(5) | 4406(4) | 24(1) |
| O(6) | 3558(2) | 3262(3) | 4817(3) | 25(1) |
| C(7) | 3721(3) | 5477(5) | 4581(4) | 28(1) |
| O(7) | 3153(2) | 5851(3) | 3958(3) | 25(1) |
| C(8) | 3348(3) | 5713(5) | 3024(4) | 22(1) |
| O(8) | 4029(2) | 6284(3) | 2803(3) | 24(1) |
| C(9) | 2776(3) | 6446(5) | 2457(4) | 27(1) |
| O(9) | 3202(2) | 7164(3) | 1833(2) | 30(1) |
| C(10) | 3884(2) | 7437(5) | 2304(3) | 25(1) |
| C(11) | 3812(3) | 8520(5) | 2955(4) | 31(1) |
| C(12) | 4479(2) | 7579(6) | 1581(3) | 33(1) |
| C(13) | 3763(3) | 2114(5) | 4392(4) | 24(1) |
| C(14) | 4433(3) | 1545(5) | 4867(4) | 32(1) |
| C(15) | 3120(3) | 1264(5) | 4363(4) | 27(1) |
| C(16) | 2446(3) | 3748(5) | 649(4) | 26(1) |
| C(17) | 2962(3) | 3850(5) | -178(4) | 29(1) |
| C(18) | 3487(3) | 2794(5) | -282(4) | 24(1) |
| C(19) | 3542(3) | 1876(5) | 349(4) | 24(1) |
| C(20) | 3103(3) | 1916(5) | 1250(4) | 26(1) |
| C(21) | 3942(3) | 2865(5) | -1152(4) | 34(2) |
| C(22) | 4029(3) | 750(5) | 276(4) | 32(1) |

**Tabelle 3: Bond lengths [Å] for *[70]a*.**

| | | | |
|---|---|---|---|
| W(1)-C(1C) | 2.036(6) | O(5)-C(13) | 1.474(6) |
| W(1)-C(1A) | 2.042(6) | C(6)-O(6) | 1.419(6) |
| W(1)-C(1D) | 2.045(6) | C(6)-C(7) | 1.524(7) |
| W(1)-C(1E) | 2.060(6) | O(6)-C(13) | 1.430(6) |
| W(1)-C(1B) | 2.088(6) | C(7)-O(7) | 1.427(6) |
| W(1)-C(1) | 2.171(5) | O(7)-C(8) | 1.407(6) |
| C(1A)-O(1A) | 1.141(6) | C(8)-O(8) | 1.417(6) |
| C(1B)-O(1B) | 1.123(7) | C(8)-C(9) | 1.541(7) |
| C(1C)-O(1C) | 1.137(7) | O(8)-C(10) | 1.461(6) |
| C(1D)-O(1D) | 1.138(7) | C(9)-O(9) | 1.418(6) |
| C(1E)-O(1E) | 1.140(7) | O(9)-C(10) | 1.443(5) |
| O(1)-C(1) | 1.309(6) | C(10)-C(11) | 1.505(7) |
| O(1)-C(4) | 1.483(5) | C(10)-C(12) | 1.512(6) |
| C(1)-C(2) | 1.528(7) | C(13)-C(15) | 1.483(7) |
| C(2)-C(20) | 1.524(7) | C(13)-C(14) | 1.526(7) |
| C(2)-C(16) | 1.538(7) | C(16)-C(17) | 1.525(7) |
| C(2)-C(3) | 1.545(7) | C(17)-C(18) | 1.491(8) |
| C(3)-C(4) | 1.522(7) | C(18)-C(19) | 1.350(7) |
| C(4)-C(8) | 1.525(7) | C(18)-C(21) | 1.509(7) |
| C(4)-C(5) | 1.537(7) | C(19)-C(22) | 1.504(7) |
| C(5)-O(5) | 1.438(6) | C(19)-C(20) | 1.530(8) |
| C(5)-C(6) | 1.522(7) | | |

**Tabelle 4: Bond angles [°] for *[70]a*.**

| | | | |
|---|---|---|---|
| C(1C)-W(1)-C(1A) | 94.3(2) | C(6)-C(5)-C(4) | 114.6(4) |
| C(1C)-W(1)-C(1D) | 91.9(3) | C(5)-O(5)-C(13) | 108.0(4) |
| C(1A)-W(1)-C(1D) | 87.9(2) | O(6)-C(6)-C(5) | 103.2(4) |
| C(1C)-W(1)-C(1E) | 85.5(2) | O(6)-C(6)-C(7) | 111.1(4) |
| C(1A)-W(1)-C(1E) | 87.7(2) | C(5)-C(6)-C(7) | 115.4(4) |
| C(1D)-W(1)-C(1E) | 174.6(2) | C(6)-O(6)-C(13) | 105.4(4) |
| C(1C)-W(1)-C(1B) | 175.6(2) | O(7)-C(7)-C(6) | 114.2(4) |
| C(1A)-W(1)-C(1B) | 90.1(2) | C(8)-O(7)-C(7) | 113.4(4) |
| C(1D)-W(1)-C(1B) | 87.7(2) | O(7)-C(8)-O(8) | 113.0(4) |
| C(1E)-W(1)-C(1B) | 95.3(2) | O(7)-C(8)-C(4) | 110.2(4) |
| C(1C)-W(1)-C(1) | 84.7(2) | O(8)-C(8)-C(4) | 106.0(4) |
| C(1A)-W(1)-C(1) | 174.5(2) | O(7)-C(8)-C(9) | 106.8(4) |
| C(1D)-W(1)-C(1) | 97.6(2) | O(8)-C(8)-C(9) | 104.1(4) |
| C(1E)-W(1)-C(1) | 86.8(2) | C(4)-C(8)-C(9) | 116.7(4) |
| C(1B)-W(1)-C(1) | 90.99(19) | C(8)-O(8)-C(10) | 108.9(3) |
| O(1A)-C(1A)-W(1) | 179.2(5) | O(9)-C(9)-C(8) | 104.6(4) |
| O(1B)-C(1B)-W(1) | 178.0(6) | C(9)-O(9)-C(10) | 106.1(4) |
| O(1C)-C(1C)-W(1) | 175.8(5) | O(9)-C(10)-O(8) | 102.4(4) |
| O(1D)-C(1D)-W(1) | 174.2(5) | O(9)-C(10)-C(11) | 112.4(4) |
| O(1E)-C(1E)-W(1) | 174.9(5) | O(8)-C(10)-C(11) | 111.4(4) |
| C(1)-O(1)-C(4) | 115.5(4) | O(9)-C(10)-C(12) | 107.8(4) |
| O(1)-C(1)-C(2) | 109.5(4) | O(8)-C(10)-C(12) | 107.4(4) |
| O(1)-C(1)-W(1) | 117.8(3) | C(11)-C(10)-C(12) | 114.6(5) |
| C(2)-C(1)-W(1) | 132.6(4) | O(6)-C(13)-O(5) | 105.0(4) |
| C(20)-C(2)-C(1) | 109.2(4) | O(6)-C(13)-C(15) | 110.0(4) |
| C(20)-C(2)-C(16) | 107.2(5) | O(5)-C(13)-C(15) | 109.5(4) |
| C(1)-C(2)-C(16) | 112.4(4) | O(6)-C(13)-C(14) | 111.1(4) |
| C(20)-C(2)-C(3) | 112.1(4) | O(5)-C(13)-C(14) | 107.8(4) |
| C(1)-C(2)-C(3) | 104.2(4) | C(15)-C(13)-C(14) | 113.0(5) |
| C(16)-C(2)-C(3) | 111.8(4) | C(17)-C(16)-C(2) | 110.4(4) |
| C(4)-C(3)-C(2) | 106.7(4) | C(18)-C(17)-C(16) | 114.6(5) |
| O(1)-C(4)-C(3) | 103.7(4) | C(19)-C(18)-C(17) | 122.6(5) |
| O(1)-C(4)-C(8) | 105.1(4) | C(19)-C(18)-C(21) | 124.2(5) |
| C(3)-C(4)-C(8) | 114.7(4) | C(17)-C(18)-C(21) | 113.2(5) |
| O(1)-C(4)-C(5) | 108.7(4) | C(18)-C(19)-C(22) | 125.9(5) |
| C(3)-C(4)-C(5) | 114.5(4) | C(18)-C(19)-C(20) | 121.2(5) |
| C(8)-C(4)-C(5) | 109.3(4) | C(22)-C(19)-C(20) | 112.9(5) |
| O(5)-C(5)-C(6) | 103.4(4) | C(2)-C(20)-C(19) | 111.8(4) |
| O(5)-C(5)-C(4) | 111.0(4) | | |

**Tabelle 5: Torsion angles [°] for *[70]a.***

| | | | |
|---|---|---|---|
| C(1C)-W(1)-C(1A)-O(1A) | -108(38) | C(1)-O(1)-C(4)-C(3) | 0.6(5) |
| C(1D)-W(1)-C(1A)-O(1A) | 161(38) | C(1)-O(1)-C(4)-C(8) | 121.4(4) |
| C(1E)-W(1)-C(1A)-O(1A) | -22(38) | C(1)-O(1)-C(4)-C(5) | -121.7(5) |
| C(1B)-W(1)-C(1A)-O(1A) | 73(38) | C(2)-C(3)-C(4)-O(1) | -4.5(5) |
| C(1)-W(1)-C(1A)-O(1A) | -28(40) | C(2)-C(3)-C(4)-C(8) | -118.6(5) |
| C(1C)-W(1)-C(1B)-O(1B) | -47(15) | C(2)-C(3)-C(4)-C(5) | 113.8(4) |
| C(1A)-W(1)-C(1B)-O(1B) | 125(13) | O(1)-C(4)-C(5)-O(5) | 45.4(5) |
| C(1D)-W(1)-C(1B)-O(1B) | 37(13) | C(3)-C(4)-C(5)-O(5) | -70.1(5) |
| C(1E)-W(1)-C(1B)-O(1B) | -148(13) | C(8)-C(4)-C(5)-O(5) | 159.6(4) |
| C(1)-W(1)-C(1B)-O(1B) | -61(13) | O(1)-C(4)-C(5)-C(6) | -71.3(5) |
| C(1A)-W(1)-C(1C)-O(1C) | 157(9) | C(3)-C(4)-C(5)-C(6) | 173.3(4) |
| C(1D)-W(1)-C(1C)-O(1C) | -115(9) | C(8)-C(4)-C(5)-C(6) | 43.0(6) |
| C(1E)-W(1)-C(1C)-O(1C) | 70(9) | C(6)-C(5)-O(5)-C(13) | 11.5(5) |
| C(1B)-W(1)-C(1C)-O(1C) | -31(11) | C(4)-C(5)-O(5)-C(13) | -111.9(4) |
| C(1)-W(1)-C(1C)-O(1C) | -18(9) | O(5)-C(5)-C(6)-O(6) | -31.1(5) |
| C(1C)-W(1)-C(1D)-O(1D) | -104(6) | C(4)-C(5)-C(6)-O(6) | 89.9(5) |
| C(1A)-W(1)-C(1D)-O(1D) | -10(6) | O(5)-C(5)-C(6)-C(7) | -152.5(4) |
| C(1E)-W(1)-C(1D)-O(1D) | -44(7) | C(4)-C(5)-C(6)-C(7) | -31.6(6) |
| C(1B)-W(1)-C(1D)-O(1D) | 80(6) | C(5)-C(6)-O(6)-C(13) | 39.5(5) |
| C(1)-W(1)-C(1D)-O(1D) | 171(6) | C(7)-C(6)-O(6)-C(13) | 163.8(4) |
| C(1C)-W(1)-C(1E)-O(1E) | 46(6) | O(6)-C(6)-C(7)-O(7) | -82.5(6) |
| C(1A)-W(1)-C(1E)-O(1E) | -49(6) | C(5)-C(6)-C(7)-O(7) | 34.6(6) |
| C(1D)-W(1)-C(1E)-O(1E) | -15(7) | C(6)-C(7)-O(7)-C(8) | -52.7(6) |
| C(1B)-W(1)-C(1E)-O(1E) | -139(6) | C(7)-O(7)-C(8)-O(8) | -52.1(6) |
| C(1)-W(1)-C(1E)-O(1E) | 131(6) | C(7)-O(7)-C(8)-C(4) | 66.3(5) |
| C(4)-O(1)-C(1)-C(2) | 3.8(6) | C(7)-O(7)-C(8)-C(9) | -166.0(4) |
| C(4)-O(1)-C(1)-W(1) | -175.1(3) | O(1)-C(4)-C(8)-O(7) | 56.8(5) |
| C(1C)-W(1)-C(1)-O(1) | 81.8(4) | C(3)-C(4)-C(8)-O(7) | 170.0(4) |
| C(1A)-W(1)-C(1)-O(1) | 2(2) | C(5)-C(4)-C(8)-O(7) | -59.8(5) |
| C(1D)-W(1)-C(1)-O(1) | 173.0(4) | O(1)-C(4)-C(8)-O(8) | 179.4(4) |
| C(1E)-W(1)-C(1)-O(1) | -4.0(4) | C(3)-C(4)-C(8)-O(8) | -67.4(5) |
| C(1B)-W(1)-C(1)-O(1) | -99.2(4) | C(5)-C(4)-C(8)-O(8) | 62.8(5) |
| C(1C)-W(1)-C(1)-C(2) | -96.8(5) | O(1)-C(4)-C(8)-C(9) | -65.2(6) |
| C(1A)-W(1)-C(1)-C(2) | -176.7(19) | C(3)-C(4)-C(8)-C(9) | 48.0(6) |
| C(1D)-W(1)-C(1)-C(2) | -5.6(5) | C(5)-C(4)-C(8)-C(9) | 178.2(4) |
| C(1E)-W(1)-C(1)-C(2) | 177.4(5) | O(7)-C(8)-O(8)-C(10) | -104.3(5) |
| C(1B)-W(1)-C(1)-C(2) | 82.1(5) | C(4)-C(8)-O(8)-C(10) | 135.0(4) |
| O(1)-C(1)-C(2)-C(20) | 113.6(5) | C(9)-C(8)-O(8)-C(10) | 11.3(5) |
| W(1)-C(1)-C(2)-C(20) | -67.7(6) | O(7)-C(8)-C(9)-O(9) | 132.0(4) |
| O(1)-C(1)-C(2)-C(16) | -127.6(4) | O(8)-C(8)-C(9)-O(9) | 12.2(5) |
| W(1)-C(1)-C(2)-C(16) | 51.1(6) | C(4)-C(8)-C(9)-O(9) | -104.3(5) |
| O(1)-C(1)-C(2)-C(3) | -6.3(6) | C(8)-C(9)-O(9)-C(10) | -31.3(5) |
| W(1)-C(1)-C(2)-C(3) | 172.4(4) | C(9)-O(9)-C(10)-O(8) | 37.9(5) |
| C(20)-C(2)-C(3)-C(4) | -111.4(5) | C(9)-O(9)-C(10)-C(11) | -81.7(5) |
| C(1)-C(2)-C(3)-C(4) | 6.5(5) | C(9)-O(9)-C(10)-C(12) | 151.1(5) |
| C(16)-C(2)-C(3)-C(4) | 128.2(4) | C(8)-O(8)-C(10)-O(9) | -30.1(5) |

| | | | |
|---|---|---|---|
| C(8)-O(8)-C(10)-C(11) | 90.2(5) | C(16)-C(17)-C(18)-C(19) | -5.3(8) |
| C(8)-O(8)-C(10)-C(12) | -143.6(4) | C(16)-C(17)-C(18)-C(21) | 174.8(5) |
| C(6)-O(6)-C(13)-O(5) | -32.5(5) | C(17)-C(18)-C(19)-C(22) | 176.8(5) |
| C(6)-O(6)-C(13)-C(15) | -150.2(4) | C(21)-C(18)-C(19)-C(22) | -3.3(8) |
| C(6)-O(6)-C(13)-C(14) | 83.8(5) | C(17)-C(18)-C(19)-C(20) | -4.5(8) |
| C(5)-O(5)-C(13)-O(6) | 12.0(5) | C(21)-C(18)-C(19)-C(20) | 175.4(5) |
| C(5)-O(5)-C(13)-C(15) | 130.0(4) | C(1)-C(2)-C(20)-C(19) | 176.1(4) |
| C(5)-O(5)-C(13)-C(14) | -106.6(4) | C(16)-C(2)-C(20)-C(19) | 54.1(6) |
| C(20)-C(2)-C(16)-C(17) | -64.0(6) | C(3)-C(2)-C(20)-C(19) | -69.0(6) |
| C(1)-C(2)-C(16)-C(17) | 176.0(4) | C(18)-C(19)-C(20)-C(2) | -21.3(8) |
| C(3)-C(2)-C(16)-C(17) | 59.2(6) | C(22)-C(19)-C(20)-C(2) | 157.6(5) |
| C(2)-C(16)-C(17)-C(18) | 39.9(6) | | |

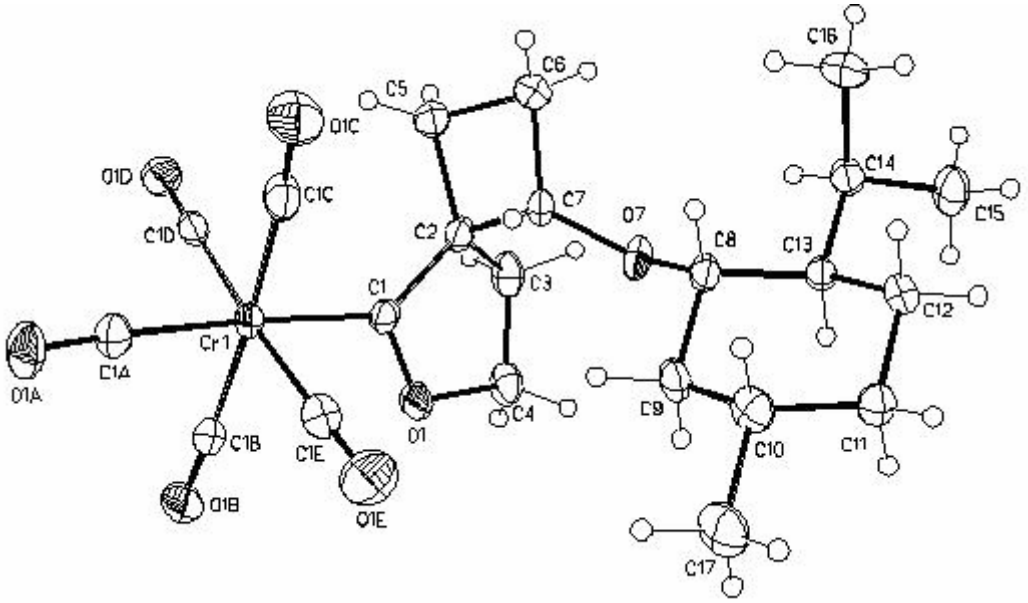
**Tabelle 6: Hydrogen bonds for *[70]a* [Å and °].**

| **D-H...A** | **d(D-H)** | **d(H...A)** | **d(D...A)** | **<(DHA)** |
|---|---|---|---|---|
| C(15)-H(15C)...O(7)[a] | 0.98 | 2.52 | 3.383(6) | 146.2 |

Symmetry transformations used to generate equivalent atoms:

[a] -x+1/2,y-1/2,-z+1

**23. (*1S,4R,1´S,2´R,5´S*)-Pentacarbonyl{1-(5´-methyl-2´-methylethylcyclohexyloxy)-6-oxaspiro[3.4]oct-5-yliden}chrom(0) *[75]a***



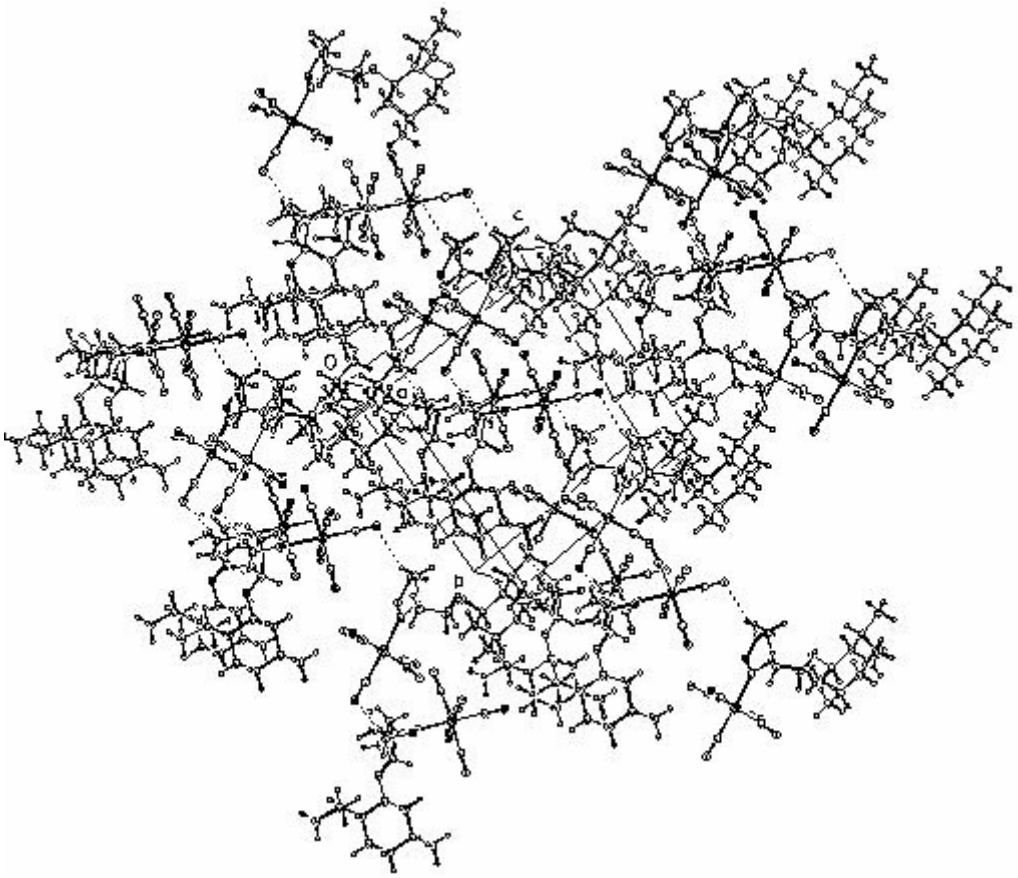



**Table 1: Crystal data and structure refinements for *[75]a***

| | |
|---|---|
| Identification code | ***[75]a*** |
| Empirical formula | $C_{22}H_{28}CrO_7$ |
| Formula weight | 456.44 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Monoclinic |
| Space group | P2(1) (No.4) |
| Unit cell dimensions | a = 7.1754(1) Å α = 90° |
| | b = 12.7609(2) Å β = 92.825(1)° |
| | c = 12.5296(2) Å γ = 90° |
| Volume | 1145.87(3) $Å^3$ |
| Z | 2 |
| Calculated density | 1.323 $mg/m^3$ |
| Absorption coefficient | 0.537 $mm^{-1}$ |
| F(000) | 480 |
| Crystal size | 0.50 x 0.40 x 0.20 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.84 to 27.48° |
| Limiting indices | $-9 \le h \le 9$, $-16 \le k \le 16$, $-16 \le l \le 16$ |
| Reflections collected / unique | 15693 / 5173 [R(int) = 0.0362] |
| Completeness to Θ = 27.48 | 99.1 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 5173 / 1 / 271 |
| Goodness-of-fit on $F^2$ | 1.052 |
| Final R indices [I>2σ(I)] | R1 = 0.0244, wR2 = 0.0607 |
| R indices (all data) | R1 = 0.0269, wR2 = 0.0617 |
| Absolute structure parameter | -0.005(10) |
| Largest diff. peak and hole | 0.220 and -0.199 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[75]a*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 8388(1) | 4824(1) | 5536(1) | 20(1) |
| C(1A) | 8801(2) | 5434(1) | 6890(1) | 26(1) |
| O(1A) | 9127(2) | 5812(1) | 7713(1) | 37(1) |
| C(1B) | 10672(2) | 4077(1) | 5810(1) | 26(1) |
| O(1B) | 12029(2) | 3653(1) | 6013(1) | 42(1) |
| C(1C) | 6127(2) | 5591(1) | 5368(1) | 29(1) |
| O(1C) | 4777(2) | 6075(1) | 5343(1) | 45(1) |
| C(1D) | 7009(2) | 3673(1) | 6087(1) | 24(1) |
| O(1D) | 6178(2) | 2993(1) | 6411(1) | 31(1) |
| C(1E) | 9712(2) | 5920(1) | 4880(1) | 30(1) |
| O(1E) | 10506(2) | 6569(1) | 4463(1) | 50(1) |
| O(1) | 9696(1) | 3815(1) | 3640(1) | 27(1) |
| C(1) | 8160(2) | 4125(1) | 4085(1) | 20(1) |
| C(2) | 6542(2) | 3885(1) | 3323(1) | 21(1) |
| C(3) | 7275(2) | 3013(1) | 2613(1) | 28(1) |
| C(4) | 9319(2) | 3295(1) | 2593(1) | 28(1) |
| C(5) | 4523(2) | 3746(2) | 3679(1) | 30(1) |
| C(6) | 3811(2) | 4475(2) | 2762(1) | 35(1) |
| C(7) | 5832(2) | 4871(2) | 2665(1) | 22(1) |
| O(7) | 6523(1) | 4928(1) | 1632(1) | 25(1) |
| C(8) | 6562(2) | 5972(1) | 1189(1) | 20(1) |
| C(9) | 8378(2) | 6511(1) | 1546(1) | 25(1) |
| C(10) | 8536(2) | 7606(1) | 1069(1) | 26(1) |
| C(11) | 8263(2) | 7550(1) | -151(1) | 23(1) |
| C(12) | 6455(2) | 6995(1) | -495(1) | 21(1) |
| C(13) | 6359(2) | 5884(1) | -27(1) | 18(1) |
| C(14) | 4635(2) | 5244(1) | -429(1) | 23(1) |
| C(15) | 4647(2) | 5034(1) | -1627(1) | 34(1) |
| C(16) | 2791(2) | 5746(2) | -146(1) | 35(1) |
| C(17) | 10397(2) | 8120(2) | 1400(1) | 38(1) |

**Tabelle 3: Bond lengths [Å] for *[75]a*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1A) | 1.8775(16) | C(2)-C(7) | 1.575(2) |
| Cr(1)-C(1C) | 1.8975(16) | C(3)-C(4) | 1.511(2) |
| Cr(1)-C(1E) | 1.8994(17) | C(5)-C(6) | 1.546(2) |
| Cr(1)-C(1B) | 1.9121(16) | C(6)-C(7) | 1.546(2) |
| Cr(1)-C(1D) | 1.9181(17) | C(7)-O(7) | 1.4111(15) |
| Cr(1)-C(1) | 2.0245(14) | O(7)-C(8) | 1.4431(19) |
| C(1A)-O(1A) | 1.1519(19) | C(8)-C(9) | 1.521(2) |
| C(1B)-O(1B) | 1.1323(19) | C(8)-C(13) | 1.5266(19) |
| C(1C)-O(1C) | 1.1474(19) | C(9)-C(10) | 1.527(2) |
| C(1D)-O(1D) | 1.1394(19) | C(10)-C(17) | 1.527(2) |
| C(1E)-O(1E) | 1.147(2) | C(10)-C(11) | 1.533(2) |
| O(1)-C(1) | 1.3199(17) | C(11)-C(12) | 1.522(2) |
| O(1)-C(4) | 1.4832(18) | C(12)-C(13) | 1.538(2) |
| C(1)-C(2) | 1.498(2) | C(13)-C(14) | 1.546(2) |
| C(2)-C(3) | 1.533(2) | C(14)-C(15) | 1.526(2) |
| C(2)-C(5) | 1.5469(19) | C(14)-C(16) | 1.528(2) |

**Tabelle 4: Bond angles [°] for *[75]a*.**

| | | | |
|---|---|---|---|
| C(1A)-Cr(1)-C(1C) | 89.01(7) | C(1)-C(2)-C(7) | 112.95(12) |
| C(1A)-Cr(1)-C(1E) | 91.48(7) | C(3)-C(2)-C(7) | 112.79(12) |
| C(1C)-Cr(1)-C(1E) | 90.87(7) | C(5)-C(2)-C(7) | 87.67(11) |
| C(1A)-Cr(1)-C(1B) | 87.04(7) | C(4)-C(3)-C(2) | 101.43(12) |
| C(1C)-Cr(1)-C(1B) | 175.95(7) | O(1)-C(4)-C(3) | 103.09(11) |
| C(1E)-Cr(1)-C(1B) | 90.11(7) | C(6)-C(5)-C(2) | 89.78(11) |
| C(1A)-Cr(1)-C(1D) | 93.04(6) | C(5)-C(6)-C(7) | 88.75(11) |
| C(1C)-Cr(1)-C(1D) | 88.96(7) | O(7)-C(7)-C(6) | 117.49(12) |
| C(1E)-Cr(1)-C(1D) | 175.48(7) | O(7)-C(7)-C(2) | 113.74(12) |
| C(1B)-Cr(1)-C(1D) | 90.36(7) | C(6)-C(7)-C(2) | 88.75(12) |
| C(1A)-Cr(1)-C(1) | 175.33(6) | C(7)-O(7)-C(8) | 114.52(12) |
| C(1C)-Cr(1)-C(1) | 95.56(6) | O(7)-C(8)-C(9) | 109.70(12) |
| C(1E)-Cr(1)-C(1) | 87.47(6) | O(7)-C(8)-C(13) | 108.22(12) |
| C(1B)-Cr(1)-C(1) | 88.41(6) | C(9)-C(8)-C(13) | 111.43(11) |
| C(1D)-Cr(1)-C(1) | 88.05(6) | C(8)-C(9)-C(10) | 112.21(12) |
| O(1A)-C(1A)-Cr(1) | 177.36(13) | C(17)-C(10)-C(9) | 111.58(13) |
| O(1B)-C(1B)-Cr(1) | 177.12(14) | C(17)-C(10)-C(11) | 110.97(13) |
| O(1C)-C(1C)-Cr(1) | 175.04(15) | C(9)-C(10)-C(11) | 109.81(12) |
| O(1D)-C(1D)-Cr(1) | 179.46(14) | C(12)-C(11)-C(10) | 111.70(12) |
| O(1E)-C(1E)-Cr(1) | 178.43(16) | C(11)-C(12)-C(13) | 112.01(12) |
| C(1)-O(1)-C(4) | 112.83(11) | C(8)-C(13)-C(12) | 107.99(12) |
| O(1)-C(1)-C(2) | 107.86(12) | C(8)-C(13)-C(14) | 113.56(11) |
| O(1)-C(1)-Cr(1) | 118.62(10) | C(12)-C(13)-C(14) | 114.40(12) |
| C(2)-C(1)-Cr(1) | 133.49(10) | C(15)-C(14)-C(16) | 110.43(13) |
| C(1)-C(2)-C(3) | 103.93(12) | C(15)-C(14)-C(13) | 111.72(12) |
| C(1)-C(2)-C(5) | 123.16(12) | C(16)-C(14)-C(13) | 113.03(13) |
| C(3)-C(2)-C(5) | 115.95(13) | | |

**Tabelle 5: Torsion angles [°] for *[75]a.***

| | | | |
|---|---|---|---|
| C(1C)-Cr(1)-C(1A)-O(1A) | -139(3) | O(1)-C(1)-C(2)-C(7) | -100.15(14) |
| C(1E)-Cr(1)-C(1A)-O(1A) | -48(3) | Cr(1)-C(1)-C(2)-C(7) | 77.82(16) |
| C(1B)-Cr(1)-C(1A)-O(1A) | 42(3) | C(1)-C(2)-C(3)-C(4) | -31.85(15) |
| C(1D)-Cr(1)-C(1A)-O(1A) | 132(3) | C(5)-C(2)-C(3)-C(4) | -170.23(13) |
| C(1)-Cr(1)-C(1A)-O(1A) | 29(4) | C(7)-C(2)-C(3)-C(4) | 90.83(14) |
| C(1A)-Cr(1)-C(1B)-O(1B) | 3(3) | C(1)-O(1)-C(4)-C(3) | -17.87(17) |
| C(1C)-Cr(1)-C(1B)-O(1B) | -9(4) | C(2)-C(3)-C(4)-O(1) | 29.67(15) |
| C(1E)-Cr(1)-C(1B)-O(1B) | 95(3) | C(1)-C(2)-C(5)-C(6) | 132.90(15) |
| C(1D)-Cr(1)-C(1B)-O(1B) | -90(3) | C(3)-C(2)-C(5)-C(6) | -97.47(15) |
| C(1)-Cr(1)-C(1B)-O(1B) | -178(100) | C(7)-C(2)-C(5)-C(6) | 16.82(12) |
| C(1A)-Cr(1)-C(1C)-O(1C) | -9.7(18) | C(2)-C(5)-C(6)-C(7) | -17.14(13) |
| C(1E)-Cr(1)-C(1C)-O(1C) | -101.2(18) | C(5)-C(6)-C(7)-O(7) | 133.09(16) |
| C(1B)-Cr(1)-C(1C)-O(1C) | 3(3) | C(5)-C(6)-C(7)-C(2) | 16.83(12) |
| C(1D)-Cr(1)-C(1C)-O(1C) | 83.4(18) | C(1)-C(2)-C(7)-O(7) | 98.27(15) |
| C(1)-Cr(1)-C(1C)-O(1C) | 171.3(18) | C(3)-C(2)-C(7)-O(7) | -19.22(17) |
| C(1A)-Cr(1)-C(1D)-O(1D) | 110(16) | C(5)-C(2)-C(7)-O(7) | -136.48(13) |
| C(1C)-Cr(1)-C(1D)-O(1D) | 21(16) | C(1)-C(2)-C(7)-C(6) | -142.08(12) |
| C(1E)-Cr(1)-C(1D)-O(1D) | -67(16) | C(3)-C(2)-C(7)-C(6) | 100.43(13) |
| C(1B)-Cr(1)-C(1D)-O(1D) | -163(100) | C(5)-C(2)-C(7)-C(6) | -16.83(12) |
| C(1)-Cr(1)-C(1D)-O(1D) | -75(16) | C(6)-C(7)-O(7)-C(8) | 102.61(17) |
| C(1A)-Cr(1)-C(1E)-O(1E) | 176(100) | C(2)-C(7)-O(7)-C(8) | -155.74(12) |
| C(1C)-Cr(1)-C(1E)-O(1E) | -95(6) | C(7)-O(7)-C(8)-C(9) | 86.50(13) |
| C(1B)-Cr(1)-C(1E)-O(1E) | 89(6) | C(7)-O(7)-C(8)-C(13) | -151.74(10) |
| C(1D)-Cr(1)-C(1E)-O(1E) | -7(6) | O(7)-C(8)-C(9)-C(10) | 177.88(11) |
| C(1)-Cr(1)-C(1E)-O(1E) | 0(6) | C(13)-C(8)-C(9)-C(10) | 58.06(16) |
| C(4)-O(1)-C(1)-C(2) | -3.00(17) | C(8)-C(9)-C(10)-C(17) | -177.78(13) |
| C(4)-O(1)-C(1)-Cr(1) | 178.67(10) | C(8)-C(9)-C(10)-C(11) | -54.29(16) |
| C(1A)-Cr(1)-C(1)-O(1) | -13.2(9) | C(17)-C(10)-C(11)-C(12) | 177.40(13) |
| C(1C)-Cr(1)-C(1)-O(1) | 154.50(12) | C(9)-C(10)-C(11)-C(12) | 53.57(16) |
| C(1E)-Cr(1)-C(1)-O(1) | 63.87(12) | C(10)-C(11)-C(12)-C(13) | -56.96(16) |
| C(1B)-Cr(1)-C(1)-O(1) | -26.31(12) | O(7)-C(8)-C(13)-C(12) | -178.49(10) |
| C(1D)-Cr(1)-C(1)-O(1) | -116.73(12) | C(9)-C(8)-C(13)-C(12) | -57.80(15) |
| C(1A)-Cr(1)-C(1)-C(2) | 169.0(8) | O(7)-C(8)-C(13)-C(14) | 53.52(15) |
| C(1C)-Cr(1)-C(1)-C(2) | -23.30(15) | C(9)-C(8)-C(13)-C(14) | 174.22(12) |
| C(1E)-Cr(1)-C(1)-C(2) | -113.94(15) | C(11)-C(12)-C(13)-C(8) | 57.71(15) |
| C(1B)-Cr(1)-C(1)-C(2) | 155.88(15) | C(11)-C(12)-C(13)-C(14) | -174.79(11) |
| C(1D)-Cr(1)-C(1)-C(2) | 65.46(14) | C(8)-C(13)-C(14)-C(15) | -171.29(13) |
| O(1)-C(1)-C(2)-C(3) | 22.43(15) | C(12)-C(13)-C(14)-C(15) | 64.11(17) |
| Cr(1)-C(1)-C(2)-C(3) | -159.60(12) | C(8)-C(13)-C(14)-C(16) | 63.43(17) |
| O(1)-C(1)-C(2)-C(5) | 156.91(15) | C(12)-C(13)-C(14)-C(16) | -61.17(17) |
| Cr(1)-C(1)-C(2)-C(5) | -25.1(2) | | |

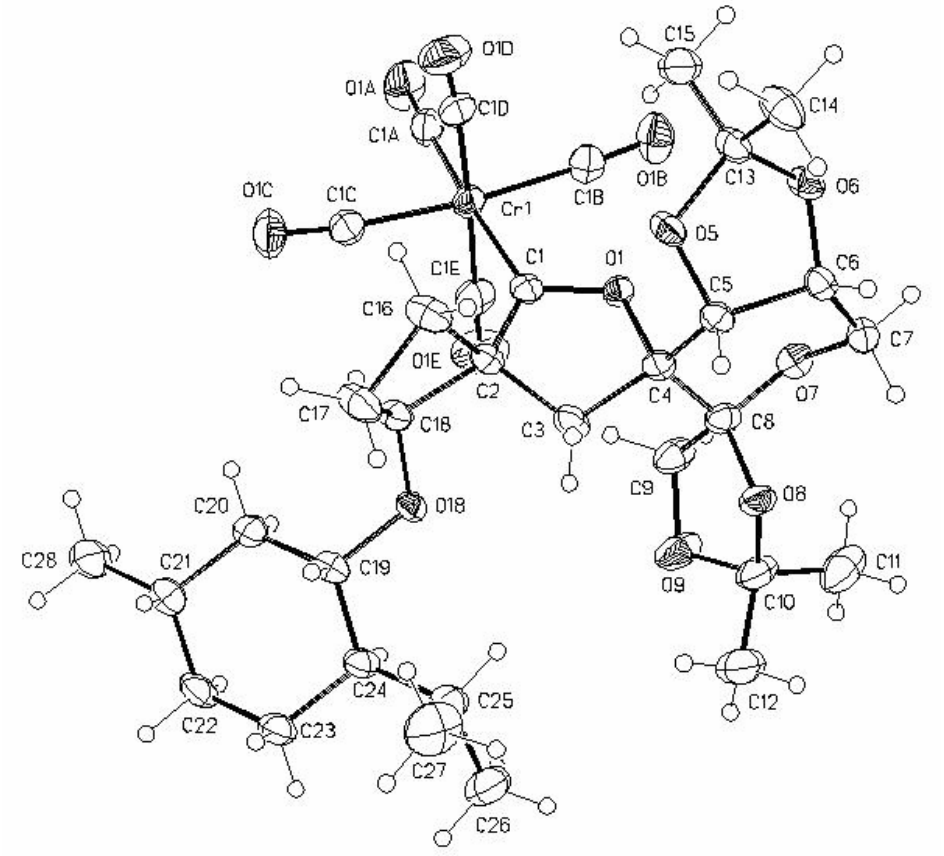
**Tabelle 6: Hydrogen bonds for *[75]a* [Å and °].**

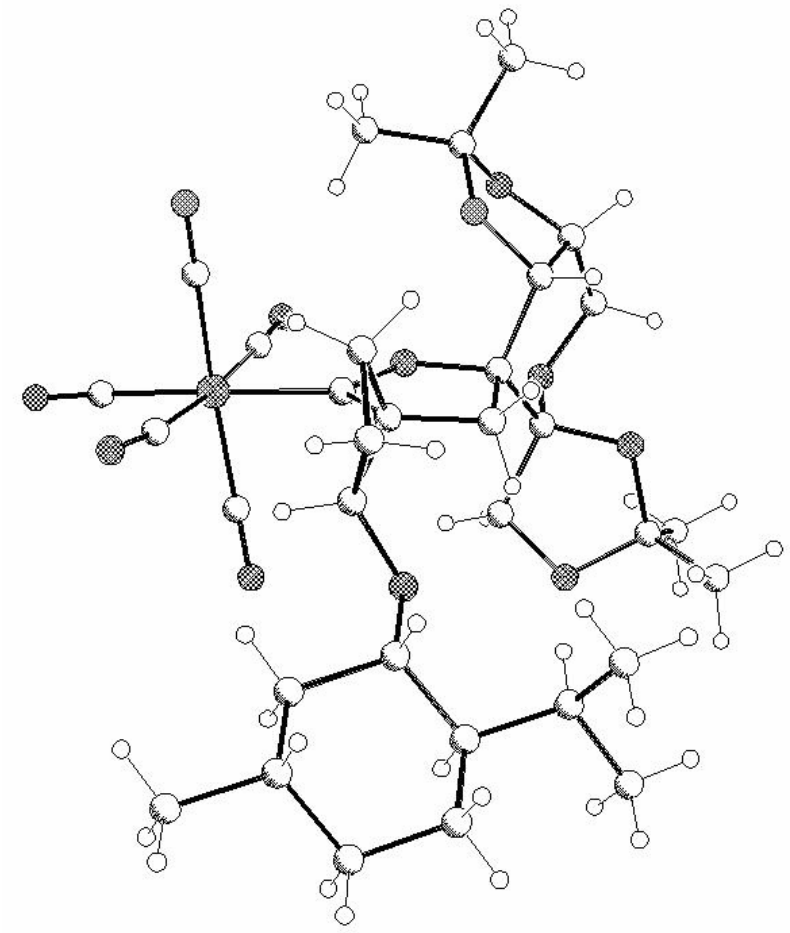
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(4)-H(4A)...O(1A)[a] | 0.99 | 2.45 | 3.387(2) | 158.7 |

Symmetry transformations used to generate equivalent atoms:

[a] -x+2,y-1/2,-z+1

**24. (*1S,4R,7R,1´R,4´S,6´R,1´´´S,2´´´R,5´´´S*)-Pentacarbonyl{1-(5´´´-methyl-2´´´-methylethylcyclohexyloxy)-8´,8´,2´´,2´´-tetramethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro-[3.4]oct]-5-yliden}chrom(0) *[77]b***





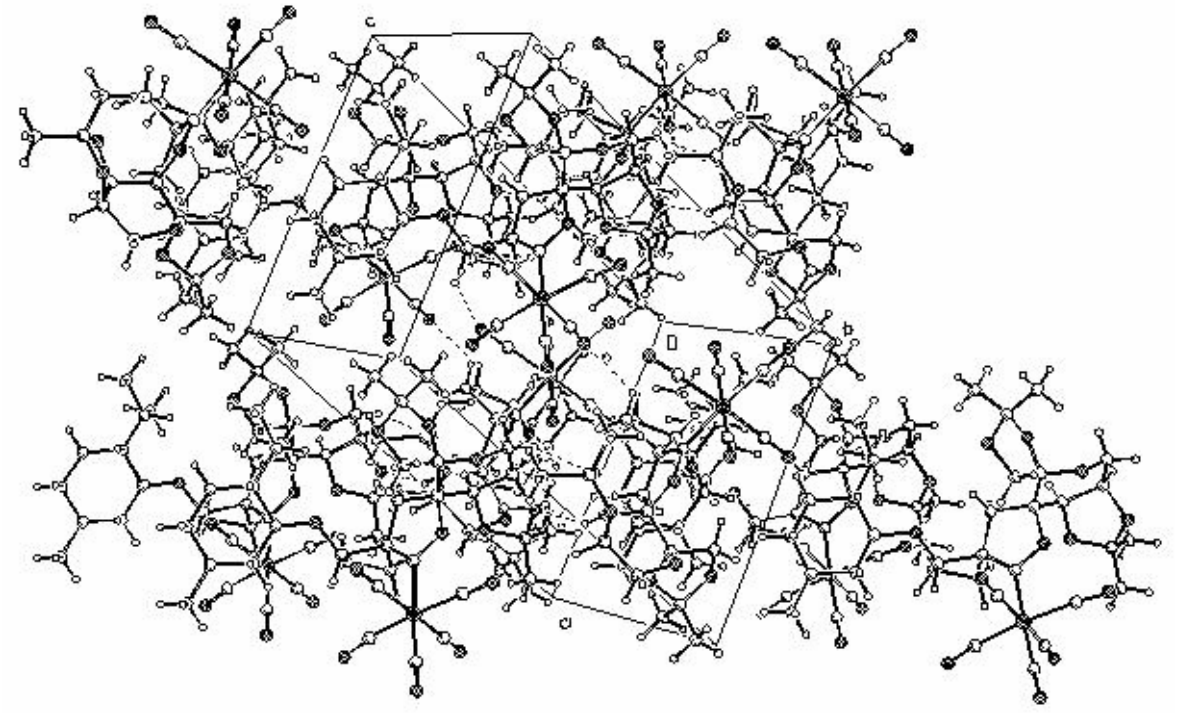


**Table 1: Crystal data and structure refinements for *[77]b***

| | |
|---|---|
| Identification code | ***[77]b*** |
| Empirical formula | $C_{33}H_{44}CrO_{12}$ |
| Formula weight | 684.68 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Monoclinic |
| Space group | P2(1) (No.4) |
| Unit cell dimensions | a = 11.2030(2) Å α = 90° |
| | b = 9.9232(3) Å β = 103.666(2)° |
| | c = 16.5231(5) Å γ = 90° |
| Volume | 1784.86(8) $Å^3$ |
| Z | 2 |
| Calculated density | 1.274 $mg/m^3$ |
| Absorption coefficient | 0.378 $mm^{-1}$ |
| F(000) | 724 |
| Crystal size | 0.35 x 0.20 x 0.15 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 3.23 to 27.44° |
| Limiting indices | $-14 \le h \le 14$, $-12 \le k \le 12$, $-21 \le l \le 21$ |
| Reflections collected / unique | 21513 / 7991 [R(int) = 0.0408] |
| Completeness to Θ = 27.44 | 98.6 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 7991 / 1 / 415 |
| Goodness-of-fit on $F^2$ | 0.911 |
| Final R indices [I>2σ(I)] | R1 = 0.0335, wR2 = 0.0549 |
| R indices (all data) | R1 = 0.0559, wR2 = 0.0593 |
| Absolute structure parameter | -0.014(11) |
| Largest diff. peak and hole | 0.247 and -0.238 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[77]b*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 4948(1) | 7699(1) | 6512(1) | 25(1) |
| C(1A) | 6244(2) | 8937(2) | 6538(2) | 35(1) |
| O(1A) | 7018(1) | 9698(2) | 6574(1) | 53(1) |
| C(1B) | 3826(2) | 9164(2) | 6206(1) | 29(1) |
| O(1B) | 3180(1) | 10057(2) | 6000(1) | 45(1) |
| C(1C) | 6128(2) | 6307(2) | 6728(1) | 32(1) |
| O(1C) | 6899(1) | 5515(2) | 6795(1) | 47(1) |
| C(1D) | 4817(2) | 7203(2) | 5387(1) | 32(1) |
| O(1D) | 4761(1) | 6804(2) | 4727(1) | 48(1) |
| C(1E) | 5050(2) | 7989(2) | 7664(2) | 36(1) |
| O(1E) | 5097(2) | 8174(2) | 8350(1) | 60(1) |
| O(1) | 2378(1) | 7067(1) | 6350(1) | 20(1) |
| C(1) | 3482(2) | 6513(2) | 6521(1) | 20(1) |
| C(2) | 3340(2) | 5036(2) | 6679(1) | 21(1) |
| C(3) | 2027(2) | 4861(2) | 6785(1) | 25(1) |
| C(4) | 1365(2) | 6144(2) | 6428(1) | 21(1) |
| C(5) | 488(2) | 5955(2) | 5571(1) | 21(1) |
| O(5) | 1164(1) | 5916(1) | 4938(1) | 25(1) |
| C(6) | -468(2) | 7066(2) | 5283(1) | 22(1) |
| O(6) | 105(1) | 7894(1) | 4776(1) | 24(1) |
| C(7) | -796(2) | 7874(2) | 5972(1) | 26(1) |
| O(7) | 209(1) | 8088(1) | 6668(1) | 26(1) |
| C(8) | 741(2) | 6880(2) | 7030(1) | 25(1) |
| O(8) | -145(1) | 5991(1) | 7222(1) | 28(1) |
| C(9) | 1592(2) | 7228(2) | 7885(1) | 35(1) |
| O(9) | 1157(1) | 6419(2) | 8460(1) | 50(1) |
| C(10) | -111(2) | 6163(3) | 8097(1) | 42(1) |
| C(11) | -904(2) | 7335(3) | 8234(1) | 52(1) |
| C(12) | -445(2) | 4838(3) | 8429(2) | 63(1) |
| C(13) | 750(2) | 6993(2) | 4351(1) | 26(1) |
| C(14) | -97(2) | 6421(2) | 3569(1) | 34(1) |
| C(15) | 1824(2) | 7737(2) | 4175(1) | 35(1) |
| C(16) | 3667(2) | 4085(2) | 6004(1) | 31(1) |
| C(17) | 4163(2) | 3048(2) | 6698(1) | 36(1) |
| C(18) | 4314(2) | 4223(2) | 7318(1) | 23(1) |
| O(18) | 4023(1) | 4011(1) | 8097(1) | 23(1) |
| C(19) | 4659(2) | 2867(2) | 8546(1) | 25(1) |
| C(20) | 6050(2) | 3074(2) | 8776(1) | 30(1) |
| C(21) | 6712(2) | 1879(2) | 9271(1) | 35(1) |
| C(22) | 6212(2) | 1662(2) | 10042(1) | 33(1) |
| C(23) | 4833(2) | 1450(2) | 9811(1) | 30(1) |
| C(24) | 4144(2) | 2637(2) | 9309(1) | 26(1) |

| | | | | |
|---|---|---|---|---|
| C(25) | 2739(2) | 2491(3) | 9094(1) | 37(1) |
| C(26) | 2234(2) | 2563(3) | 9879(1) | 52(1) |
| C(27) | 2279(2) | 1214(3) | 8602(2) | 58(1) |
| C(28) | 8105(2) | 2077(3) | 9481(1) | 54(1) |

**Tabelle 3: Bond lengths [Å] for *[77]b*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1C) | 1.887(2) | C(7)-O(7) | 1.422(2) |
| Cr(1)-C(1A) | 1.895(2) | O(7)-C(8) | 1.407(2) |
| Cr(1)-C(1D) | 1.895(2) | C(8)-O(8) | 1.418(2) |
| Cr(1)-C(1E) | 1.902(2) | C(8)-C(9) | 1.544(3) |
| Cr(1)-C(1B) | 1.910(2) | O(8)-C(10) | 1.448(2) |
| Cr(1)-C(1) | 2.0242(18) | C(9)-O(9) | 1.416(2) |
| C(1A)-O(1A) | 1.141(2) | O(9)-C(10) | 1.427(2) |
| C(1B)-O(1B) | 1.144(2) | C(10)-C(12) | 1.506(3) |
| C(1C)-O(1C) | 1.154(2) | C(10)-C(11) | 1.513(3) |
| C(1D)-O(1D) | 1.148(2) | C(13)-C(15) | 1.497(3) |
| C(1E)-O(1E) | 1.138(2) | C(13)-C(14) | 1.520(3) |
| O(1)-C(1) | 1.322(2) | C(16)-C(17) | 1.543(3) |
| O(1)-C(4) | 1.487(2) | C(17)-C(18) | 1.535(3) |
| C(1)-C(2) | 1.503(3) | C(18)-O(18) | 1.4172(19) |
| C(2)-C(3) | 1.531(2) | O(18)-C(19) | 1.448(2) |
| C(2)-C(18) | 1.552(3) | C(19)-C(24) | 1.523(2) |
| C(2)-C(16) | 1.569(2) | C(19)-C(20) | 1.528(2) |
| C(3)-C(4) | 1.520(3) | C(20)-C(21) | 1.529(3) |
| C(4)-C(8) | 1.531(2) | C(21)-C(22) | 1.523(3) |
| C(4)-C(5) | 1.532(2) | C(21)-C(28) | 1.529(3) |
| C(5)-O(5) | 1.4296(19) | C(22)-C(23) | 1.515(3) |
| C(5)-C(6) | 1.532(2) | C(23)-C(24) | 1.539(3) |
| O(5)-C(13) | 1.444(2) | C(24)-C(25) | 1.537(3) |
| C(6)-O(6) | 1.430(2) | C(25)-C(27) | 1.527(3) |
| C(6)-C(7) | 1.508(2) | C(25)-C(26) | 1.536(2) |
| O(6)-C(13) | 1.434(2) | | |

**Tabelle 4: Bond angles [°] for *[77]b*.**

| | | | |
|---|---|---|---|
| C(1C)-Cr(1)-C(1A) | 88.37(8) | O(7)-C(7)-C(6) | 113.65(14) |
| C(1C)-Cr(1)-C(1D) | 83.33(9) | C(8)-O(7)-C(7) | 113.05(14) |
| C(1A)-Cr(1)-C(1D) | 94.13(9) | O(7)-C(8)-O(8) | 112.16(15) |
| C(1C)-Cr(1)-C(1E) | 92.74(10) | O(7)-C(8)-C(4) | 110.01(14) |
| C(1A)-Cr(1)-C(1E) | 90.85(9) | O(8)-C(8)-C(4) | 107.40(16) |
| C(1D)-Cr(1)-C(1E) | 173.55(10) | O(7)-C(8)-C(9) | 107.86(16) |
| C(1C)-Cr(1)-C(1B) | 174.32(9) | O(8)-C(8)-C(9) | 104.39(15) |
| C(1A)-Cr(1)-C(1B) | 88.03(9) | C(4)-C(8)-C(9) | 114.99(16) |
| C(1D)-Cr(1)-C(1B) | 92.55(9) | C(8)-O(8)-C(10) | 107.15(15) |
| C(1E)-Cr(1)-C(1B) | 91.71(9) | O(9)-C(9)-C(8) | 104.53(15) |
| C(1C)-Cr(1)-C(1) | 96.10(8) | C(9)-O(9)-C(10) | 106.55(15) |
| C(1A)-Cr(1)-C(1) | 174.86(9) | O(9)-C(10)-O(8) | 103.26(14) |
| C(1D)-Cr(1)-C(1) | 88.93(8) | O(9)-C(10)-C(12) | 108.1(2) |
| C(1E)-Cr(1)-C(1) | 86.40(8) | O(8)-C(10)-C(12) | 108.1(2) |
| C(1B)-Cr(1)-C(1) | 87.70(8) | O(9)-C(10)-C(11) | 111.2(2) |
| O(1A)-C(1A)-Cr(1) | 178.1(2) | O(8)-C(10)-C(11) | 111.22(18) |
| O(1B)-C(1B)-Cr(1) | 177.21(18) | C(12)-C(10)-C(11) | 114.30(17) |
| O(1C)-C(1C)-Cr(1) | 172.85(18) | O(6)-C(13)-O(5) | 104.57(13) |
| O(1D)-C(1D)-Cr(1) | 174.71(19) | O(6)-C(13)-C(15) | 107.96(16) |
| O(1E)-C(1E)-Cr(1) | 179.0(2) | O(5)-C(13)-C(15) | 110.48(16) |
| C(1)-O(1)-C(4) | 114.82(14) | O(6)-C(13)-C(14) | 111.30(16) |
| O(1)-C(1)-C(2) | 108.10(15) | O(5)-C(13)-C(14) | 109.37(16) |
| O(1)-C(1)-Cr(1) | 118.46(13) | C(15)-C(13)-C(14) | 112.85(16) |
| C(2)-C(1)-Cr(1) | 133.44(14) | C(17)-C(16)-C(2) | 88.67(14) |
| C(1)-C(2)-C(3) | 105.90(15) | C(18)-C(17)-C(16) | 87.26(14) |
| C(1)-C(2)-C(18) | 122.56(16) | O(18)-C(18)-C(17) | 118.70(16) |
| C(3)-C(2)-C(18) | 113.83(14) | O(18)-C(18)-C(2) | 114.77(14) |
| C(1)-C(2)-C(16) | 114.26(14) | C(17)-C(18)-C(2) | 89.56(14) |
| C(3)-C(2)-C(16) | 113.85(16) | C(18)-O(18)-C(19) | 113.01(13) |
| C(18)-C(2)-C(16) | 85.74(14) | O(18)-C(19)-C(24) | 107.74(15) |
| C(4)-C(3)-C(2) | 104.77(14) | O(18)-C(19)-C(20) | 111.80(16) |
| O(1)-C(4)-C(3) | 103.70(13) | C(24)-C(19)-C(20) | 112.16(15) |
| O(1)-C(4)-C(8) | 103.71(14) | C(19)-C(20)-C(21) | 111.49(16) |
| C(3)-C(4)-C(8) | 113.81(15) | C(22)-C(21)-C(20) | 109.24(16) |
| O(1)-C(4)-C(5) | 109.35(13) | C(22)-C(21)-C(28) | 112.69(18) |
| C(3)-C(4)-C(5) | 113.81(16) | C(20)-C(21)-C(28) | 111.23(18) |
| C(8)-C(4)-C(5) | 111.55(14) | C(23)-C(22)-C(21) | 111.29(17) |
| O(5)-C(5)-C(4) | 110.19(14) | C(22)-C(23)-C(24) | 112.39(16) |
| O(5)-C(5)-C(6) | 104.12(13) | C(19)-C(24)-C(25) | 112.91(15) |
| C(4)-C(5)-C(6) | 116.59(15) | C(19)-C(24)-C(23) | 108.70(16) |
| C(5)-O(5)-C(13) | 109.30(13) | C(25)-C(24)-C(23) | 114.13(17) |
| O(6)-C(6)-C(7) | 111.11(15) | C(27)-C(25)-C(26) | 110.04(19) |
| O(6)-C(6)-C(5) | 102.63(13) | C(27)-C(25)-C(24) | 113.61(19) |
| C(7)-C(6)-C(5) | 115.21(15) | C(26)-C(25)-C(24) | 111.19(17) |
| C(6)-O(6)-C(13) | 106.04(13) | | |

**Tabelle 5: Torsion angles [°] for *[77]b.***

| | | | |
|---|---|---|---|
| C(1C)-Cr(1)-C(1A)-O(1A) | -112(6) | C(1)-O(1)-C(4)-C(3) | 6.06(19) |
| C(1D)-Cr(1)-C(1A)-O(1A) | 164(6) | C(1)-O(1)-C(4)-C(8) | 125.22(15) |
| C(1E)-Cr(1)-C(1A)-O(1A) | -20(6) | C(1)-O(1)-C(4)-C(5) | -115.67(16) |
| C(1B)-Cr(1)-C(1A)-O(1A) | 72(6) | C(2)-C(3)-C(4)-O(1) | -13.67(18) |
| C(1)-Cr(1)-C(1A)-O(1A) | 38(7) | C(2)-C(3)-C(4)-C(8) | -125.65(16) |
| C(1C)-Cr(1)-C(1B)-O(1B) | -5(5) | C(2)-C(3)-C(4)-C(5) | 105.03(16) |
| C(1A)-Cr(1)-C(1B)-O(1B) | 46(4) | O(1)-C(4)-C(5)-O(5) | 36.99(19) |
| C(1D)-Cr(1)-C(1B)-O(1B) | -48(4) | C(3)-C(4)-C(5)-O(5) | -78.44(17) |
| C(1E)-Cr(1)-C(1B)-O(1B) | 137(4) | C(8)-C(4)-C(5)-O(5) | 151.12(14) |
| C(1)-Cr(1)-C(1B)-O(1B) | -137(4) | O(1)-C(4)-C(5)-C(6) | -81.36(17) |
| C(1A)-Cr(1)-C(1C)-O(1C) | -47.2(17) | C(3)-C(4)-C(5)-C(6) | 163.22(14) |
| C(1D)-Cr(1)-C(1C)-O(1C) | 47.2(17) | C(8)-C(4)-C(5)-C(6) | 32.8(2) |
| C(1E)-Cr(1)-C(1C)-O(1C) | -138.0(17) | C(4)-C(5)-O(5)-C(13) | -120.56(16) |
| C(1B)-Cr(1)-C(1C)-O(1C) | 4(3) | C(6)-C(5)-O(5)-C(13) | 5.20(18) |
| C(1)-Cr(1)-C(1C)-O(1C) | 135.4(17) | O(5)-C(5)-C(6)-O(6) | -25.31(17) |
| C(1C)-Cr(1)-C(1D)-O(1D) | 29.9(19) | C(4)-C(5)-C(6)-O(6) | 96.28(16) |
| C(1A)-Cr(1)-C(1D)-O(1D) | 117.8(19) | O(5)-C(5)-C(6)-C(7) | -146.20(15) |
| C(1E)-Cr(1)-C(1D)-O(1D) | -23(2) | C(4)-C(5)-C(6)-C(7) | -24.6(2) |
| C(1B)-Cr(1)-C(1D)-O(1D) | -154.0(19) | C(7)-C(6)-O(6)-C(13) | 160.21(15) |
| C(1)-Cr(1)-C(1D)-O(1D) | -66.3(19) | C(5)-C(6)-O(6)-C(13) | 36.55(17) |
| C(1C)-Cr(1)-C(1E)-O(1E) | -170(13) | O(6)-C(6)-C(7)-O(7) | -80.88(19) |
| C(1A)-Cr(1)-C(1E)-O(1E) | 102(13) | C(5)-C(6)-C(7)-O(7) | 35.3(2) |
| C(1D)-Cr(1)-C(1E)-O(1E) | -117(13) | C(6)-C(7)-O(7)-C(8) | -58.3(2) |
| C(1B)-Cr(1)-C(1E)-O(1E) | 14(13) | C(7)-O(7)-C(8)-O(8) | -52.19(19) |
| C(1)-Cr(1)-C(1E)-O(1E) | -74(13) | C(7)-O(7)-C(8)-C(4) | 67.27(17) |
| C(4)-O(1)-C(1)-C(2) | 4.7(2) | C(7)-O(7)-C(8)-C(9) | -166.59(13) |
| C(4)-O(1)-C(1)-Cr(1) | -176.02(11) | O(1)-C(4)-C(8)-O(7) | 64.86(17) |
| C(1C)-Cr(1)-C(1)-O(1) | -176.78(14) | C(3)-C(4)-C(8)-O(7) | 176.83(14) |
| C(1A)-Cr(1)-C(1)-O(1) | 33.1(11) | C(5)-C(4)-C(8)-O(7) | -52.72(19) |
| C(1D)-Cr(1)-C(1)-O(1) | -93.60(14) | O(1)-C(4)-C(8)-O(8) | -172.81(14) |
| C(1E)-Cr(1)-C(1)-O(1) | 90.85(14) | C(3)-C(4)-C(8)-O(8) | -60.84(19) |
| C(1B)-Cr(1)-C(1)-O(1) | -1.01(14) | C(5)-C(4)-C(8)-O(8) | 69.60(19) |
| C(1C)-Cr(1)-C(1)-C(2) | 2.33(19) | O(1)-C(4)-C(8)-C(9) | -57.13(19) |
| C(1A)-Cr(1)-C(1)-C(2) | -147.8(10) | C(3)-C(4)-C(8)-C(9) | 54.8(2) |
| C(1D)-Cr(1)-C(1)-C(2) | 85.51(18) | C(5)-C(4)-C(8)-C(9) | -174.72(16) |
| C(1E)-Cr(1)-C(1)-C(2) | -90.04(18) | O(7)-C(8)-O(8)-C(10) | -97.95(18) |
| C(1B)-Cr(1)-C(1)-C(2) | 178.10(18) | C(4)-C(8)-O(8)-C(10) | 141.07(16) |
| O(1)-C(1)-C(2)-C(3) | -13.42(19) | C(9)-C(8)-O(8)-C(10) | 18.6(2) |
| Cr(1)-C(1)-C(2)-C(3) | 167.41(14) | O(7)-C(8)-C(9)-O(9) | 123.87(16) |
| O(1)-C(1)-C(2)-C(18) | -146.29(15) | O(8)-C(8)-C(9)-O(9) | 4.4(2) |
| Cr(1)-C(1)-C(2)-C(18) | 34.5(2) | C(4)-C(8)-C(9)-O(9) | -112.98(17) |
| O(1)-C(1)-C(2)-C(16) | 112.70(17) | C(8)-C(9)-O(9)-C(10) | -26.1(2) |
| Cr(1)-C(1)-C(2)-C(16) | -66.5(2) | C(9)-O(9)-C(10)-O(8) | 37.8(2) |
| C(1)-C(2)-C(3)-C(4) | 16.58(19) | C(9)-O(9)-C(10)-C(12) | 152.19(19) |
| C(18)-C(2)-C(3)-C(4) | 154.11(15) | C(9)-O(9)-C(10)-C(11) | -81.6(2) |
| C(16)-C(2)-C(3)-C(4) | -109.79(17) | C(8)-O(8)-C(10)-O(9) | -34.9(2) |

| | | | |
|---|---|---|---|
| C(8)-O(8)-C(10)-C(12) | -149.25(17) | C(2)-C(18)-O(18)-C(19) | 157.68(15) |
| C(8)-O(8)-C(10)-C(11) | 84.49(19) | C(18)-O(18)-C(19)-C(24) | -172.64(15) |
| C(6)-O(6)-C(13)-O(5) | -33.92(18) | C(18)-O(18)-C(19)-C(20) | 63.7(2) |
| C(6)-O(6)-C(13)-C(15) | -151.59(15) | O(18)-C(19)-C(20)-C(21) | 178.33(15) |
| C(6)-O(6)-C(13)-C(14) | 84.05(17) | C(24)-C(19)-C(20)-C(21) | 57.2(2) |
| C(5)-O(5)-C(13)-O(6) | 16.93(18) | C(19)-C(20)-C(21)-C(22) | -56.4(2) |
| C(5)-O(5)-C(13)-C(15) | 132.85(16) | C(19)-C(20)-C(21)-C(28) | 178.61(17) |
| C(5)-O(5)-C(13)-C(14) | -102.35(16) | C(20)-C(21)-C(22)-C(23) | 56.7(2) |
| C(1)-C(2)-C(16)-C(17) | 145.88(18) | C(28)-C(21)-C(22)-C(23) | -179.2(2) |
| C(3)-C(2)-C(16)-C(17) | -92.27(18) | C(21)-C(22)-C(23)-C(24) | -57.7(2) |
| C(18)-C(2)-C(16)-C(17) | 21.94(15) | O(18)-C(19)-C(24)-C(25) | 54.0(2) |
| C(2)-C(16)-C(17)-C(18) | -22.17(15) | C(20)-C(19)-C(24)-C(25) | 177.42(19) |
| C(16)-C(17)-C(18)-O(18) | 140.67(17) | O(18)-C(19)-C(24)-C(23) | -178.34(16) |
| C(16)-C(17)-C(18)-C(2) | 22.41(14) | C(20)-C(19)-C(24)-C(23) | -54.9(2) |
| C(1)-C(2)-C(18)-O(18) | 100.07(19) | C(22)-C(23)-C(24)-C(19) | 55.4(2) |
| C(3)-C(2)-C(18)-O(18) | -29.5(2) | C(22)-C(23)-C(24)-C(25) | -177.54(17) |
| C(16)-C(2)-C(18)-O(18) | -143.75(16) | C(19)-C(24)-C(25)-C(27) | 67.3(3) |
| C(1)-C(2)-C(18)-C(17) | -138.24(17) | C(23)-C(24)-C(25)-C(27) | -57.5(2) |
| C(3)-C(2)-C(18)-C(17) | 92.17(17) | C(19)-C(24)-C(25)-C(26) | -167.9(2) |
| C(16)-C(2)-C(18)-C(17) | -22.05(14) | C(23)-C(24)-C(25)-C(26) | 67.3(3) |
| C(17)-C(18)-O(18)-C(19) | 53.6(2) | | |

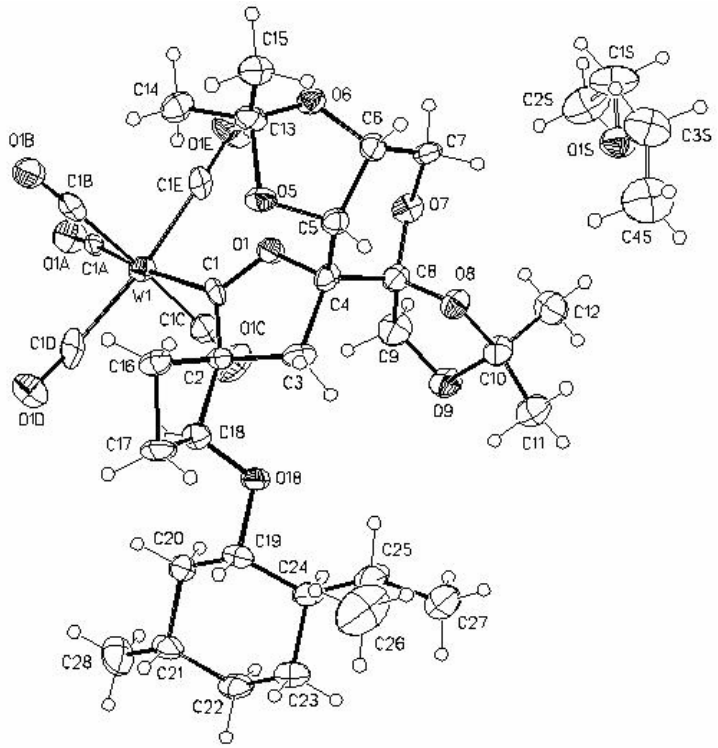
**Tabelle 6: Hydrogen bonds for *[77]b* [Å and °].**

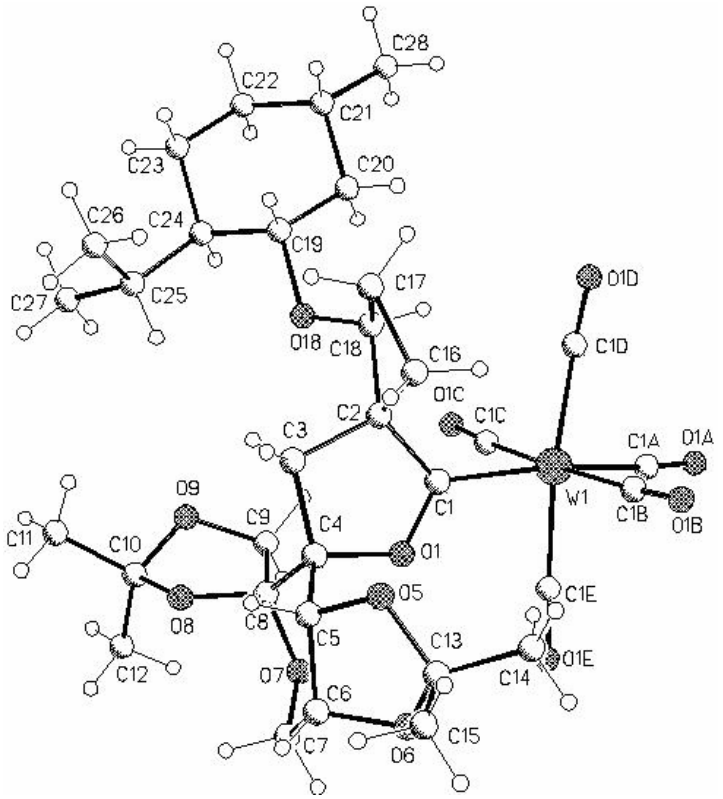
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(17)-H(17B)...O(1D)[a] | 0.99 | 2.52 | 3.141(2) | 120.2 |
| C(7)-H(7A)...O(5)[b] | 0.99 | 2.42 | 3.354(2) | 158.0 |
| C(5)-H(5)...O(6)[c] | 1.00 | 2.24 | 3.133(2) | 148.6 |

Symmetry transformations used to generate equivalent atoms:

[a] -x+1,y-1/2,-z+1 [b] -x,y+1/2,-z+1 [c] -x,y-1/2,-z+1

**25.** **(*1S,4R,7R,1´R,4´S,6´R,1´´´S,2´´´R,5´´´S*)-Pentacarbonyl{1-(5´´´-methyl-2´´´-methylethylcyclohexyloxy)-8´,8´,2´´,2´´-tetramethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro-[3.4]oct]-5-yliden}wolfram(0)** ***[79]b***



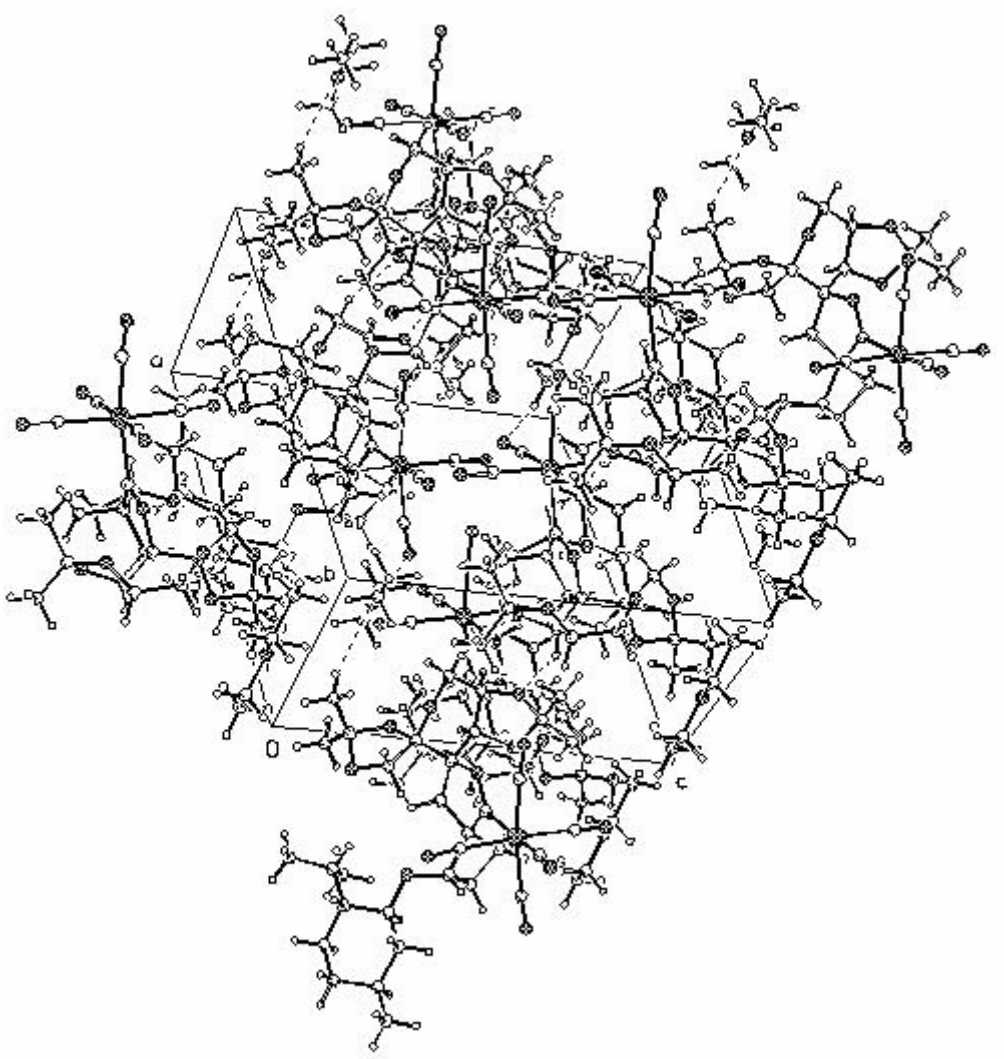

a
b
0
c

**Table 1: Crystal data and structure refinements for *[79]b***

| | |
|---|---|
| Identification code | ***[79]b*** |
| Empirical formula | $C_{33}H_{44}O_{12}W$ - $Et_2O$ |
| Formula weight | 890.65 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Monoclinic |
| Space group | P2(1) (No.4) |
| Unit cell dimensions | a = 14.3246(4) Å α = 90° |
| | b = 10.0658(3) Å β = 103.744(2)° |
| | c = 14.4063(4) Å γ = 90° |
| Volume | 2017.75(10) $Å^3$ |
| Z | 2 |
| Calculated density | 1.466 $mg/m^3$ |
| Absorption coefficient | 2.922 $mm^{-1}$ |
| F(000) | 908 |
| Crystal size | 0.30 x 0.20 x 0.10 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.71 to 25.00° |
| Limiting indices | $-16 \le h \le 17$, $-11 \le k \le 11$, $-17 \le l \le 17$ |
| Reflections collected / unique | 21095 / 7081 [R(int) = 0.0632] |
| Completeness to Θ = 25.00 | 99.8 % |
| Absorption correction | Empirical from multiple refl |
| Max. and min. transmission | 0.55351 and 0.50274 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 7081 / 64 / 460 |
| Goodness-of-fit on $F^2$ | 0.932 |
| Final R indices [I>2σ(I)] | R1 = 0.0348, wR2 = 0.0572 |
| R indices (all data) | R1 = 0.0510, wR2 = 0.0603 |
| Absolute structure parameter | -0.026(7) |
| Largest diff. peak and hole | 1.392 and -0.814 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[79]b*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| W(1) | 5996(1) | 4151(1) | 3964(1) | 25(1) |
| C(1A) | 5035(6) | 5623(9) | 3901(7) | 20(2) |
| O(1A) | 4444(4) | 6445(6) | 3787(4) | 43(2) |
| C(1B) | 6312(3) | 4183(14) | 5425(4) | 25(1) |
| O(1B) | 6467(2) | 4158(11) | 6245(2) | 37(1) |
| C(1C) | 5592(3) | 4172(15) | 2502(4) | 33(1) |
| O(1C) | 5323(3) | 4120(12) | 1690(3) | 57(1) |
| C(1D) | 4955(7) | 2821(12) | 4054(7) | 37(3) |
| O(1D) | 4390(4) | 2085(7) | 4163(4) | 44(2) |
| C(1E) | 6991(6) | 5602(9) | 4009(7) | 26(2) |
| O(1E) | 7596(3) | 6392(5) | 4117(4) | 48(1) |
| O(1) | 7954(3) | 3221(4) | 3782(3) | 26(1) |
| C(1) | 7134(6) | 2763(10) | 3851(6) | 22(2) |
| C(2) | 7114(4) | 1243(6) | 3732(4) | 24(2) |
| C(3) | 8031(4) | 906(6) | 3396(4) | 28(2) |
| C(4) | 8625(4) | 2211(6) | 3565(4) | 24(1) |
| C(5) | 9511(4) | 2120(6) | 4397(4) | 24(1) |
| O(5) | 9221(2) | 2080(4) | 5279(3) | 25(1) |
| C(6) | 10199(4) | 3293(6) | 4504(4) | 24(2) |
| O(6) | 9868(2) | 4111(8) | 5158(2) | 25(1) |
| C(7) | 10217(3) | 4027(12) | 3590(3) | 29(2) |
| O(7) | 9303(2) | 4038(8) | 2886(2) | 28(1) |
| C(8) | 8896(4) | 2770(7) | 2675(4) | 26(2) |
| O(8) | 9539(2) | 1852(4) | 2412(3) | 27(1) |
| C(9) | 8077(4) | 2884(7) | 1773(4) | 37(2) |
| O(9) | 8286(3) | 1921(4) | 1132(3) | 34(1) |
| C(10) | 9305(4) | 1749(7) | 1385(4) | 30(2) |
| C(11) | 9543(4) | 342(7) | 1129(4) | 40(2) |
| C(12) | 9798(4) | 2819(7) | 943(4) | 40(2) |
| C(13) | 9581(4) | 3248(6) | 5821(4) | 28(2) |
| C(14) | 8787(3) | 3906(9) | 6170(4) | 31(2) |
| C(15) | 10429(4) | 2859(6) | 6623(4) | 32(2) |
| C(16) | 7010(4) | 470(6) | 4634(4) | 27(2) |
| C(17) | 6404(4) | -580(7) | 3957(4) | 32(2) |
| C(18) | 6206(4) | 480(6) | 3173(4) | 25(2) |
| O(18) | 6235(2) | 164(4) | 2228(3) | 27(1) |
| C(19) | 5575(3) | -914(13) | 1816(3) | 26(1) |
| C(20) | 4531(4) | -453(6) | 1692(4) | 30(2) |
| C(21) | 3825(4) | -1531(6) | 1200(4) | 35(2) |
| C(22) | 4060(4) | -1967(7) | 268(4) | 36(2) |
| C(23) | 5109(4) | -2387(6) | 414(4) | 36(2) |
| C(24) | 5799(4) | -1269(6) | 865(4) | 32(2) |

| | | | | |
|---|---|---|---|---|
| C(25) | 6865(4) | -1599(7) | 945(5) | 45(2) |
| C(26) | 7196(5) | -2878(9) | 1497(6) | 80(3) |
| C(27) | 7096(5) | -1634(8) | -29(5) | 67(3) |
| C(28) | 2785(4) | -1026(14) | 1030(4) | 61(3) |
| C(2S) | 11193(5) | 6356(7) | 1720(5) | 60(2) |
| C(1S) | 12036(6) | 5639(8) | 2308(6) | 54(2) |
| O(1S) | 11884(3) | 4267(6) | 2162(3) | 42(1) |
| C(3S) | 12643(5) | 3467(7) | 2675(5) | 55(2) |
| C(4S) | 12388(5) | 2033(8) | 2491(6) | 62(3) |

**Tabelle 3: Bond lengths [Å] for *[79]b*.**

| | | | |
|---|---|---|---|
| W(1)-C(1A) | 2.009(9) | C(8)-O(8) | 1.419(6) |
| W(1)-C(1D) | 2.031(11) | C(8)-C(9) | 1.534(7) |
| W(1)-C(1E) | 2.031(9) | O(8)-C(10) | 1.441(6) |
| W(1)-C(1B) | 2.045(5) | C(9)-O(9) | 1.419(7) |
| W(1)-C(1C) | 2.047(5) | O(9)-C(10) | 1.428(6) |
| W(1)-C(1) | 2.183(9) | C(10)-C(12) | 1.508(8) |
| C(1A)-O(1A) | 1.167(10) | C(10)-C(11) | 1.522(8) |
| C(1B)-O(1B) | 1.149(5) | C(13)-C(14) | 1.502(8) |
| C(1C)-O(1C) | 1.143(5) | C(13)-C(15) | 1.516(8) |
| C(1D)-O(1D) | 1.136(11) | C(16)-C(17) | 1.556(8) |
| C(1E)-O(1E) | 1.159(10) | C(17)-C(18) | 1.531(8) |
| O(1)-C(1) | 1.287(9) | C(18)-O(18) | 1.408(6) |
| O(1)-C(4) | 1.483(6) | O(18)-C(19) | 1.469(10) |
| C(1)-C(2) | 1.539(11) | C(19)-C(24) | 1.521(7) |
| C(2)-C(3) | 1.542(7) | C(19)-C(20) | 1.536(7) |
| C(2)-C(16) | 1.552(8) | C(20)-C(21) | 1.536(7) |
| C(2)-C(18) | 1.559(8) | C(21)-C(22) | 1.525(8) |
| C(3)-C(4) | 1.552(8) | C(21)-C(28) | 1.538(8) |
| C(4)-C(5) | 1.527(7) | C(22)-C(23) | 1.527(8) |
| C(4)-C(8) | 1.532(8) | C(23)-C(24) | 1.537(7) |
| C(5)-O(5) | 1.428(6) | C(24)-C(25) | 1.540(7) |
| C(5)-C(6) | 1.522(7) | C(25)-C(27) | 1.517(8) |
| O(5)-C(13) | 1.438(6) | C(25)-C(26) | 1.528(10) |
| C(6)-O(6) | 1.415(7) | C(2S)-C(1S) | 1.487(8) |
| C(6)-C(7) | 1.516(9) | C(1S)-O(1S) | 1.406(7) |
| O(6)-C(13) | 1.421(8) | O(1S)-C(3S) | 1.412(6) |
| C(7)-O(7) | 1.453(5) | C(3S)-C(4S) | 1.496(8) |
| O(7)-C(8) | 1.406(9) | | |

**Tabelle 4: Bond angles [°] for *[79]b*.**

| | | | |
|---|---|---|---|
| C(1A)-W(1)-C(1D) | 89.0(2) | C(8)-O(7)-C(7) | 113.8(7) |
| C(1A)-W(1)-C(1E) | 86.5(4) | O(7)-C(8)-O(8) | 112.8(4) |
| C(1D)-W(1)-C(1E) | 172.9(4) | O(7)-C(8)-C(4) | 109.0(5) |
| C(1A)-W(1)-C(1B) | 91.0(4) | O(8)-C(8)-C(4) | 107.1(5) |
| C(1D)-W(1)-C(1B) | 86.0(4) | O(7)-C(8)-C(9) | 108.0(5) |
| C(1E)-W(1)-C(1B) | 88.5(4) | O(8)-C(8)-C(9) | 103.7(4) |
| C(1A)-W(1)-C(1C) | 85.6(4) | C(4)-C(8)-C(9) | 116.2(5) |
| C(1D)-W(1)-C(1C) | 92.3(4) | C(8)-O(8)-C(10) | 108.2(4) |
| C(1E)-W(1)-C(1C) | 92.9(4) | O(9)-C(9)-C(8) | 105.3(5) |
| C(1B)-W(1)-C(1C) | 176.2(3) | C(9)-O(9)-C(10) | 106.1(4) |
| C(1A)-W(1)-C(1) | 169.9(4) | O(9)-C(10)-O(8) | 103.2(4) |
| C(1D)-W(1)-C(1) | 99.0(4) | O(9)-C(10)-C(12) | 111.0(5) |
| C(1E)-W(1)-C(1) | 86.1(2) | O(8)-C(10)-C(12) | 111.3(5) |
| C(1B)-W(1)-C(1) | 95.7(3) | O(9)-C(10)-C(11) | 109.0(5) |
| C(1C)-W(1)-C(1) | 87.9(4) | O(8)-C(10)-C(11) | 107.7(5) |
| O(1A)-C(1A)-W(1) | 174.3(8) | C(12)-C(10)-C(11) | 114.0(5) |
| O(1B)-C(1B)-W(1) | 177.3(11) | O(6)-C(13)-O(5) | 105.0(5) |
| O(1C)-C(1C)-W(1) | 175.5(11) | O(6)-C(13)-C(14) | 108.6(5) |
| O(1D)-C(1D)-W(1) | 175.8(9) | O(5)-C(13)-C(14) | 109.8(5) |
| O(1E)-C(1E)-W(1) | 173.8(8) | O(6)-C(13)-C(15) | 111.3(5) |
| C(1)-O(1)-C(4) | 114.8(6) | O(5)-C(13)-C(15) | 108.7(5) |
| O(1)-C(1)-C(2) | 110.0(7) | C(14)-C(13)-C(15) | 113.1(5) |
| O(1)-C(1)-W(1) | 119.2(6) | C(2)-C(16)-C(17) | 88.0(4) |
| C(2)-C(1)-W(1) | 130.5(5) | C(18)-C(17)-C(16) | 88.1(5) |
| C(1)-C(2)-C(3) | 104.9(5) | O(18)-C(18)-C(17) | 120.8(5) |
| C(1)-C(2)-C(16) | 114.1(5) | O(18)-C(18)-C(2) | 113.5(4) |
| C(3)-C(2)-C(16) | 114.3(5) | C(17)-C(18)-C(2) | 88.7(4) |
| C(1)-C(2)-C(18) | 122.6(6) | C(18)-O(18)-C(19) | 113.2(4) |
| C(3)-C(2)-C(18) | 113.4(5) | O(18)-C(19)-C(24) | 106.5(5) |
| C(16)-C(2)-C(18) | 87.2(4) | O(18)-C(19)-C(20) | 109.8(8) |
| C(2)-C(3)-C(4) | 104.0(5) | C(24)-C(19)-C(20) | 112.0(4) |
| O(1)-C(4)-C(5) | 109.5(4) | C(19)-C(20)-C(21) | 110.9(6) |
| O(1)-C(4)-C(8) | 103.3(4) | C(22)-C(21)-C(20) | 110.6(5) |
| C(5)-C(4)-C(8) | 110.5(5) | C(22)-C(21)-C(28) | 111.3(5) |
| O(1)-C(4)-C(3) | 104.7(4) | C(20)-C(21)-C(28) | 110.3(6) |
| C(5)-C(4)-C(3) | 113.2(5) | C(21)-C(22)-C(23) | 112.0(5) |
| C(8)-C(4)-C(3) | 114.7(5) | C(22)-C(23)-C(24) | 111.6(5) |
| O(5)-C(5)-C(6) | 104.5(4) | C(19)-C(24)-C(23) | 107.5(5) |
| O(5)-C(5)-C(4) | 109.7(4) | C(19)-C(24)-C(25) | 113.6(5) |
| C(6)-C(5)-C(4) | 115.5(5) | C(23)-C(24)-C(25) | 113.6(5) |
| C(5)-O(5)-C(13) | 108.5(4) | C(27)-C(25)-C(26) | 110.6(6) |
| O(6)-C(6)-C(7) | 111.8(6) | C(27)-C(25)-C(24) | 111.5(6) |
| O(6)-C(6)-C(5) | 102.0(4) | C(26)-C(25)-C(24) | 113.6(6) |
| C(7)-C(6)-C(5) | 115.7(5) | O(1S)-C(1S)-C(2S) | 108.4(5) |
| C(6)-O(6)-C(13) | 106.7(6) | C(1S)-O(1S)-C(3S) | 114.1(6) |
| O(7)-C(7)-C(6) | 114.3(5) | O(1S)-C(3S)-C(4S) | 109.4(6) |

**Tabelle 5: Torsion angles [°] for *[79]b.***

| | | | |
|---|---|---|---|
| C(1D)-W(1)-C(1A)-O(1A) | -75(8) | C(1)-O(1)-C(4)-C(5) | -116.4(6) |
| C(1E)-W(1)-C(1A)-O(1A) | 110(8) | C(1)-O(1)-C(4)-C(8) | 125.7(6) |
| C(1B)-W(1)-C(1A)-O(1A) | -161(8) | C(1)-O(1)-C(4)-C(3) | 5.3(6) |
| C(1C)-W(1)-C(1A)-O(1A) | 17(8) | C(2)-C(3)-C(4)-O(1) | -10.8(5) |
| C(1)-W(1)-C(1A)-O(1A) | 67(9) | C(2)-C(3)-C(4)-C(5) | 108.5(5) |
| C(1A)-W(1)-C(1B)-O(1B) | 100(19) | C(2)-C(3)-C(4)-C(8) | -123.4(5) |
| C(1D)-W(1)-C(1B)-O(1B) | 11(19) | O(1)-C(4)-C(5)-O(5) | 45.6(6) |
| C(1E)-W(1)-C(1B)-O(1B) | -173(19) | C(8)-C(4)-C(5)-O(5) | 158.8(5) |
| C(1C)-W(1)-C(1B)-O(1B) | 75(26) | C(3)-C(4)-C(5)-O(5) | -70.9(6) |
| C(1)-W(1)-C(1B)-O(1B) | -88(19) | O(1)-C(4)-C(5)-C(6) | -72.2(6) |
| C(1A)-W(1)-C(1C)-O(1C) | -93(14) | C(8)-C(4)-C(5)-C(6) | 41.1(7) |
| C(1D)-W(1)-C(1C)-O(1C) | -4(14) | C(3)-C(4)-C(5)-C(6) | 171.3(4) |
| C(1E)-W(1)-C(1C)-O(1C) | -179(100) | C(6)-C(5)-O(5)-C(13) | 8.2(5) |
| C(1B)-W(1)-C(1C)-O(1C) | -68(21) | C(4)-C(5)-O(5)-C(13) | -116.2(5) |
| C(1)-W(1)-C(1C)-O(1C) | 95(14) | O(5)-C(5)-C(6)-O(6) | -27.3(5) |
| C(1A)-W(1)-C(1D)-O(1D) | -90(13) | C(4)-C(5)-C(6)-O(6) | 93.3(5) |
| C(1E)-W(1)-C(1D)-O(1D) | -39(15) | O(5)-C(5)-C(6)-C(7) | -148.9(5) |
| C(1B)-W(1)-C(1D)-O(1D) | 1(13) | C(4)-C(5)-C(6)-C(7) | -28.3(7) |
| C(1C)-W(1)-C(1D)-O(1D) | -176(13) | C(7)-C(6)-O(6)-C(13) | 161.2(4) |
| C(1)-W(1)-C(1D)-O(1D) | 96(13) | C(5)-C(6)-O(6)-C(13) | 36.9(5) |
| C(1A)-W(1)-C(1E)-O(1E) | 113(7) | O(6)-C(6)-C(7)-O(7) | -84.5(9) |
| C(1D)-W(1)-C(1E)-O(1E) | 62(9) | C(5)-C(6)-C(7)-O(7) | 31.8(10) |
| C(1B)-W(1)-C(1E)-O(1E) | 22(7) | C(6)-C(7)-O(7)-C(8) | -52.2(8) |
| C(1C)-W(1)-C(1E)-O(1E) | -162(7) | C(7)-O(7)-C(8)-O(8) | -52.9(6) |
| C(1)-W(1)-C(1E)-O(1E) | -74(7) | C(7)-O(7)-C(8)-C(4) | 65.9(5) |
| C(4)-O(1)-C(1)-C(2) | 2.9(8) | C(7)-O(7)-C(8)-C(9) | -166.9(4) |
| C(4)-O(1)-C(1)-W(1) | -171.3(4) | O(1)-C(4)-C(8)-O(7) | 58.0(5) |
| C(1A)-W(1)-C(1)-O(1) | 38(3) | C(5)-C(4)-C(8)-O(7) | -59.1(6) |
| C(1D)-W(1)-C(1)-O(1) | -179.6(7) | C(3)-C(4)-C(8)-O(7) | 171.4(4) |
| C(1E)-W(1)-C(1)-O(1) | -4.7(5) | O(1)-C(4)-C(8)-O(8) | -179.7(4) |
| C(1B)-W(1)-C(1)-O(1) | -92.7(7) | C(5)-C(4)-C(8)-O(8) | 63.2(6) |
| C(1C)-W(1)-C(1)-O(1) | 88.4(7) | C(3)-C(4)-C(8)-O(8) | -66.3(6) |
| C(1A)-W(1)-C(1)-C(2) | -134(2) | O(1)-C(4)-C(8)-C(9) | -64.4(6) |
| C(1D)-W(1)-C(1)-C(2) | 7.6(7) | C(5)-C(4)-C(8)-C(9) | 178.5(5) |
| C(1E)-W(1)-C(1)-C(2) | -177.5(9) | C(3)-C(4)-C(8)-C(9) | 49.0(7) |
| C(1B)-W(1)-C(1)-C(2) | 94.5(8) | O(7)-C(8)-O(8)-C(10) | -99.9(5) |
| C(1C)-W(1)-C(1)-C(2) | -84.4(8) | C(4)-C(8)-O(8)-C(10) | 140.2(5) |
| O(1)-C(1)-C(2)-C(3) | -9.9(8) | C(9)-C(8)-O(8)-C(10) | 16.8(6) |
| W(1)-C(1)-C(2)-C(3) | 163.4(5) | O(7)-C(8)-C(9)-O(9) | 126.0(5) |
| O(1)-C(1)-C(2)-C(16) | 116.0(6) | O(8)-C(8)-C(9)-O(9) | 6.1(6) |
| W(1)-C(1)-C(2)-C(16) | -70.7(8) | C(4)-C(8)-C(9)-O(9) | -111.2(6) |
| O(1)-C(1)-C(2)-C(18) | -141.2(6) | C(8)-C(9)-O(9)-C(10) | -26.7(6) |
| W(1)-C(1)-C(2)-C(18) | 32.2(10) | C(9)-O(9)-C(10)-O(8) | 37.0(6) |
| C(1)-C(2)-C(3)-C(4) | 12.3(6) | C(9)-O(9)-C(10)-C(12) | -82.3(6) |
| C(16)-C(2)-C(3)-C(4) | -113.4(6) | C(9)-O(9)-C(10)-C(11) | 151.3(5) |
| C(18)-C(2)-C(3)-C(4) | 148.6(5) | C(8)-O(8)-C(10)-O(9) | -33.4(6) |

| | | | |
|---|---|---|---|
| C(8)-O(8)-C(10)-C(12) | 85.6(5) | C(18)-O(18)-C(19)-C(24) | -171.8(5) |
| C(8)-O(8)-C(10)-C(11) | -148.7(5) | C(18)-O(18)-C(19)-C(20) | 66.7(6) |
| C(6)-O(6)-C(13)-O(5) | -32.7(5) | O(18)-C(19)-C(20)-C(21) | 176.3(5) |
| C(6)-O(6)-C(13)-C(14) | -150.2(4) | C(24)-C(19)-C(20)-C(21) | 58.1(9) |
| C(6)-O(6)-C(13)-C(15) | 84.7(5) | C(19)-C(20)-C(21)-C(22) | -53.4(7) |
| C(5)-O(5)-C(13)-O(6) | 14.0(5) | C(19)-C(20)-C(21)-C(28) | -176.8(5) |
| C(5)-O(5)-C(13)-C(14) | 130.6(5) | C(20)-C(21)-C(22)-C(23) | 53.4(7) |
| C(5)-O(5)-C(13)-C(15) | -105.1(5) | C(28)-C(21)-C(22)-C(23) | 176.3(7) |
| C(1)-C(2)-C(16)-C(17) | 145.6(6) | C(21)-C(22)-C(23)-C(24) | -57.1(7) |
| C(3)-C(2)-C(16)-C(17) | -93.7(5) | O(18)-C(19)-C(24)-C(23) | -179.4(5) |
| C(18)-C(2)-C(16)-C(17) | 20.9(4) | C(20)-C(19)-C(24)-C(23) | -59.3(9) |
| C(2)-C(16)-C(17)-C(18) | -21.3(4) | O(18)-C(19)-C(24)-C(25) | 54.0(9) |
| C(16)-C(17)-C(18)-O(18) | 137.9(5) | C(20)-C(19)-C(24)-C(25) | 174.1(6) |
| C(16)-C(17)-C(18)-C(2) | 21.2(4) | C(22)-C(23)-C(24)-C(19) | 58.5(7) |
| C(1)-C(2)-C(18)-O(18) | 98.4(7) | C(22)-C(23)-C(24)-C(25) | -174.9(5) |
| C(3)-C(2)-C(18)-O(18) | -29.2(7) | C(19)-C(24)-C(25)-C(27) | -167.0(7) |
| C(16)-C(2)-C(18)-O(18) | -144.5(5) | C(23)-C(24)-C(25)-C(27) | 69.7(7) |
| C(1)-C(2)-C(18)-C(17) | -138.3(6) | C(19)-C(24)-C(25)-C(26) | 67.2(9) |
| C(3)-C(2)-C(18)-C(17) | 94.1(5) | C(23)-C(24)-C(25)-C(26) | -56.1(7) |
| C(16)-C(2)-C(18)-C(17) | -21.2(4) | C(2S)-C(1S)-O(1S)-C(3S) | 179.3(6) |
| C(17)-C(18)-O(18)-C(19) | 57.0(7) | C(1S)-O(1S)-C(3S)-C(4S) | 178.1(6) |
| C(2)-C(18)-O(18)-C(19) | 160.2(6) | | |

**Tabelle 6: Hydrogen bonds for *[79]b* [Å and °].**

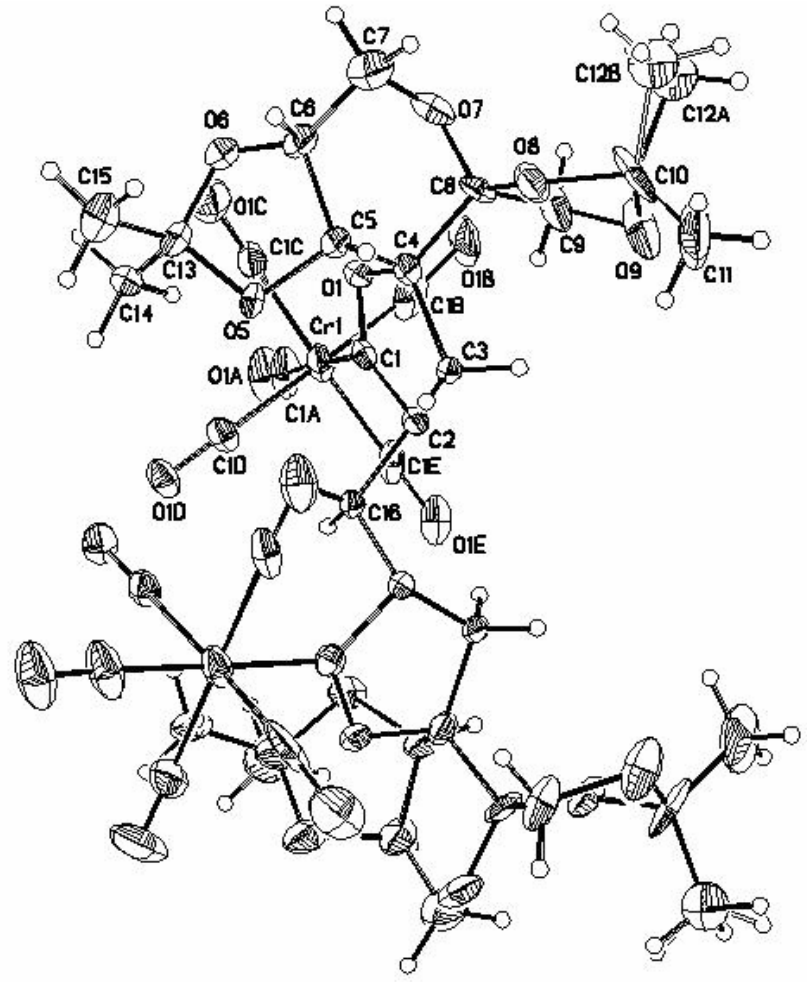
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(16)-H(16A)...O(1A)[a] | 0.99 | 2.62 | 3.567(8) | 161.3 |
| C(22)-H(22A)...O(1C)[b] | 0.99 | 2.65 | 3.334(8) | 126.6 |
| C(4S)-H(4S1)...O(1D)[c] | 0.98 | 2.56 | 3.278(9) | 130.5 |
| C(15)-H(15C)...O(1E)[d] | 0.98 | 2.60 | 3.571(7) | 171.1 |
| C(17)-H(17A)...O(1E)[e] | 0.99 | 2.55 | 3.474(9) | 155.7 |
| C(7)-H(7A)...O(5)[f] | 0.99 | 2.54 | 3.481(11) | 159.2 |
| C(5)-H(5)...O(6)[d] | 1.00 | 2.31 | 3.179(10) | 144.3 |

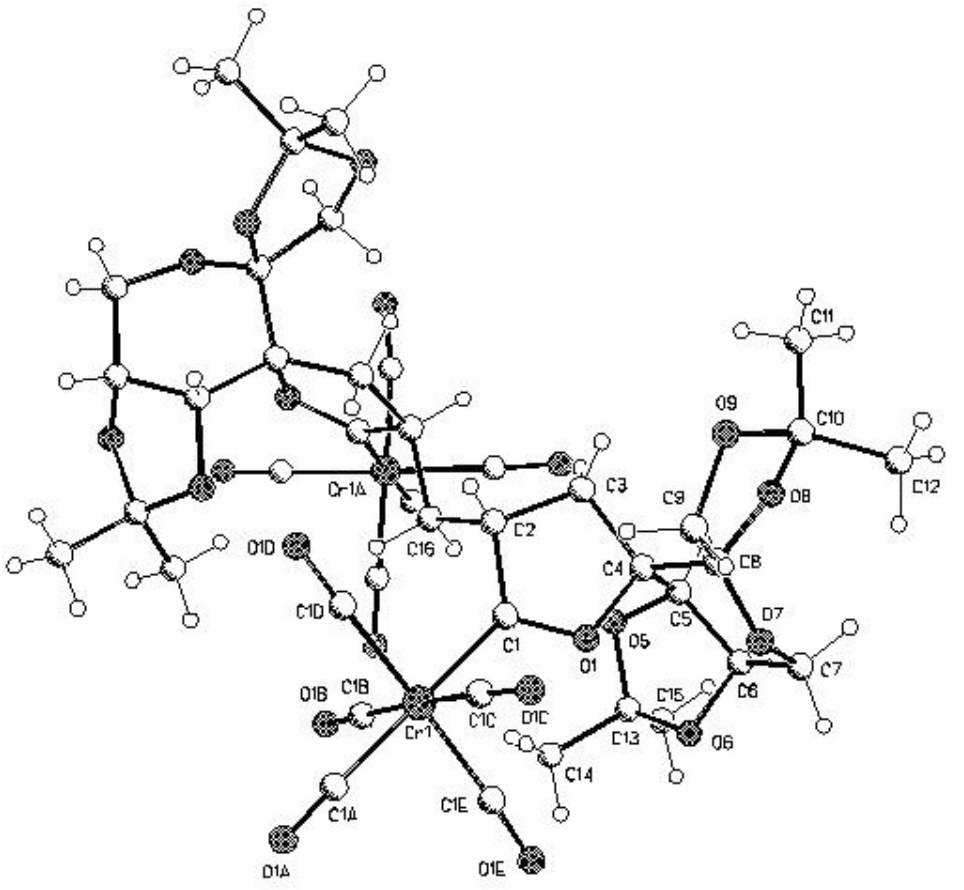
Symmetry transformations used to generate equivalent atoms:

[a] -x+1,y-1/2,-z+1 [b] -x+1,y-1/2,-z [c] x+1,y,z [d] -x+2,y-1/2,-z+1 [e] x,y-1,z

[f] -x+2,y+1/2,-z+1

**26. Bis-[pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-(*1´R,4´S,-5´R,6´R*)-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,4-(1R)-3-oxacyclo-pent]-2-yliden}chrom(0)]methan *[82]a***




C11
O9
C10
C12
O8
C9
C3
Cr1A
C2
C16
O1D
C4
O8
C1D
C5
O7
C1
O1
O5
C7
O1B
C1B
C15
O6
Cr1
C1C
O1C
C13
O6
C14
C1A
C1E
O1A
O1E

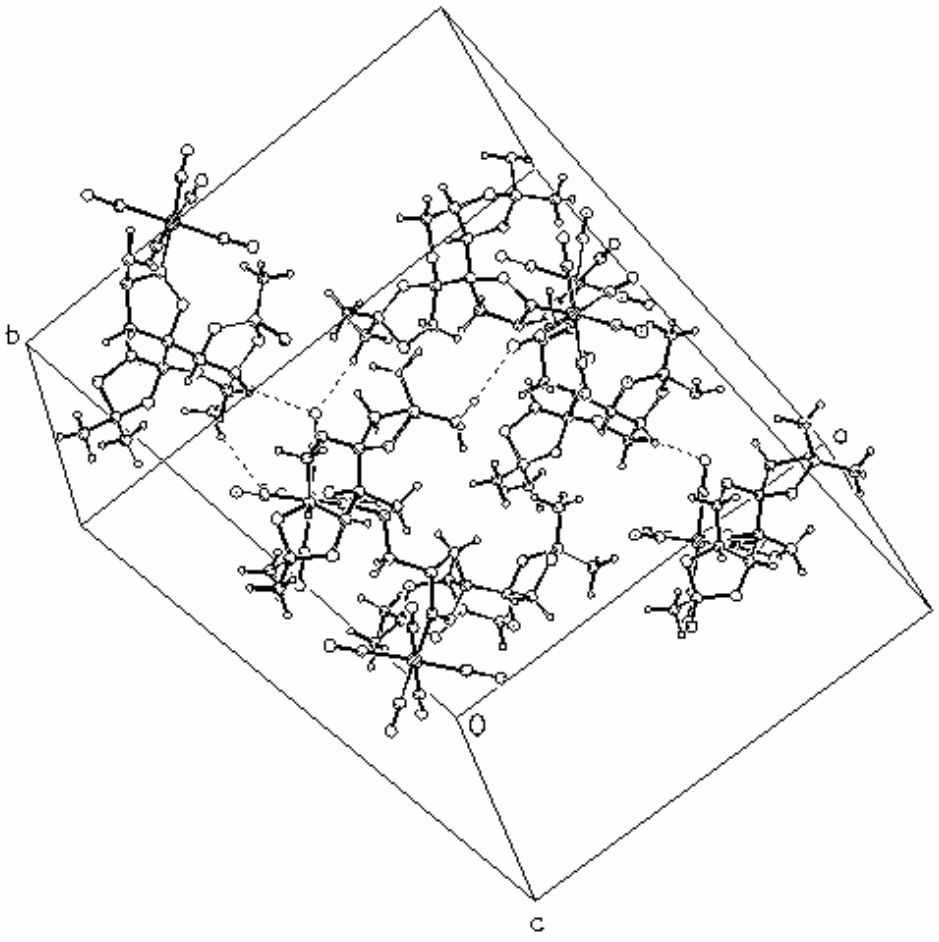

b
a
0
c

**Table 1: Crystal data and structure refinements for *[82]a***

| | |
|---|---|
| Identification code | ***[82]a*** |
| Empirical formula | $C_{41}H_{42}Cr_2O_{22}$ |
| Formula weight | 990.75 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoK$\alpha$) |
| Crystal system | Orthorhombic |
| Space group | C222(1) (No.20) |
| Unit cell dimensions | a = 17.9122(7) Å $\alpha$ = 90° |
| | b = 25.7447(13) Å $\beta$ = 90° |
| | c = 10.3164(5) Å $\gamma$ = 90° |
| Volume | 4757.3(4) $Å^3$ |
| Z | 4 |
| Calculated density | 1.383 $mg/m^3$ |
| Absorption coefficient | 0.536 $mm^{-1}$ |
| F(000) | 2048 |
| Crystal size | 0.40 x 0.30 x 0.30 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 3.01 to 27.48° |
| Limiting indices | $-15 \le h \le 23$, $-304 \le k \le 32$, $-11 \le l \le 13$ |
| Reflections collected / unique | 14693 / 4869 [R(int) = 0.0781] |
| Completeness to $\Theta$ = 27.48 | 93.1 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 4869 / 1 / 293 |
| Goodness-of-fit on $F^2$ | 0.938 |
| Final R indices [I>2$\sigma$(I)] | R1 = 0.0814, wR2 = 0.2087 |
| R indices (all data) | R1 = 0.1510, wR2 = 0.2338 |
| Absolute structure parameter | 0.01(5) |
| Largest diff. peak and hole | 1.195 and -0.399 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[82]a*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cr(1) | 8210(1) | 5712(1) | 2919(1) | 49(1) |
| C(1A) | 8788(4) | 5615(5) | 4451(9) | 73(3) |
| O(1A) | 9132(3) | 5573(4) | 5377(6) | 90(3) |
| C(1B) | 7520(6) | 6078(5) | 3872(10) | 88(4) |
| O(1B) | 7079(4) | 6376(3) | 4383(9) | 115(3) |
| C(1C) | 8718(4) | 6342(4) | 2646(10) | 67(3) |
| O(1C) | 9022(4) | 6709(3) | 2522(9) | 110(3) |
| C(1D) | 8949(4) | 5339(3) | 1967(7) | 43(2) |
| O(1D) | 9402(3) | 5113(2) | 1437(5) | 53(2) |
| C(1E) | 7688(4) | 5088(4) | 3304(7) | 50(2) |
| O(1E) | 7359(3) | 4715(3) | 3552(5) | 63(2) |
| O(1) | 7490(3) | 6271(2) | 790(5) | 47(1) |
| C(1) | 7568(4) | 5797(3) | 1351(7) | 39(2) |
| C(2) | 7061(3) | 5430(3) | 643(6) | 33(2) |
| C(3) | 6612(3) | 5744(3) | -342(7) | 38(2) |
| C(4) | 6920(4) | 6305(3) | -242(8) | 47(2) |
| C(5) | 7290(4) | 6488(3) | -1522(9) | 56(2) |
| O(5) | 7952(3) | 6202(2) | -1736(6) | 59(2) |
| C(6) | 7538(5) | 7040(3) | -1441(11) | 68(3) |
| O(6) | 8268(3) | 6996(2) | -954(7) | 74(2) |
| C(7) | 7061(6) | 7399(4) | -692(13) | 95(4) |
| O(7) | 6742(4) | 7173(2) | 487(7) | 76(2) |
| C(8) | 6382(4) | 6718(3) | 258(8) | 51(2) |
| O(8) | 5817(3) | 6747(2) | -739(6) | 59(2) |
| C(9) | 5909(4) | 6588(4) | 1474(9) | 72(3) |
| O(9) | 5157(4) | 6445(3) | 983(6) | 92(2) |
| C(10) | 5071(6) | 6760(5) | -150(9) | 79(4) |
| C(11) | 4549(6) | 6432(6) | -925(15) | 126(5) |
| C(12A) | 4777(11) | 7275(7) | 290(30) | 81(5) |
| C(12B) | 4853(16) | 7343(7) | -450(40) | 81(5) |
| C(13) | 8576(5) | 6548(4) | -1552(10) | 68(3) |
| C(14) | 9167(5) | 6320(4) | -746(11) | 70(3) |
| C(15) | 8907(6) | 6699(5) | -2918(12) | 94(4) |
| C(16) | 7532(5) | 5000 | 0 | 28(2) |

**Tabelle 3: Bond lengths [Å] for *[82]a*.**

| | | | |
|---|---|---|---|
| Cr(1)-C(1B) | 1.840(12) | C(5)-O(5) | 1.414(9) |
| Cr(1)-C(1C) | 1.882(11) | C(5)-C(6) | 1.489(12) |
| Cr(1)-C(1E) | 1.899(10) | O(5)-C(13) | 1.442(9) |
| Cr(1)-C(1A) | 1.906(9) | C(6)-O(6) | 1.406(11) |
| Cr(1)-C(1D) | 1.907(7) | C(6)-C(7) | 1.477(14) |
| Cr(1)-C(1) | 1.997(8) | O(6)-C(13) | 1.420(11) |
| C(1A)-O(1A) | 1.141(10) | C(7)-O(7) | 1.465(12) |
| C(1B)-O(1B) | 1.222(13) | O(7)-C(8) | 1.358(10) |
| C(1C)-O(1C) | 1.097(11) | C(8)-O(8) | 1.445(10) |
| C(1D)-O(1D) | 1.138(8) | C(8)-C(9) | 1.550(11) |
| C(1E)-O(1E) | 1.157(10) | O(8)-C(10) | 1.467(10) |
| O(1)-C(1) | 1.358(9) | C(9)-O(9) | 1.485(12) |
| O(1)-C(4) | 1.476(9) | O(9)-C(10) | 1.431(11) |
| C(1)-C(2) | 1.500(10) | C(10)-C(11) | 1.492(17) |
| C(2)-C(3) | 1.527(9) | C(10)-C(12A) | 1.499(16) |
| C(2)-C(16) | 1.542(8) | C(10)-C(12B) | 1.582(19) |
| C(3)-C(4) | 1.548(10) | C(13)-C(14) | 1.469(14) |
| C(4)-C(8) | 1.527(10) | C(13)-C(15) | 1.577(14) |
| C(4)-C(5) | 1.551(12) | C(16)-C(2)[a] | 1.542(8) |

Symmetry transformations used to generate equivalent atoms:

[a] x,-y+1,-z

**Tabelle 4: Bond angles [°] for *[82]a*.**

| | | | |
|---|---|---|---|
| C(1B)-Cr(1)-C(1C) | 87.9(4) | O(5)-C(5)-C(4) | 109.4(6) |
| C(1B)-Cr(1)-C(1E) | 89.4(4) | C(6)-C(5)-C(4) | 111.8(8) |
| C(1C)-Cr(1)-C(1E) | 176.4(4) | C(5)-O(5)-C(13) | 107.8(6) |
| C(1B)-Cr(1)-C(1A) | 89.3(4) | O(6)-C(6)-C(7) | 113.6(9) |
| C(1C)-Cr(1)-C(1A) | 88.5(5) | O(6)-C(6)-C(5) | 102.7(7) |
| C(1E)-Cr(1)-C(1A) | 89.1(4) | C(7)-C(6)-C(5) | 117.0(8) |
| C(1B)-Cr(1)-C(1D) | 178.3(4) | C(6)-O(6)-C(13) | 105.7(7) |
| C(1C)-Cr(1)-C(1D) | 91.3(4) | O(7)-C(7)-C(6) | 114.3(8) |
| C(1E)-Cr(1)-C(1D) | 91.4(3) | C(8)-O(7)-C(7) | 112.5(7) |
| C(1A)-Cr(1)-C(1D) | 89.1(3) | O(7)-C(8)-O(8) | 114.4(7) |
| C(1B)-Cr(1)-C(1) | 89.4(4) | O(7)-C(8)-C(4) | 111.1(6) |
| C(1C)-Cr(1)-C(1) | 93.5(4) | O(8)-C(8)-C(4) | 103.8(6) |
| C(1E)-Cr(1)-C(1) | 88.8(3) | O(7)-C(8)-C(9) | 107.8(8) |
| C(1A)-Cr(1)-C(1) | 177.6(4) | O(8)-C(8)-C(9) | 101.8(6) |
| C(1D)-Cr(1)-C(1) | 92.2(3) | C(4)-C(8)-C(9) | 117.9(7) |
| O(1A)-C(1A)-Cr(1) | 178.0(11) | C(8)-O(8)-C(10) | 110.1(6) |
| O(1B)-C(1B)-Cr(1) | 171.0(12) | O(9)-C(9)-C(8) | 105.8(7) |
| O(1C)-C(1C)-Cr(1) | 177.9(10) | C(10)-O(9)-C(9) | 103.6(8) |
| O(1D)-C(1D)-Cr(1) | 177.7(7) | O(9)-C(10)-O(8) | 103.1(6) |
| O(1E)-C(1E)-Cr(1) | 178.6(7) | O(9)-C(10)-C(11) | 100.6(11) |
| C(1)-O(1)-C(4) | 115.6(5) | O(8)-C(10)-C(11) | 109.6(8) |
| O(1)-C(1)-C(2) | 107.2(6) | O(9)-C(10)-C(12A) | 106.9(12) |
| O(1)-C(1)-Cr(1) | 120.3(5) | O(8)-C(10)-C(12A) | 117.7(13) |
| C(2)-C(1)-Cr(1) | 132.3(6) | C(11)-C(10)-C(12A) | 116.4(11) |
| C(1)-C(2)-C(3) | 108.0(6) | O(9)-C(10)-C(12B) | 136.4(16) |
| C(1)-C(2)-C(16) | 109.3(5) | O(8)-C(10)-C(12B) | 99.5(14) |
| C(3)-C(2)-C(16) | 112.4(5) | C(11)-C(10)-C(12B) | 106.1(14) |
| C(2)-C(3)-C(4) | 105.1(6) | C(12A)-C(10)-C(12B) | 29.8(10) |
| O(1)-C(4)-C(8) | 103.5(6) | O(6)-C(13)-O(5) | 105.0(6) |
| O(1)-C(4)-C(3) | 103.9(5) | O(6)-C(13)-C(14) | 111.0(8) |
| C(8)-C(4)-C(3) | 116.6(6) | O(5)-C(13)-C(14) | 112.7(8) |
| O(1)-C(4)-C(5) | 109.7(6) | O(6)-C(13)-C(15) | 109.5(8) |
| C(8)-C(4)-C(5) | 110.1(6) | O(5)-C(13)-C(15) | 109.1(8) |
| C(3)-C(4)-C(5) | 112.3(7) | C(14)-C(13)-C(15) | 109.5(8) |
| O(5)-C(5)-C(6) | 104.9(6) | C(2)[a]-C(16)-C(2) | 113.7(7) |

Symmetry transformations used to generate equivalent atoms:

[a] x,-y+1,-z

**Tabelle 5: Torsion angles [°] for *[82]a.***

| | | | |
|---|---|---|---|
| C(1B)-Cr(1)-C(1A)-O(1A) | -46(22) | C(2)-C(3)-C(4)-O(1) | -1.5(7) |
| C(1C)-Cr(1)-C(1A)-O(1A) | 41(22) | C(2)-C(3)-C(4)-C(8) | -114.7(7) |
| C(1E)-Cr(1)-C(1A)-O(1A) | -136(22) | C(2)-C(3)-C(4)-C(5) | 116.9(6) |
| C(1D)-Cr(1)-C(1A)-O(1A) | 133(22) | O(1)-C(4)-C(5)-O(5) | 46.8(9) |
| C(1)-Cr(1)-C(1A)-O(1A) | -105(25) | C(8)-C(4)-C(5)-O(5) | 160.1(7) |
| C(1C)-Cr(1)-C(1B)-O(1B) | 35(5) | C(3)-C(4)-C(5)-O(5) | -68.2(8) |
| C(1E)-Cr(1)-C(1B)-O(1B) | -148(5) | O(1)-C(4)-C(5)-C(6) | -68.9(8) |
| C(1A)-Cr(1)-C(1B)-O(1B) | 123(5) | C(8)-C(4)-C(5)-C(6) | 44.4(9) |
| C(1D)-Cr(1)-C(1B)-O(1B) | 95(18) | C(3)-C(4)-C(5)-C(6) | 176.1(6) |
| C(1)-Cr(1)-C(1B)-O(1B) | -59(5) | C(6)-C(5)-O(5)-C(13) | 10.1(10) |
| C(1B)-Cr(1)-C(1C)-O(1C) | 78(23) | C(4)-C(5)-O(5)-C(13) | -109.9(8) |
| C(1E)-Cr(1)-C(1C)-O(1C) | 37(26) | O(5)-C(5)-C(6)-O(6) | -29.3(10) |
| C(1A)-Cr(1)-C(1C)-O(1C) | -11(23) | C(4)-C(5)-C(6)-O(6) | 89.1(8) |
| C(1D)-Cr(1)-C(1C)-O(1C) | -100(23) | O(5)-C(5)-C(6)-C(7) | -154.4(9) |
| C(1)-Cr(1)-C(1C)-O(1C) | 167(23) | C(4)-C(5)-C(6)-C(7) | -36.0(12) |
| C(1B)-Cr(1)-C(1D)-O(1D) | 34(30) | C(7)-C(6)-O(6)-C(13) | 165.1(8) |
| C(1C)-Cr(1)-C(1D)-O(1D) | 94(19) | C(5)-C(6)-O(6)-C(13) | 37.8(9) |
| C(1E)-Cr(1)-C(1D)-O(1D) | -84(19) | O(6)-C(6)-C(7)-O(7) | -81.0(10) |
| C(1A)-Cr(1)-C(1D)-O(1D) | 5(19) | C(5)-C(6)-C(7)-O(7) | 38.5(14) |
| C(1)-Cr(1)-C(1D)-O(1D) | -173(100) | C(6)-C(7)-O(7)-C(8) | -52.3(11) |
| C(1B)-Cr(1)-C(1E)-O(1E) | 1(30) | C(7)-O(7)-C(8)-O(8) | -53.9(9) |
| C(1C)-Cr(1)-C(1E)-O(1E) | 42(33) | C(7)-O(7)-C(8)-C(4) | 63.2(9) |
| C(1A)-Cr(1)-C(1E)-O(1E) | 90(30) | C(7)-O(7)-C(8)-C(9) | -166.2(7) |
| C(1D)-Cr(1)-C(1E)-O(1E) | 179(100) | O(1)-C(4)-C(8)-O(7) | 57.5(8) |
| C(1)-Cr(1)-C(1E)-O(1E) | -89(30) | C(3)-C(4)-C(8)-O(7) | 170.8(7) |
| C(4)-O(1)-C(1)-C(2) | 4.0(7) | C(5)-C(4)-C(8)-O(7) | -59.7(9) |
| C(4)-O(1)-C(1)-Cr(1) | -171.5(4) | O(1)-C(4)-C(8)-O(8) | -179.1(6) |
| C(1B)-Cr(1)-C(1)-O(1) | 69.9(6) | C(3)-C(4)-C(8)-O(8) | -65.8(8) |
| C(1C)-Cr(1)-C(1)-O(1) | -17.9(5) | C(5)-C(4)-C(8)-O(8) | 63.7(8) |
| C(1E)-Cr(1)-C(1)-O(1) | 159.3(5) | O(1)-C(4)-C(8)-C(9) | -67.6(9) |
| C(1A)-Cr(1)-C(1)-O(1) | 128(10) | C(3)-C(4)-C(8)-C(9) | 45.8(10) |
| C(1D)-Cr(1)-C(1)-O(1) | -109.3(5) | C(5)-C(4)-C(8)-C(9) | 175.2(7) |
| C(1B)-Cr(1)-C(1)-C(2) | -104.3(7) | O(7)-C(8)-O(8)-C(10) | -103.4(8) |
| C(1C)-Cr(1)-C(1)-C(2) | 167.9(6) | C(4)-C(8)-O(8)-C(10) | 135.3(8) |
| C(1E)-Cr(1)-C(1)-C(2) | -14.9(6) | C(9)-C(8)-O(8)-C(10) | 12.4(9) |
| C(1A)-Cr(1)-C(1)-C(2) | -46(10) | O(7)-C(8)-C(9)-O(9) | 132.7(8) |
| C(1D)-Cr(1)-C(1)-C(2) | 76.5(6) | O(8)-C(8)-C(9)-O(9) | 12.1(9) |
| O(1)-C(1)-C(2)-C(3) | -4.8(7) | C(4)-C(8)-C(9)-O(9) | -100.6(9) |
| Cr(1)-C(1)-C(2)-C(3) | 170.0(5) | C(8)-C(9)-O(9)-C(10) | -32.4(9) |
| O(1)-C(1)-C(2)-C(16) | 117.8(5) | C(9)-O(9)-C(10)-O(8) | 39.3(10) |
| Cr(1)-C(1)-C(2)-C(16) | -67.4(7) | C(9)-O(9)-C(10)-C(11) | 152.6(8) |
| C(1)-C(2)-C(3)-C(4) | 3.8(7) | C(9)-O(9)-C(10)-C(12A) | -85.4(11) |
| C(16)-C(2)-C(3)-C(4) | -116.9(6) | C(9)-O(9)-C(10)-C(12B) | -80(2) |
| C(1)-O(1)-C(4)-C(8) | 120.7(6) | C(8)-O(8)-C(10)-O(9) | -33.3(11) |
| C(1)-O(1)-C(4)-C(3) | -1.6(7) | C(8)-O(8)-C(10)-C(11) | -139.8(9) |
| C(1)-O(1)-C(4)-C(5) | -121.8(6) | C(8)-O(8)-C(10)-C(12A) | 84.1(13) |

| | | | |
|---|---|---|---|
| C(8)-O(8)-C(10)-C(12B) | 109.2(15) | C(5)-O(5)-C(13)-C(14) | 133.7(8) |
| C(6)-O(6)-C(13)-O(5) | -32.0(9) | C(5)-O(5)-C(13)-C(15) | -104.6(8) |
| C(6)-O(6)-C(13)-C(14) | -154.1(7) | C(1)-C(2)-C(16)-C(2)[a] | 174.3(6) |
| C(6)-O(6)-C(13)-C(15) | 84.9(8) | C(3)-C(2)-C(16)-C(2)[a] | -65.8(4) |
| C(5)-O(5)-C(13)-O(6) | 12.7(9) | | |

Symmetry transformations used to generate equivalent atoms:

[a] x,-y+1,-z

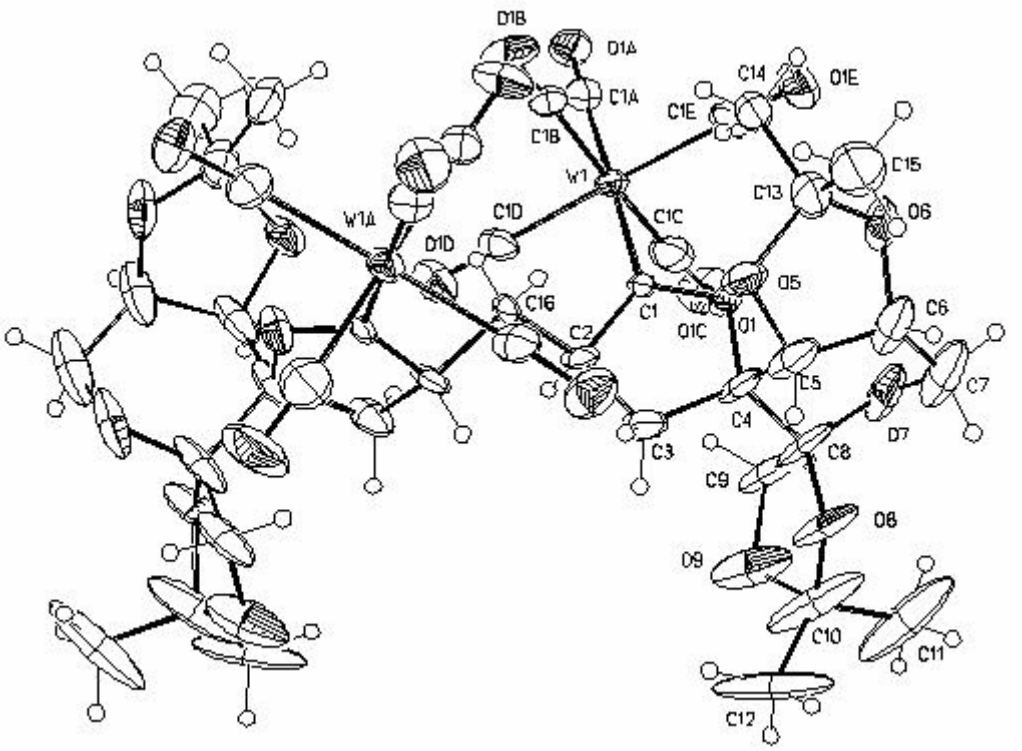
**Tabelle 6: Hydrogen bonds for *[82]a* [Å and °].**

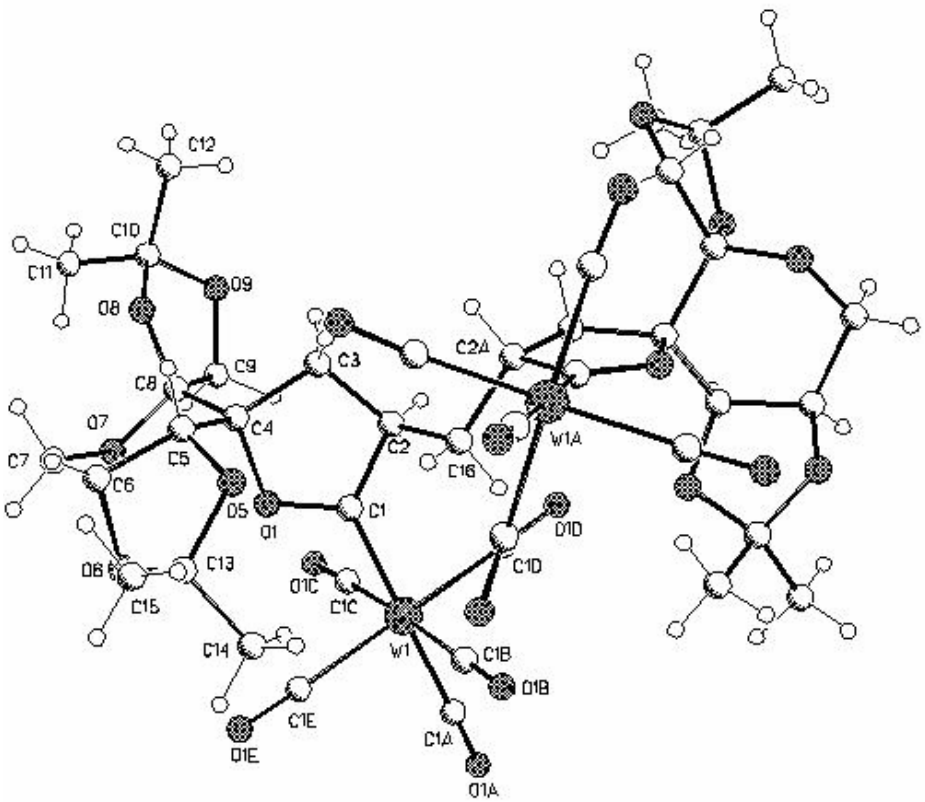
| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(15)-H(15A)...O(1A)[a] | 0.98 | 2.49 | 3.416(15) | 158.2 |
| C(7)-H(7A)...O(1B)[b] | 0.99 | 2.57 | 3.510(14) | 158.6 |
| C(11)-H(11B)...O(1B)[c] | 0.98 | 2.35 | 3.325(12) | 170.7 |
| C(7)-H(7B)...O(1C)[b] | 0.99 | 2.64 | 3.525(14) | 148.7 |
| C(11)-H(11A)...O(8)[d] | 0.98 | 2.66 | 3.597(16) | 159.9 |

Symmetry transformations used to generate equivalent atoms:

[a] x,y,z-1 [b] -x+3/2,-y+3/2,z-1/2 [c] -x+1,y,-z+1/2 [d] -x+1,y,-z-1/2

**27. Bis-[pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-(*1´R,4´S,-5´R,6´R*)-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,4-(1R)-3-oxacyclopent]-2-yliden}wolfram(0)]methan *[84]***



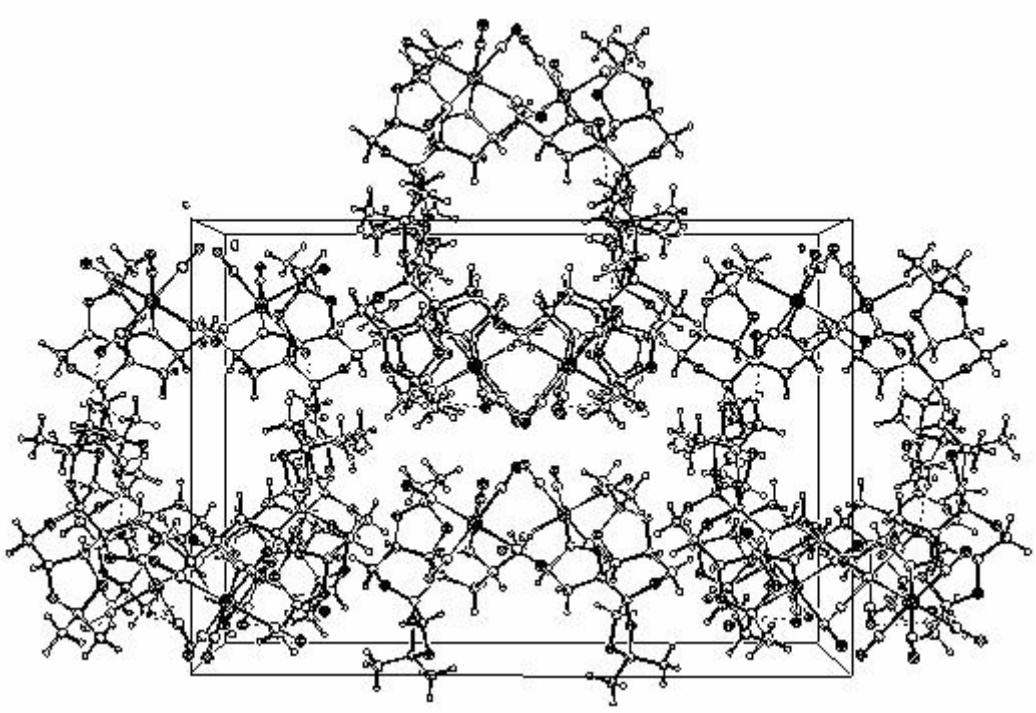


**Table 1: Crystal data and structure refinements for *[84]***

| | |
|---|---|
| Identification code | ***[84]*** |
| Empirical formula | $C_{41}H_{44}O_{22}W_2$ |
| Formula weight | 1256.46 |
| Temperature | 123(2) K |
| Wavelength | 0.71073 Å (MoKα) |
| Crystal system | Orthorhombic |
| Space group | C222(1) (No.20) |
| Unit cell dimensions | a = 17.9603(3) Å α = 90° |
| | b = 25.9305(4) Å β = 90° |
| | c = 10.4851(2) Å γ = 90° |
| Volume | 4883.12(14) $Å^3$ |
| Z | 4 |
| Calculated density | 1.709 $mg/m^3$ |
| Absorption coefficient | 4.785 $mm^{-1}$ |
| F(000) | 2456 |
| Crystal size | 0.35 x 0.25 x 0.10 mm |
| Diffractometer | Nonius KappaCCD |
| Theta range for data collection | 2.99 to 25.00° |
| Limiting indices | $-21 \le h \le 21$, $-30 \le k \le 30$, $-11 \le l \le 12$ |
| Reflections collected / unique | 20336 / 4302 [R(int) = 0.0654] |
| Completeness to Θ = 25.00 | 99.1 % |
| Absorption correction | Empirical from multiple refl |
| Max. and min. transmission | 0.41842 and 0.31339 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 4302 / 0 / 294 |
| Goodness-of-fit on $F^2$ | 1.026 |
| Final R indices [I>2σ(I)] | R1 = 0.0185, wR2 = 0.0496 |
| R indices (all data) | R1 = 0.0375, wR2 = 0.0812 |
| Absolute structure parameter | -0.018(11) |
| Largest diff. peak and hole | 1.450 and -1.626 $eÅ^{-3}$ |

**Tabelle 2: Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for *[84]*. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| W(1) | 1772(1) | 712(1) | 2050(1) | 30(1) |
| C(1A) | 1147(4) | 611(3) | 439(8) | 40(2) |
| O(1A) | 802(3) | 570(2) | -474(5) | 46(1) |
| C(1B) | 988(4) | 299(3) | 3057(7) | 33(2) |
| O(1B) | 548(3) | 65(2) | 3593(5) | 48(1) |
| C(1C) | 2507(5) | 1128(3) | 995(10) | 57(2) |
| O(1C) | 2912(4) | 1382(3) | 433(8) | 93(3) |
| C(1D) | 2329(4) | 57(3) | 1673(7) | 34(2) |
| O(1D) | 2678(3) | -312(2) | 1419(5) | 48(1) |
| C(1E) | 1212(4) | 1389(3) | 2407(7) | 42(2) |
| O(1E) | 921(3) | 1767(2) | 2573(6) | 56(2) |
| O(1) | 2527(3) | 1266(2) | 4257(5) | 39(1) |
| C(1) | 2458(3) | 811(2) | 3719(7) | 30(2) |
| C(2) | 2965(3) | 433(3) | 4410(7) | 32(2) |
| C(3) | 3401(4) | 747(3) | 5367(7) | 44(2) |
| C(4) | 3100(4) | 1289(3) | 5286(7) | 42(2) |
| C(5) | 2725(5) | 1490(3) | 6517(8) | 48(2) |
| O(5) | 2063(3) | 1206(2) | 6741(5) | 43(1) |
| C(6) | 2483(6) | 2034(3) | 6428(9) | 56(3) |
| O(6) | 1738(4) | 1994(2) | 5946(5) | 49(1) |
| C(7) | 2982(7) | 2393(4) | 5647(9) | 77(4) |
| O(7) | 3287(4) | 2162(2) | 4534(5) | 58(2) |
| C(8) | 3672(5) | 1695(3) | 4783(8) | 54(3) |
| O(8) | 4195(3) | 1743(2) | 5779(5) | 59(2) |
| C(9) | 4094(5) | 1557(4) | 3618(8) | 56(3) |
| O(9) | 4840(4) | 1453(3) | 4035(6) | 82(2) |
| C(10) | 4948(7) | 1744(6) | 5170(9) | 108(6) |
| C(11) | 5169(9) | 2293(6) | 4869(11) | 143(8) |
| C(12) | 5485(5) | 1454(8) | 6012(11) | 132(7) |
| C(13) | 1438(5) | 1562(3) | 6558(7) | 45(2) |
| C(14) | 863(5) | 1302(3) | 5732(9) | 50(2) |
| C(15) | 1114(5) | 1703(4) | 7879(9) | 61(2) |
| C(16) | 2480(5) | 0 | 5000 | 33(2) |

**Tabelle 3: Bond lengths [Å] for *[84]*.**

| | | | |
|---|---|---|---|
| W(1)-C(1D) | 2.011(8) | C(5)-O(5) | 1.419(8) |
| W(1)-C(1C) | 2.032(10) | C(5)-C(6) | 1.480(13) |
| W(1)-C(1A) | 2.044(8) | O(5)-C(13) | 1.467(9) |
| W(1)-C(1E) | 2.057(8) | C(6)-O(6) | 1.434(10) |
| W(1)-C(1B) | 2.059(8) | C(6)-C(7) | 1.530(12) |
| W(1)-C(1) | 2.156(7) | O(6)-C(13) | 1.398(9) |
| C(1A)-O(1A) | 1.146(9) | C(7)-O(7) | 1.421(12) |
| C(1B)-O(1B) | 1.144(8) | O(7)-C(8) | 1.418(11) |
| C(1C)-O(1C) | 1.145(10) | C(8)-O(8) | 1.410(8) |
| C(1D)-O(1D) | 1.175(9) | C(8)-C(9) | 1.481(14) |
| C(1E)-O(1E) | 1.125(9) | O(8)-C(10) | 1.497(13) |
| O(1)-C(1) | 1.313(8) | C(9)-O(9) | 1.435(10) |
| O(1)-C(4) | 1.492(8) | O(9)-C(10) | 1.423(14) |
| C(1)-C(2) | 1.520(8) | C(10)-C(12) | 1.508(17) |
| C(2)-C(3) | 1.510(10) | C(10)-C(11) | 1.510(18) |
| C(2)-C(16) | 1.550(8) | C(13)-C(14) | 1.506(10) |
| C(3)-C(4) | 1.510(11) | C(13)-C(15) | 1.547(12) |
| C(4)-C(5) | 1.545(13) | C(16)-C(2)[a] | 1.550(8) |
| C(4)-C(8) | 1.563(9) | | |

Symmetry transformations used to generate equivalent atoms:

[a] x,-y,-z+1

**Tabelle 4: Bond angles [°] for *[84].***

| | | | |
|---|---|---|---|
| C(1D)-W(1)-C(1C) | 91.0(3) | C(5)-C(4)-C(8) | 110.0(6) |
| C(1D)-W(1)-C(1A) | 90.1(3) | O(5)-C(5)-C(6) | 105.0(7) |
| C(1C)-W(1)-C(1A) | 88.6(3) | O(5)-C(5)-C(4) | 109.2(6) |
| C(1D)-W(1)-C(1E) | 178.9(3) | C(6)-C(5)-C(4) | 113.4(7) |
| C(1C)-W(1)-C(1E) | 87.9(3) | C(5)-O(5)-C(13) | 107.0(6) |
| C(1A)-W(1)-C(1E) | 89.5(3) | O(6)-C(6)-C(5) | 103.1(6) |
| C(1D)-W(1)-C(1B) | 90.1(3) | O(6)-C(6)-C(7) | 113.8(7) |
| C(1C)-W(1)-C(1B) | 177.3(3) | C(5)-C(6)-C(7) | 116.1(9) |
| C(1A)-W(1)-C(1B) | 88.9(3) | C(13)-O(6)-C(6) | 104.8(5) |
| C(1E)-W(1)-C(1B) | 91.0(3) | O(7)-C(7)-C(6) | 114.2(7) |
| C(1D)-W(1)-C(1) | 88.6(3) | C(8)-O(7)-C(7) | 113.4(6) |
| C(1C)-W(1)-C(1) | 90.4(3) | O(8)-C(8)-O(7) | 112.6(7) |
| C(1A)-W(1)-C(1) | 178.3(3) | O(8)-C(8)-C(9) | 107.0(7) |
| C(1E)-W(1)-C(1) | 91.7(3) | O(7)-C(8)-C(9) | 107.7(6) |
| C(1B)-W(1)-C(1) | 92.1(3) | O(8)-C(8)-C(4) | 104.3(6) |
| O(1A)-C(1A)-W(1) | 177.8(7) | O(7)-C(8)-C(4) | 108.4(7) |
| O(1B)-C(1B)-W(1) | 178.6(6) | C(9)-C(8)-C(4) | 116.9(7) |
| O(1C)-C(1C)-W(1) | 176.8(9) | C(8)-O(8)-C(10) | 106.6(6) |
| O(1D)-C(1D)-W(1) | 176.8(6) | O(9)-C(9)-C(8) | 105.7(6) |
| O(1E)-C(1E)-W(1) | 177.7(7) | C(10)-O(9)-C(9) | 106.4(9) |
| C(1)-O(1)-C(4) | 114.4(5) | O(9)-C(10)-O(8) | 103.4(6) |
| O(1)-C(1)-C(2) | 108.5(5) | O(9)-C(10)-C(12) | 108.2(14) |
| O(1)-C(1)-W(1) | 120.6(4) | O(8)-C(10)-C(12) | 109.1(7) |
| C(2)-C(1)-W(1) | 130.7(5) | O(9)-C(10)-C(11) | 111.2(8) |
| C(3)-C(2)-C(1) | 106.3(6) | O(8)-C(10)-C(11) | 109.1(13) |
| C(3)-C(2)-C(16) | 114.6(5) | C(12)-C(10)-C(11) | 115.2(10) |
| C(1)-C(2)-C(16) | 108.7(5) | O(6)-C(13)-O(5) | 105.7(6) |
| C(4)-C(3)-C(2) | 106.1(5) | O(6)-C(13)-C(14) | 111.1(6) |
| O(1)-C(4)-C(3) | 104.4(5) | O(5)-C(13)-C(14) | 108.5(6) |
| O(1)-C(4)-C(5) | 108.5(6) | O(6)-C(13)-C(15) | 111.5(7) |
| C(3)-C(4)-C(5) | 115.0(6) | O(5)-C(13)-C(15) | 108.6(6) |
| O(1)-C(4)-C(8) | 103.7(5) | C(14)-C(13)-C(15) | 111.3(7) |
| C(3)-C(4)-C(8) | 114.2(7) | C(2)-C(16)-C(2)[a] | 111.7(7) |

Symmetry transformations used to generate equivalent atoms:

[a] x,-y,-z+1

**Tabelle 5: Torsion angles [°] for *[84].***

| | | | |
|---|---|---|---|
| C(1D)-W(1)-C(1A)-O(1A) | -130(17) | C(2)-C(3)-C(4)-O(1) | -2.0(7) |
| C(1C)-W(1)-C(1A)-O(1A) | -39(17) | C(2)-C(3)-C(4)-C(5) | 116.8(6) |
| C(1E)-W(1)-C(1A)-O(1A) | 49(17) | C(2)-C(3)-C(4)-C(8) | -114.6(6) |
| C(1B)-W(1)-C(1A)-O(1A) | 140(17) | O(1)-C(4)-C(5)-O(5) | 49.3(7) |
| C(1)-W(1)-C(1A)-O(1A) | -91(20) | C(3)-C(4)-C(5)-O(5) | -67.2(7) |
| C(1D)-W(1)-C(1B)-O(1B) | -69(27) | C(8)-C(4)-C(5)-O(5) | 162.1(6) |
| C(1C)-W(1)-C(1B)-O(1B) | 46(30) | O(1)-C(4)-C(5)-C(6) | -67.4(8) |
| C(1A)-W(1)-C(1B)-O(1B) | 22(27) | C(3)-C(4)-C(5)-C(6) | 176.1(6) |
| C(1E)-W(1)-C(1B)-O(1B) | 111(27) | C(8)-C(4)-C(5)-C(6) | 45.4(8) |
| C(1)-W(1)-C(1B)-O(1B) | -157(27) | C(6)-C(5)-O(5)-C(13) | 9.8(8) |
| C(1D)-W(1)-C(1C)-O(1C) | -156(14) | C(4)-C(5)-O(5)-C(13) | -112.1(6) |
| C(1A)-W(1)-C(1C)-O(1C) | 114(14) | O(5)-C(5)-C(6)-O(6) | -29.3(9) |
| C(1E)-W(1)-C(1C)-O(1C) | 24(14) | C(4)-C(5)-C(6)-O(6) | 89.8(8) |
| C(1B)-W(1)-C(1C)-O(1C) | 90(17) | O(5)-C(5)-C(6)-C(7) | -154.4(7) |
| C(1)-W(1)-C(1C)-O(1C) | -68(14) | C(4)-C(5)-C(6)-C(7) | -35.3(10) |
| C(1C)-W(1)-C(1D)-O(1D) | 0(12) | C(5)-C(6)-O(6)-C(13) | 38.4(9) |
| C(1A)-W(1)-C(1D)-O(1D) | 89(12) | C(7)-C(6)-O(6)-C(13) | 165.1(8) |
| C(1E)-W(1)-C(1D)-O(1D) | 17(24) | O(6)-C(6)-C(7)-O(7) | -81.3(11) |
| C(1B)-W(1)-C(1D)-O(1D) | 178(100) | C(5)-C(6)-C(7)-O(7) | 38.1(11) |
| C(1)-W(1)-C(1D)-O(1D) | -90(12) | C(6)-C(7)-O(7)-C(8) | -53.9(11) |
| C(1D)-W(1)-C(1E)-O(1E) | -8(29) | C(7)-O(7)-C(8)-O(8) | -50.2(9) |
| C(1C)-W(1)-C(1E)-O(1E) | 9(18) | C(7)-O(7)-C(8)-C(9) | -168.0(7) |
| C(1A)-W(1)-C(1E)-O(1E) | -80(18) | C(7)-O(7)-C(8)-C(4) | 64.7(8) |
| C(1B)-W(1)-C(1E)-O(1E) | -169(18) | O(1)-C(4)-C(8)-O(8) | 176.8(7) |
| C(1)-W(1)-C(1E)-O(1E) | 99(18) | C(3)-C(4)-C(8)-O(8) | -70.2(9) |
| C(4)-O(1)-C(1)-C(2) | 4.1(7) | C(5)-C(4)-C(8)-O(8) | 60.9(9) |
| C(4)-O(1)-C(1)-W(1) | -171.9(4) | O(1)-C(4)-C(8)-O(7) | 56.5(8) |
| C(1D)-W(1)-C(1)-O(1) | 160.1(5) | C(3)-C(4)-C(8)-O(7) | 169.5(6) |
| C(1C)-W(1)-C(1)-O(1) | 69.1(6) | C(5)-C(4)-C(8)-O(7) | -59.4(7) |
| C(1A)-W(1)-C(1)-O(1) | 121(10) | O(1)-C(4)-C(8)-C(9) | -65.4(8) |
| C(1E)-W(1)-C(1)-O(1) | -18.9(6) | C(3)-C(4)-C(8)-C(9) | 47.7(9) |
| C(1B)-W(1)-C(1)-O(1) | -109.9(6) | C(5)-C(4)-C(8)-C(9) | 178.8(7) |
| C(1D)-W(1)-C(1)-C(2) | -14.9(6) | O(7)-C(8)-O(8)-C(10) | -106.5(9) |
| C(1C)-W(1)-C(1)-C(2) | -105.9(6) | C(9)-C(8)-O(8)-C(10) | 11.7(9) |
| C(1A)-W(1)-C(1)-C(2) | -54(10) | C(4)-C(8)-O(8)-C(10) | 136.1(9) |
| C(1E)-W(1)-C(1)-C(2) | 166.2(6) | O(8)-C(8)-C(9)-O(9) | 8.7(9) |
| C(1B)-W(1)-C(1)-C(2) | 75.2(6) | O(7)-C(8)-C(9)-O(9) | 130.0(7) |
| O(1)-C(1)-C(2)-C(3) | -5.2(7) | C(4)-C(8)-C(9)-O(9) | -107.7(7) |
| W(1)-C(1)-C(2)-C(3) | 170.2(5) | C(8)-C(9)-O(9)-C(10) | -26.8(9) |
| O(1)-C(1)-C(2)-C(16) | 118.6(6) | C(9)-O(9)-C(10)-O(8) | 33.4(10) |
| W(1)-C(1)-C(2)-C(16) | -66.0(7) | C(9)-O(9)-C(10)-C(12) | 149.0(7) |
| C(1)-C(2)-C(3)-C(4) | 4.2(7) | C(9)-O(9)-C(10)-C(11) | -83.6(12) |
| C(16)-C(2)-C(3)-C(4) | -115.8(7) | C(8)-O(8)-C(10)-O(9) | -27.9(11) |
| C(1)-O(1)-C(4)-C(3) | -1.3(8) | C(8)-O(8)-C(10)-C(12) | -142.8(11) |
| C(1)-O(1)-C(4)-C(5) | -124.5(6) | C(8)-O(8)-C(10)-C(11) | 90.6(9) |
| C(1)-O(1)-C(4)-C(8) | 118.6(7) | C(6)-O(6)-C(13)-O(5) | -32.7(7) |

| | | | |
|---|---|---|---|
| C(6)-O(6)-C(13)-C(14) | -150.1(7) | C(5)-O(5)-C(13)-C(15) | -105.9(7) |
| C(6)-O(6)-C(13)-C(15) | 85.2(8) | C(3)-C(2)-C(16)-C(2)[a] | -67.7(5) |
| C(5)-O(5)-C(13)-O(6) | 13.9(7) | C(1)-C(2)-C(16)-C(2)[a] | 173.5(7) |
| C(5)-O(5)-C(13)-C(14) | 133.0(7) | | |

Symmetry transformations used to generate equivalent atoms:

[a] x,-y,-z+1

**Tabelle 6: Hydrogen bonds for *[84]* [Å and °].**

| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(15)-H(15B)...O(1A)[a] | 0.98 | 2.49 | 3.455(11) | 167.3 |
| C(12)-H(12A)...O(1C)[b] | 0.98 | 2.29 | 3.260(12) | 169.4 |
| C(7)-H(7B)...O(1E)[c] | 0.99 | 2.66 | 3.564(11) | 152.0 |
| C(12)-H(12C)...O(8)[d] | 0.98 | 2.56 | 3.494(14) | 159.6 |

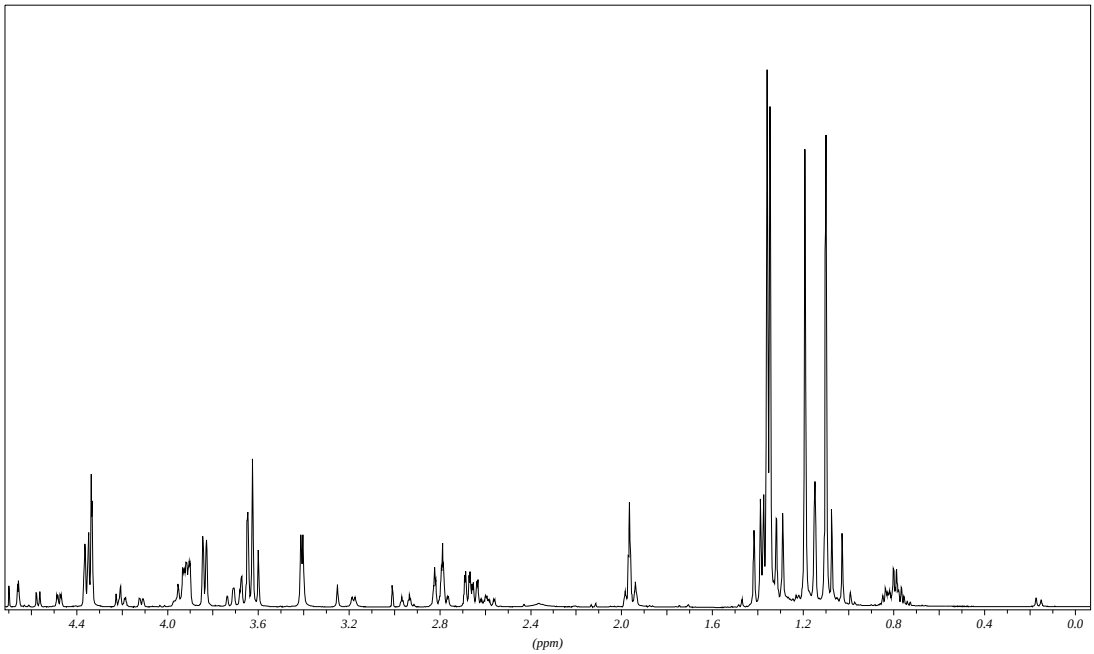
Symmetry transformations used to generate equivalent atoms:

[a] x,y,z+1 [b] -x+1,y,-z+1/2 [c] -x+1/2,-y+1/2,z+1/2 [d] -x+1,y,-z+3/2

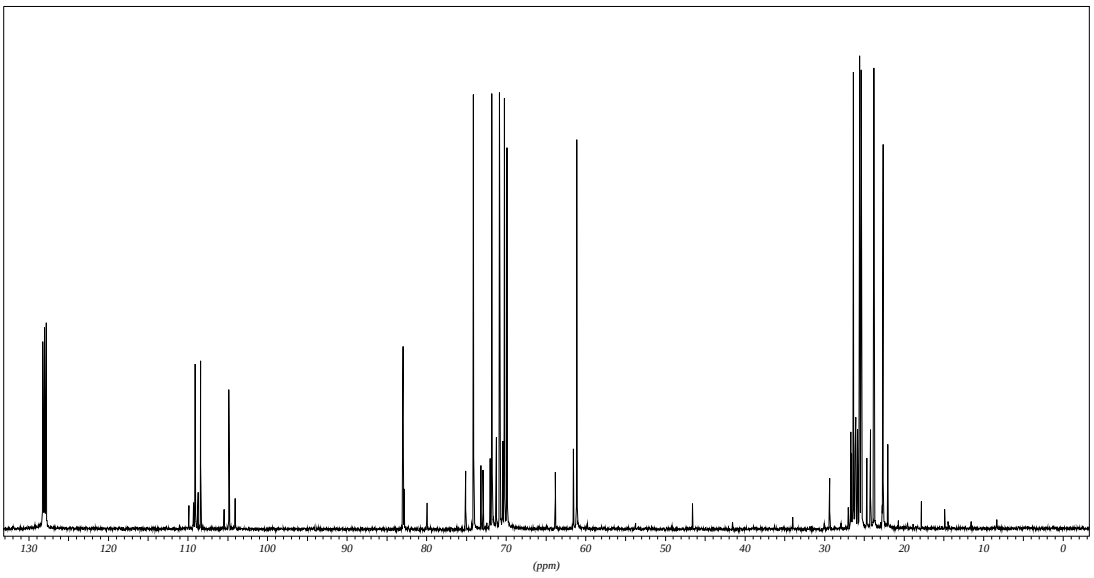
# I Ausgewählte NMR-Spektren

**(*1S,1´R,2´S,6´S,9´R*)-1-(4´,4´,11´,11´-Tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´-yl)-but-3-in-1-ol *[5]S***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

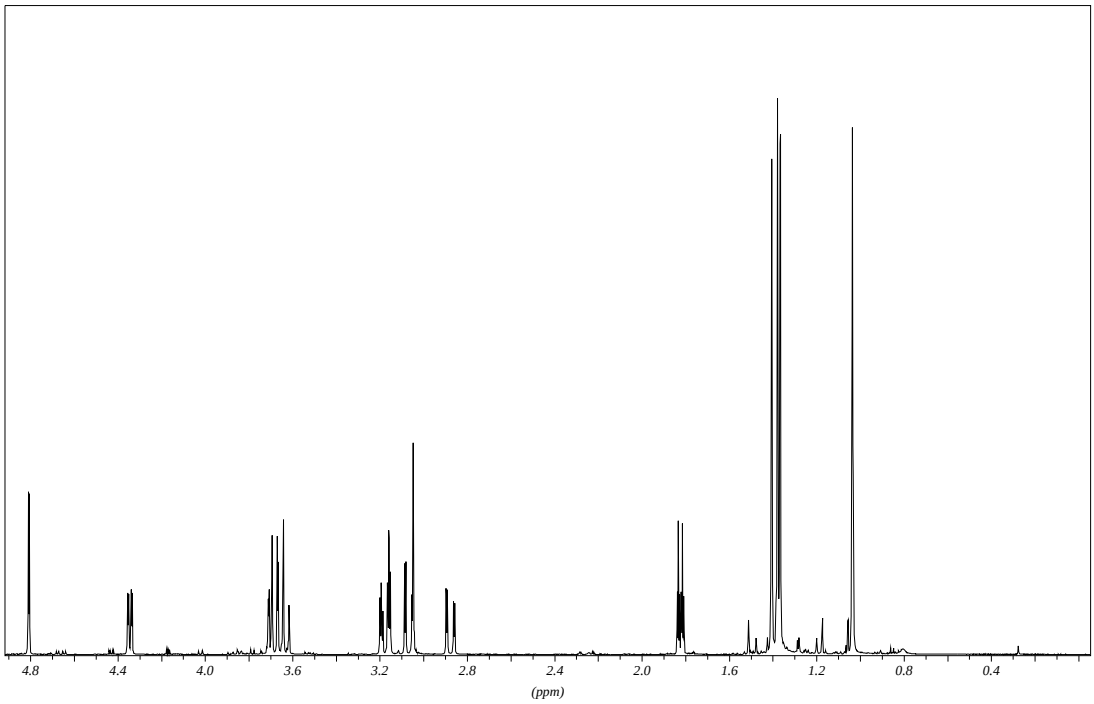



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$)

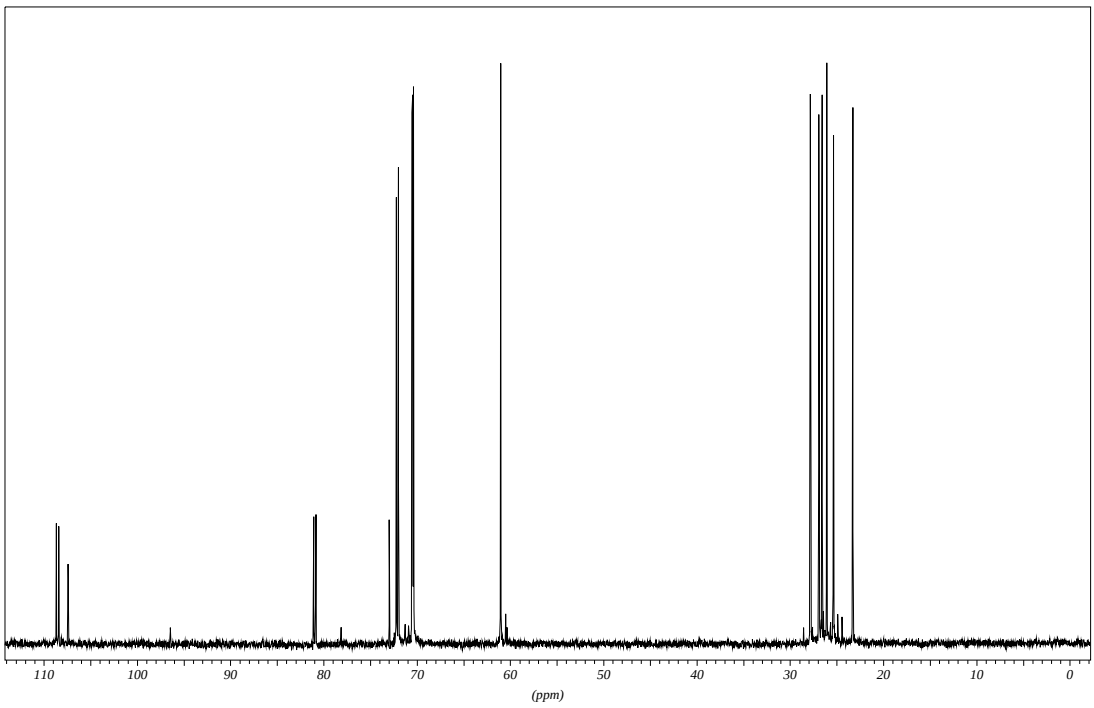


**(*1´R,2´S,6´S,9´R*)-4-(4´,4´,11´,11´-Tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo-[7.3.0.0$^{2,6}$]dodec-6´-yl)-hepta-1,6-diin-4-ol *[7]***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

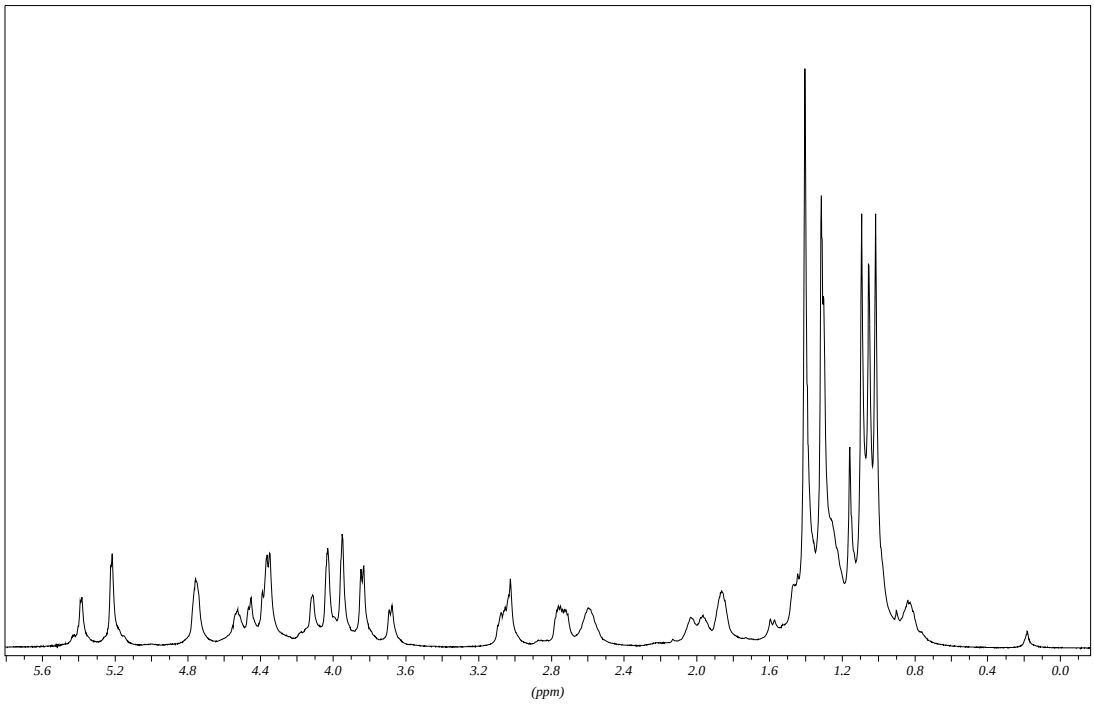



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$)

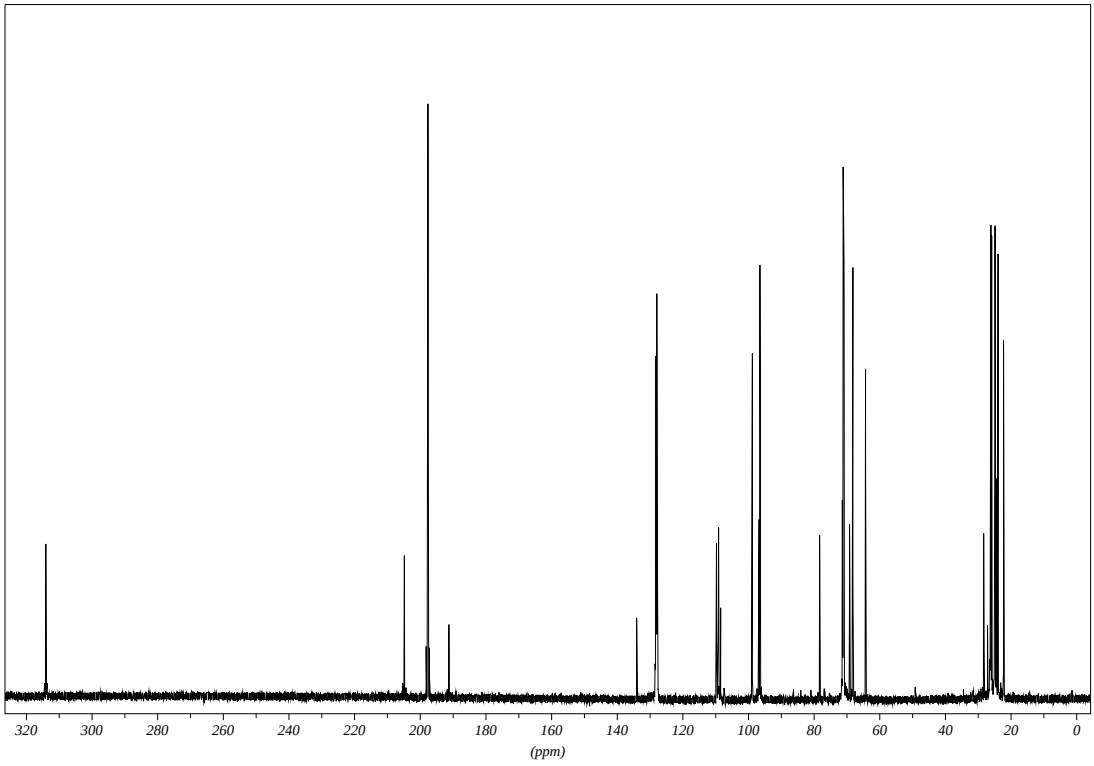


**(*3R/S,1´S,2´R,6´R,8´R,9´S*)-Pentacarbonyl{3-(4´,4´,11´,11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-8´-yl)-2-oxacyclopent-yliden}wolfram(0) *[11]R/S***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

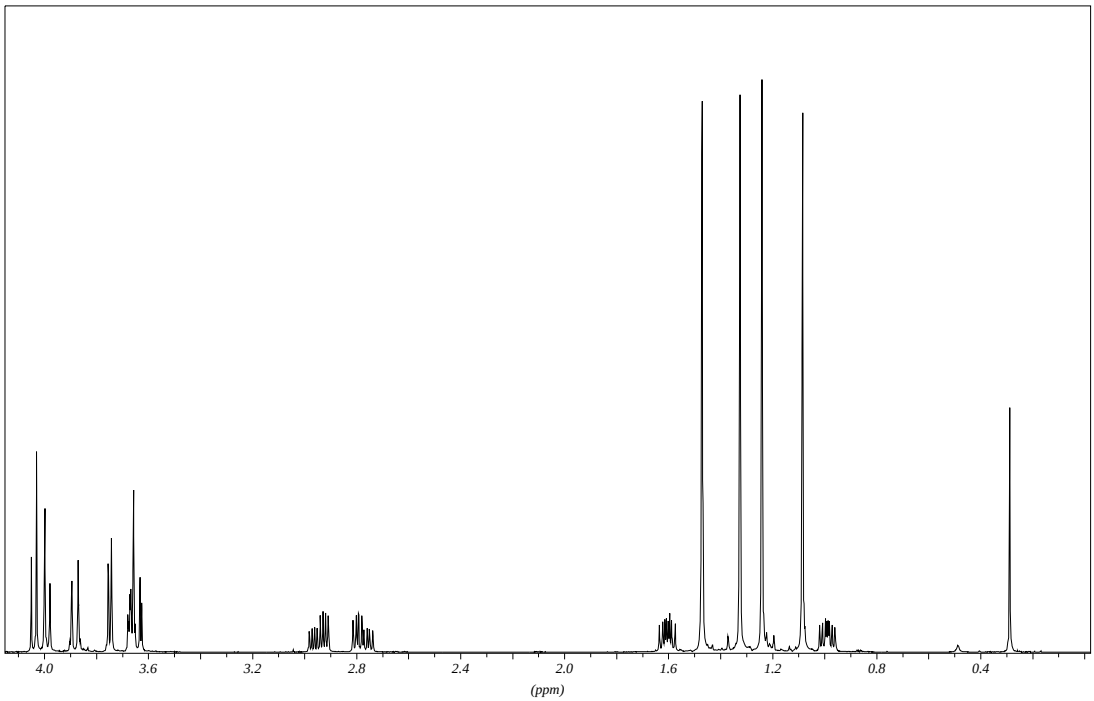



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

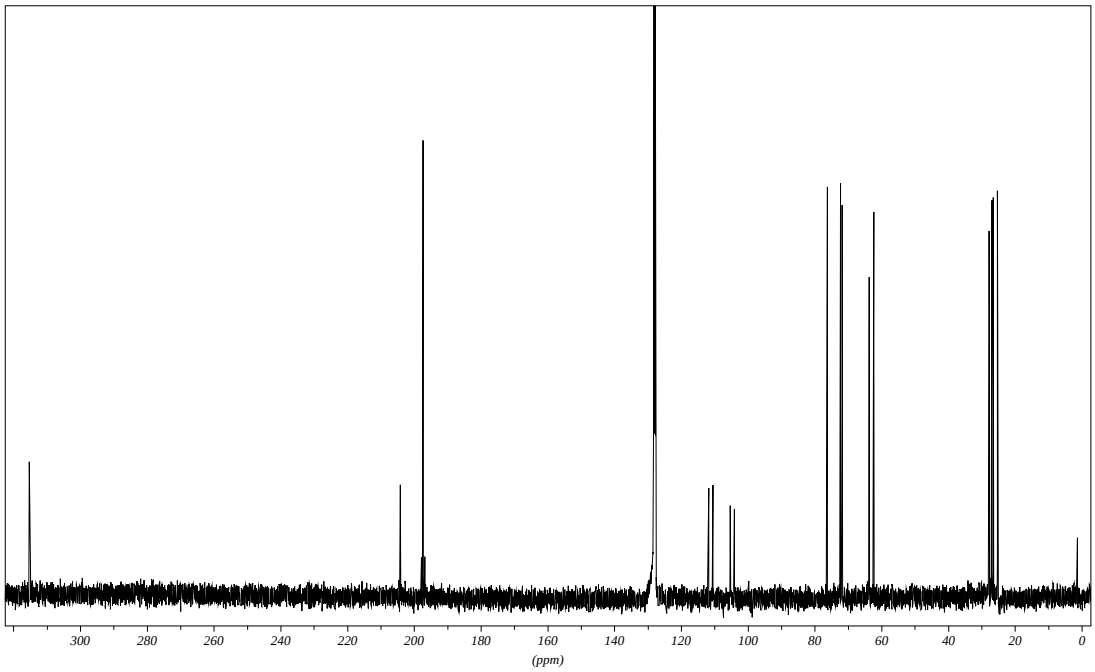


**(*3R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-dispiro[3,7,9-trioxabicyclo-[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,3-2-oxacyclo-pent]-5-yliden}wolfram(0)** ***[15]***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

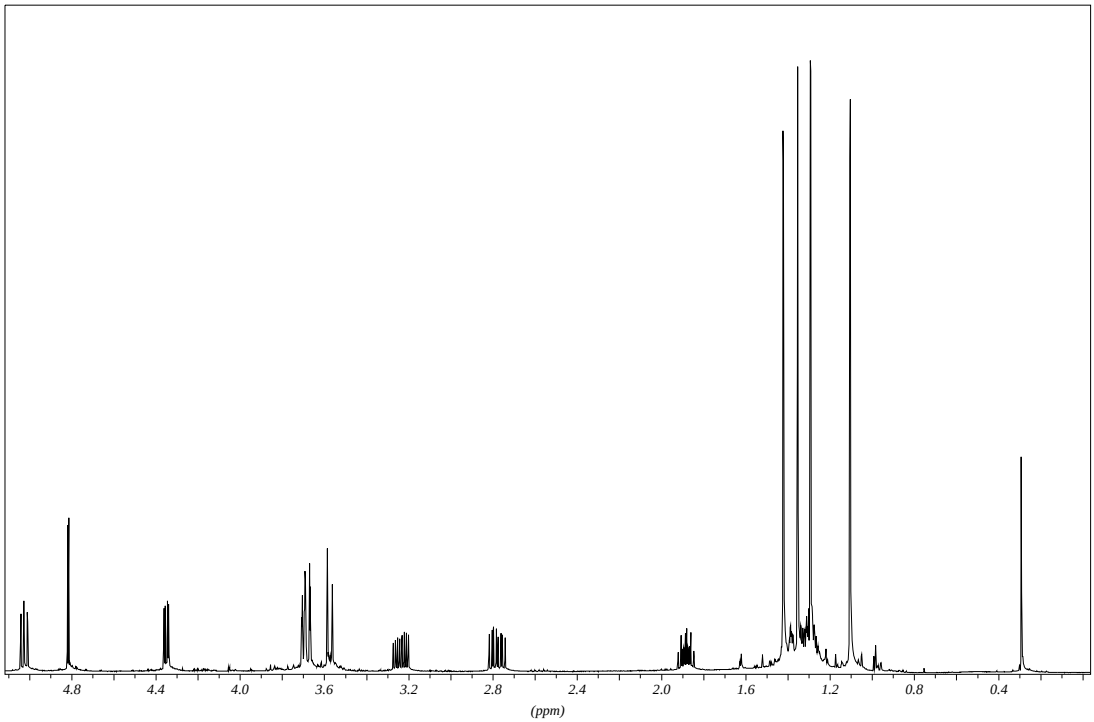



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

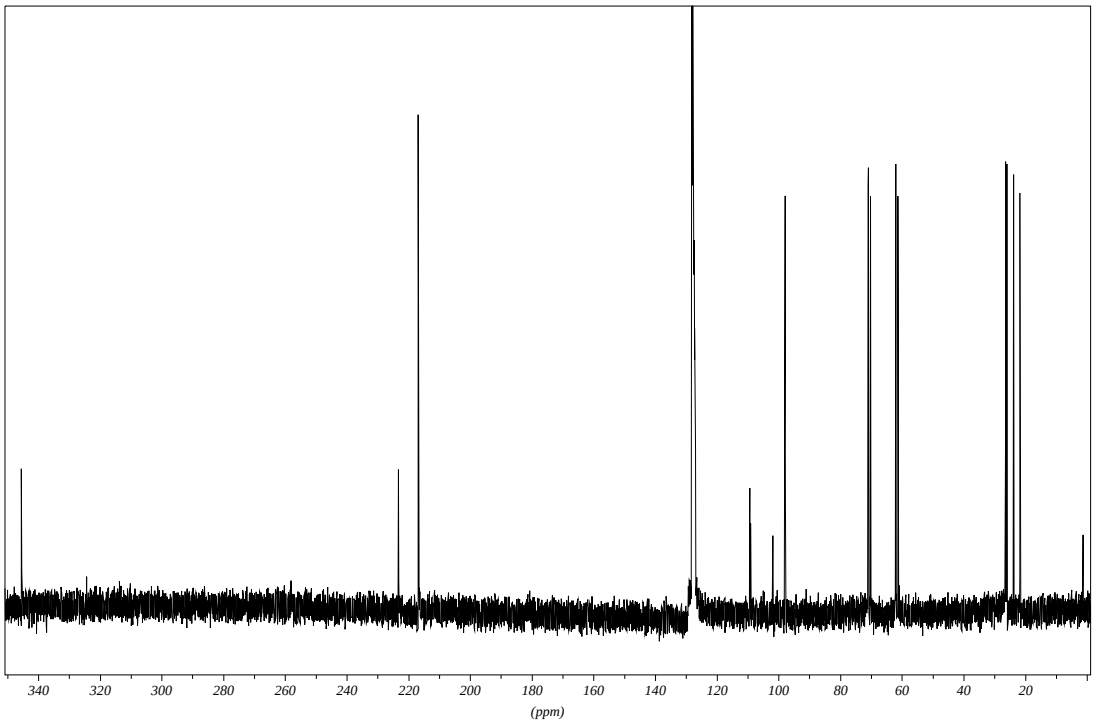


**(*3R,1´R,2´S,6´S,9´R*)-Pentacarbonyl{3-(4´,4´,11´,11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´-yl)-2-oxacyclo-pentyliden}chrom(0) *[16]R***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

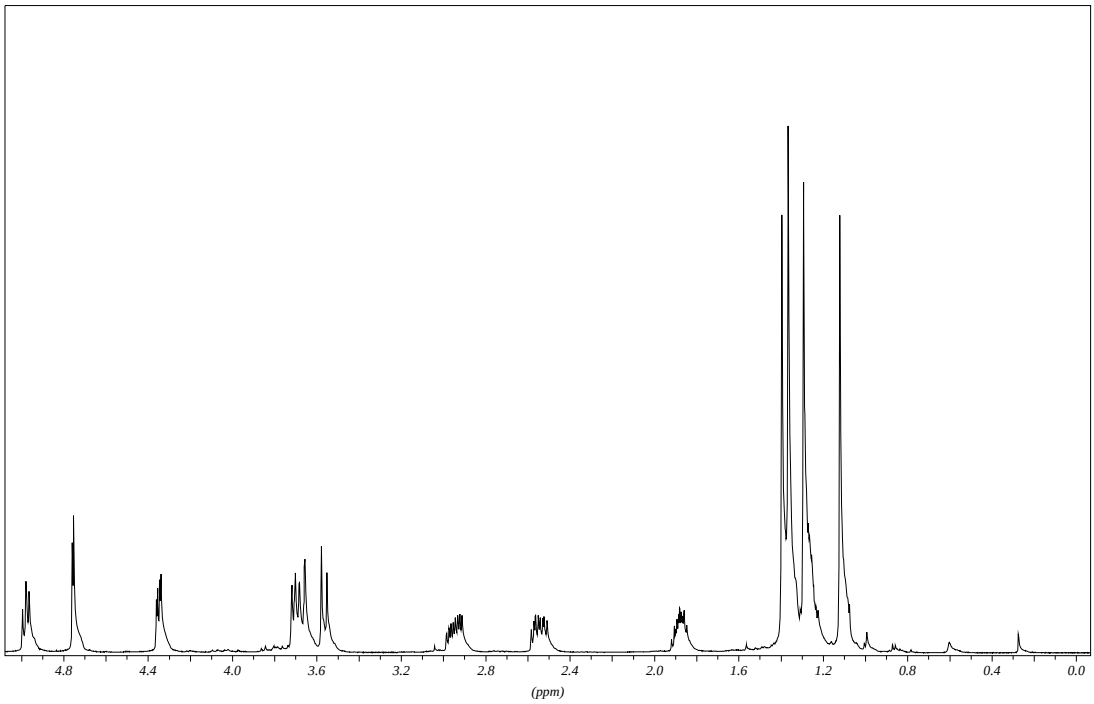



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

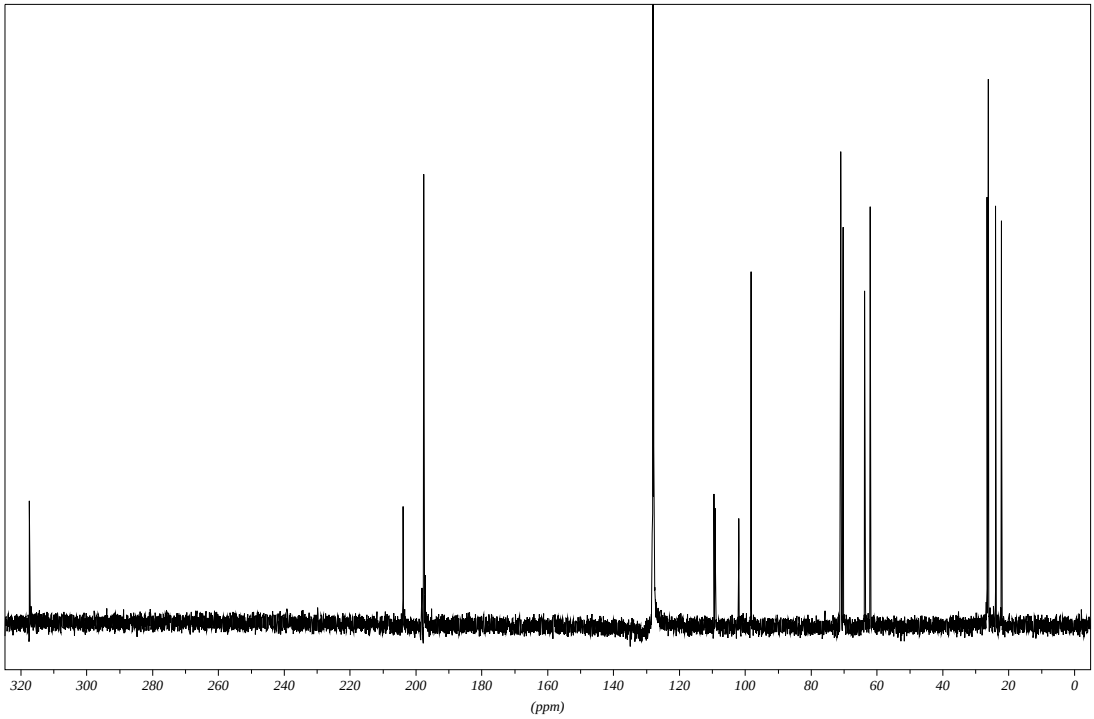


**(*3R,1´R,2´S,6´S,9´R*)-Pentacarbonyl{3-(4´,4´,11´,11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´-yl)-2-oxacyclo-pentyliden}wolfram(0) *[17]R***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

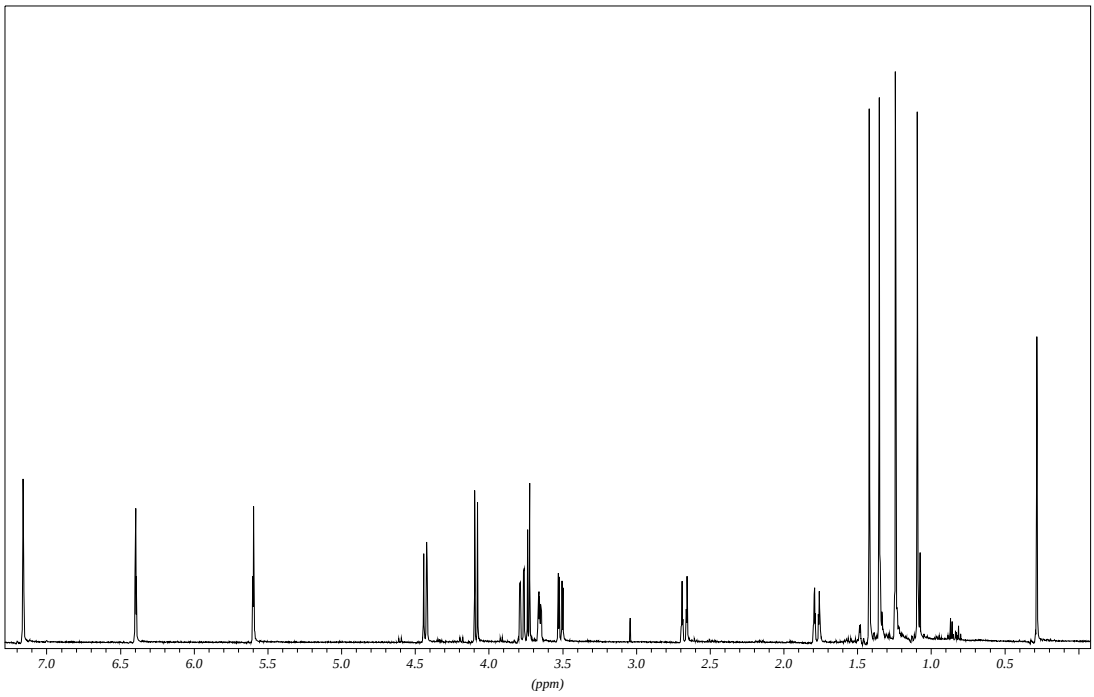



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

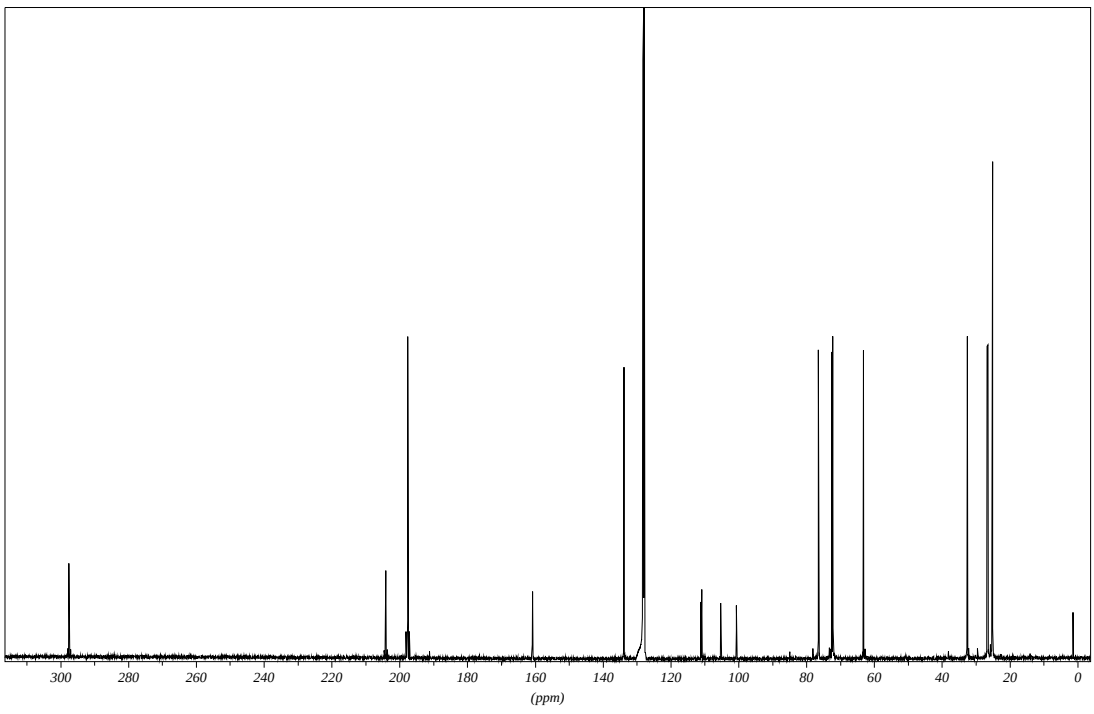


**(*3R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-5-methylen-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´, 4´´-1,3-dioxacyclopentan-5´,3-2-oxacyclopent]-1-yliden}-wolfram(0)** ***[29]***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

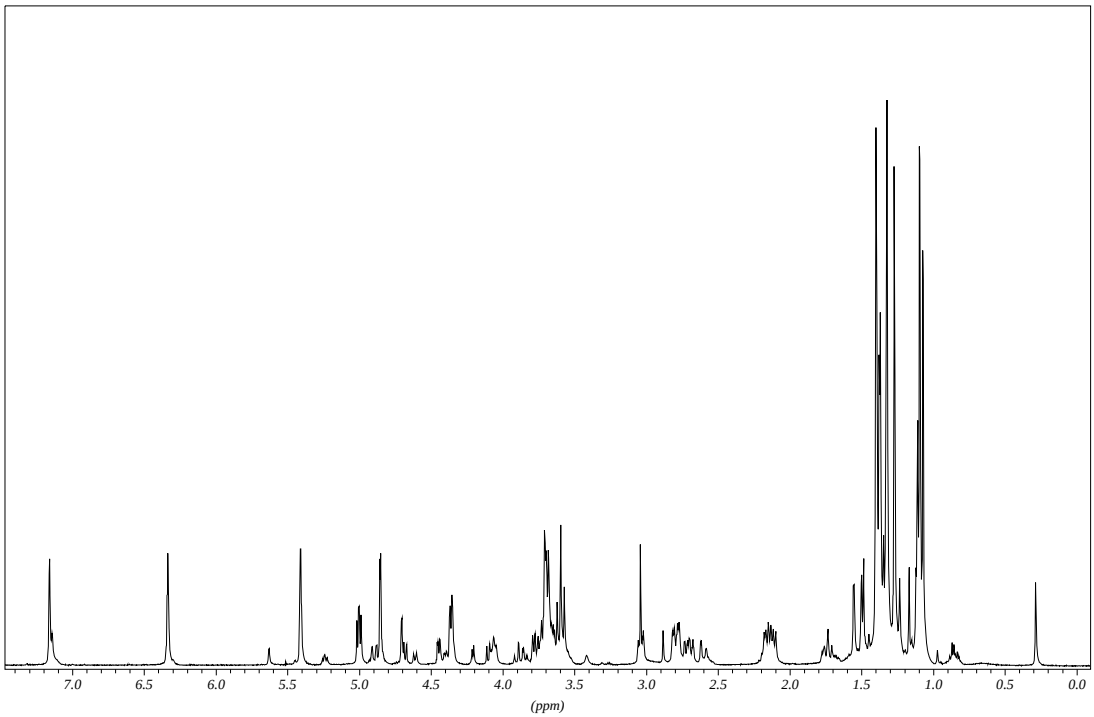



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

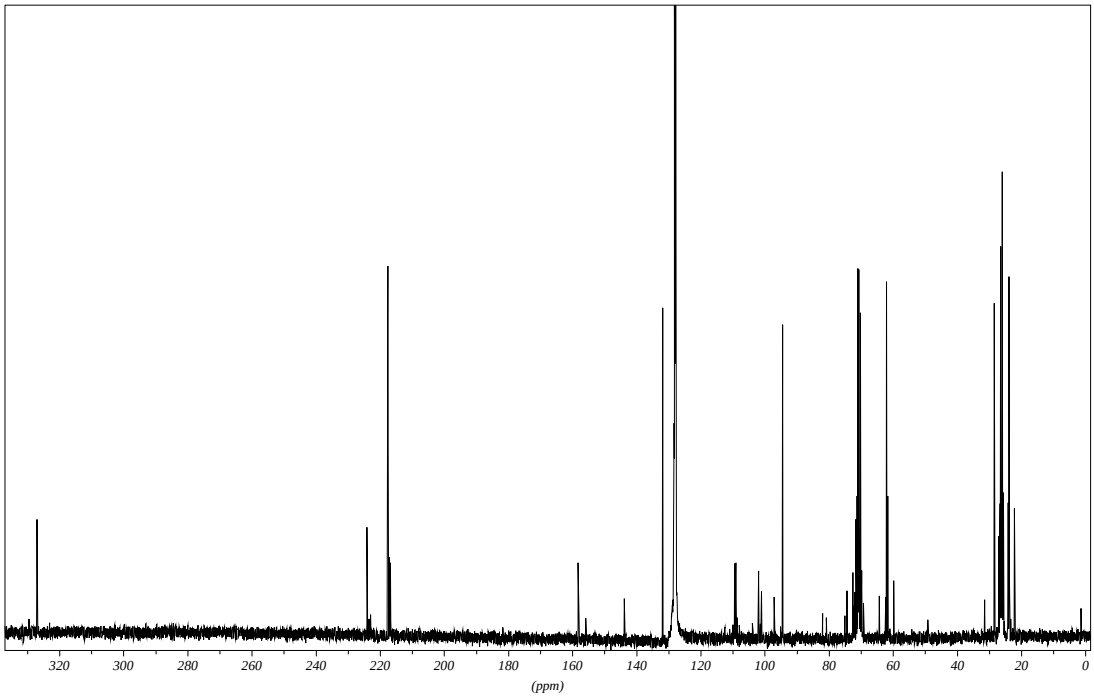


**(*3R,1´R,2´S,6´S,9´R*)-Pentacarbonyl{3-(4´,4´,11´,11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodecan-6´-yl)-5-methylen-2-oxacyclopent-1-yliden}-chrom(0)** ***[30]***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

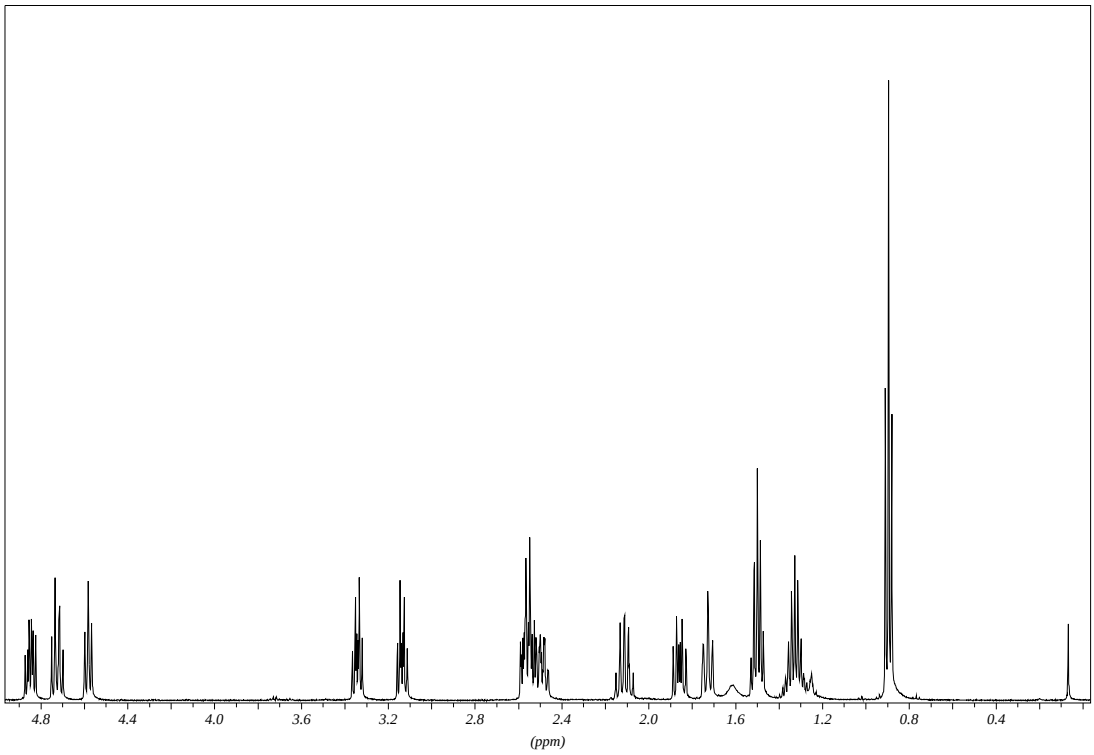



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

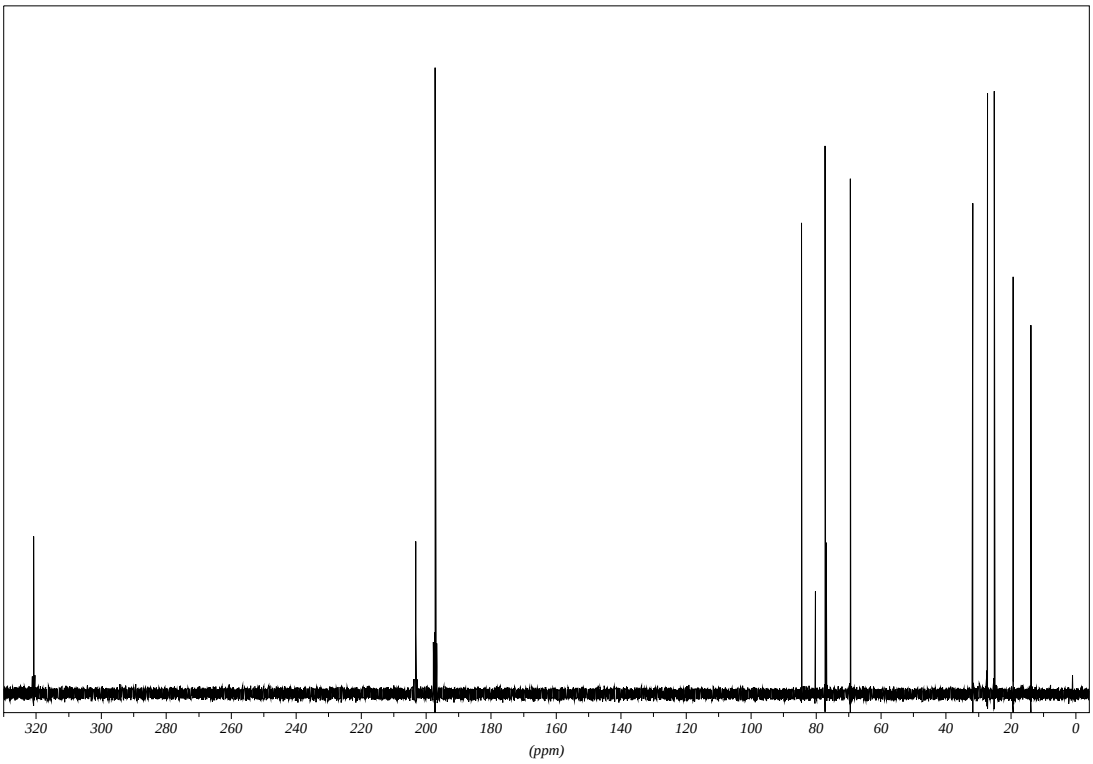


**(*1R**,*4S**)-Pentacarbonyl{(1-*n*-butoxy)-6-oxaspiro[3.4]oct-5-yliden}wolfram(0)** ***[32]***

$^{1}$H-NMR-Spektrum (500 MHz, $CDCl_3$):

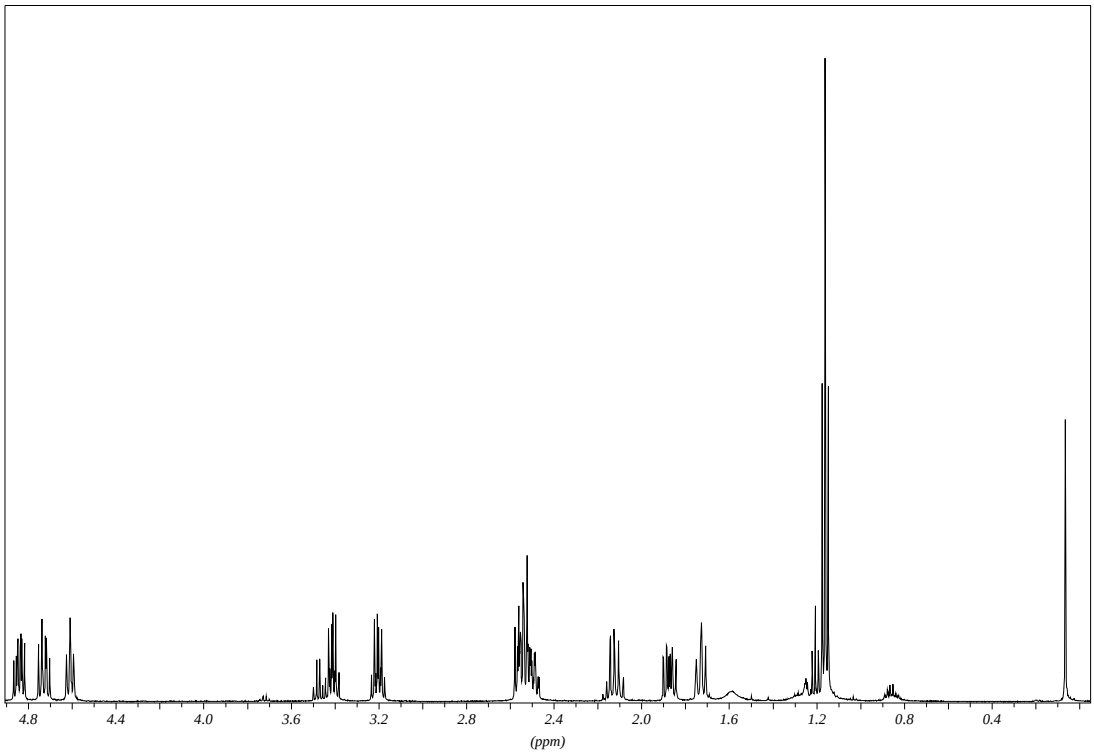



$^{13}$C-NMR-Spektrum (500 MHz, $CDCl_3$):

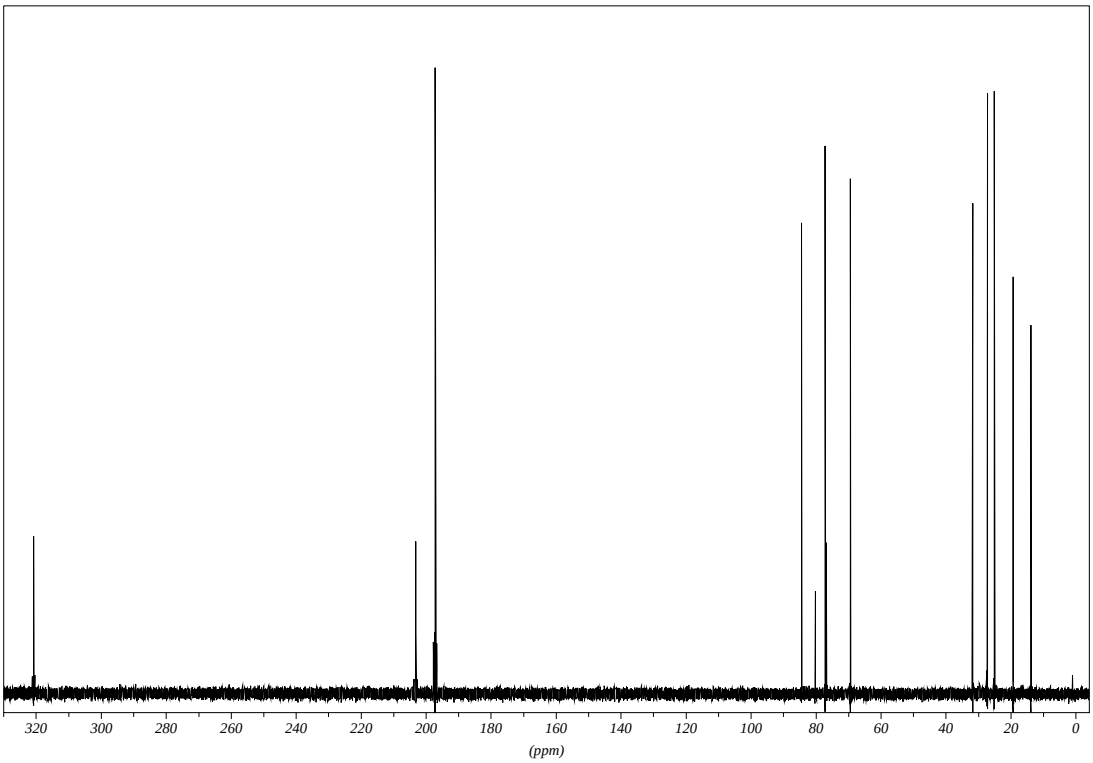


**(*1R**,*4S**)-Pentacarbonyl{(1-ethoxy)-6-oxaspiro[3.4]oct-5-yliden}-wolfram(0)** ***[33]***

$^{1}$H-NMR-Spektrum (500 MHz, $CDCl_3$):

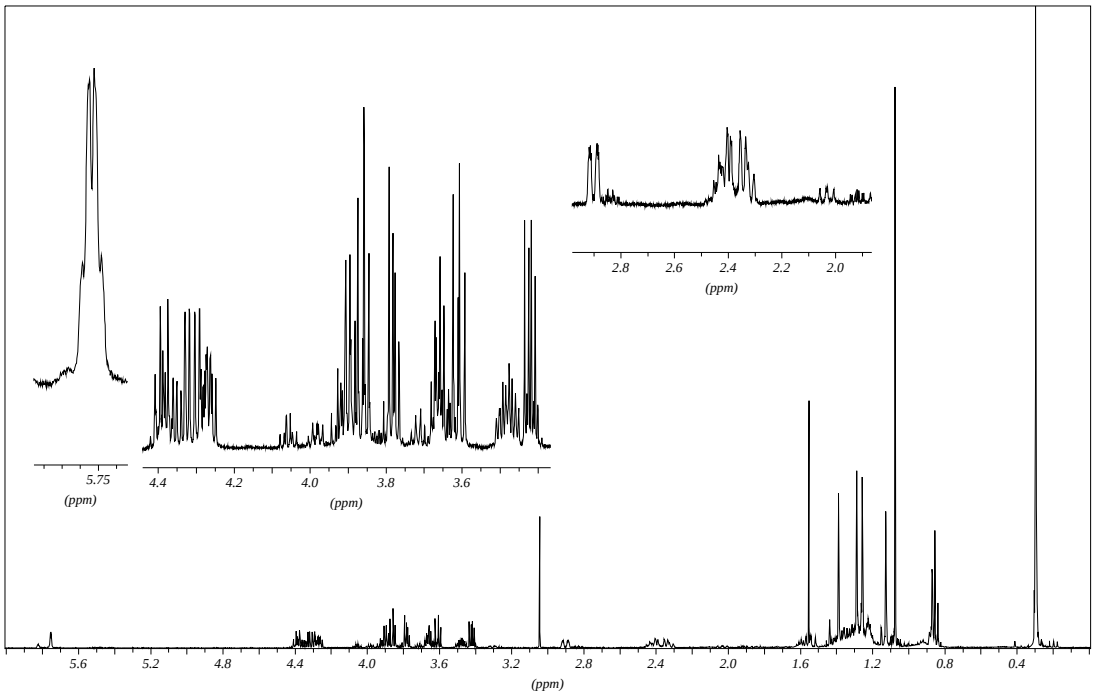



$^{13}$C-NMR-Spektrum (500 MHz, $CDCl_3$):

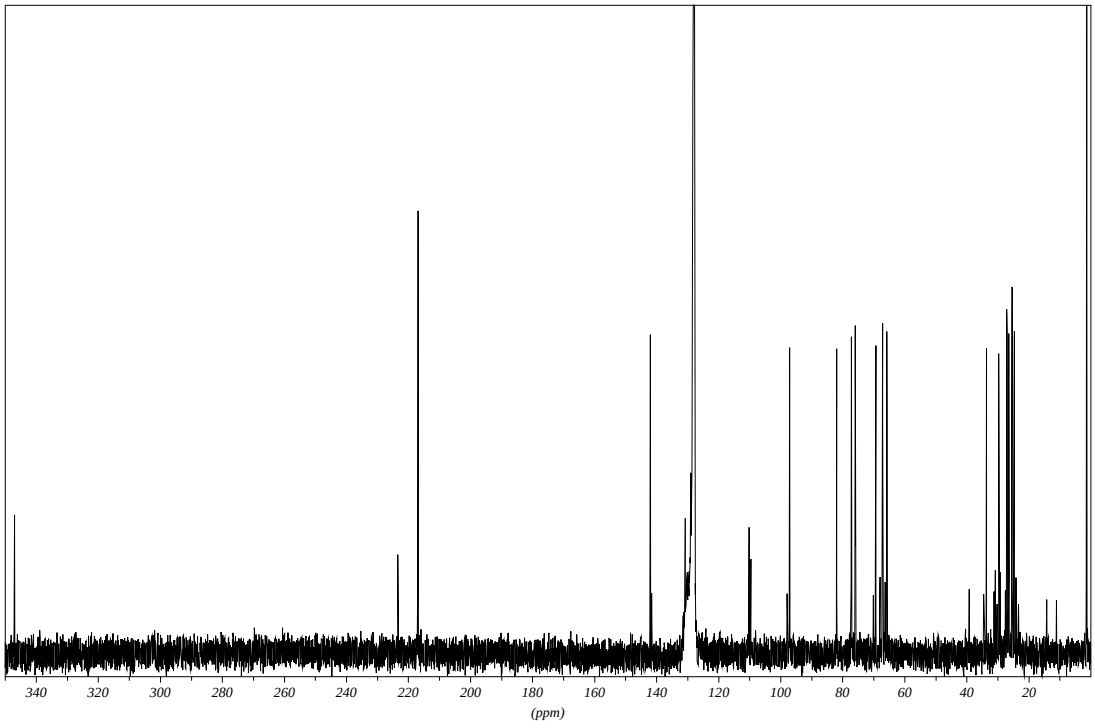


**(*3S,5R/S,4´R,5´´S,4´´´R*)-Pentacarbonyl{2´,2´,2´´´,2´´´-tetramethyl-3-(1,3-dioxacyclopent-4´-yl)-5-[5´´-(1,3-dioxacyclopent-4´´´-yl)-oxacyclo-pent-2-en-3´´-ylmethyl]-2-oxacyclopent-1-yliden}chrom(0) *[35]a/b***

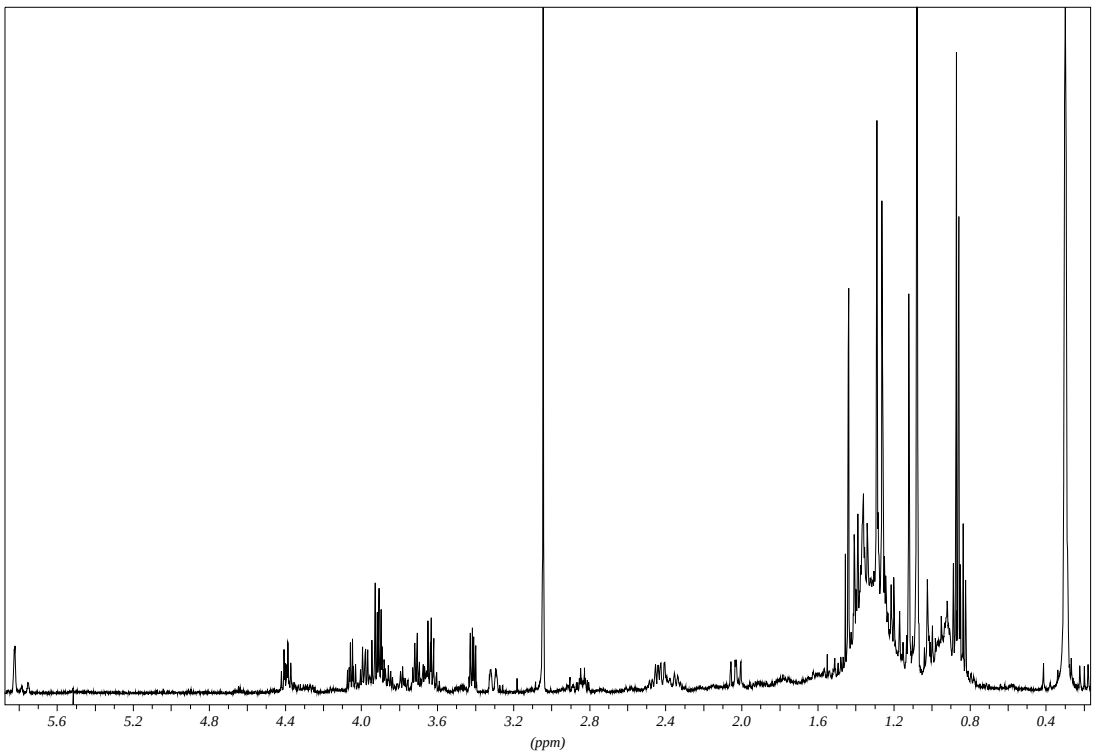
$^{1}$H-NMR-Spektrum von ***[35]a*** (500 MHz, $C_6D_6$):



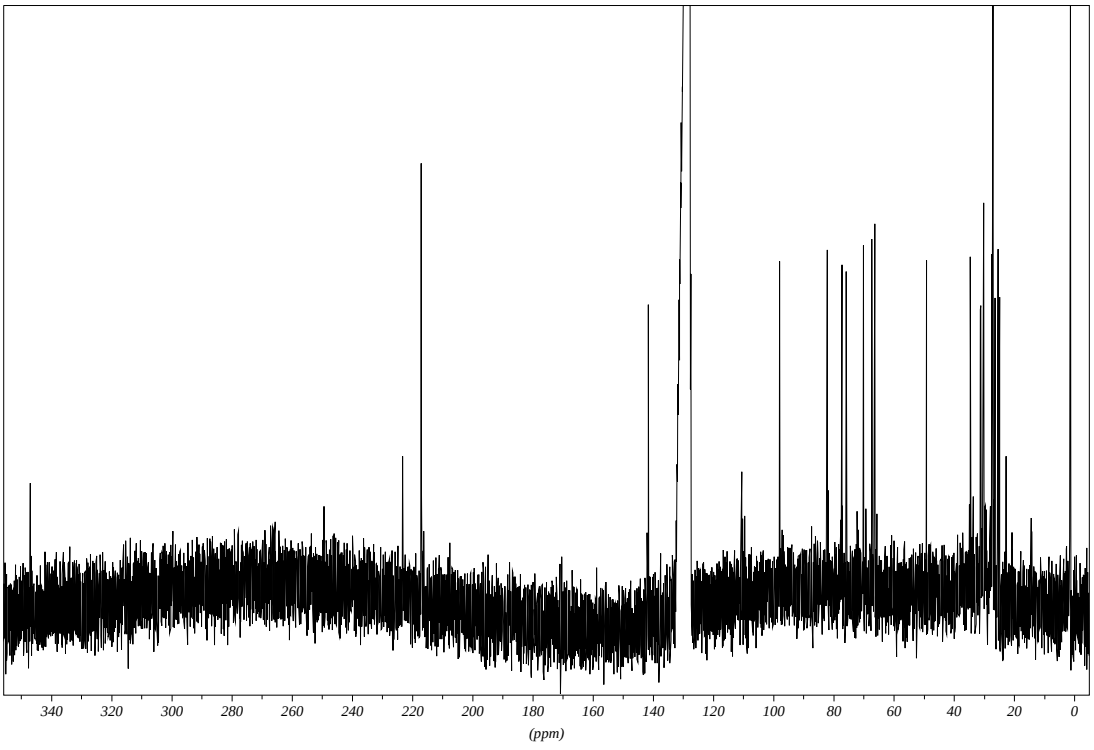

$^{13}$C-NMR-Spektrum von ***[35]a*** (500 MHz, $C_6D_6$):



$^{1}$H-NMR-Spektrum von ***[35]b*** (500 MHz, $C_6D_6$):

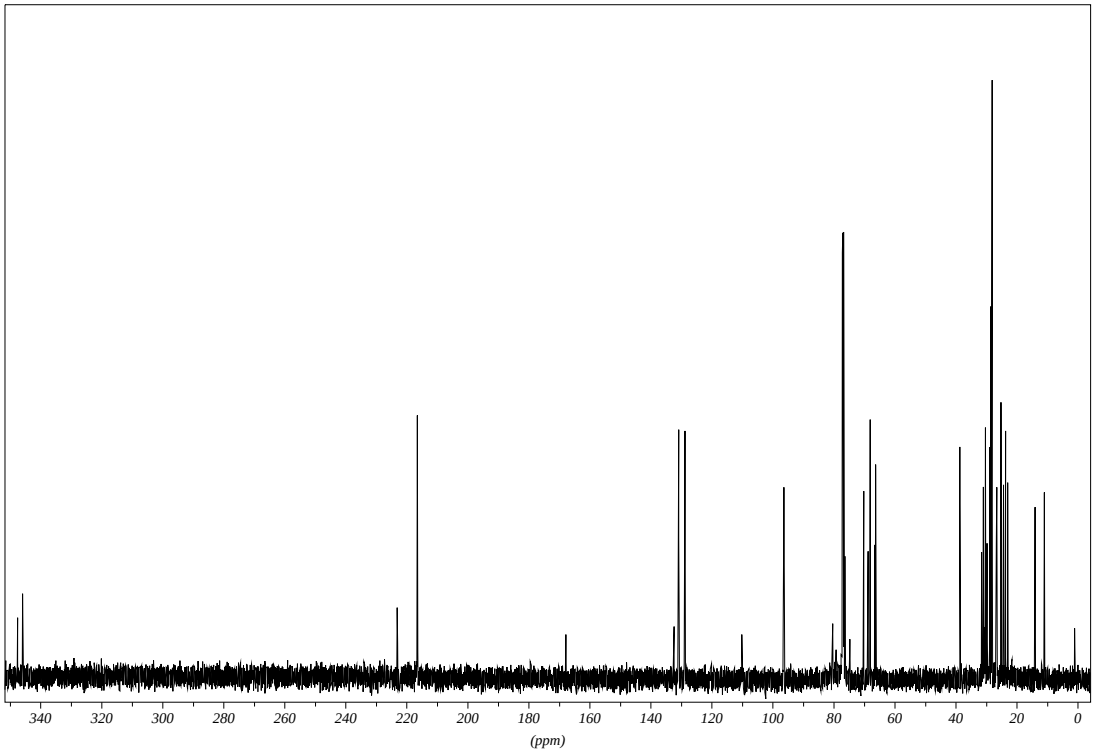



$^{13}$C-NMR-Spektrum von ***[35]b*** (500 MHz, $C_6D_6$):



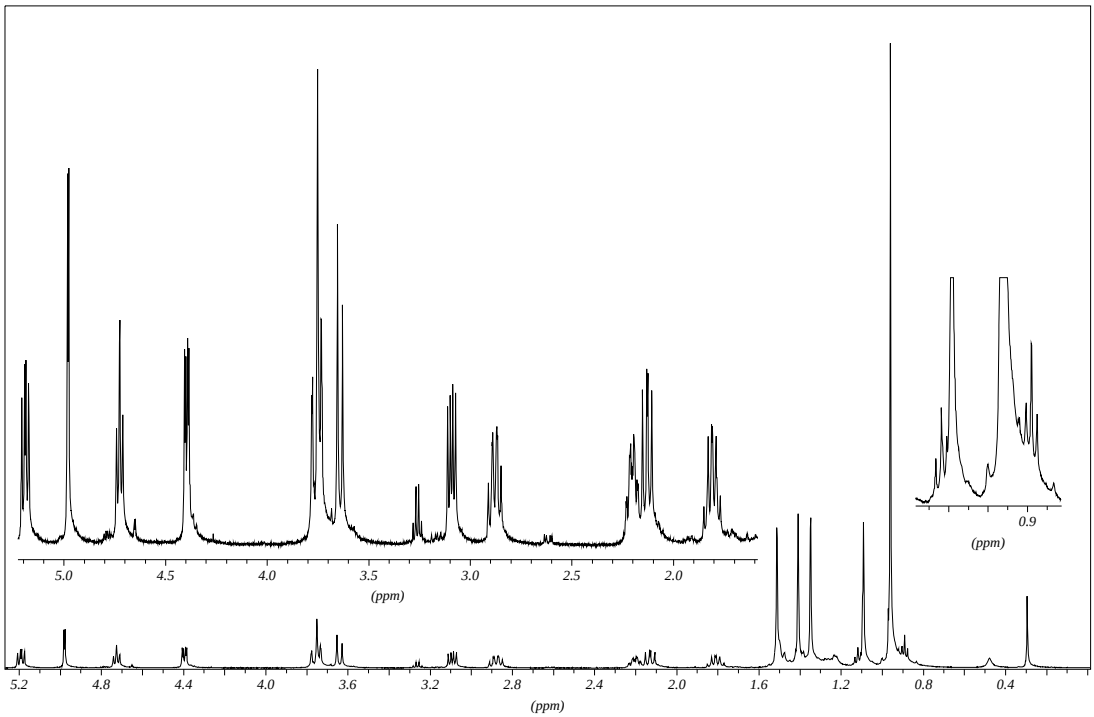
**(*1R/S,4S/R,7S,4´R*)-Pentacarbonyl{7-(2´,2´-dimethyl-1,3-dioxa-cyclopent-4´-yl)-6-oxaspiro[3.4]oct-5-yliden}chrom(0) *[36]a/b***

$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

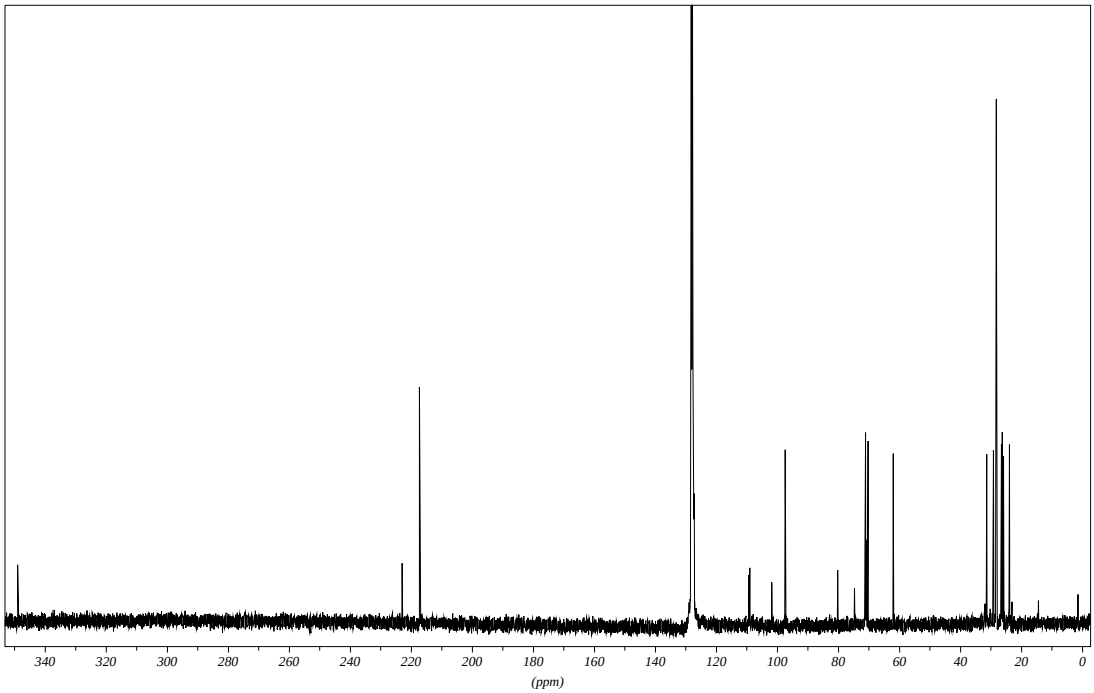


**(*1S,4R,7R,1´R,2´S,6´S,9´R*)-Pentacarbonyl{1-(dimethylethoxy)-7-(4´,4´,11´,11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]-dodec-6´-yl)-6-oxaspiro[3.4]oct-5-yliden}chrom(0) *[40]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

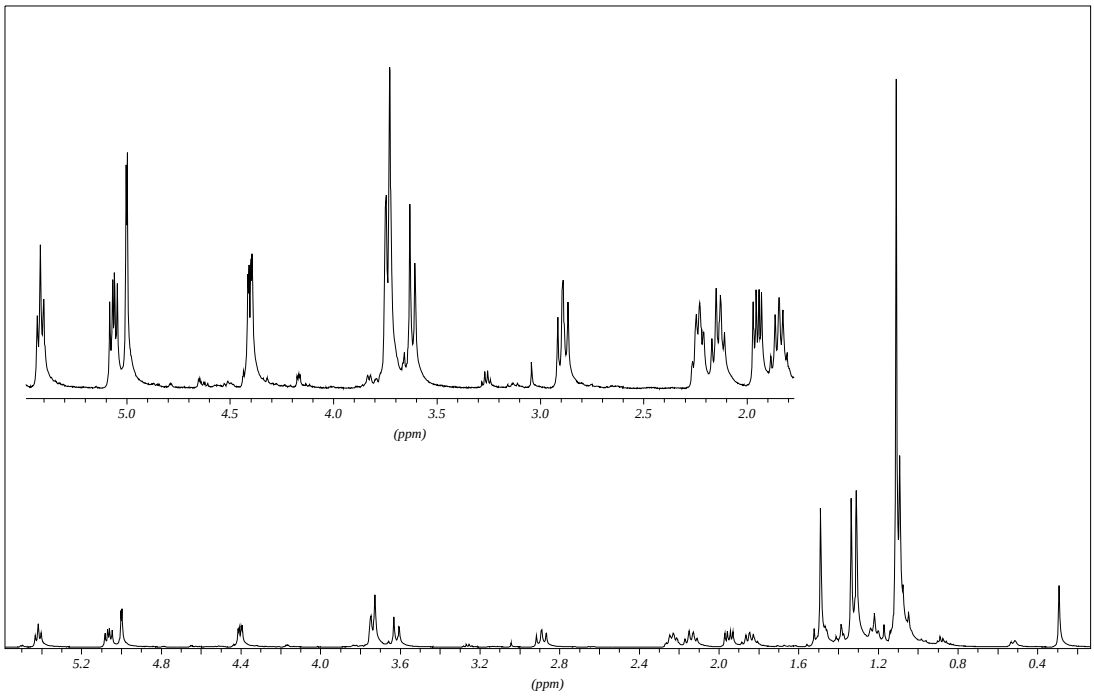



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

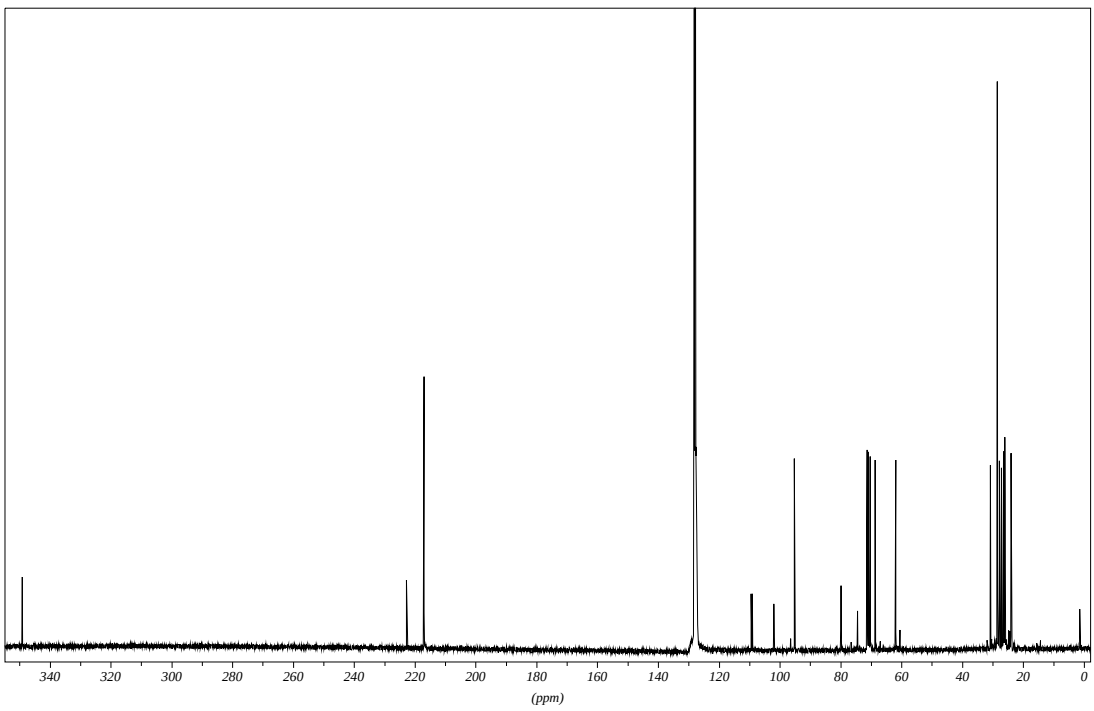


**(*1R,4S,7R,1´R,2´S,6´S,9´R*)-Pentacarbonyl{1-(dimethylethoxy)-7-(4´,4´,11´,11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]-dodec-6´-yl)-6-oxaspiro[3.4]oct-5-yliden}chrom(0) *[40]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

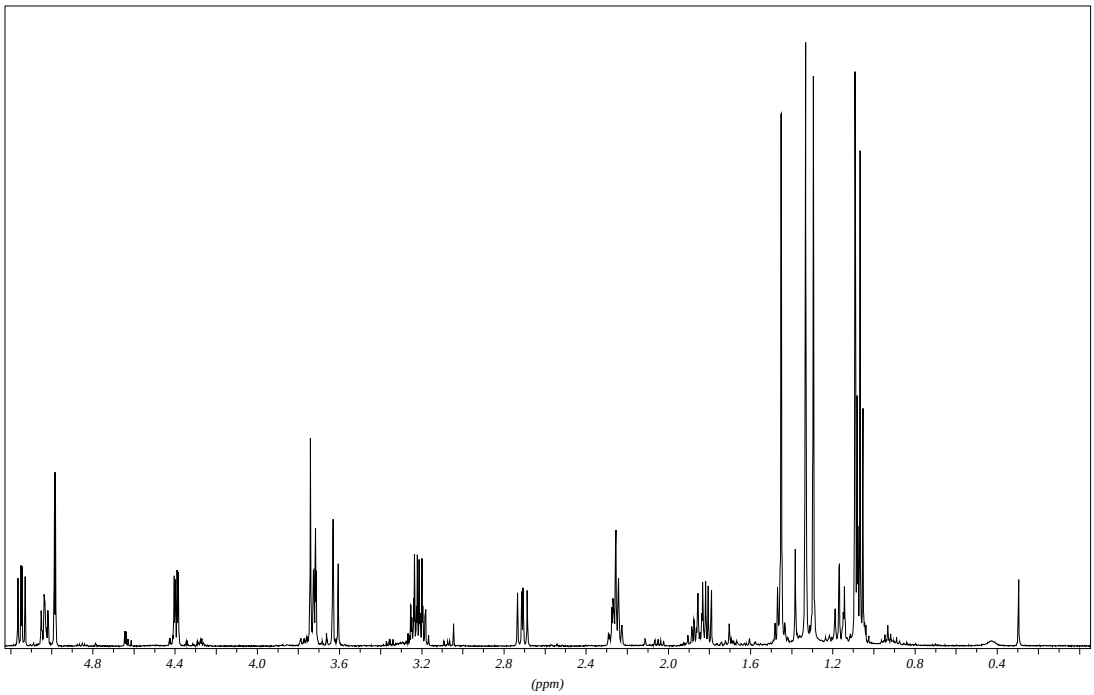



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

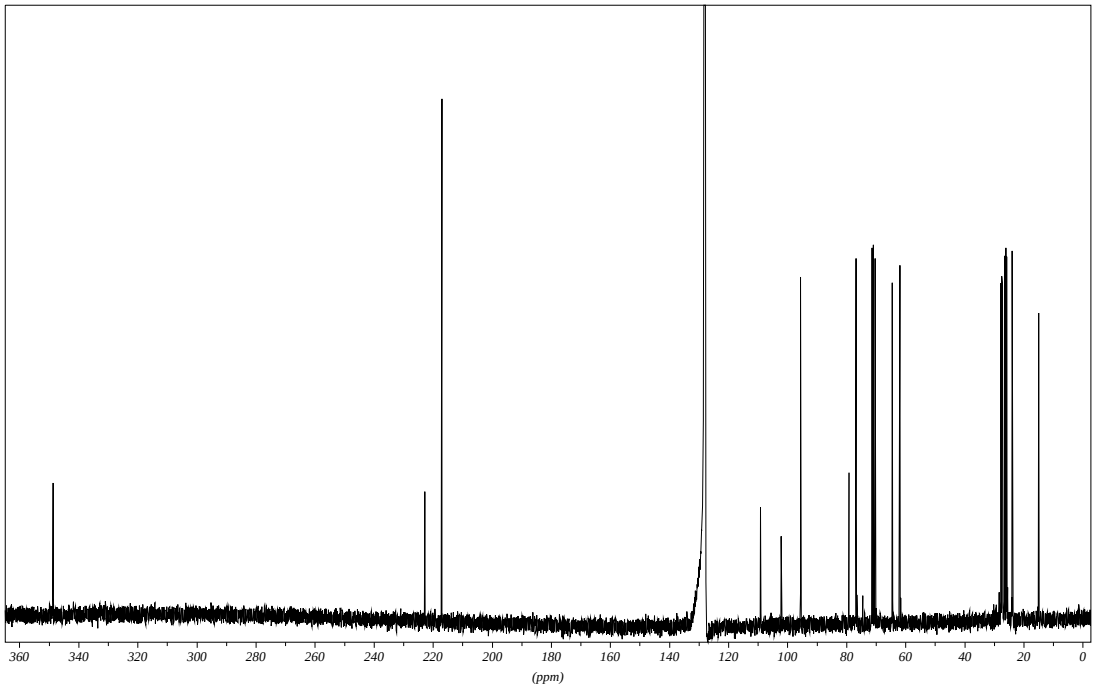


**(*1R,4S,7R,1´R,2´S,6´S,9´R*)-Pentacarbonyl{1-ethoxy-7-(4´,4´,11´,-11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´-yl)-6-oxaspiro[3.4]oct-5-yliden}chrom(0) *[42]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

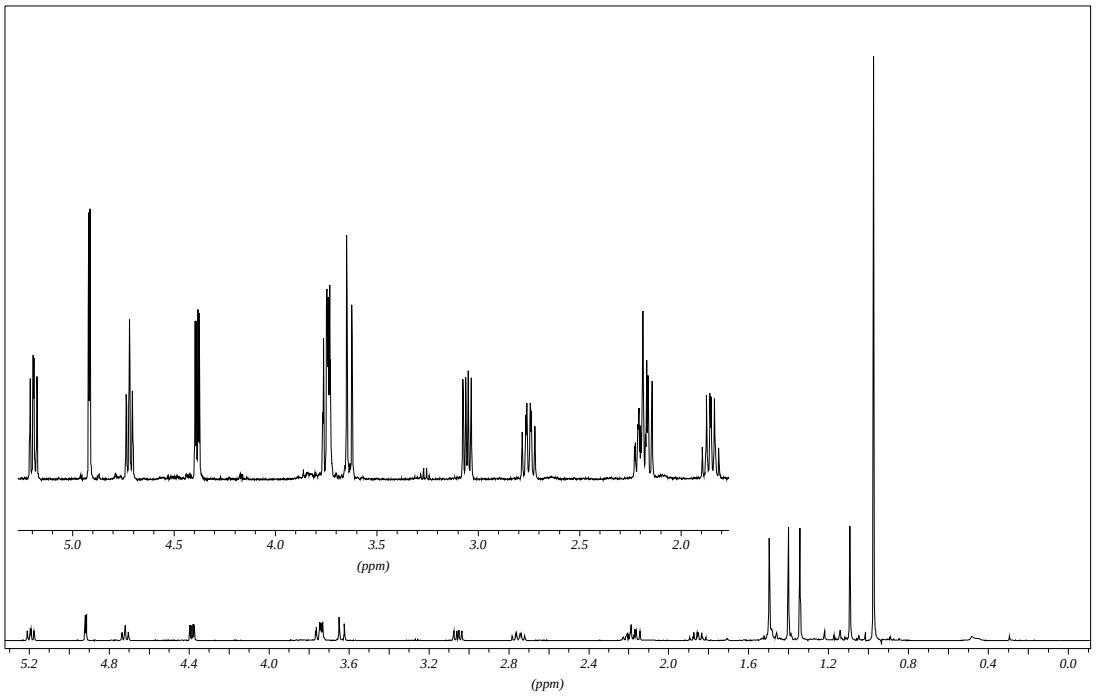



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

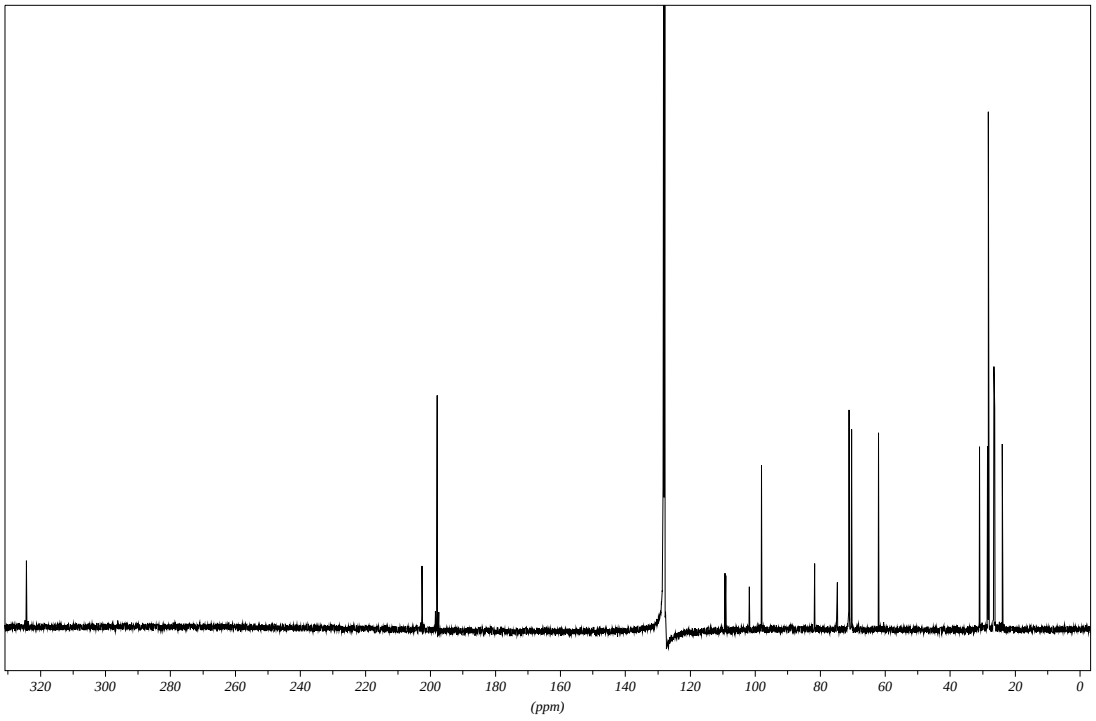


**(*1S,4R,7R,1´R,2´S,6´S,9´R*)-Pentacarbonyl{1-(dimethylethoxy)-7-(4´,4´,11´,11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]-dodec-6´-yl)-6-oxaspiro[3.4]oct-5-yliden}wolfram(0) *[43]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

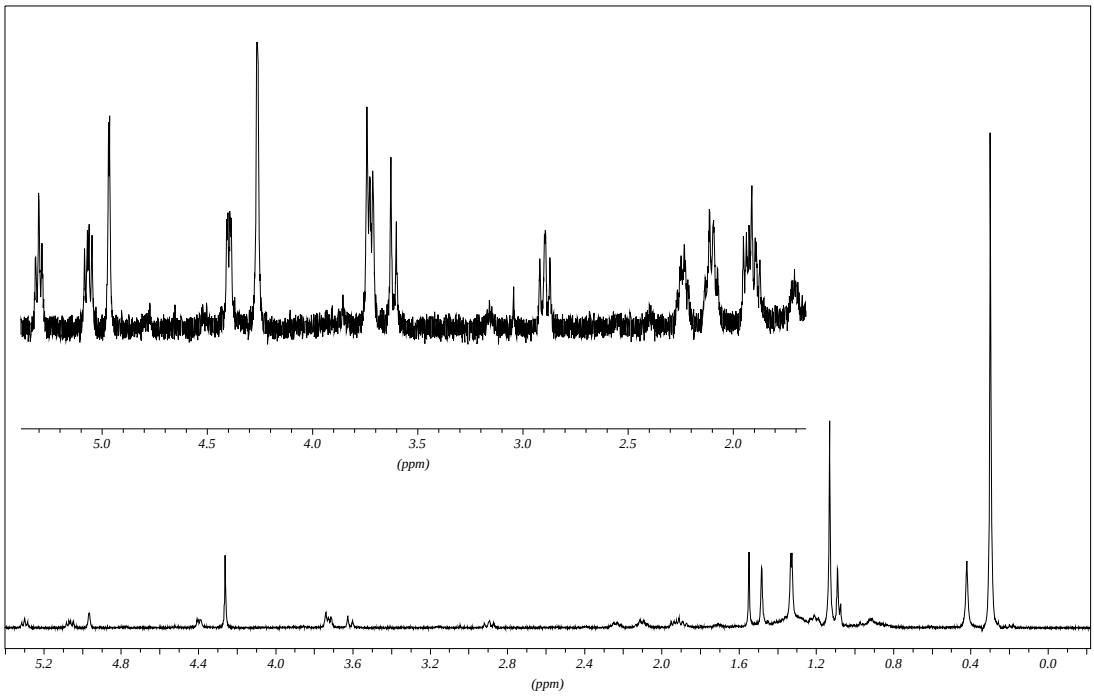



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

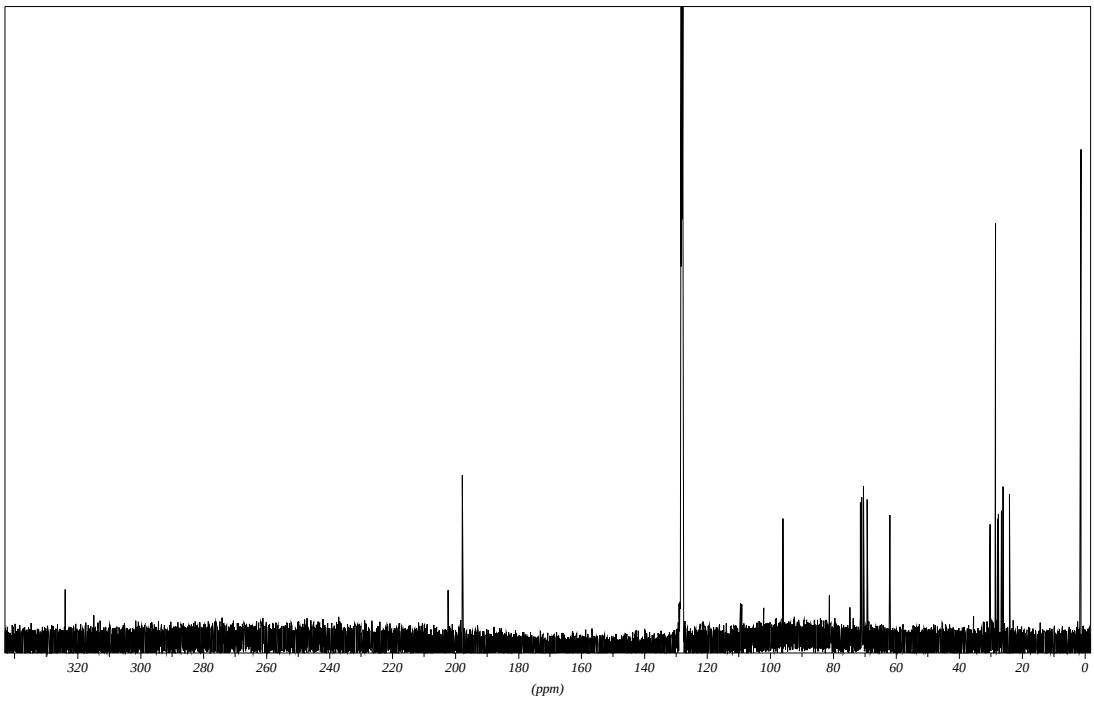


**(*1R,4S,7R,1´R,2´S,6´S,9´R*)-Pentacarbonyl{1-(dimethylethoxy)-7-(4´,4´,11´,11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]-dodec-6´-yl)-6-oxaspiro[3.4]oct-5-yliden}wolfram(0) *[43]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

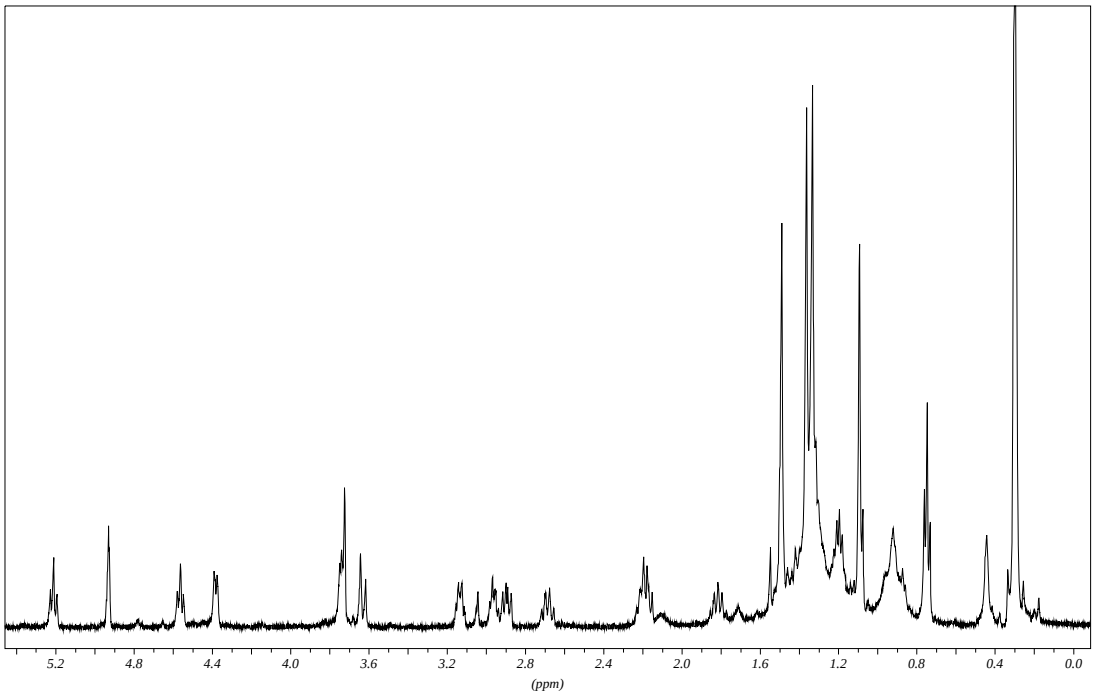



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

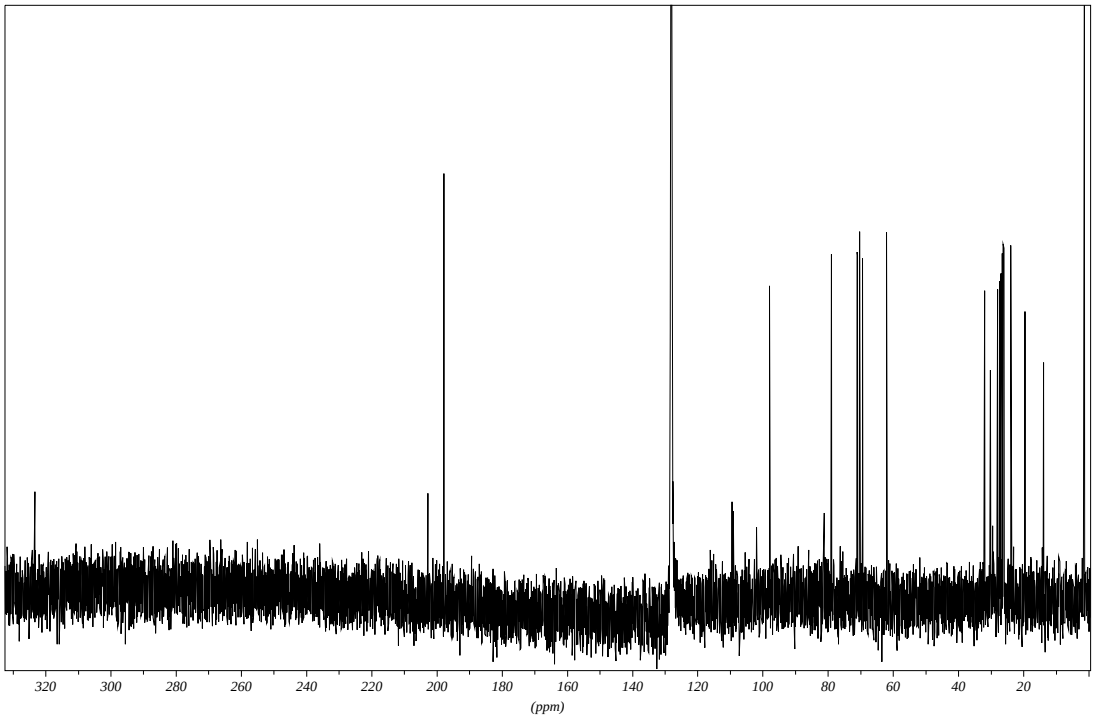


**(*1S,4R,7R,1´R,2´S,6´S,9´R*)-Pentacarbonyl{1-butoxy-7-(4´,4´,11´,-11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´-yl)-6-oxaspiro[3.4]oct-5-yliden}wolfram(0) *[44]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

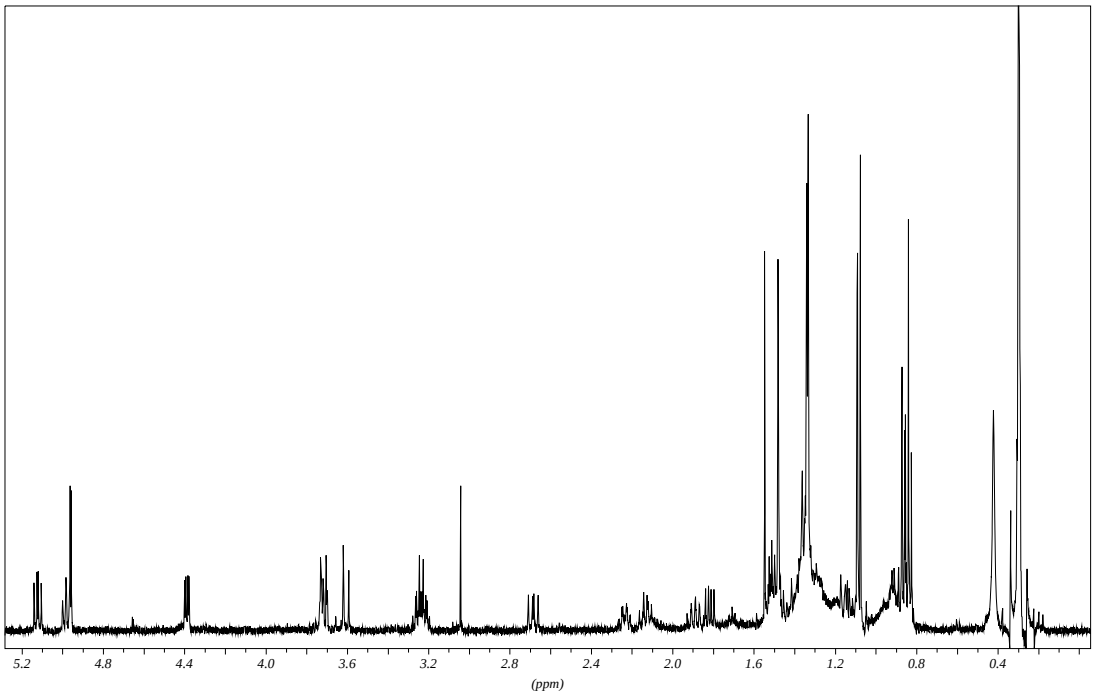



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

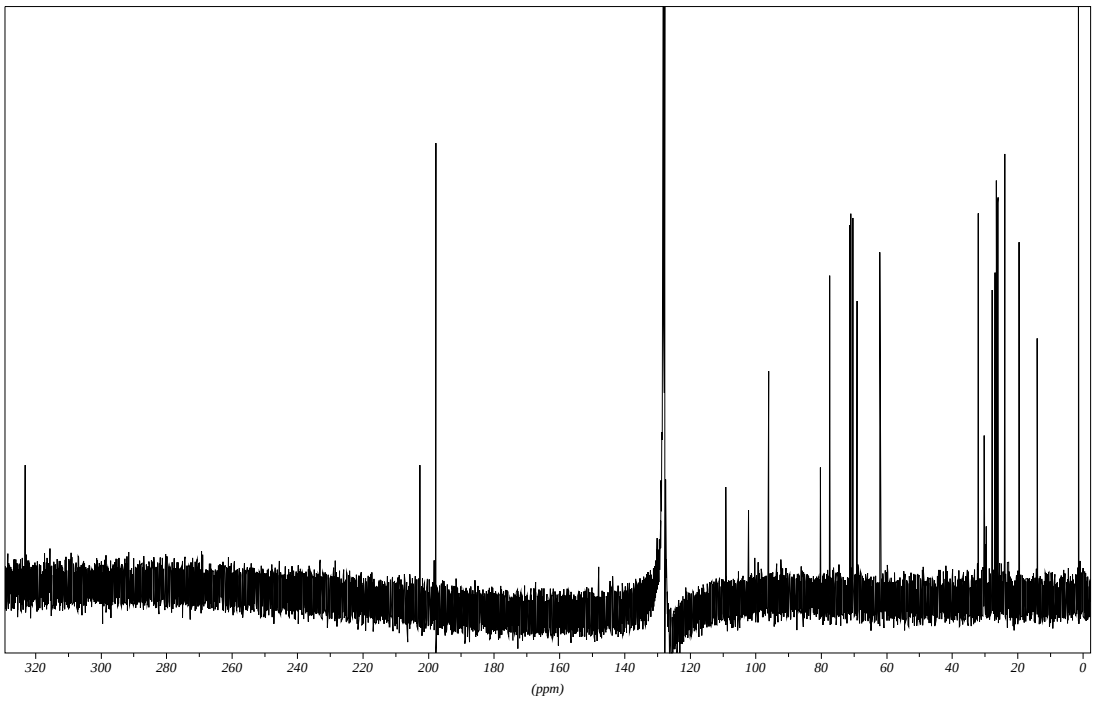


**(*1R,4S,7R,1´R,2´S,6´S,9´R*)-Pentacarbonyl{1-butoxy-7-(4´,4´,11´,-11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´-yl)-6-oxaspiro[3.4]oct-5-yliden}wolfram(0) *[44]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

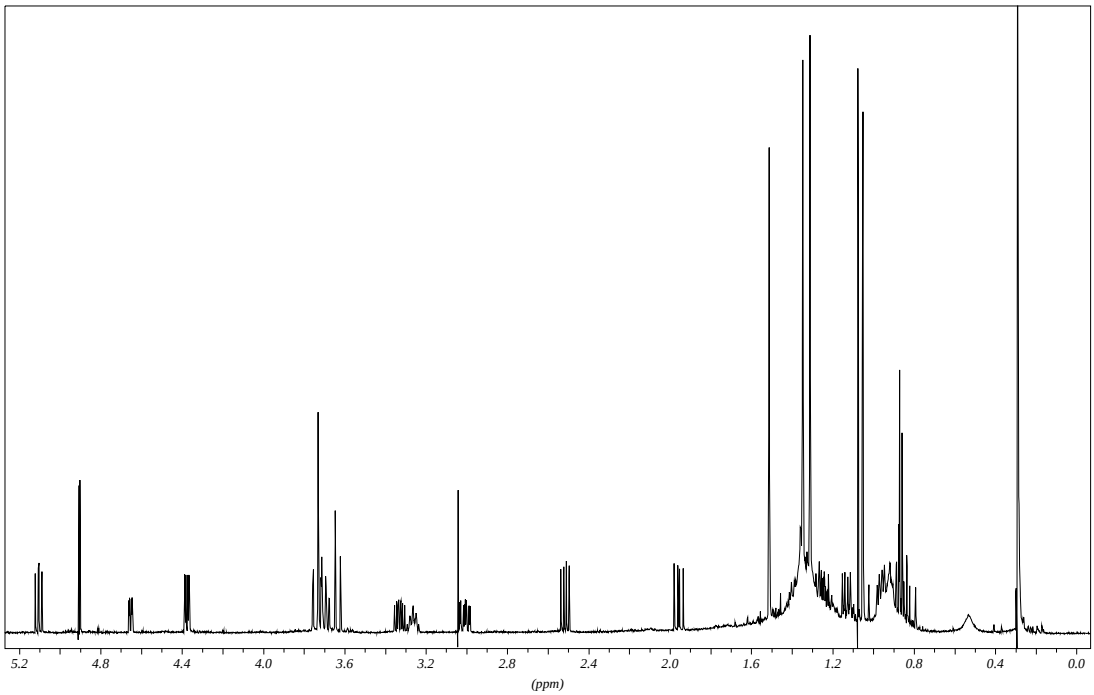



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

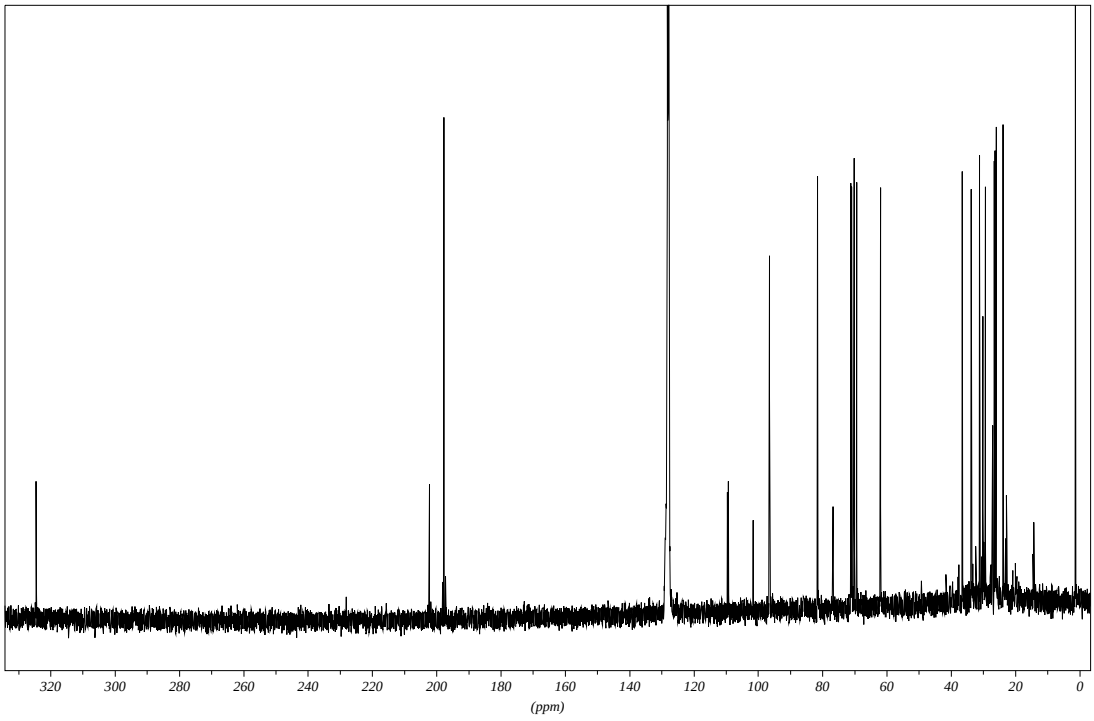


**(*3R,5R,1´S,5´R,1´´R,2´´S,6´´S,9´´R*)-Pentacarbonyl{3-(4´´,4´´,-11´´,11´´-tetramethyl-3,5,7,10,12-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´´-yl)-spiro[2-oxabicyclo[3.2.0]heptan-7´,5-2-oxacyclopent]-1-yliden}wolfram(0) *[45]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

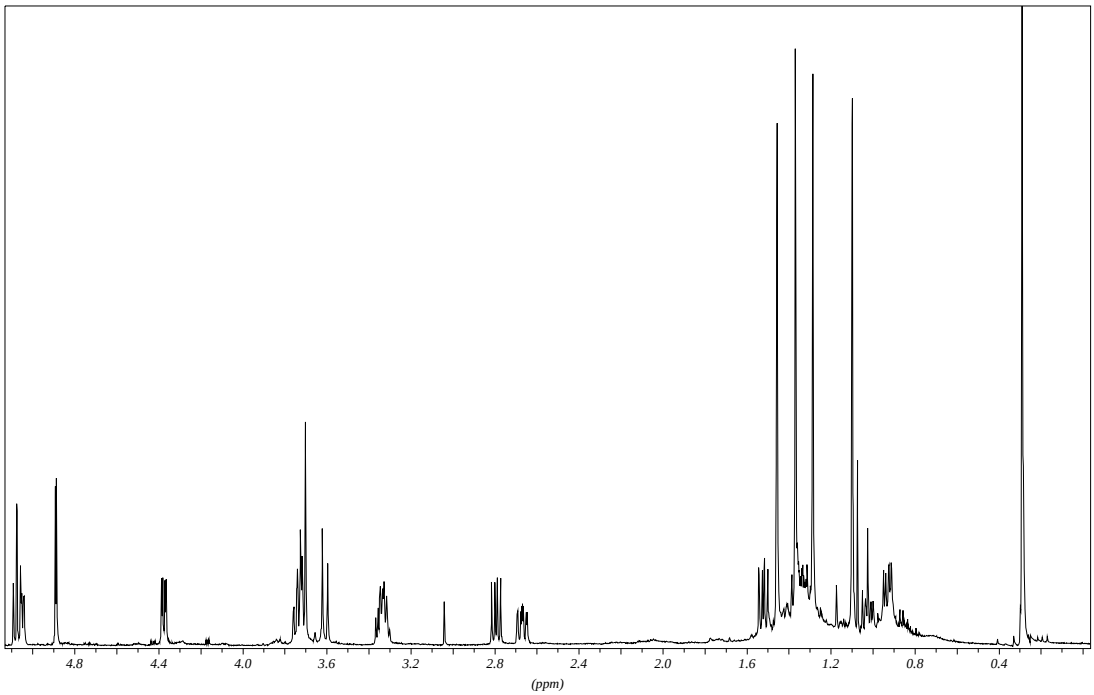



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

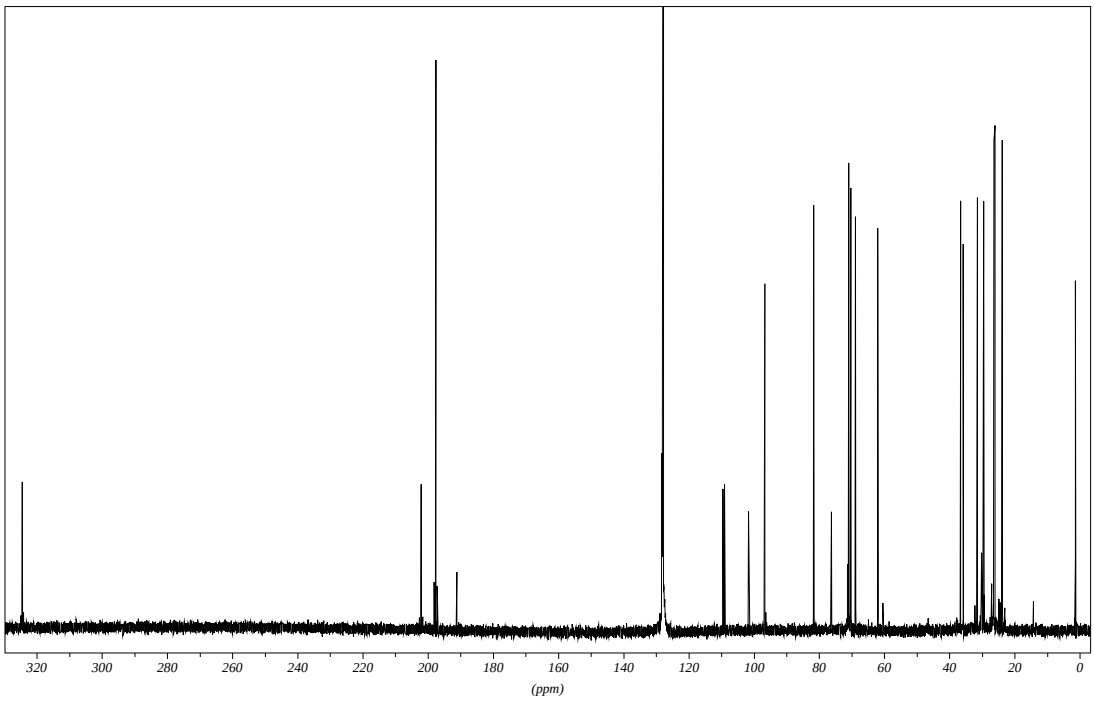


**(*3R,5S,1´R,5´S,1´´R,2´´S,6´´S,9´´R*)-Pentacarbonyl{3-(4´´,4´´,-11´´,11´´-tetramethyl-3,5,7,10,12-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´´-yl)-spiro[2-oxabicyclo[3.2.0]heptan-7´,5-2-oxacyclopent]-1-yliden}wolf-ram(0) *[45]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

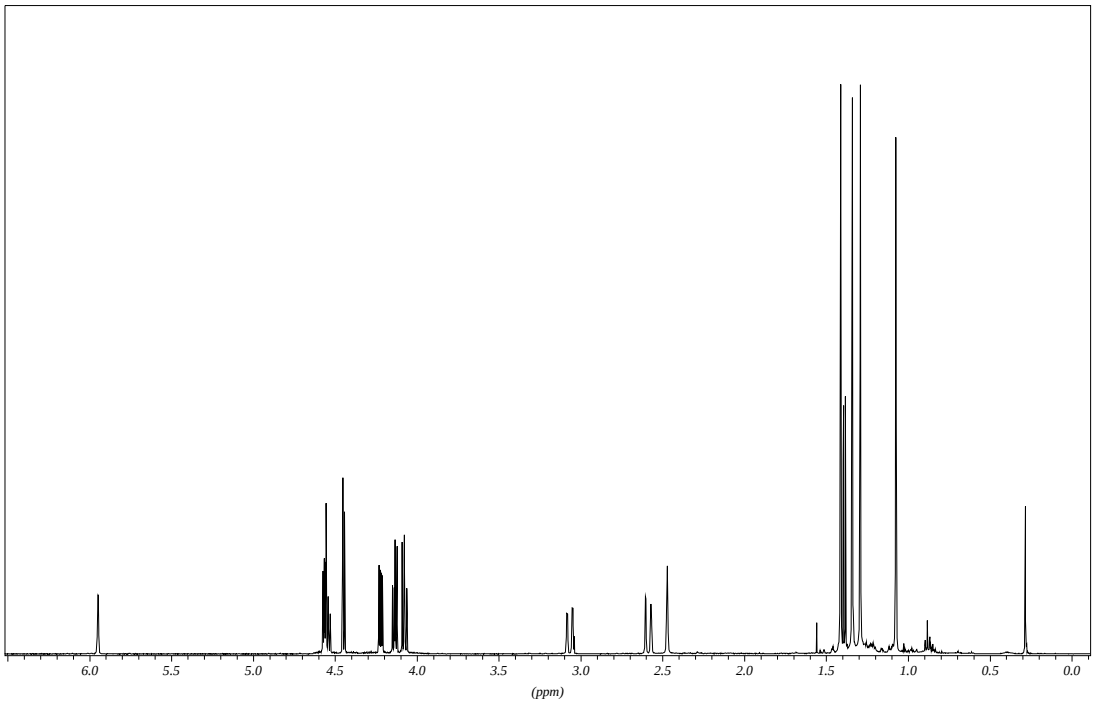



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

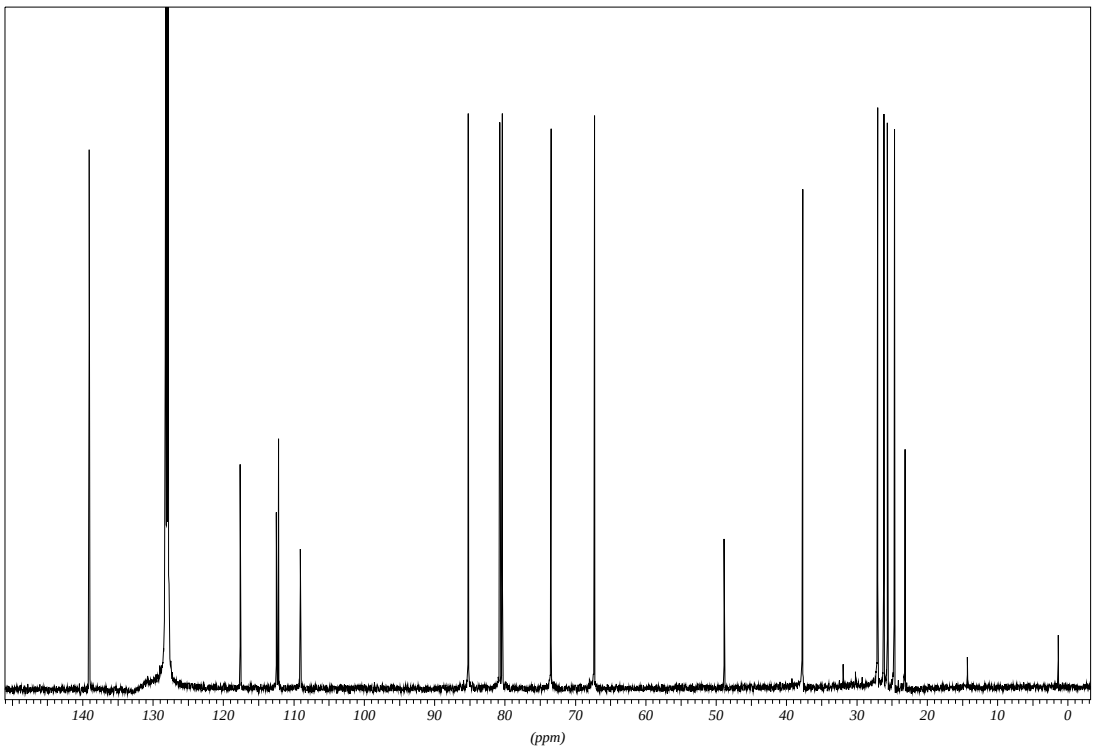


**Bis{(*5S,1´S,5´R,6´R,4´´R*)-3´,3´-dimethyl-6´-(2´´,2´´-dimethyl-1,3-dioxacyclopent-4´´-yl)-spiro[2,4,7-trioxabicyclo[3.3.0]octan-8´,5-oxacyclopent-2-en-3-yl]}methan *[46]***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

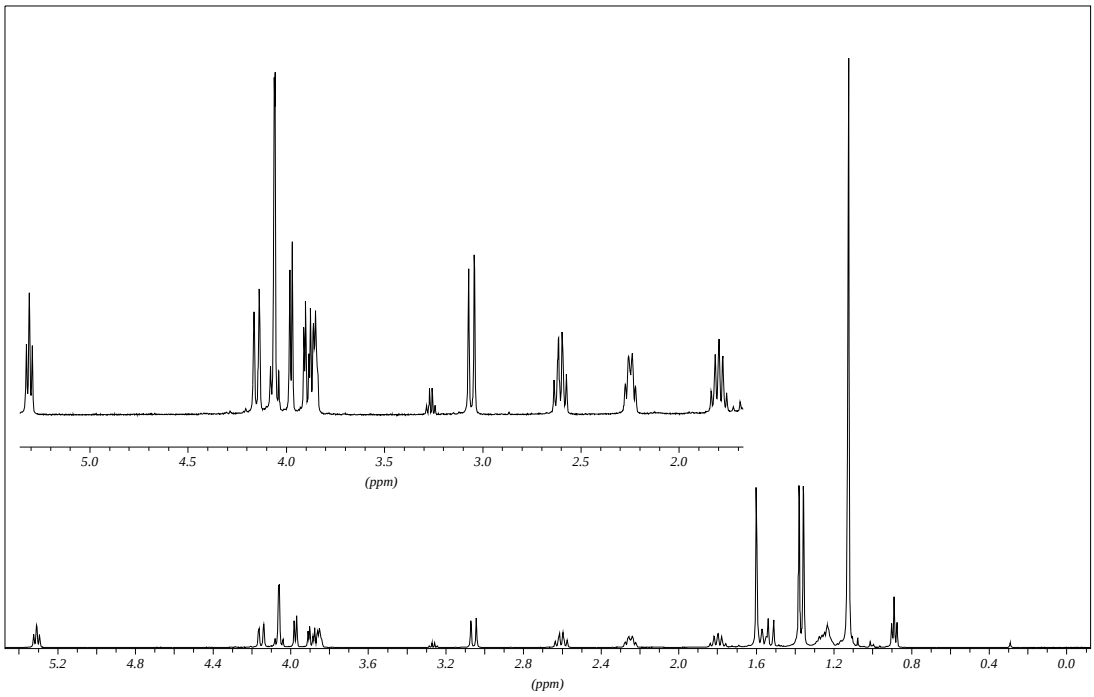



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

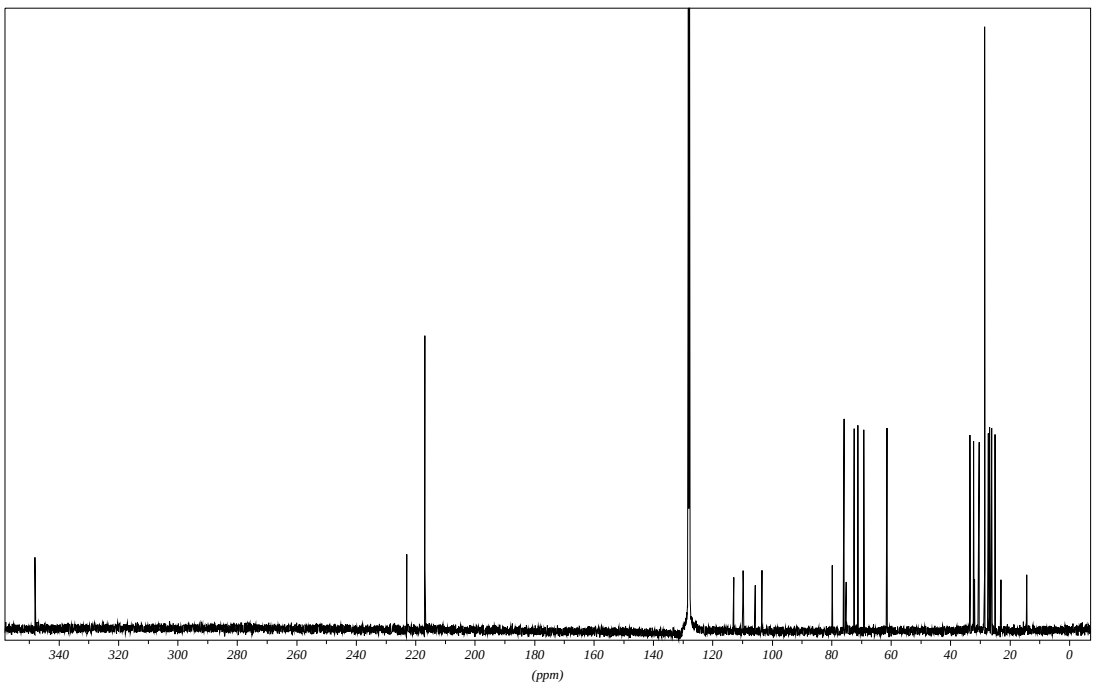


**(*1R,4S,7R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-1-(dimethyleth-oxy)-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxa-spiro-[3.4]oct]-5-yliden}chrom(0)** ***[47]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

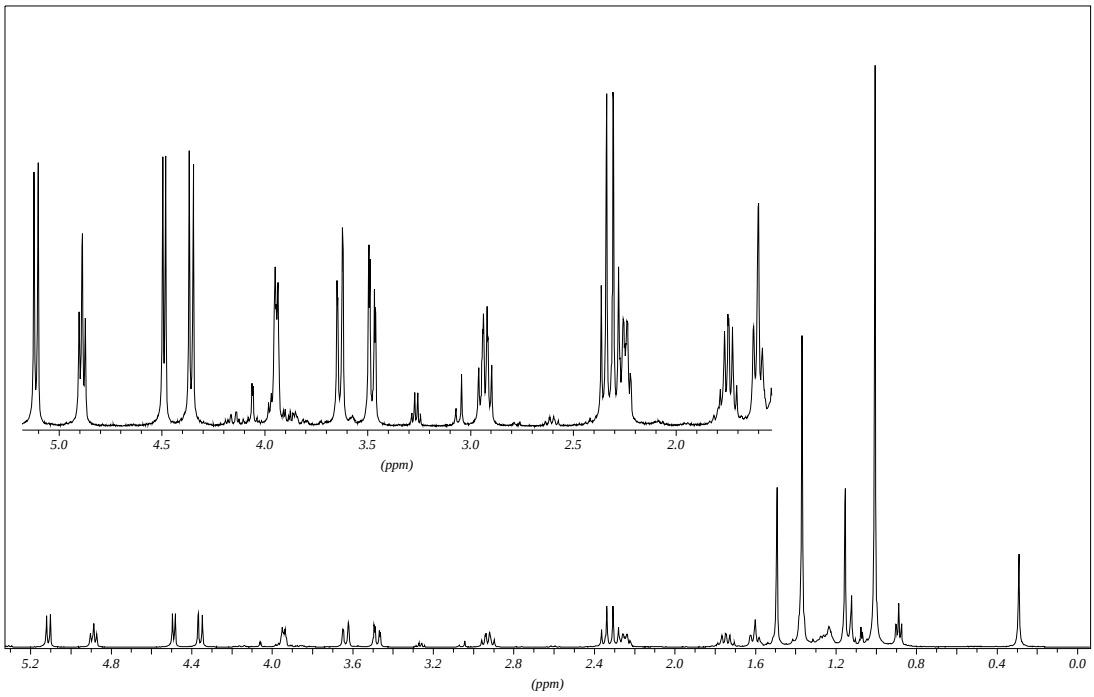



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

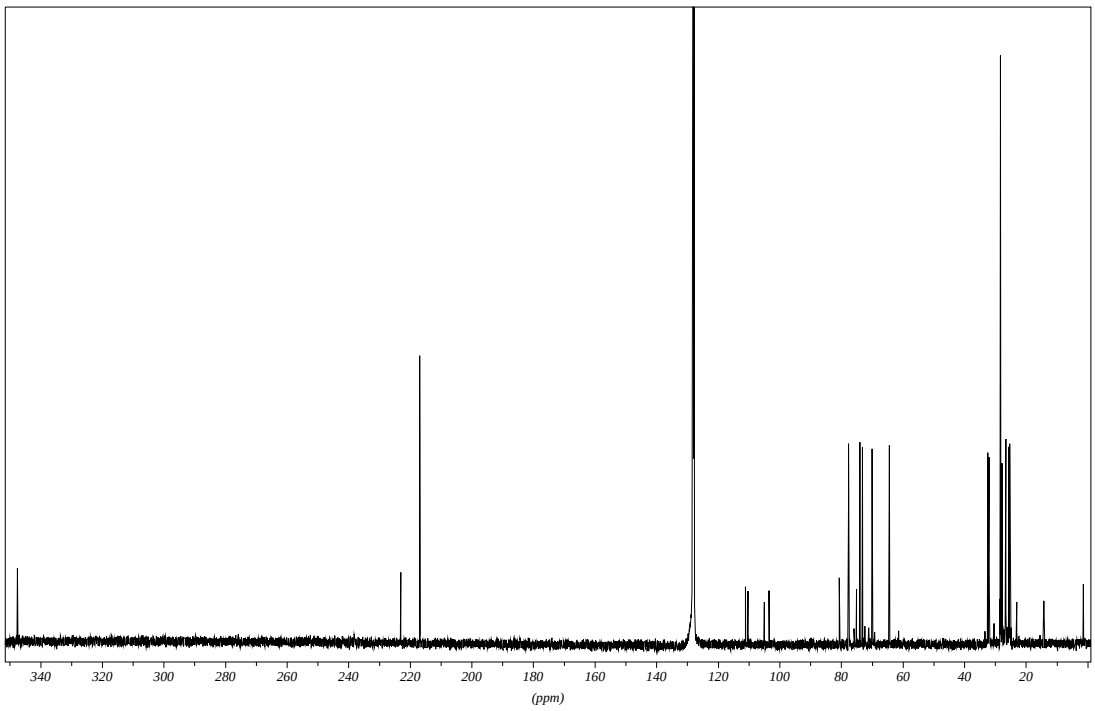


**(*1S,4R,7R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-1-(dimethyleth-oxy)-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxa-spiro-[3.4]oct]-5-yliden}chrom(0)** ***[47]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

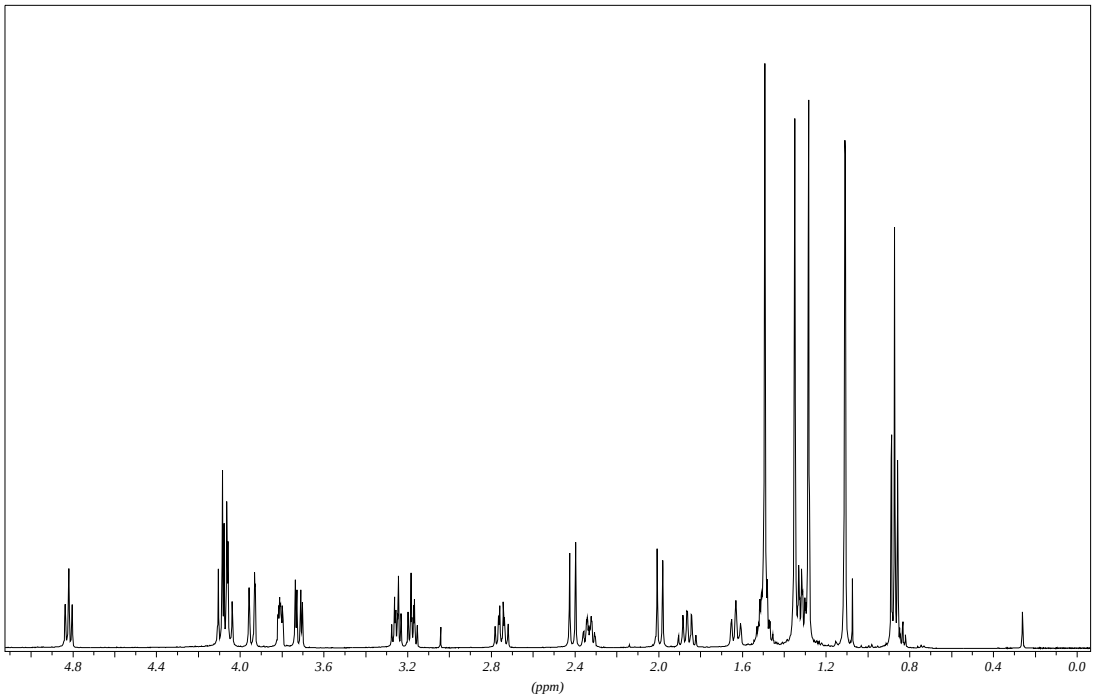



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

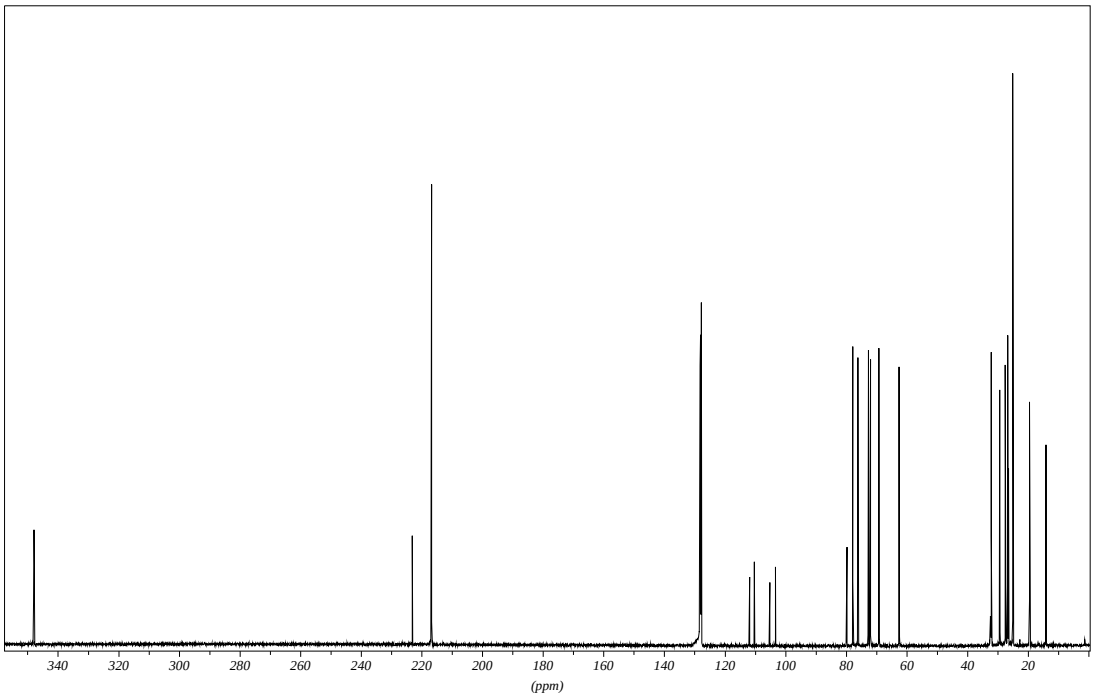


**(*1R,4S,7R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-1-*n*-butoxy-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro-[3.4]oct]-5-yliden}chrom(0) *[48]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

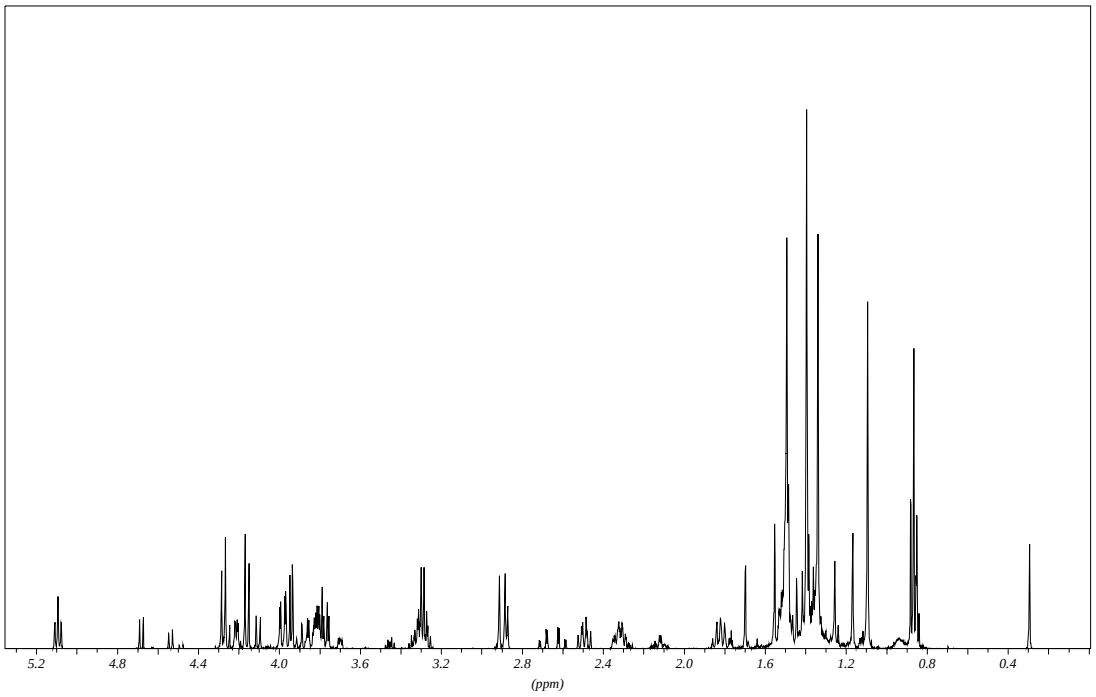



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

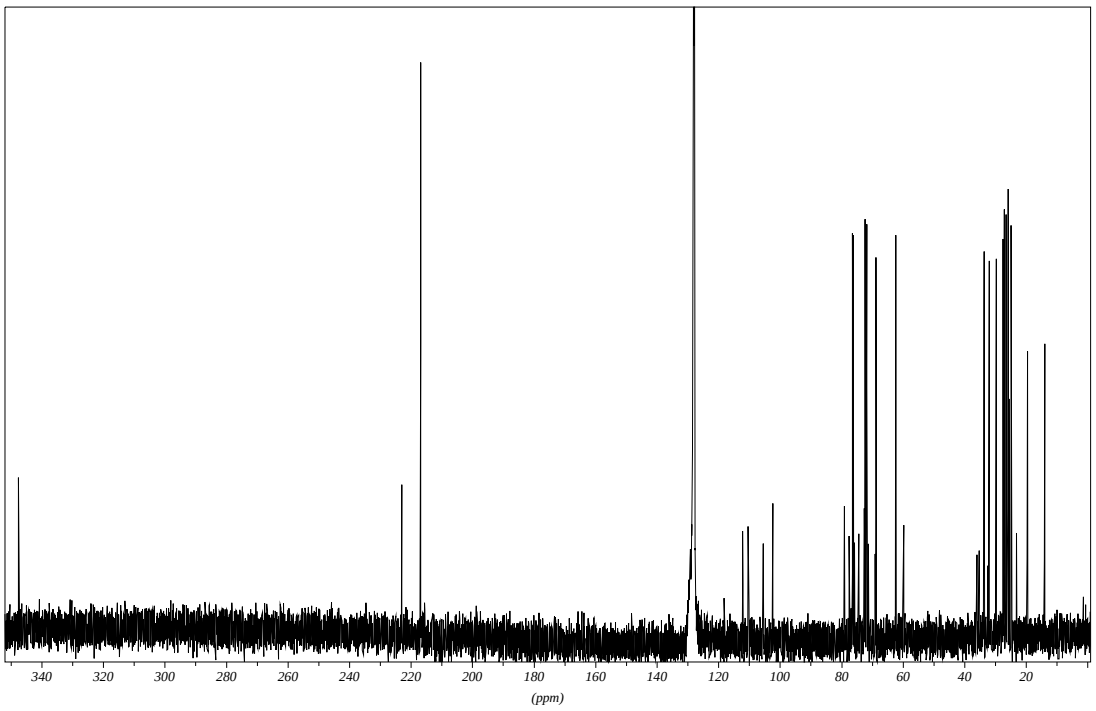


**(*1S,4R,7R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-1-*n*-butoxy-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro-[3.4]oct]-5-yliden}chrom(0) *[48]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

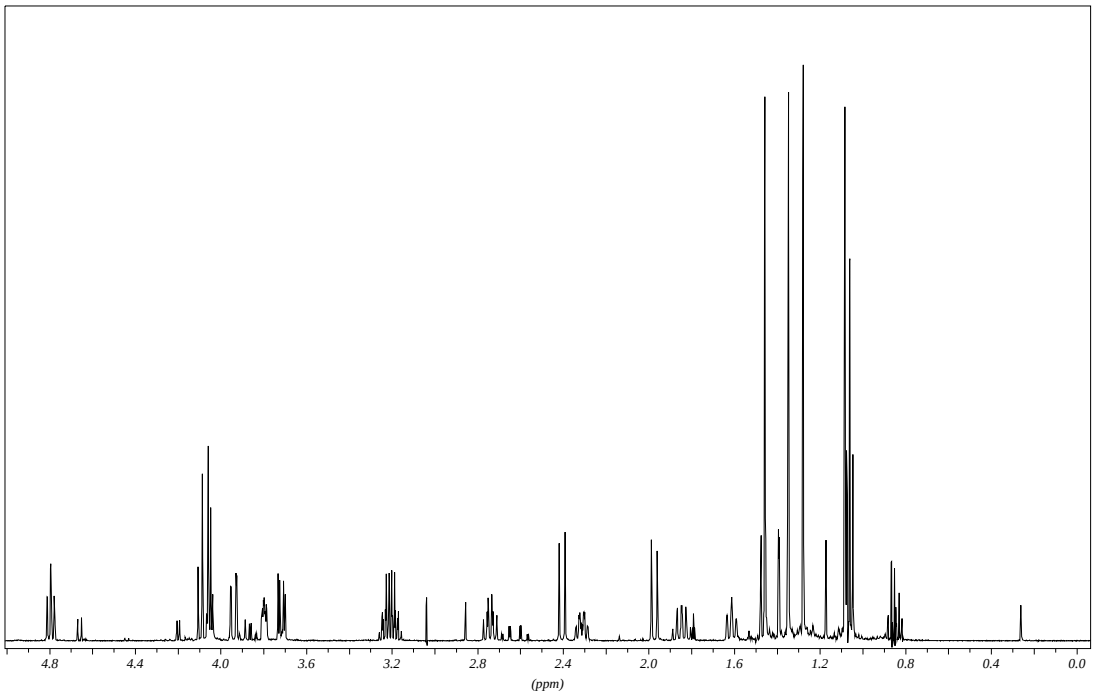



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

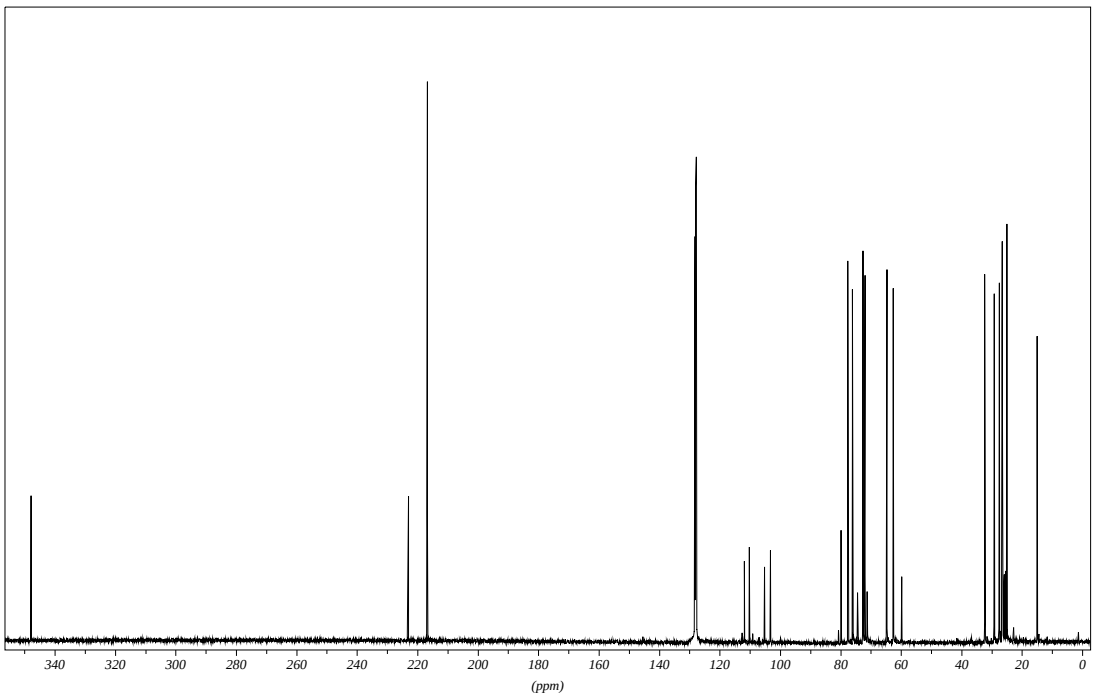


**(*1R,4S,7R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-1-ethoxy-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro[3.4]oct]-5-yliden}chrom(0) *[49]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

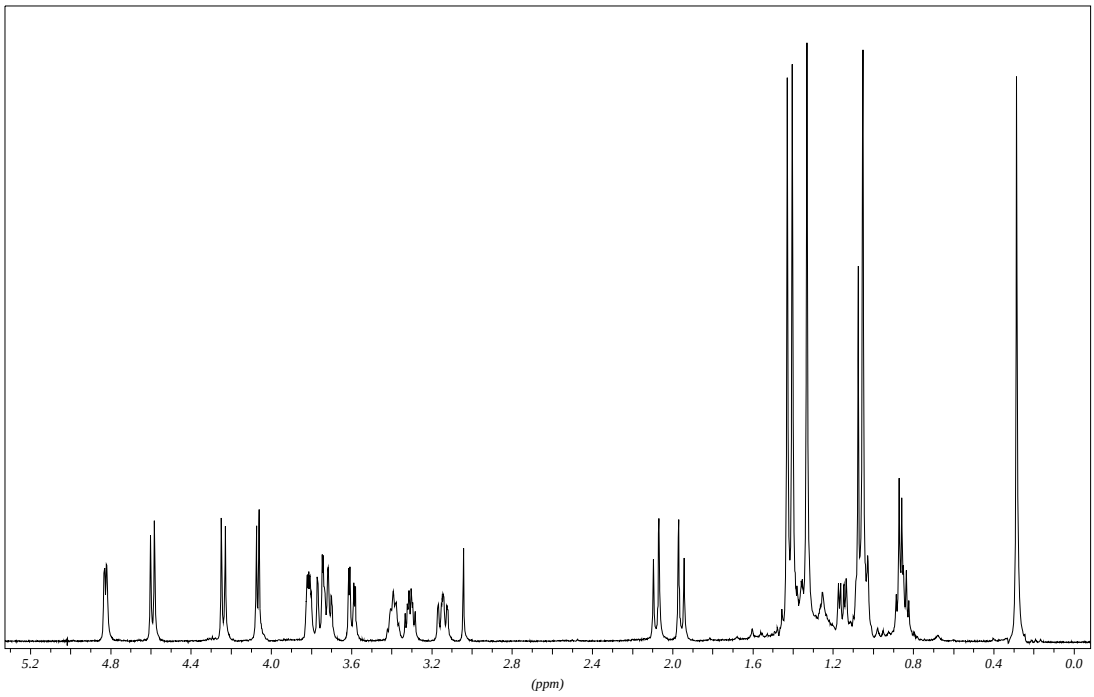



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

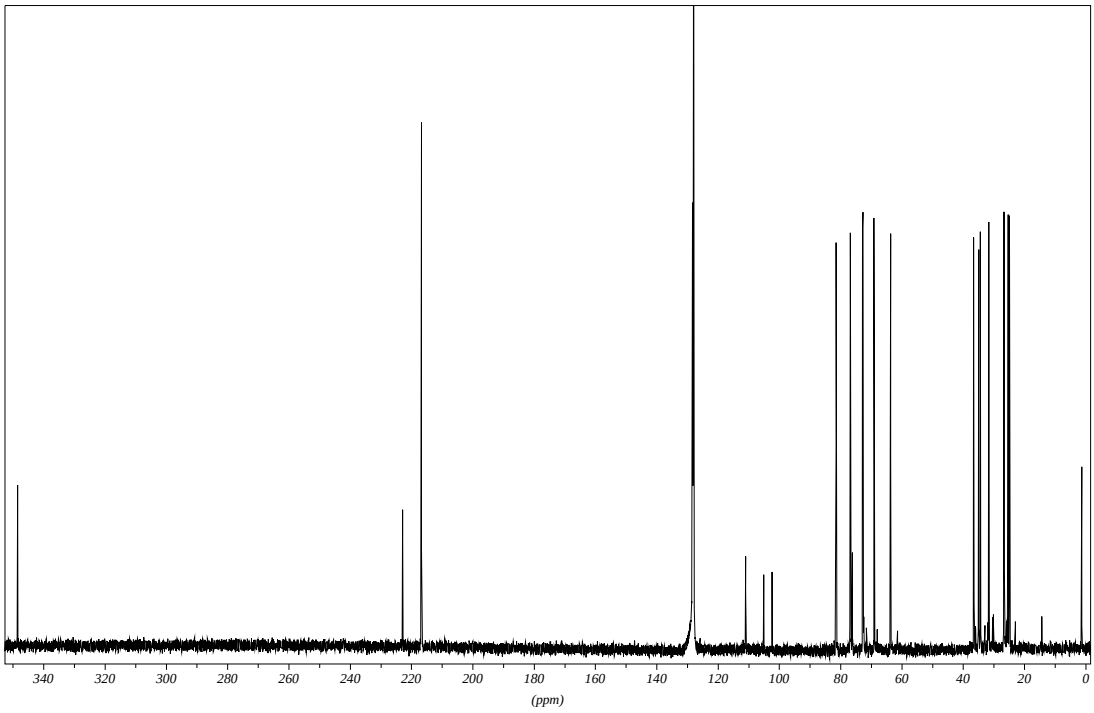


**(*3R,5S,1´R,5´S,1´´R,4´´S,6´´R*)-Pentacarbonyl{8´´,8´´,2´´´,2´´´-tetramethyl-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´´,4´´´-1,3-dioxacyclopentan-5´´,3]-spiro[2-oxabicyclo[3.2.0]heptan-7´,5]-2-oxacyclopent-1-yliden}chrom(0)** ***[50]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

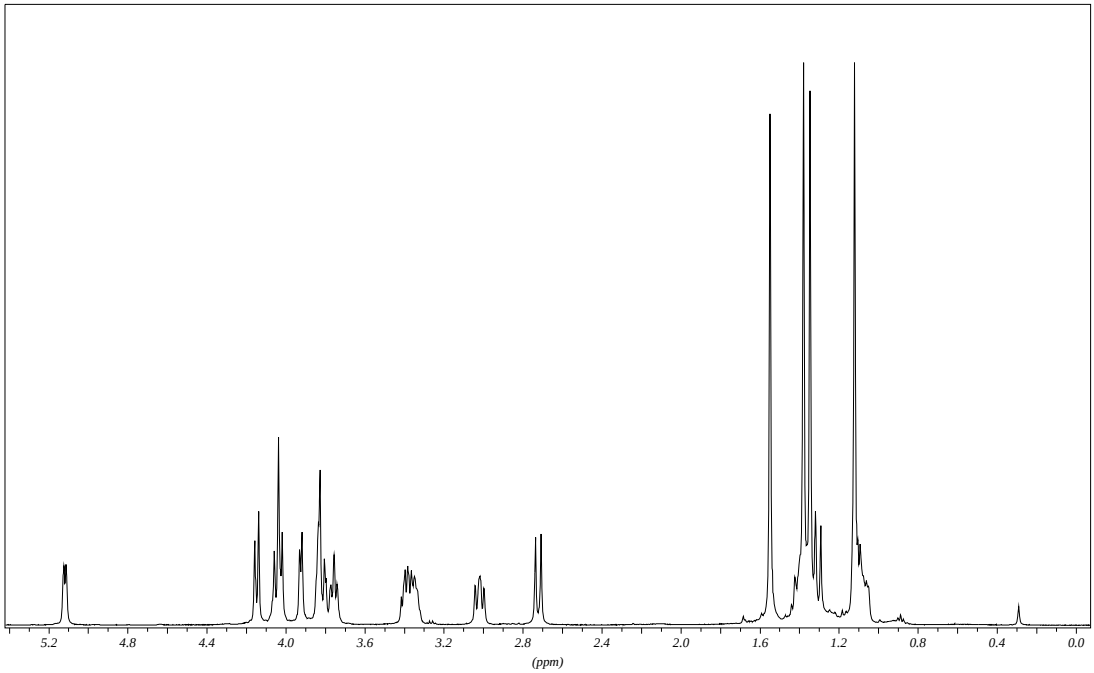



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

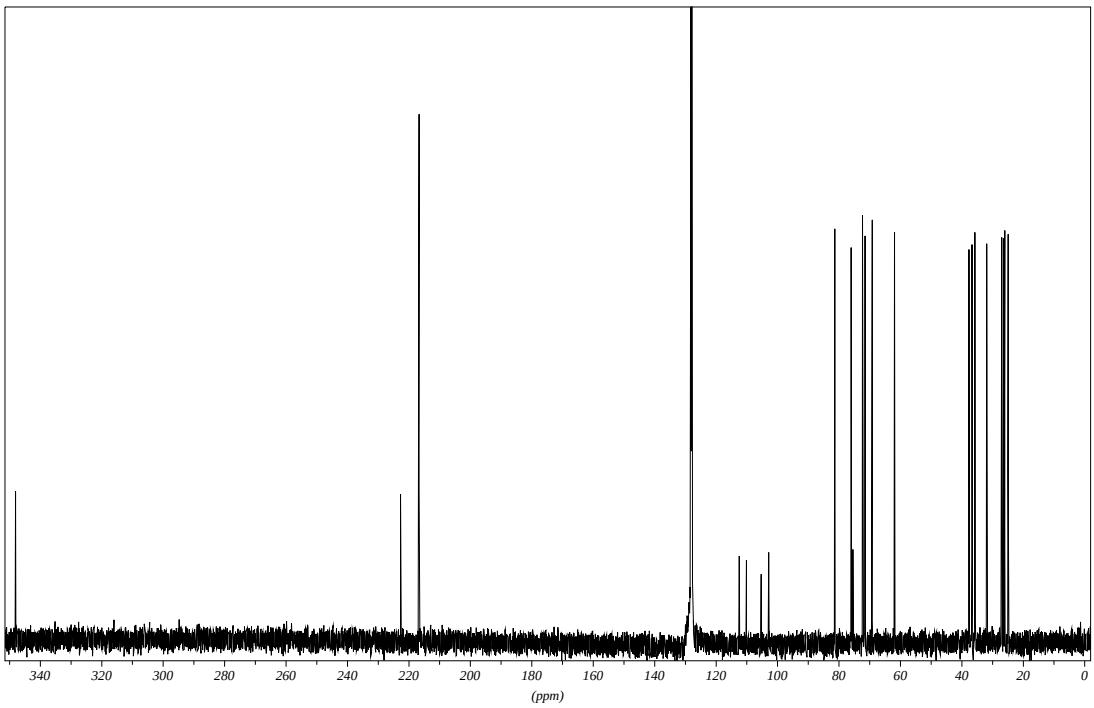


**(*3R,5R,1´S,5´R,1´´R,4´´S,6´´R*)-Pentacarbonyl{8´´,8´´,2´´´,2´´´-tetramethyl-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´´,4´´´-1,3-dioxacyclopentan-5´´,3]-spiro[2-oxabicyclo[3.2.0]heptan-7´,5]-2-oxacyclopent-1-yliden}chrom(0)** ***[50]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

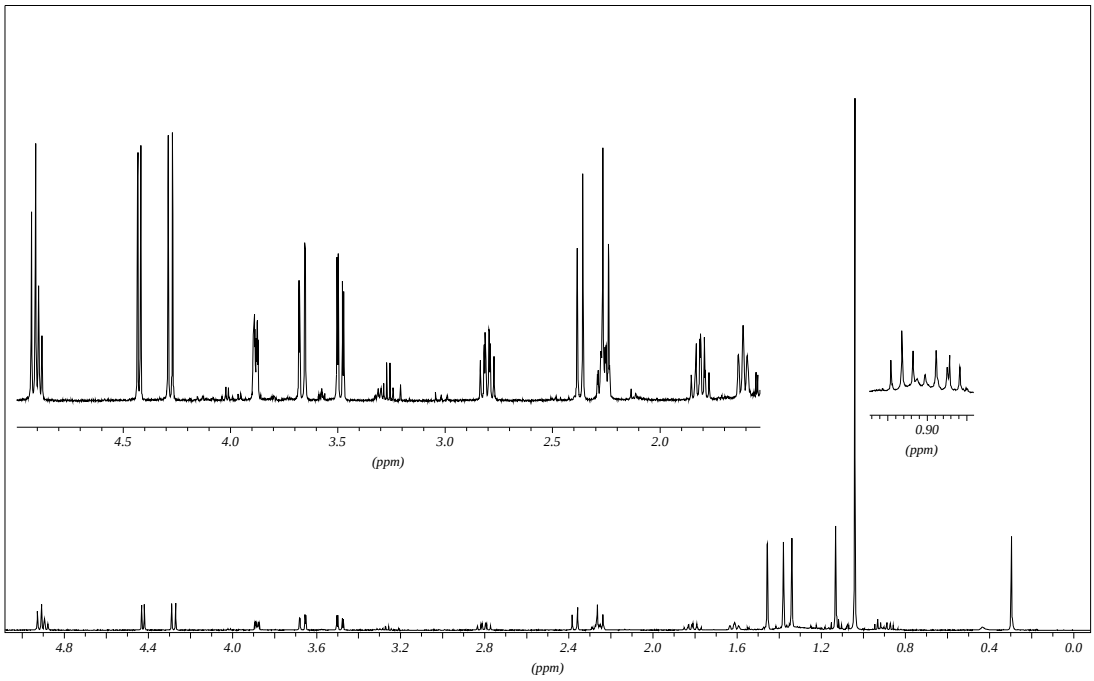



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

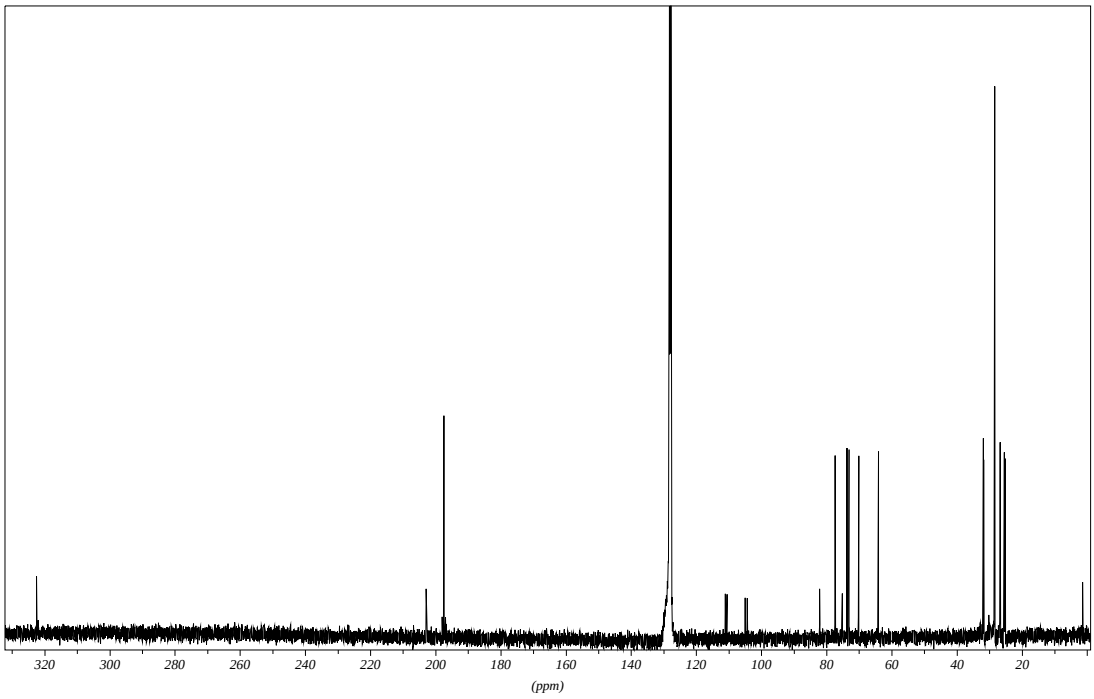


**(*1R,4S,7R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-1-(dimethylethoxy)-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro-[3.4]oct]-5-yliden}wolfram(0)** ***[51]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

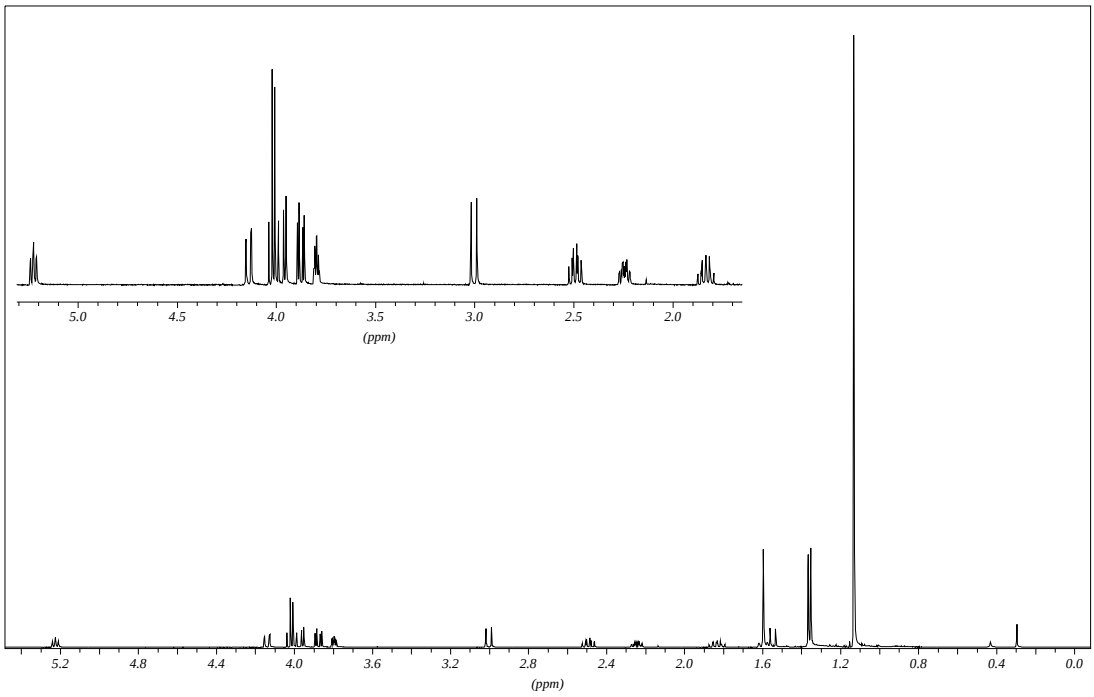



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

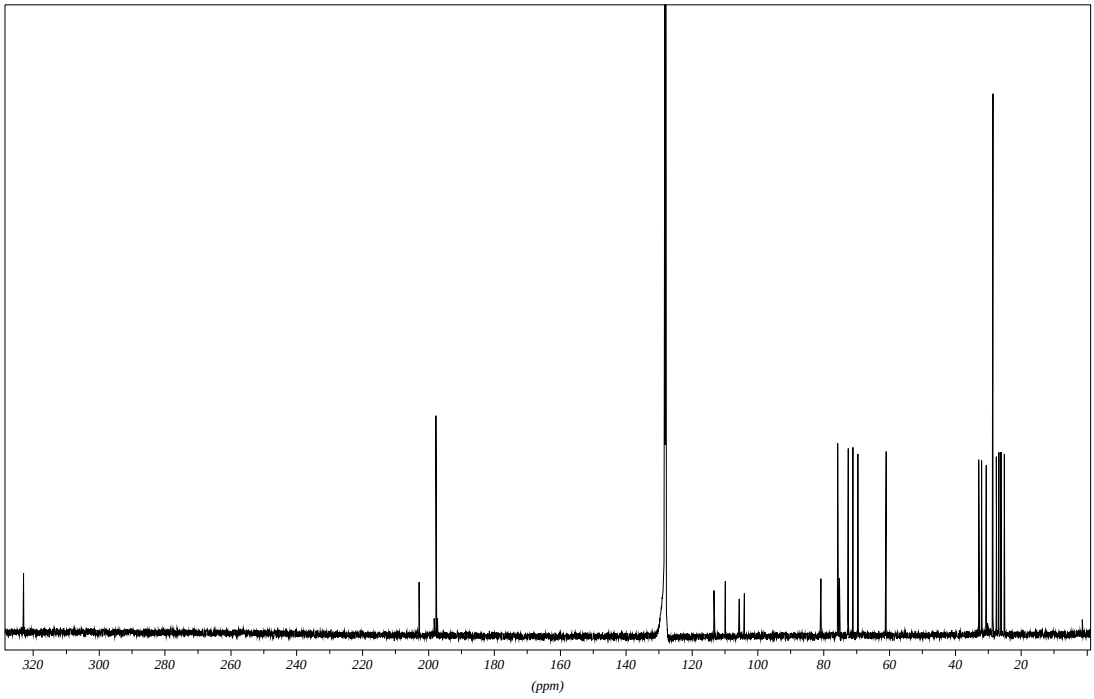


**(*1S,4R,7R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-1-(dimethylethoxy)-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro-[3.4]oct]-5-yliden}wolfram(0)** ***[51]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

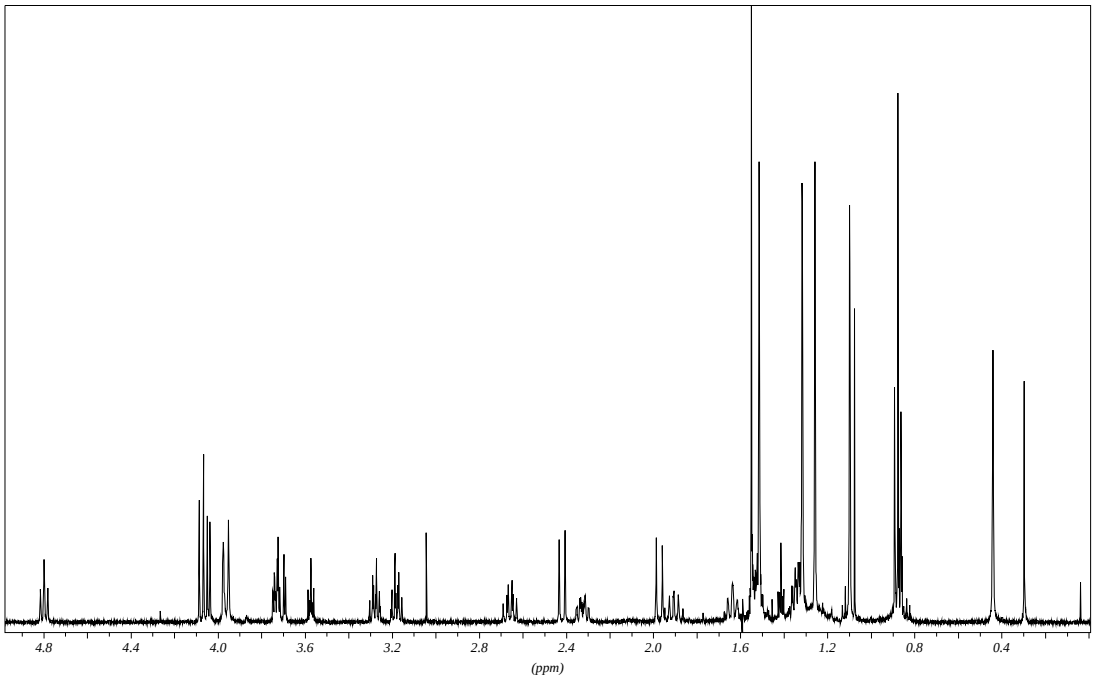



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

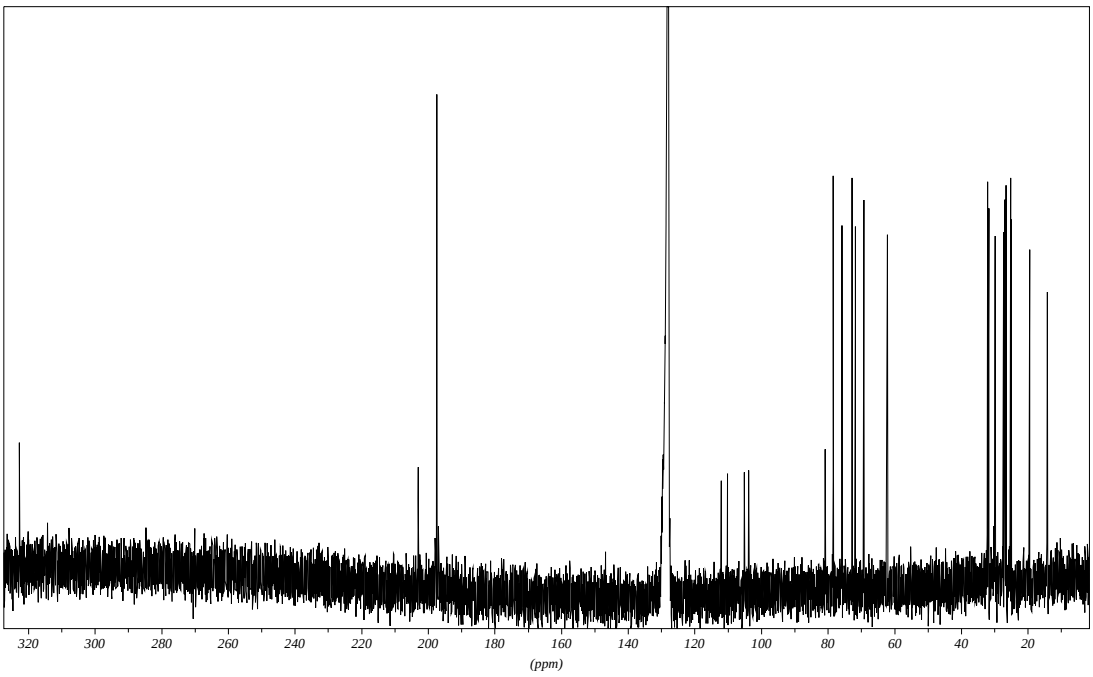


**(*1R,4S,7R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-1-(*n*-butoxy)-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro[3.4]oct]-5-yliden}wolfram(0)** ***[52]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

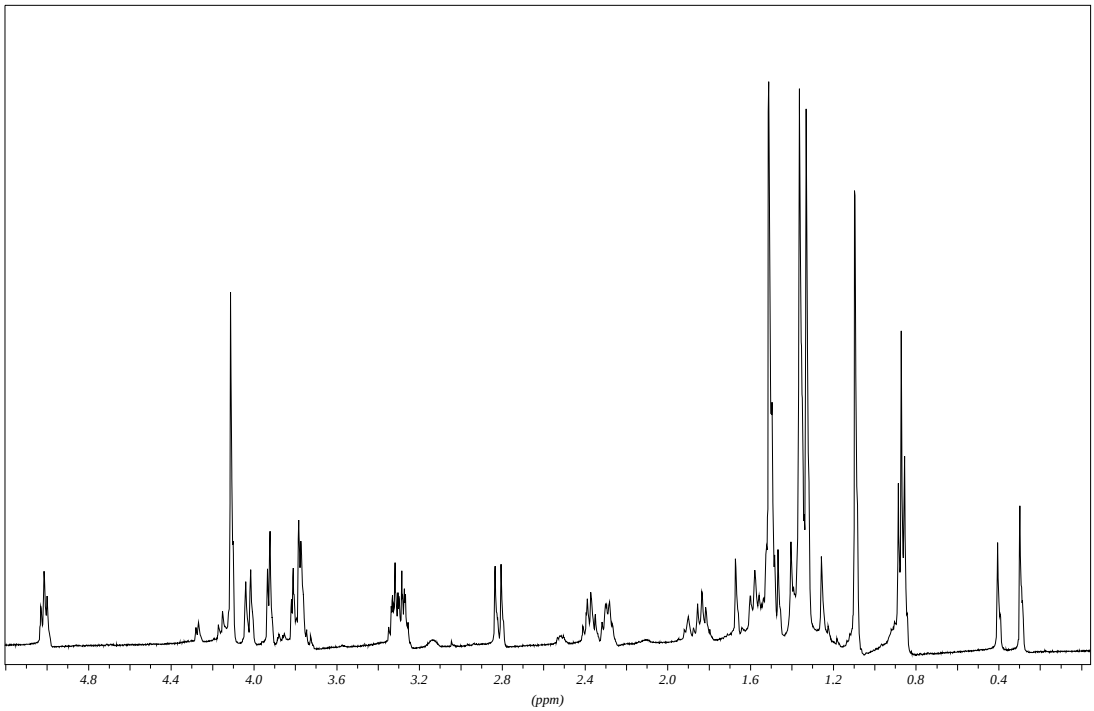



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

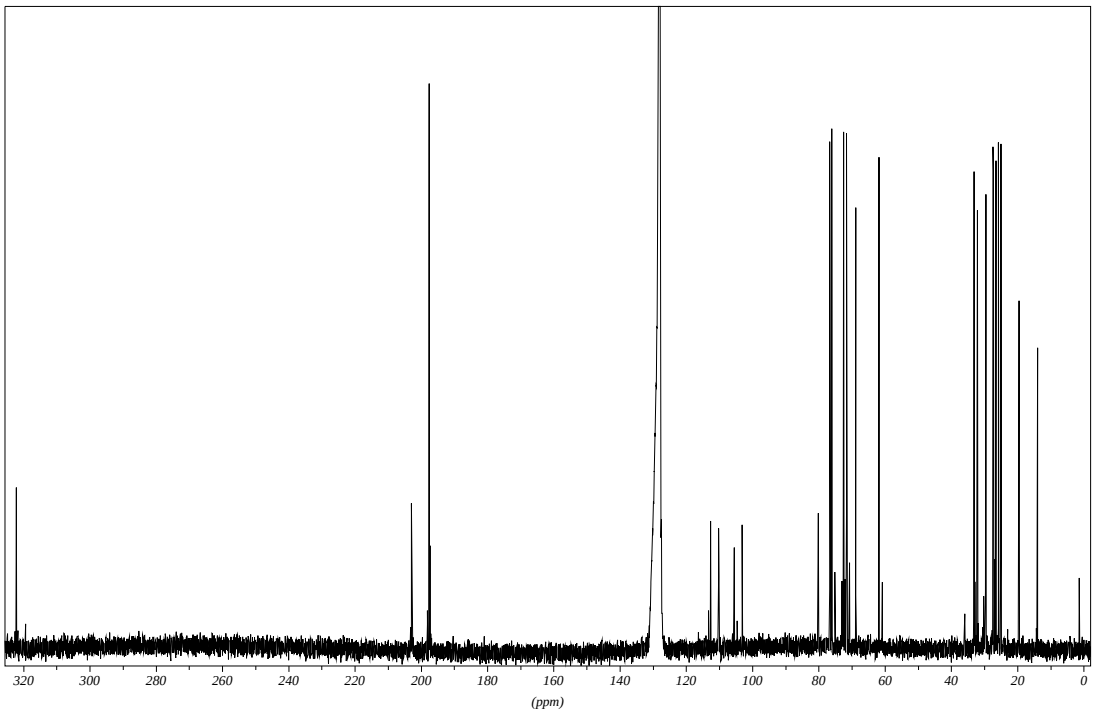


**(*1S,4R,7R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-1-(*n*-butoxy)-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro[3.4]oct]-5-yliden}wolfram(0)** ***[52]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

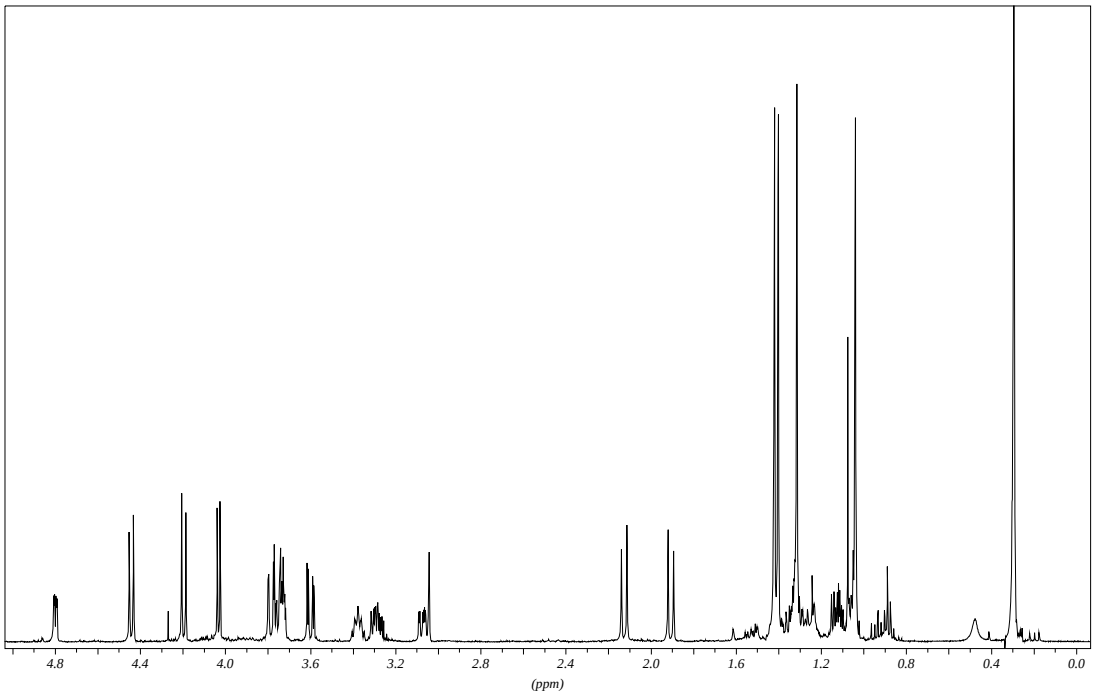



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

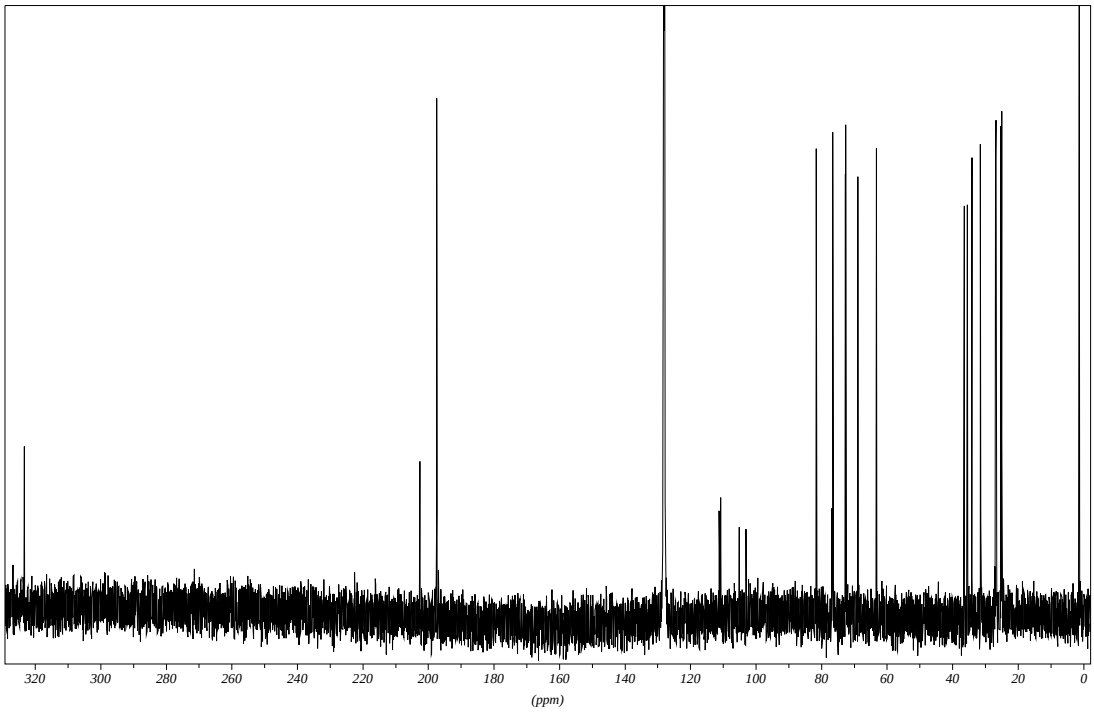


**(*3R,5S,1´R,5´S,1´´R,4´´S,6´´R*)-Pentacarbonyl{8´´,8´´,2´´´,2´´´-tetramethyl-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´´,4´´´-1,3-dioxacyclopentan-5´´,3]-spiro[2-oxabicyclo[3.2.0]heptan-7´,5]-2-oxacyclopent-1-yliden}wolfram(0)** ***[53]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

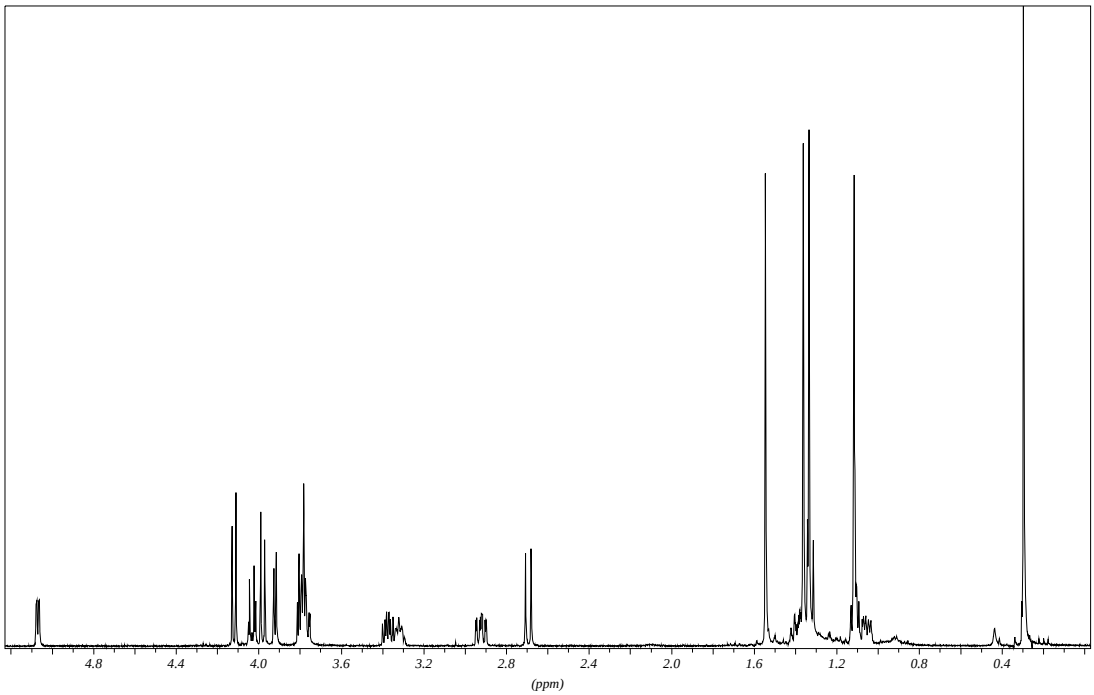



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

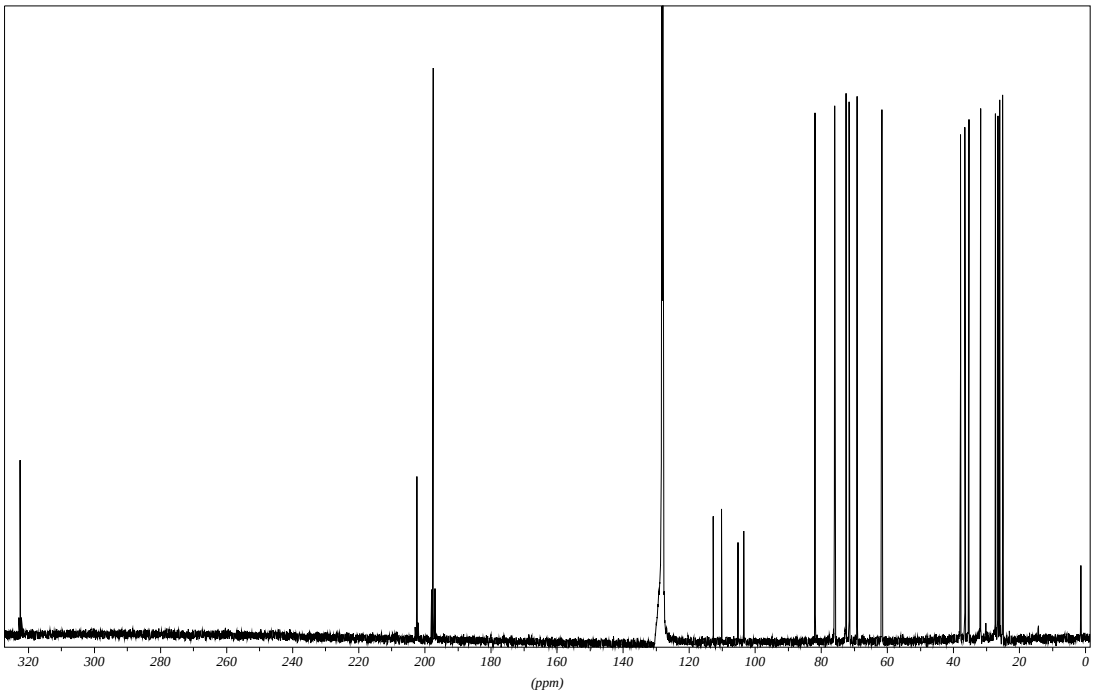


**(*3R,5R,1´S,5´R,1´´R,4´´S,6´´R*)-Pentacarbonyl{8´´,8´´,2´´´,2´´´-tetramethyl-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´´,4´´´-1,3-dioxacyclopentan-5´´,3]-spiro[2-oxabicyclo[3.2.0]heptan-7´,5]-2-oxacyclopent-1-yliden}wolfram(0)** ***[53]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

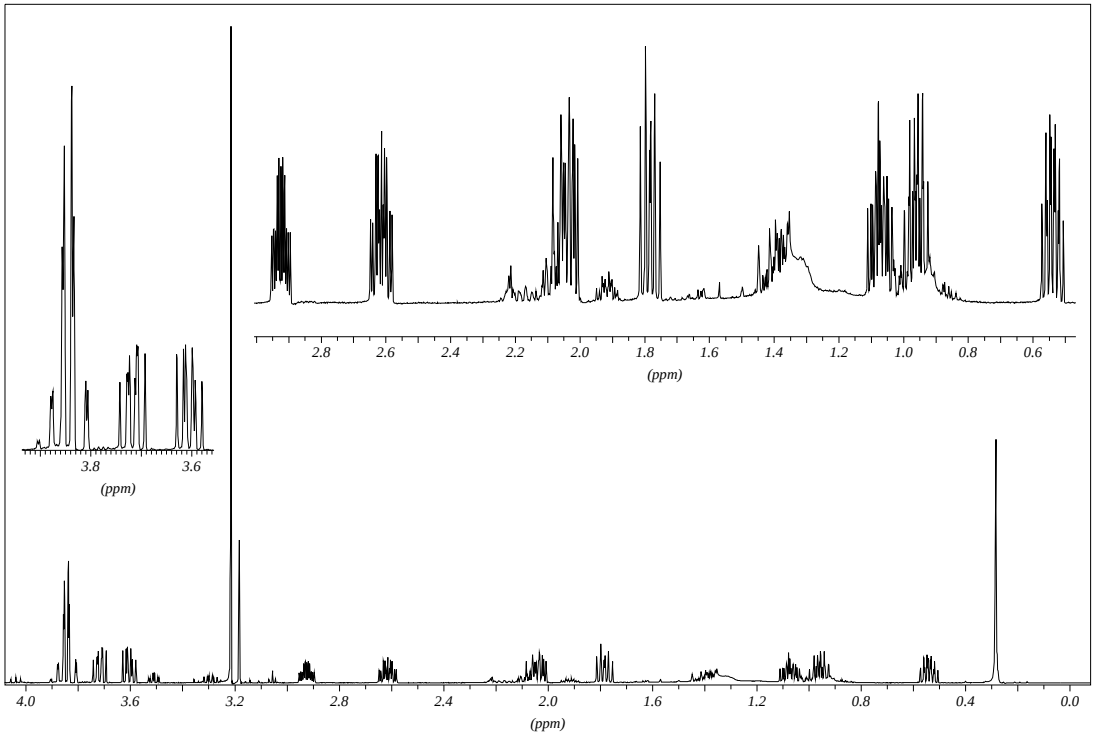



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

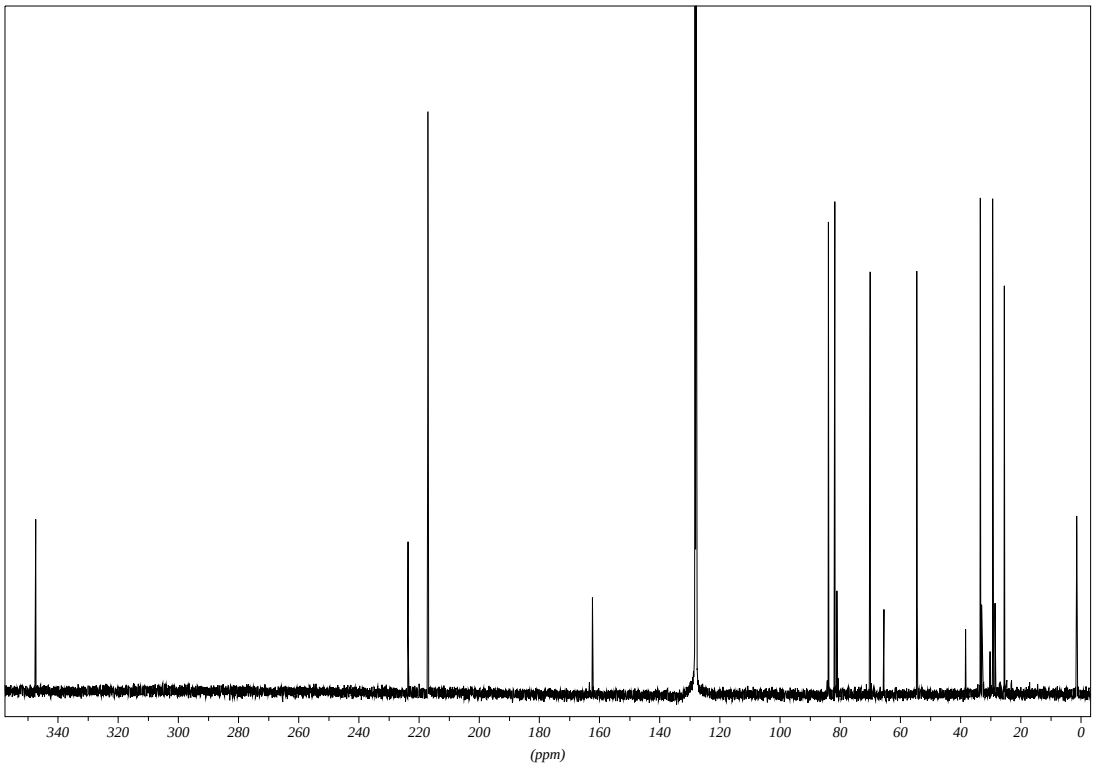


**Pentacarbonyl[5-(3´-methoxybut-3´-enyl)-2-oxacyclopentyliden]chrom(0)** ***[54]***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

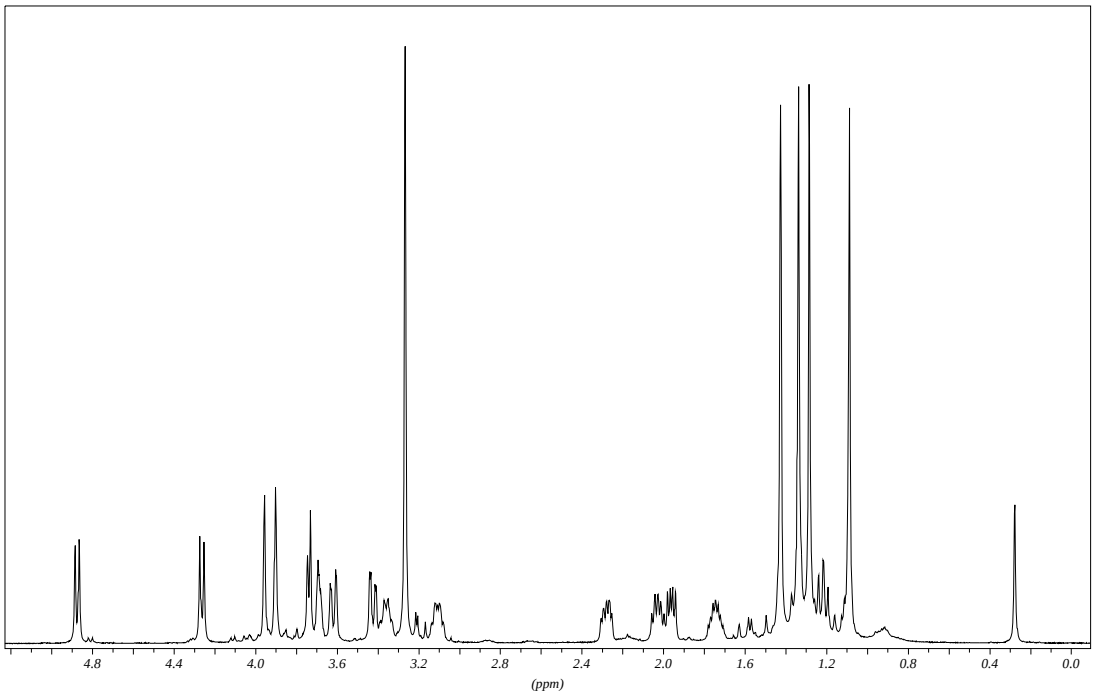



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

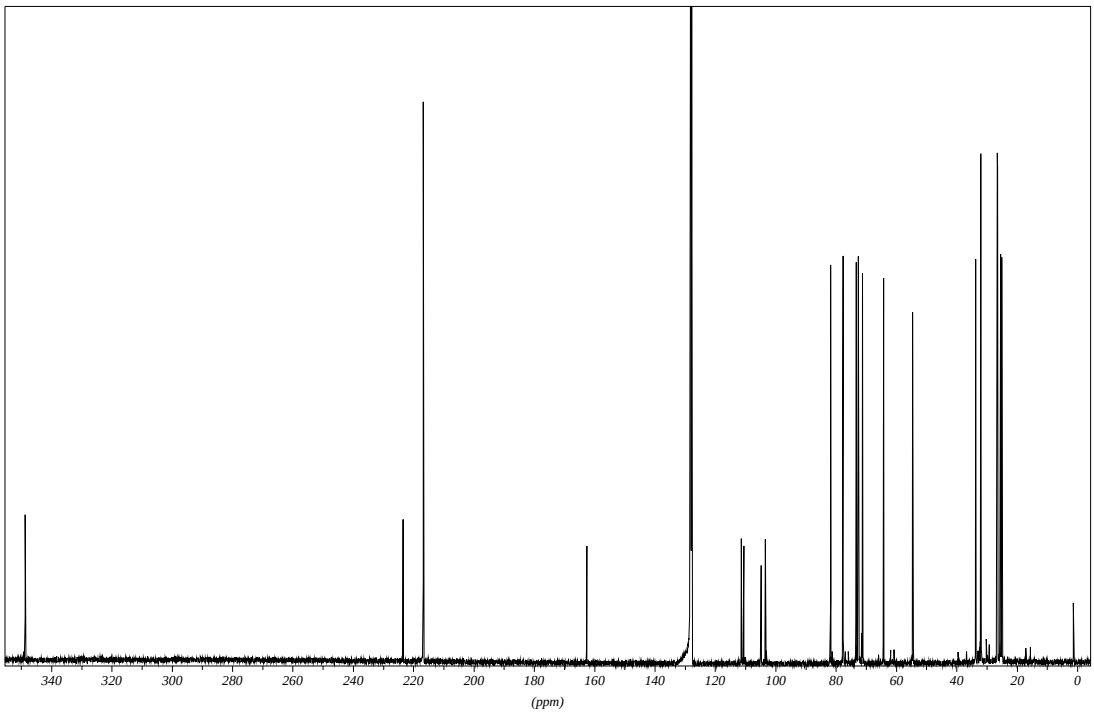


**(*3R,5S,1´R,4´S,6´R*)-Pentacarbonyl{5-(3´´´-methoxybut-3-enyl)-8´,8´,2´´,2´´-tetramethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,3-2-oxacyclopent]-1-yliden}chrom(0) *[56]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

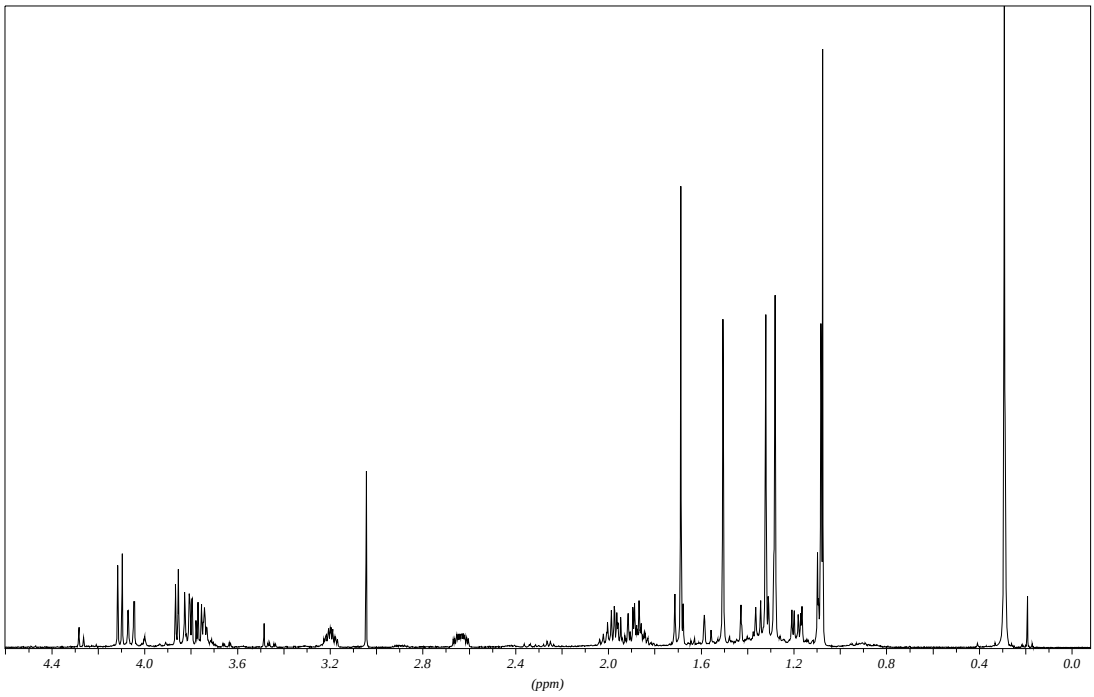



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

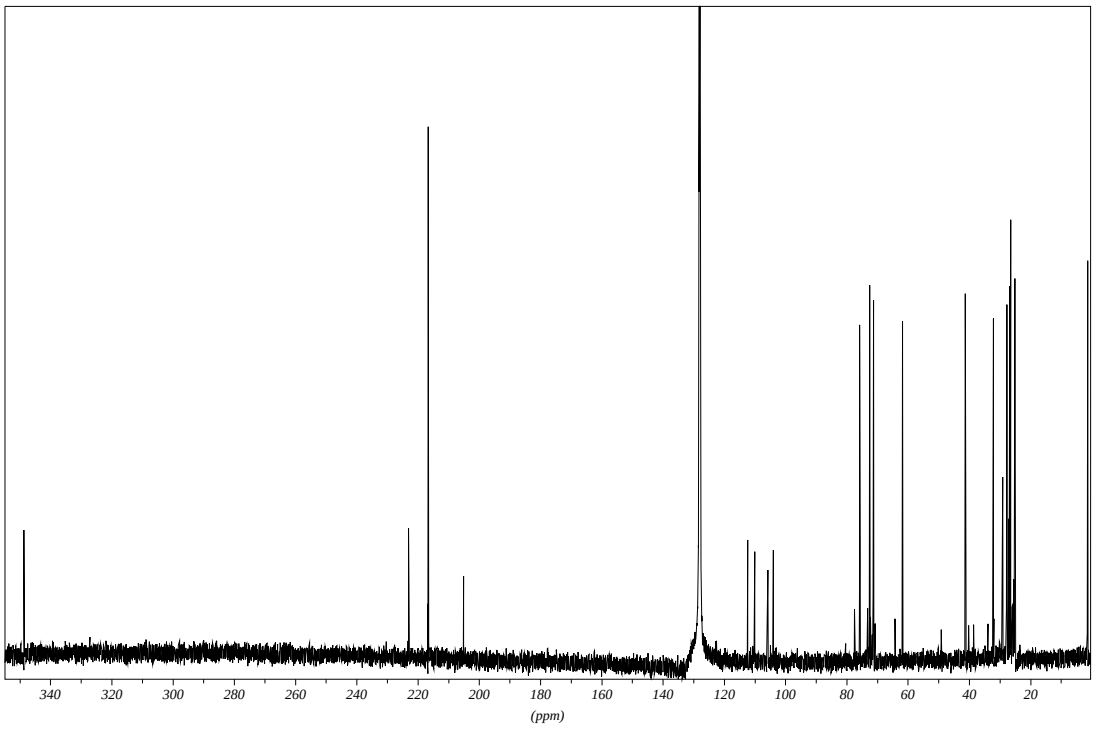


**(*3R,5R,1´R,4´S,6´R*)-Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl-5-(3´´´-oxobutyl)-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,3-2-oxacyclopent]-1-yliden}chrom(0)** ***[58]***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

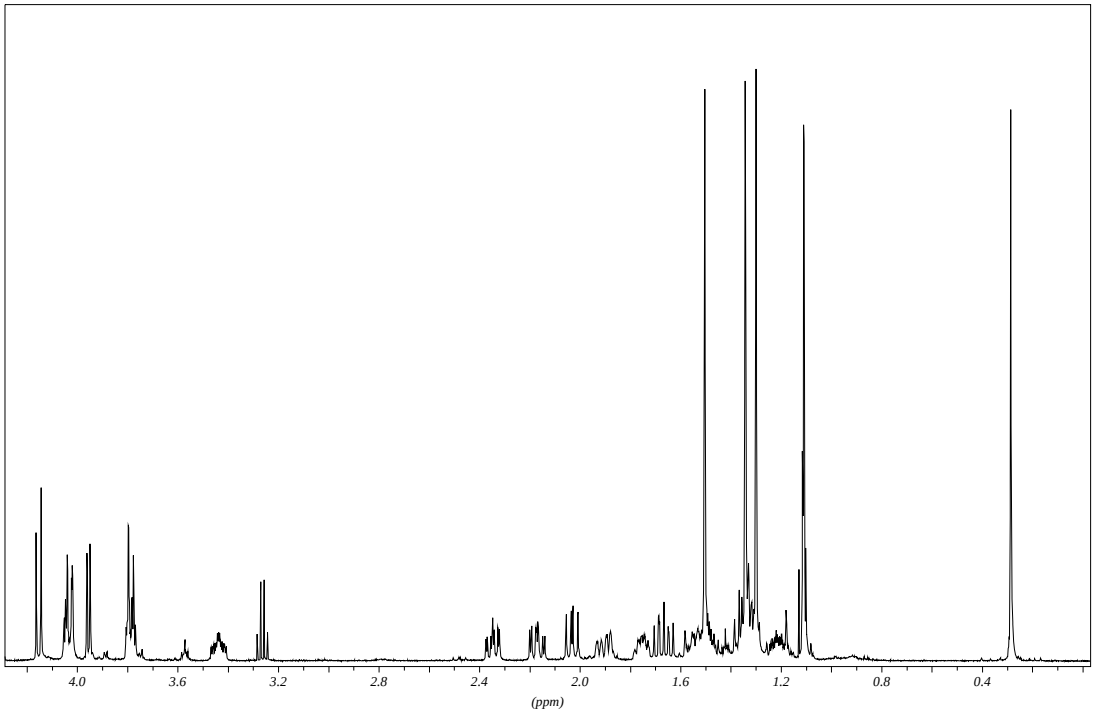



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

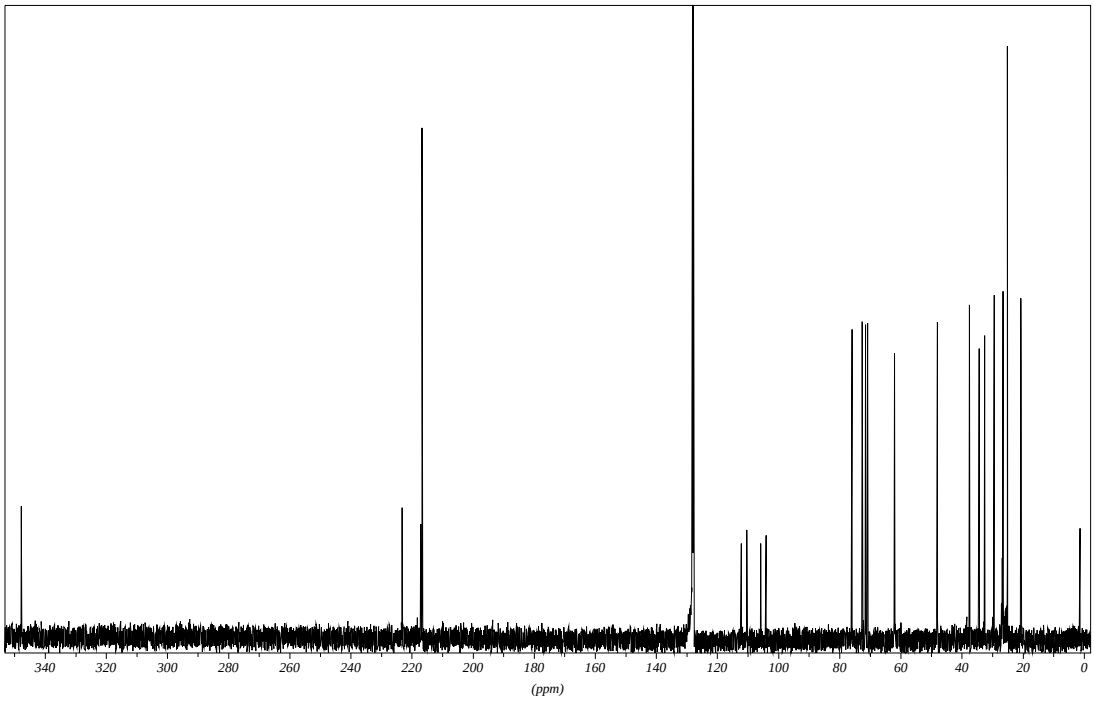


**(*3R,5R,1´R,4´S,6´R,1´´´S*)-Pentacarbonyl{5-(2´´´-oxacyclopentylmethyl)-8´,8´,2´´,2´´-tetramethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]-nonan-4´,4´´-1,3-dioxacyclopentan-5´,3-2-oxapent]-1-yliden}chrom(0) *[62]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

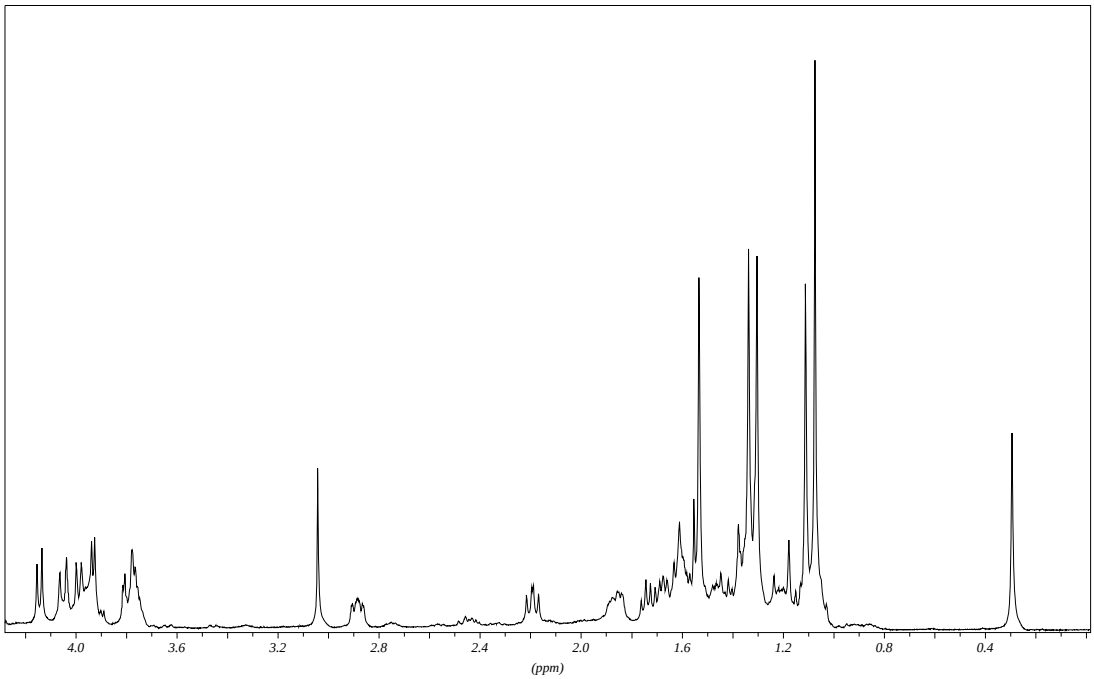



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

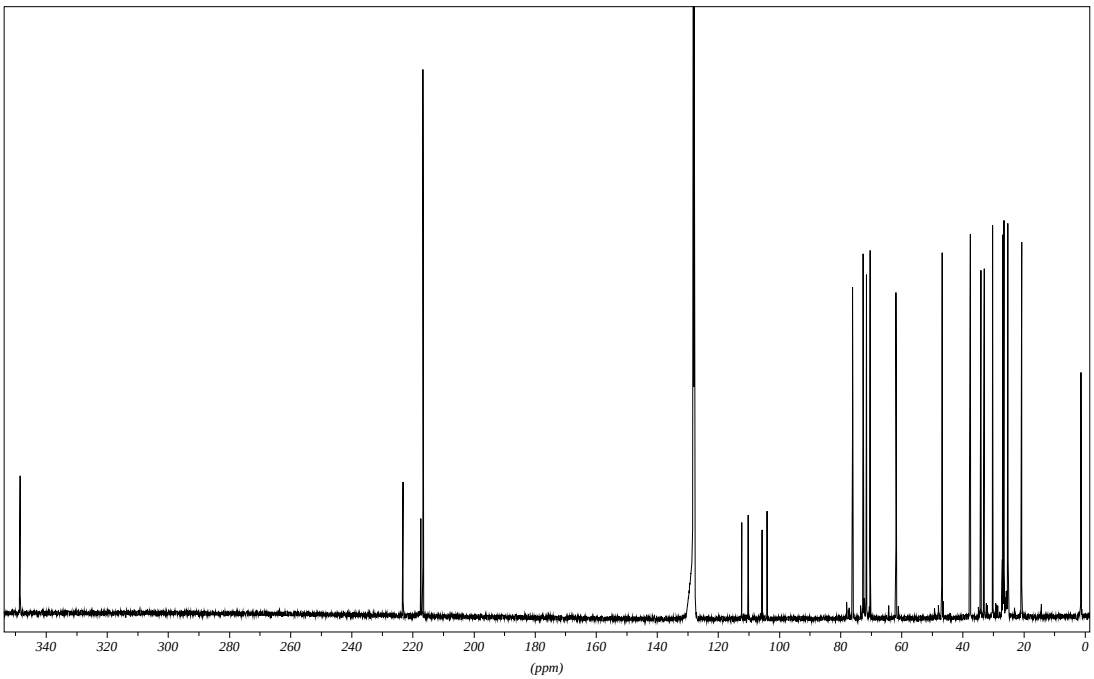


**(*3R,5S,1´R,4´S,6´R,1´´´R*)-Pentacarbonyl{5-(2´´´-oxacyclopentylmethyl)-8´,8´,2´´,2´´-tetramethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]-nonan-4´,4´´-1,3-dioxacyclopentan-5´,3-2-oxapent]-1-yliden}chrom(0) *[62]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

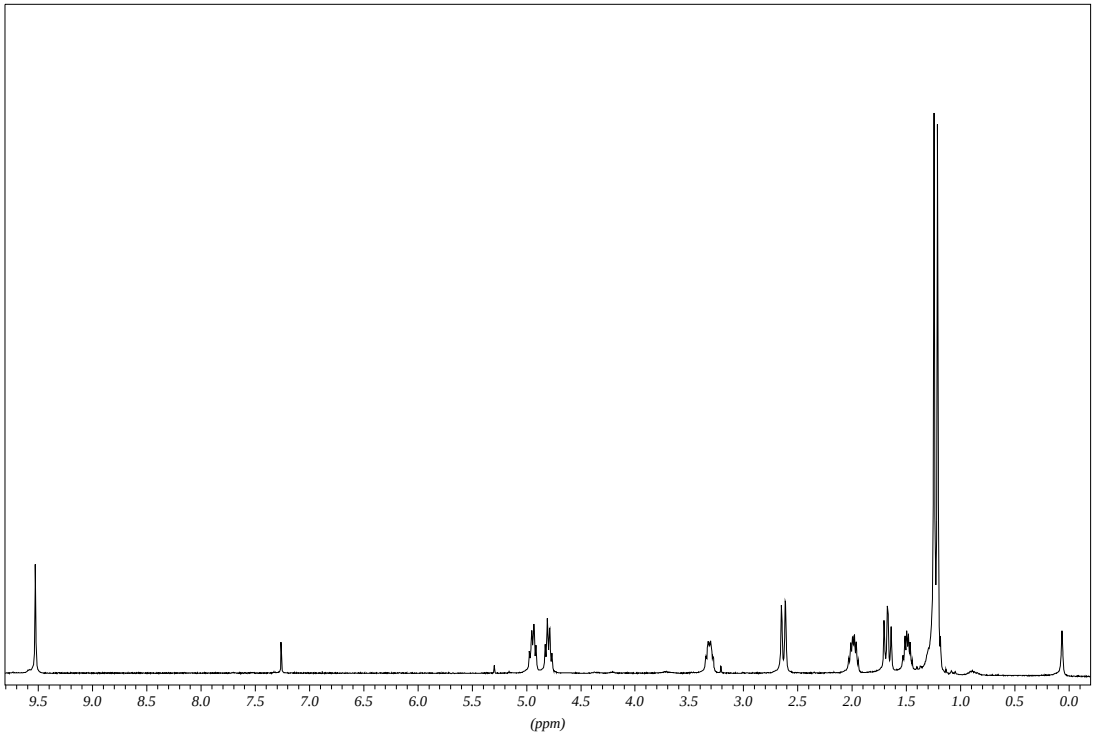



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

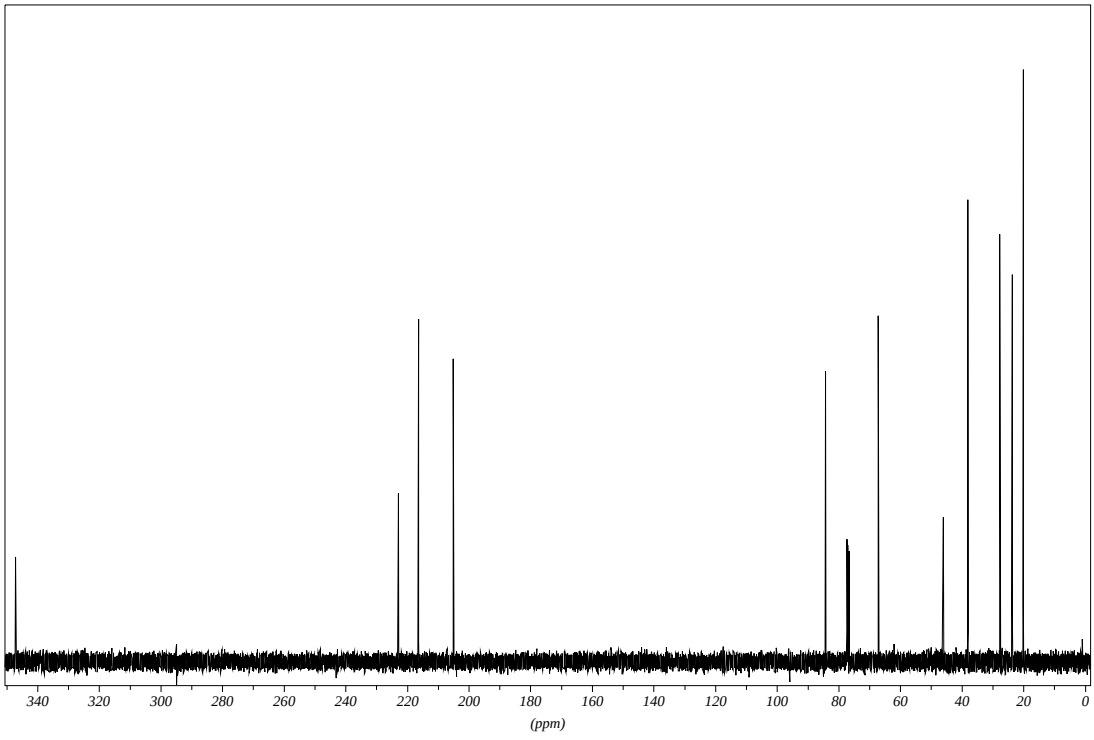


**Pentacarbonyl[5-(2´,2´-dimethyl-3´-oxopropyl)-2-oxacyclopentyliden]chrom(0)** ***[63]***

$^{1}$H-NMR-Spektrum (400 MHz, $C_6D_6$):

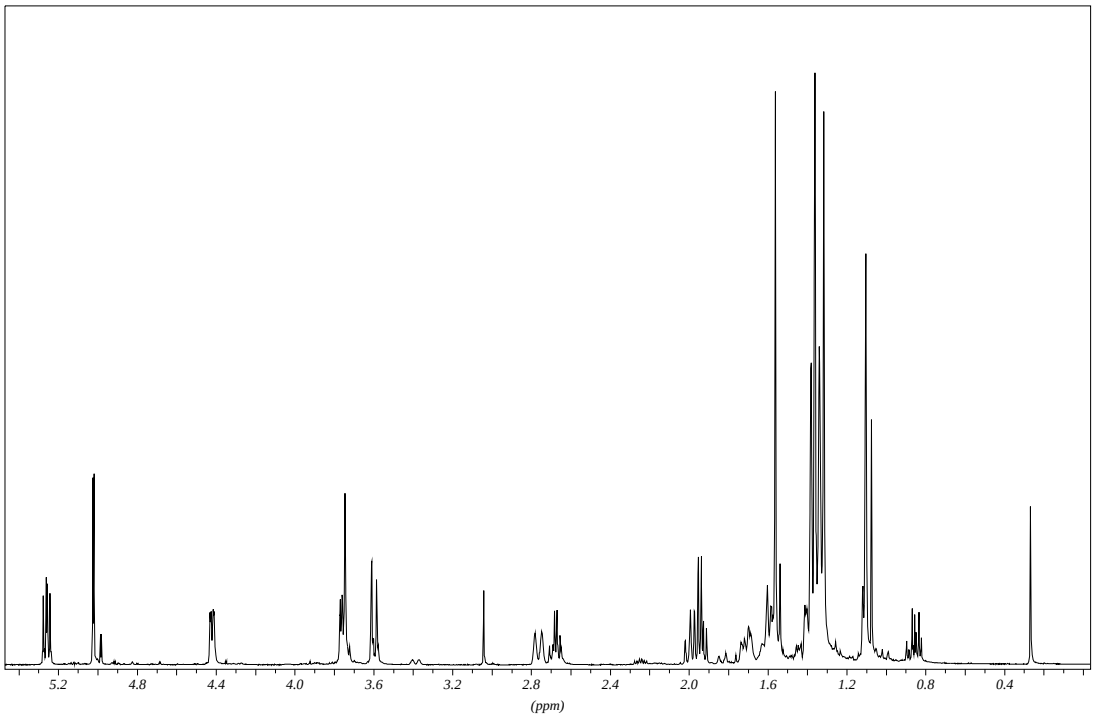



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

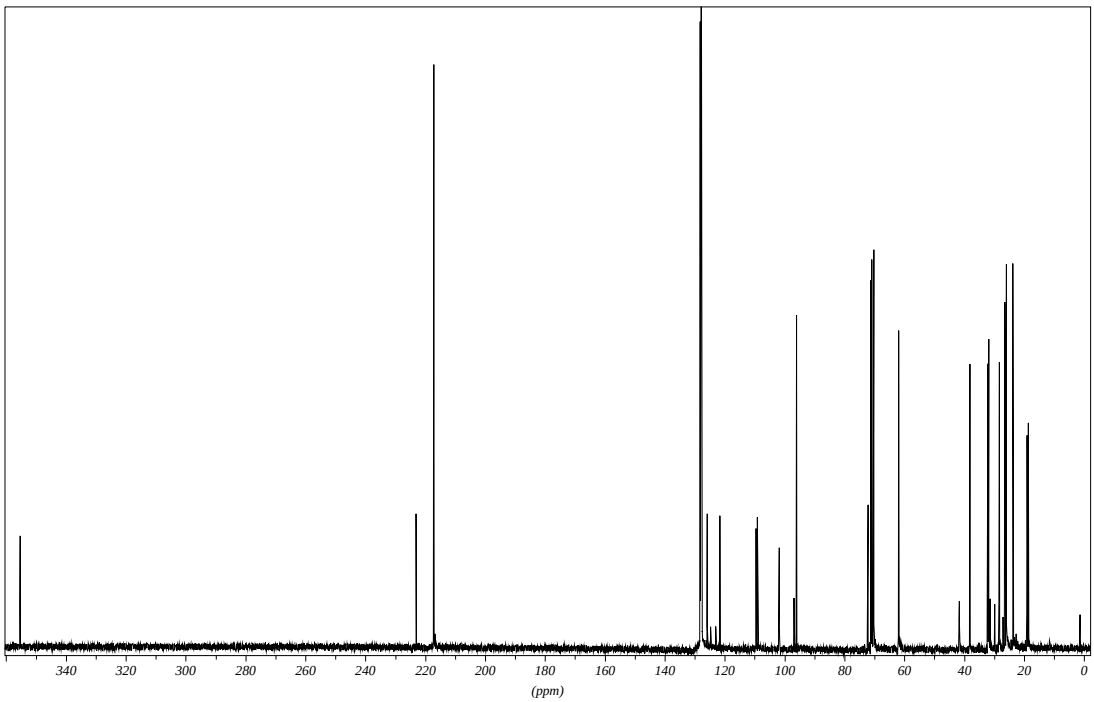


**(*3R,5S/R,1´R,2´S,6´S,9´R*)-Pentacarbonyl{7,8-dimethyl-3-(4´,4´,11´,11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´-yl)-2-oxaspiro[4.5]dec-7-en-1-yliden}chrom(0) *[65]a/b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

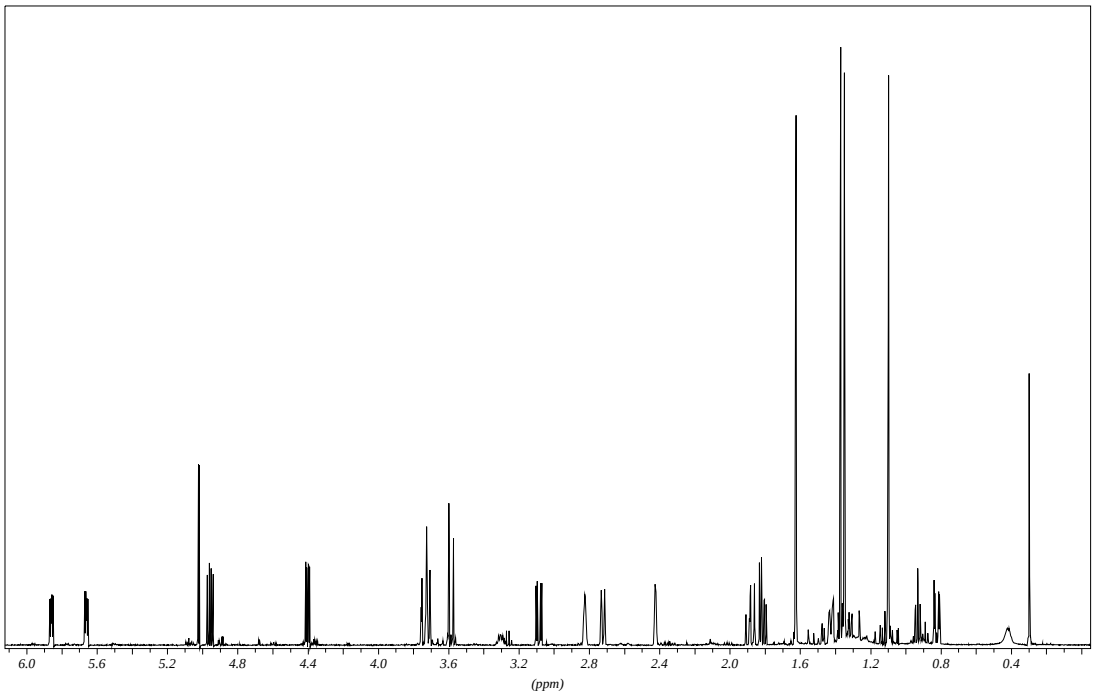



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

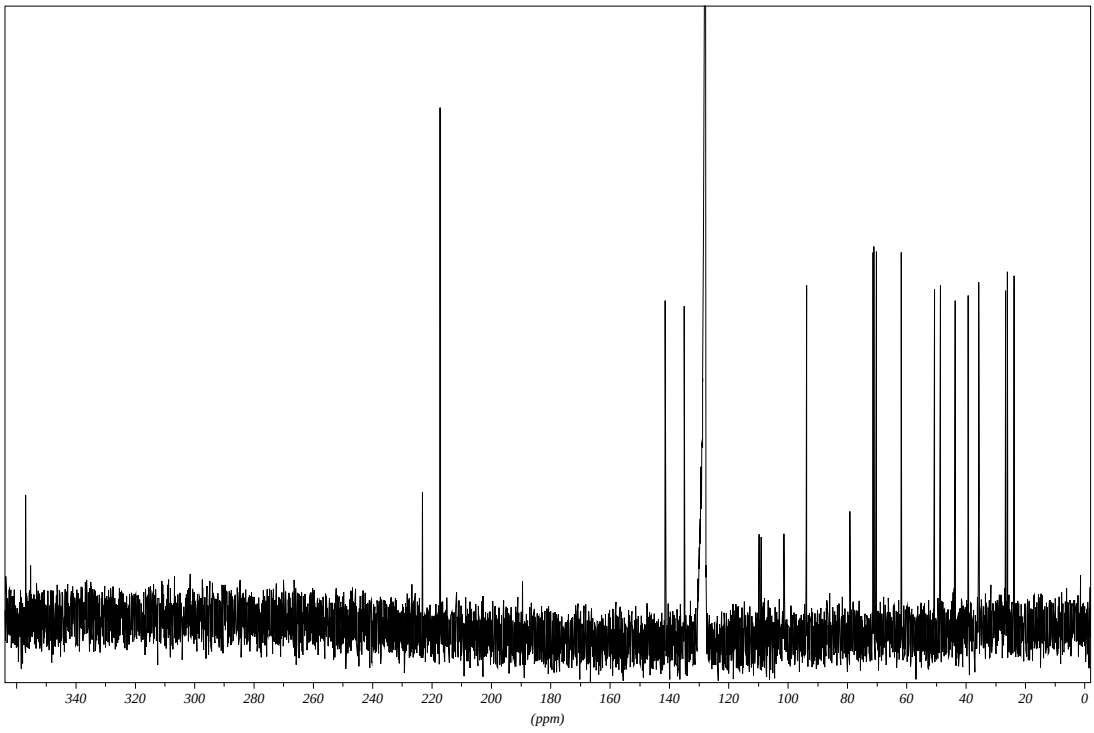


**(*3R,5R,1´R,4´S,1´´R,2´´S,5´´S,9´´R*)-Pentacarbonyl{3-(4´´,4´´,11´´,-11´´-tetramethyl-3´´,5´´,7´´,10´´,12´´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´´-yl)-spiro[bicyclo[2.2.1]hept-2´-en-5´,5-2-oxacyclopent]-1-yliden}chrom(0)** ***[66]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

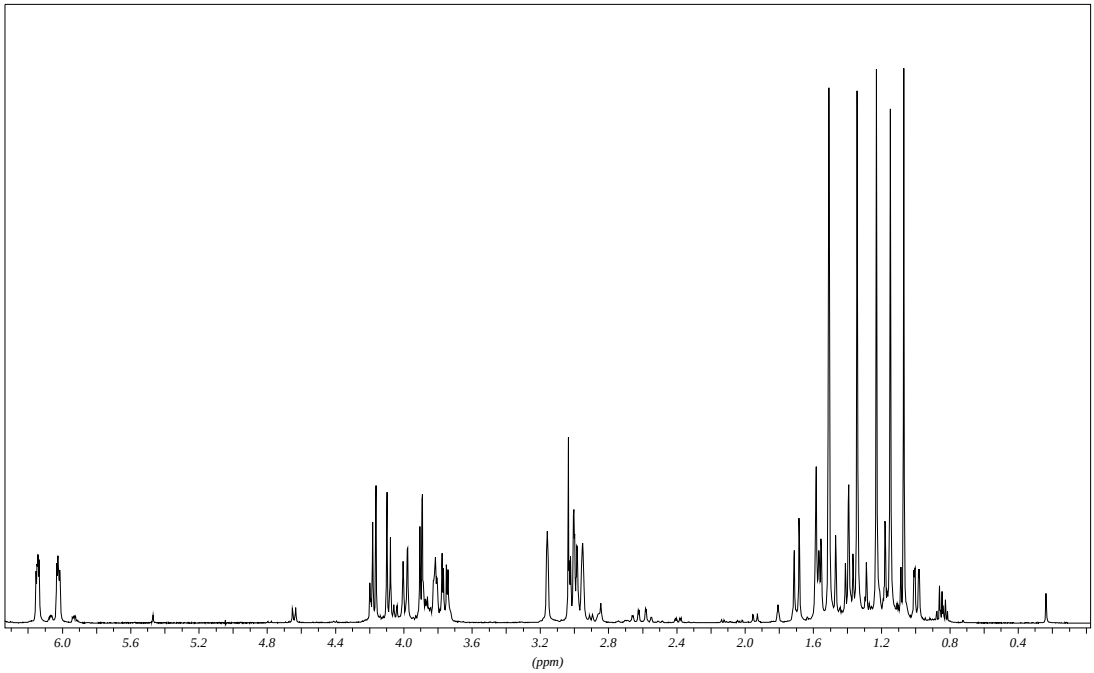



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

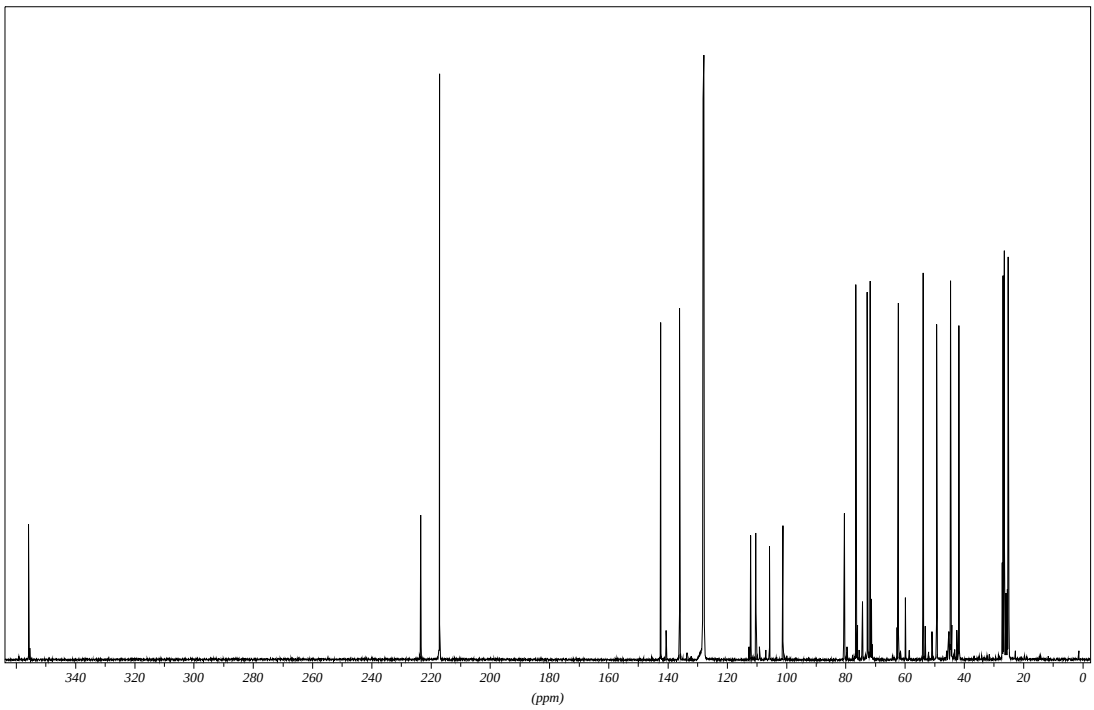


**(*3R,5S/R, 1´S/R, 4´R/S,1´´R,4´´S,6´´R*)-Pentacarbonyl{8´´,8´´,2´´´,2´´´-tetramethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´´,4´´´-1,3-dioxacyclopentan-5´´,3]-spiro[bicyclo[2.2.1]hept-2-en-5´,5]-2-oxacyclopent-1-yliden}chrom(0) *[67]a/b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

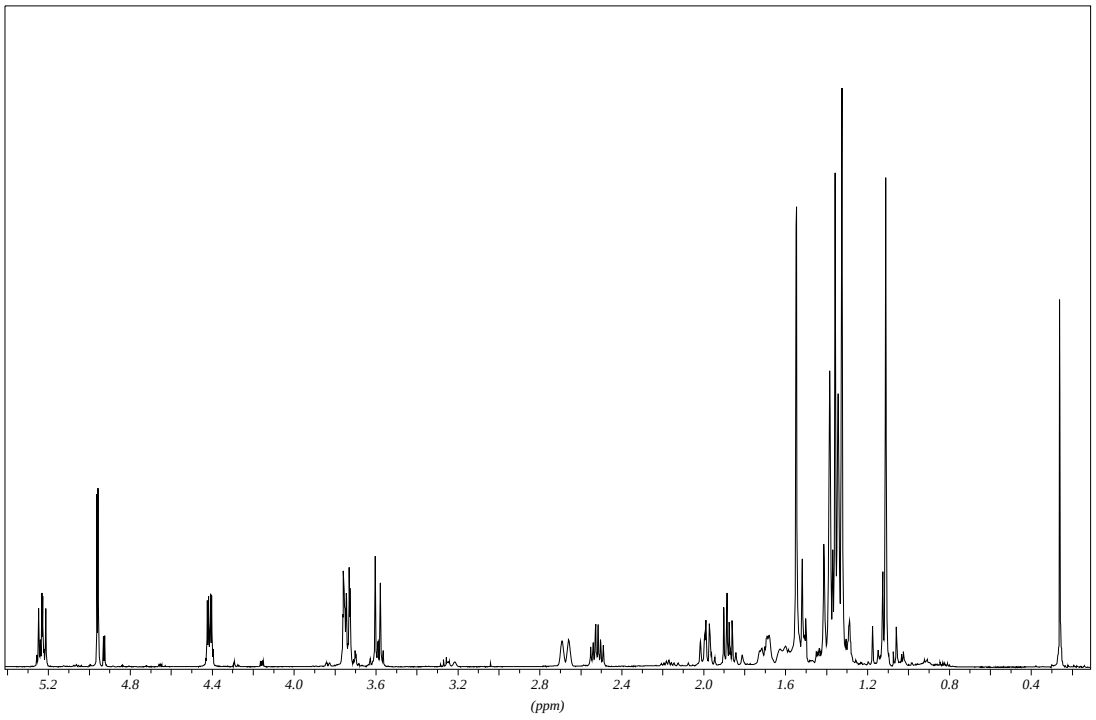



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

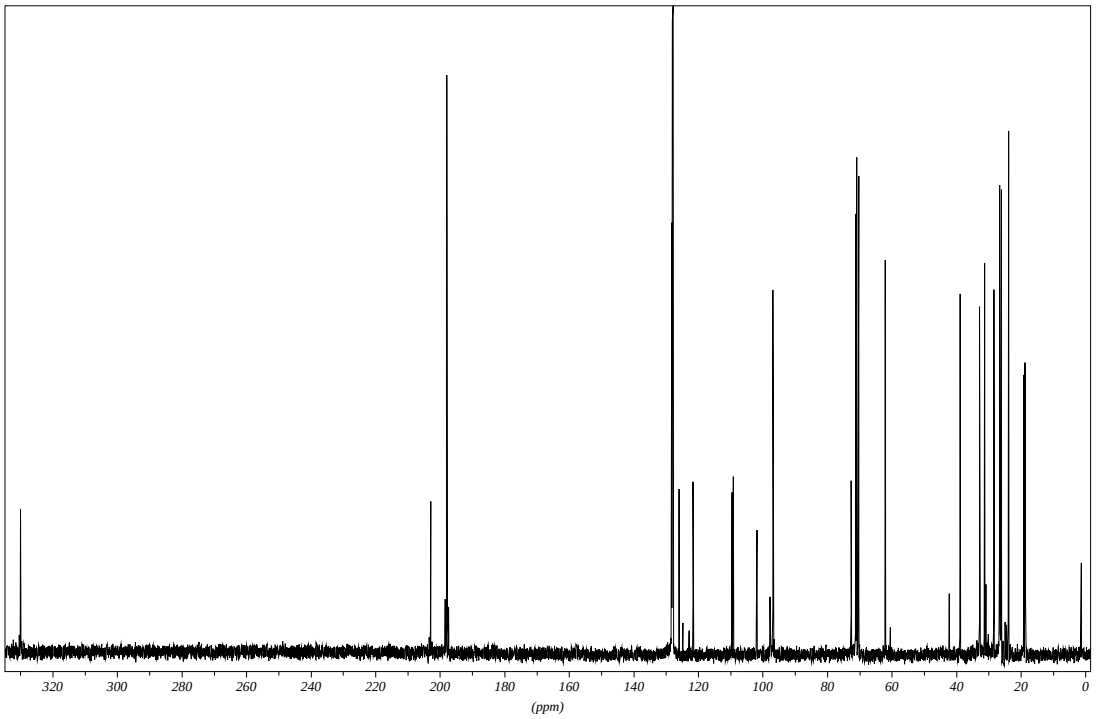


**(*3R,5S,1´R,2´S,6´S,9´R*)-Pentacarbonyl{7,8-dimethyl-3-(4´,4´,11´,11´-tetramethyl-3´,5´,7´,10´,12´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´-yl)-2-oxaspiro[4.5]dec-7-en-1-yliden}wolfram(0) *[68]a/b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

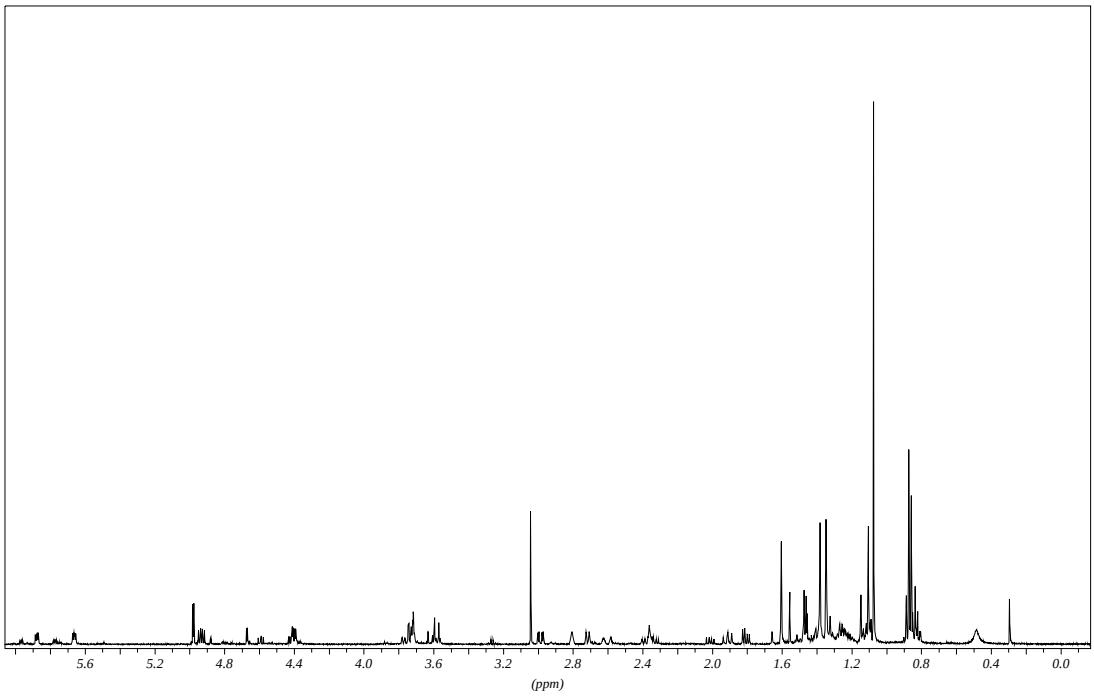



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

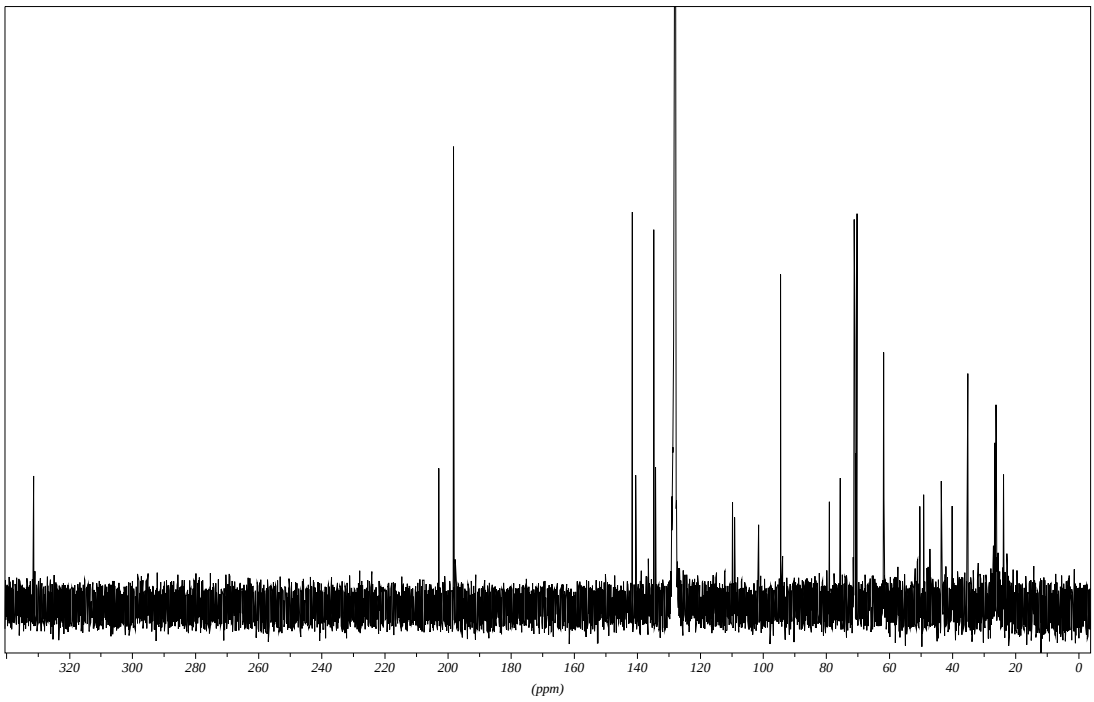


**(*3R,5R/S,1´R/S,4´S/R,1´´R,2´´S,5´´S,9´´R*)-Pentacarbonyl{3-(4´´,4´´,11´´,11´´-tetramethyl-3´´,5´´,7´´,10´´,12´´-pentaoxatricyclo[7.3.0.0$^{2,6}$]dodec-6´´-yl)-spiro[bicyclo-[2.2.1]hept-2´-en-5´,5-2-oxacyclopent]-1-yliden}wolfram(0) *[69]a/b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

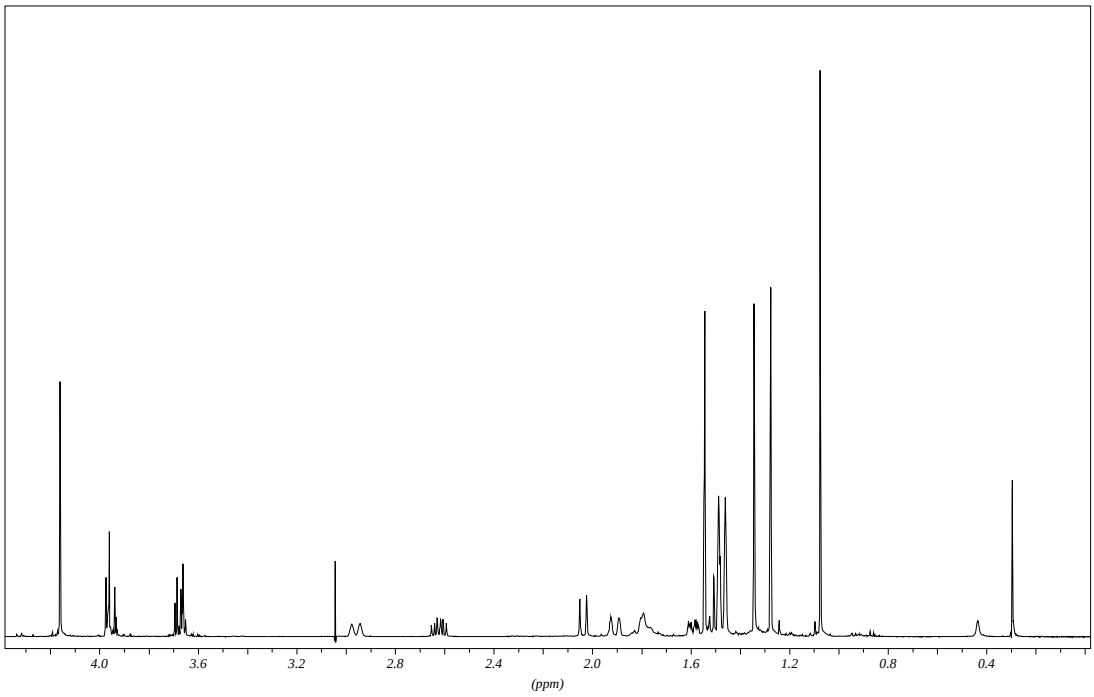



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

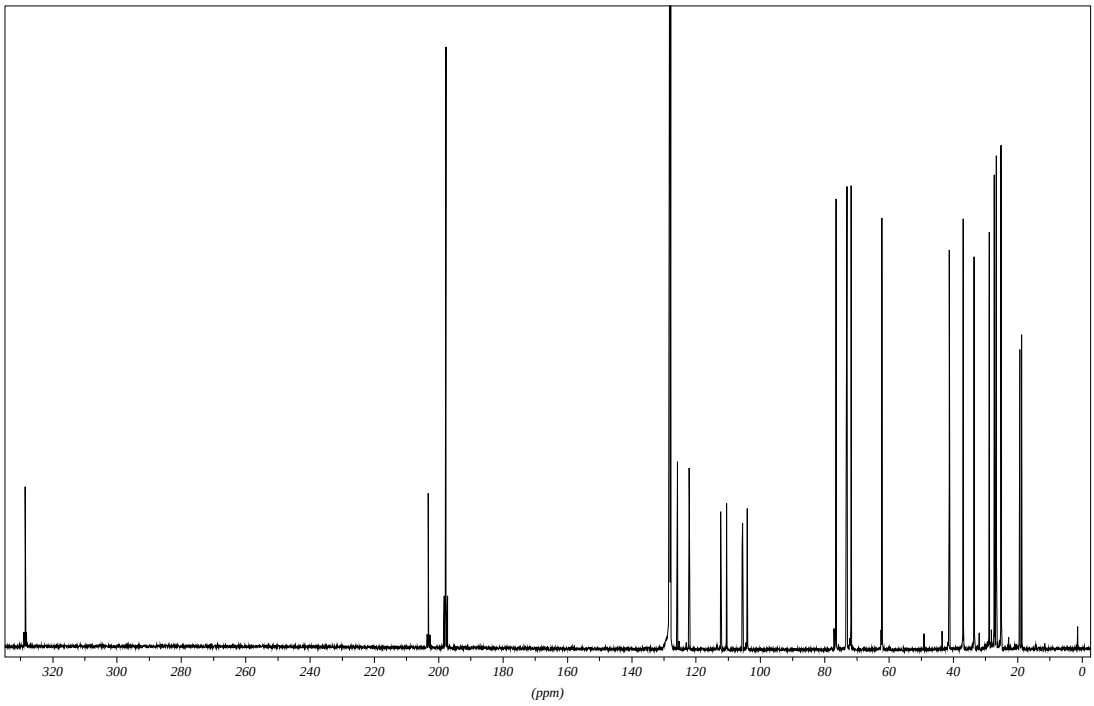


**(*3R,5R/S,1´R,4´S,6´R*)-Pentacarbonyl{7,8,8´,8´,2´´,2´´-hexamethyl-dispiro[3,7,9-trioxa-bicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,3-2-oxaspiro[4.5]dec-7-en-1-yliden}wolfram(0) *[70]a/b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

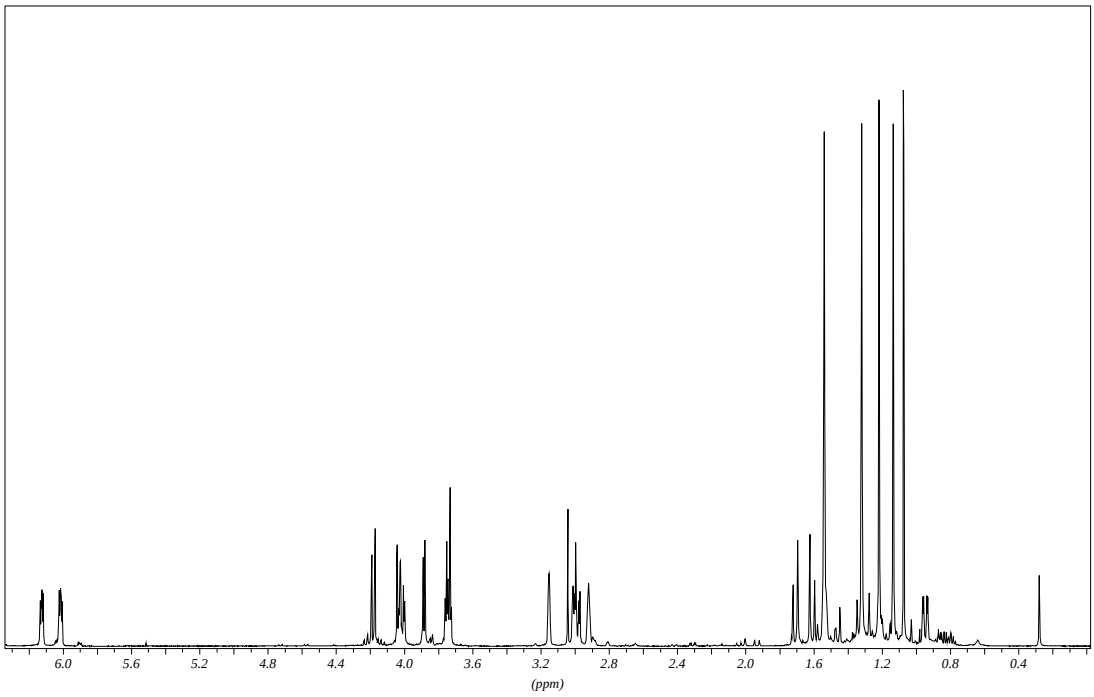



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

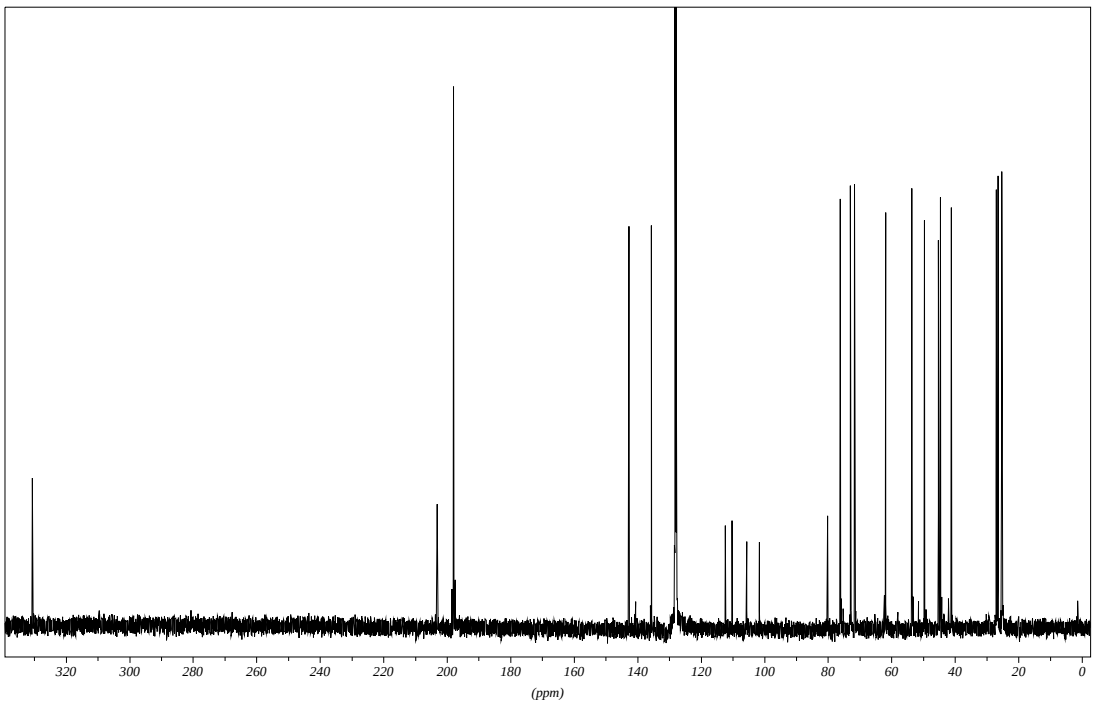


**(*3R,5S/R, 1´S/R, 4´R/S,1´´R,4´´S,6´´R*)-Pentacarbonyl{8´´,8´´,2´´´,2´´´-tetra-methyl-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´´,4´´´-1,3-dioxacyclopentan-5´´,3]-spiro[bi-cyclo[2.2.1]hept-2-en-5´,5]- 2-oxacyclopent-1-yliden}wolfram(0) *[71]a/b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

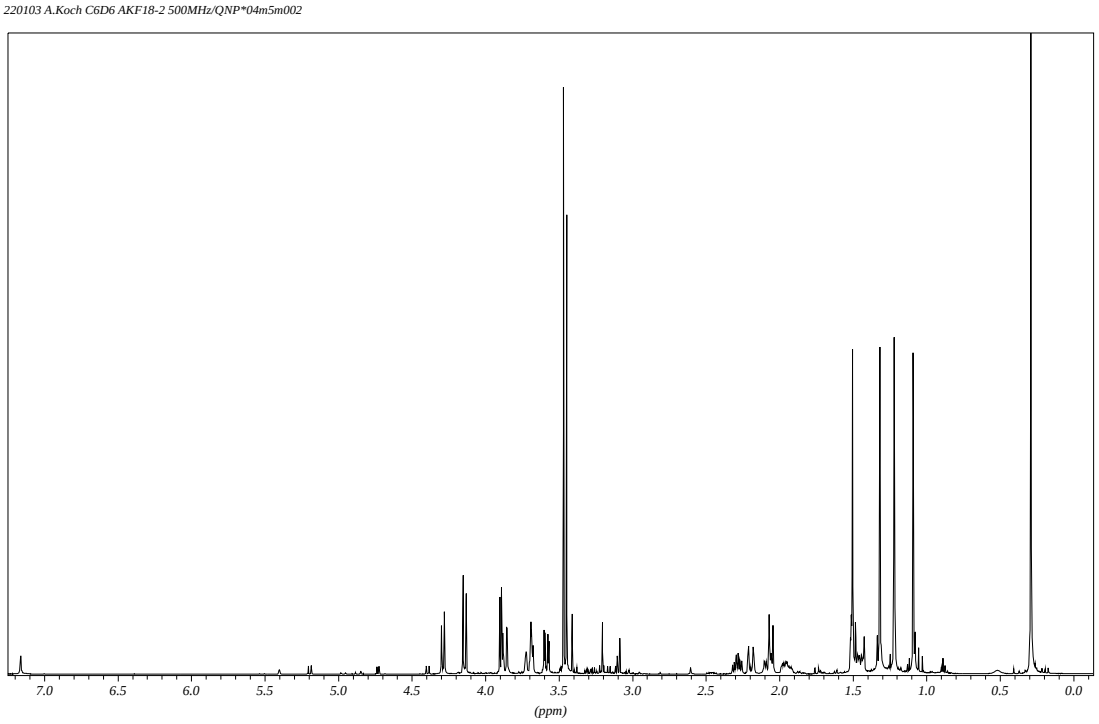



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

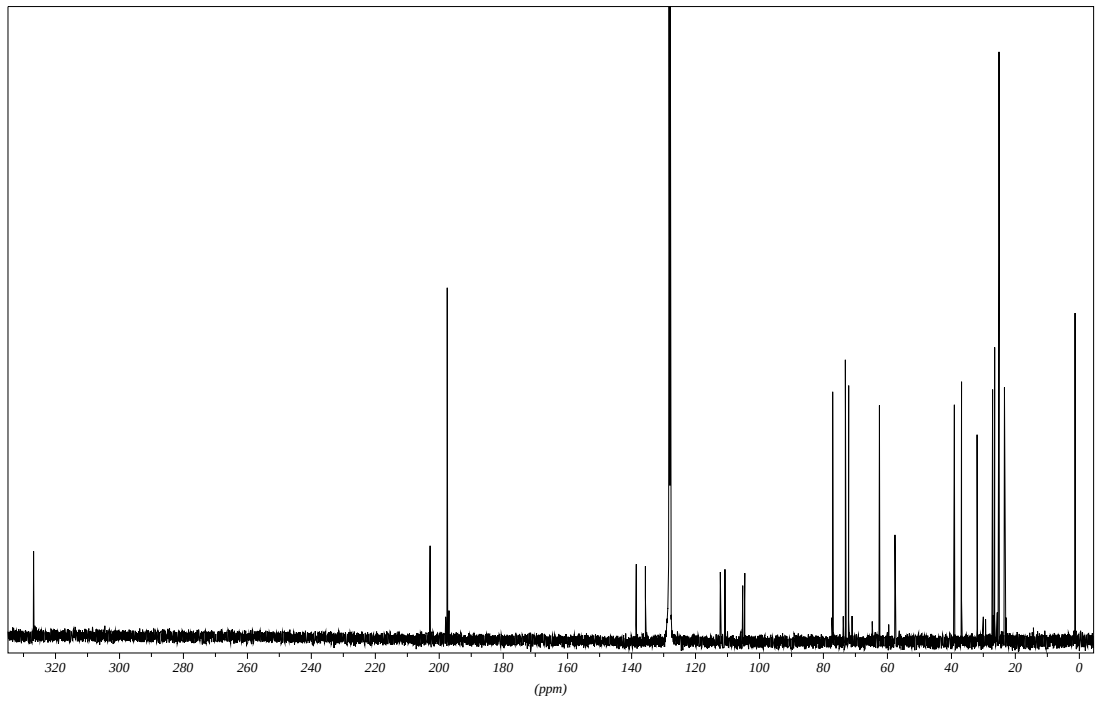


**(*3R,5R/S, 1´R,4´S,6´R*)-Pentacarbonyl{7,8-dimethoxy-8´,8´,2´´,2´´-tetramethyl-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,3-2-oxaspiro[4.5]dec-7-en-1-yliden}wolfram(0)** ***[72]a/b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

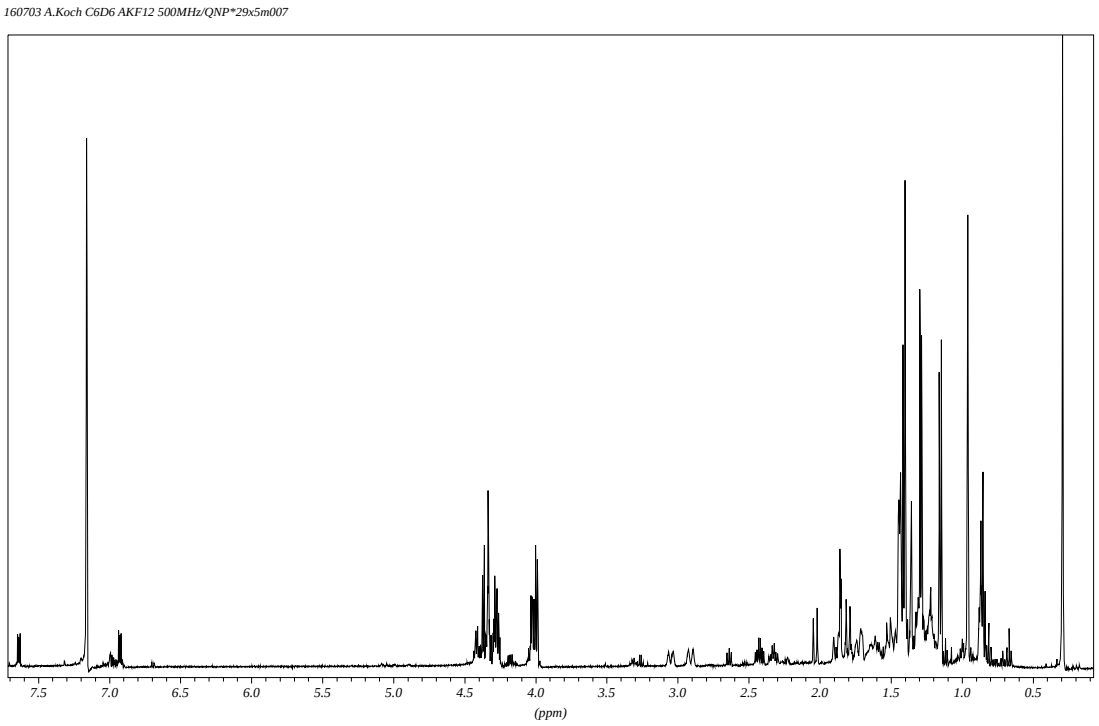



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

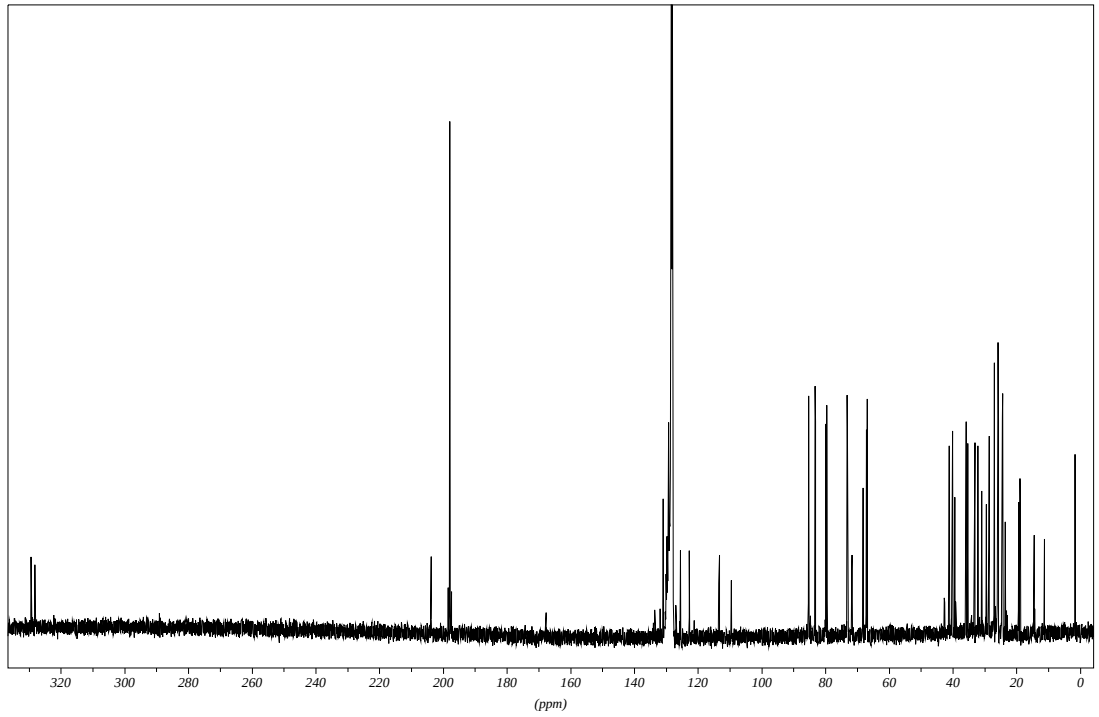


**(*3S,5R/S,1´S,5´R,6´R,4´´R*)-Pentacarbonyl{3´,3´-dimethyl-6´-(2´´,2´´-dimethyl-1,3-dioxacyclopent-4´´-yl)-spiro[2,4,7-trioxabicyclo[3.3.0]octan-8´,3-2-oxaspiro[4.5]dec-7-en]-1-yliden}wolfram(0)** ***[73]a/b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

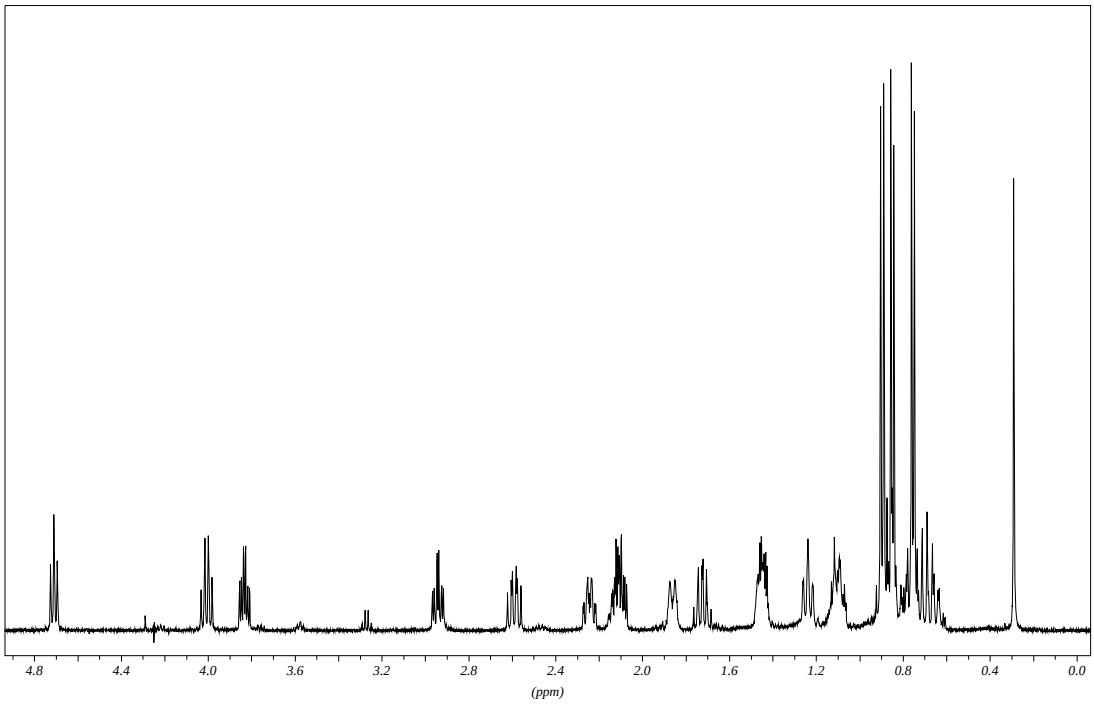



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

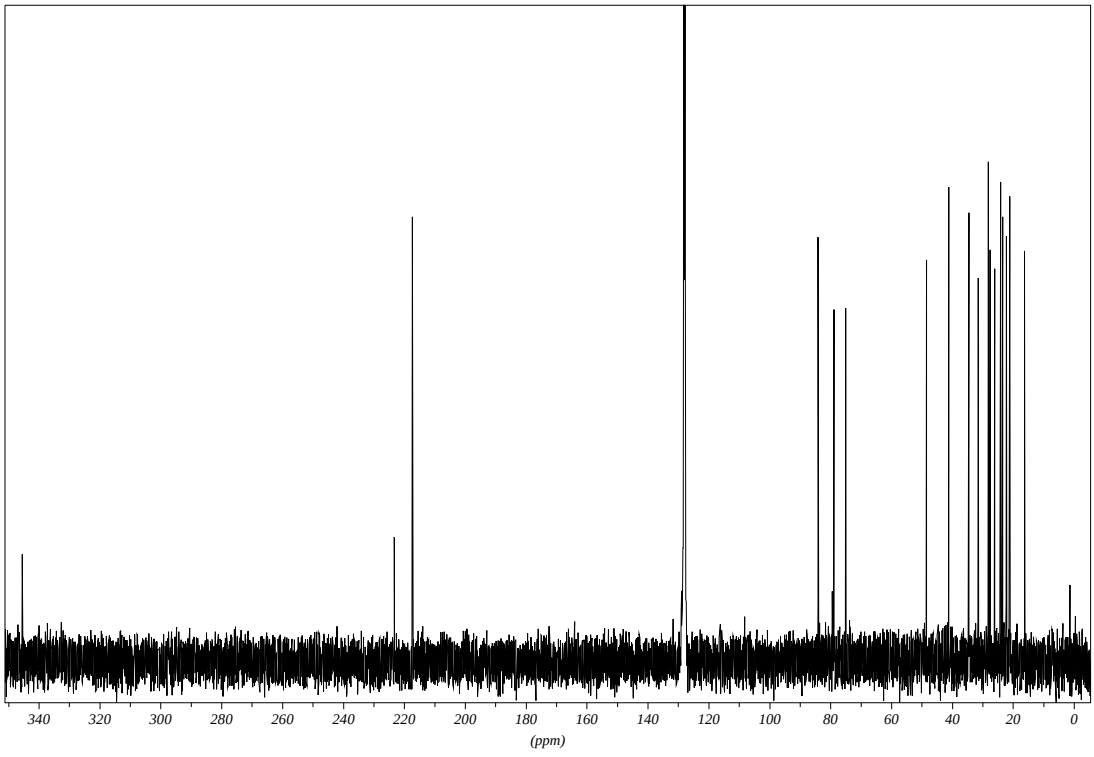


**(*1S,4R,1´S,2´R,5´S*)-Pentacarbonyl{1-(5´-methyl-2´-methylethylcyclohexyloxy)-6-oxa-spiro[3.4]oct-5-yliden}chrom(0)** ***[75]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

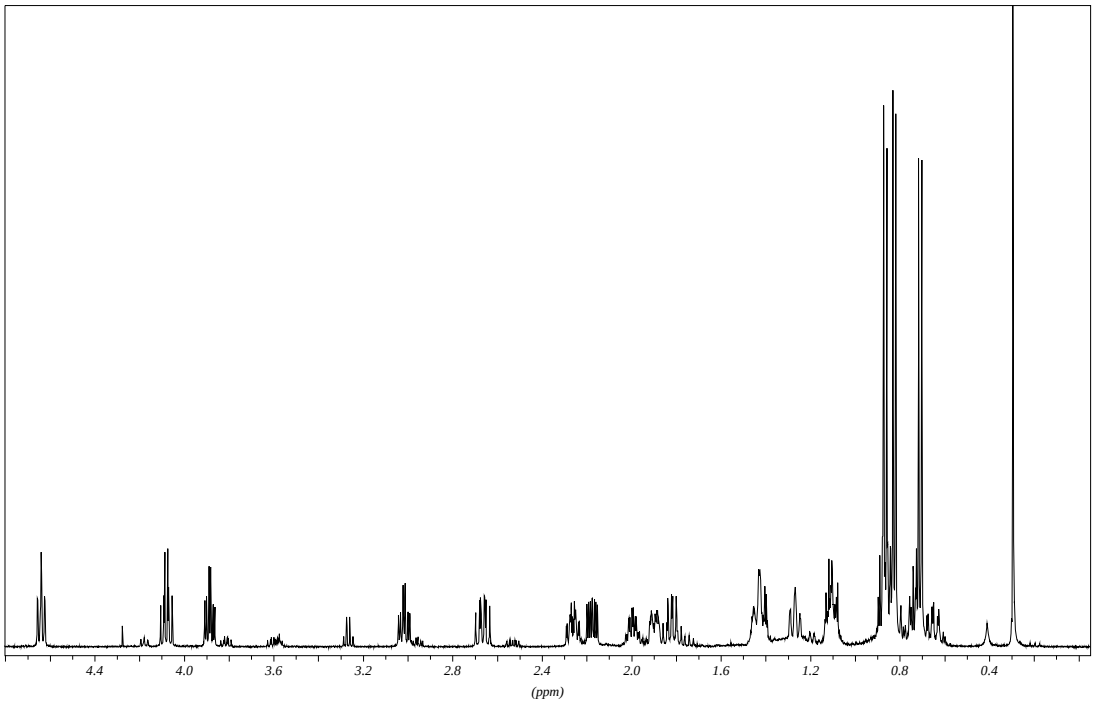



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

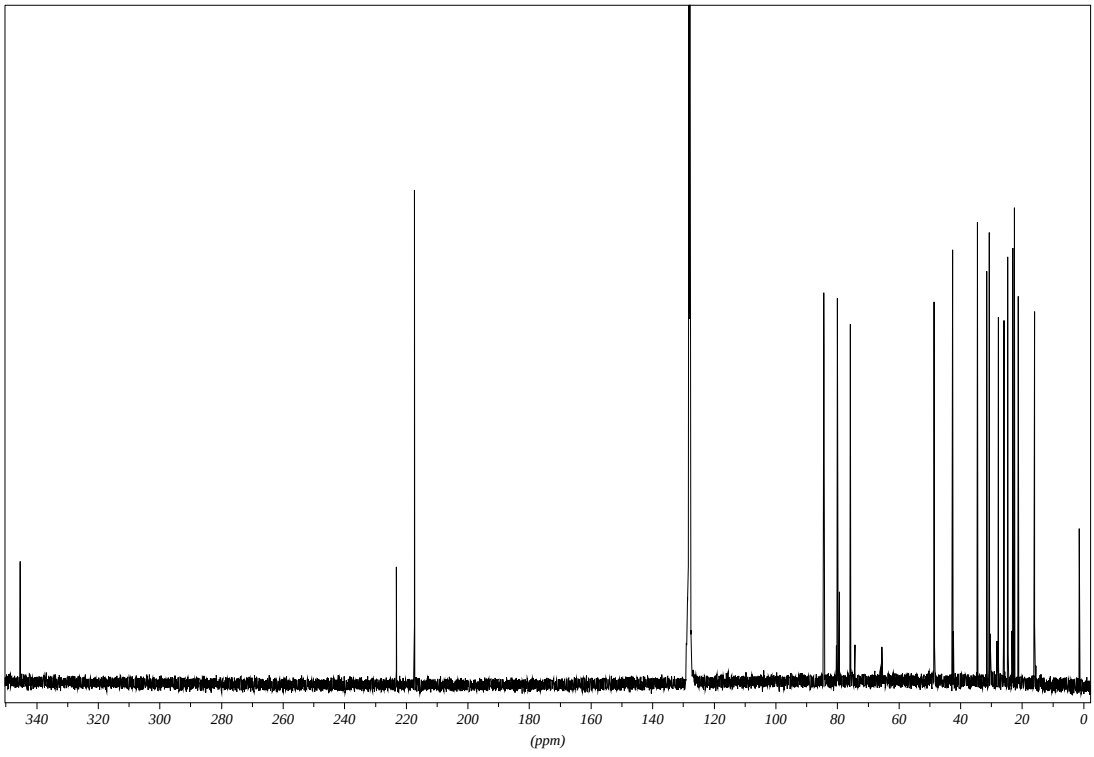


**(*1R,4S,1´S,2´R,5´S*)-Pentacarbonyl{1-(5´-methyl-2´-methylethylcyclohexyloxy)-6-oxa-spiro[3.4]oct-5-yliden}chrom(0)** ***[75]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

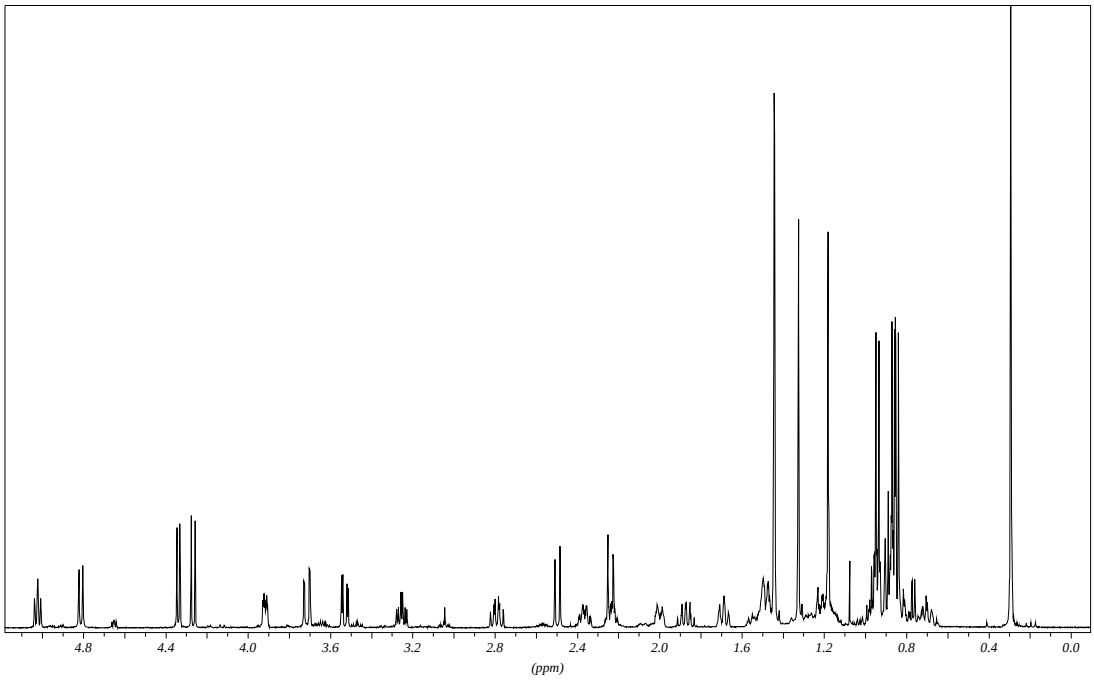



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

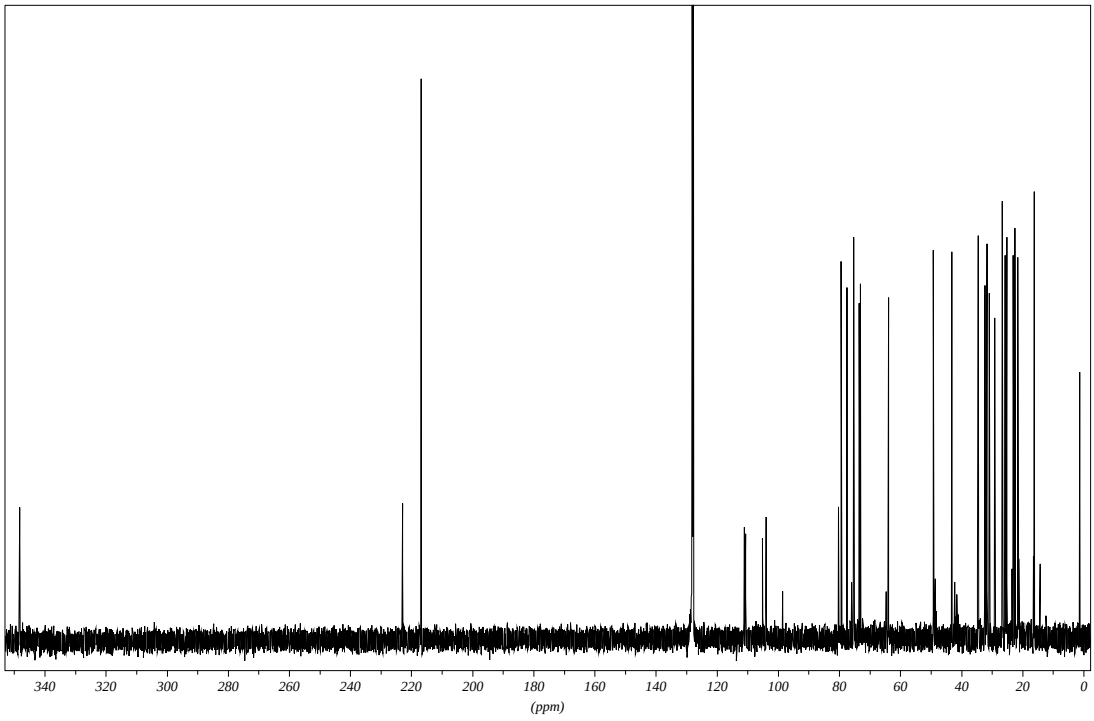


**(*1R,4S,7R,1´R,4´S,6´R,1´´´S,2´´´R,5´´´S*)-Pentacarbonyl{1-(5´´´-methyl-2´´´-methyl-ethylcyclohexyloxy)-8´,8´,2´´,2´´-tetramethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro[3.4]oct]-5-yliden}chrom(0)** ***[77]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

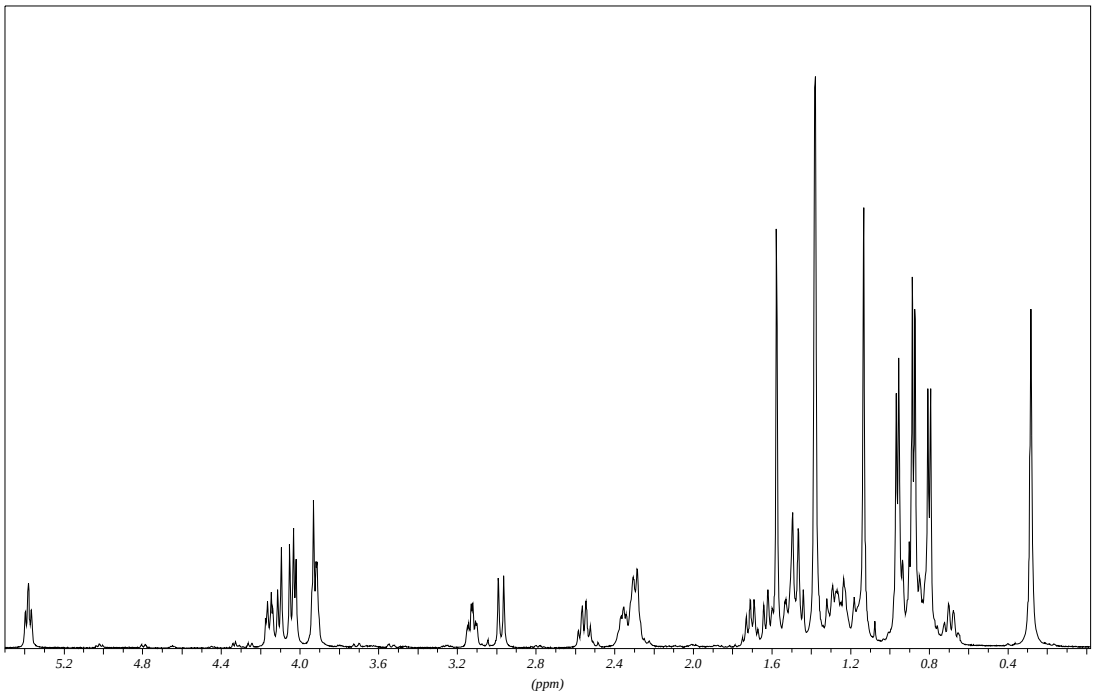



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

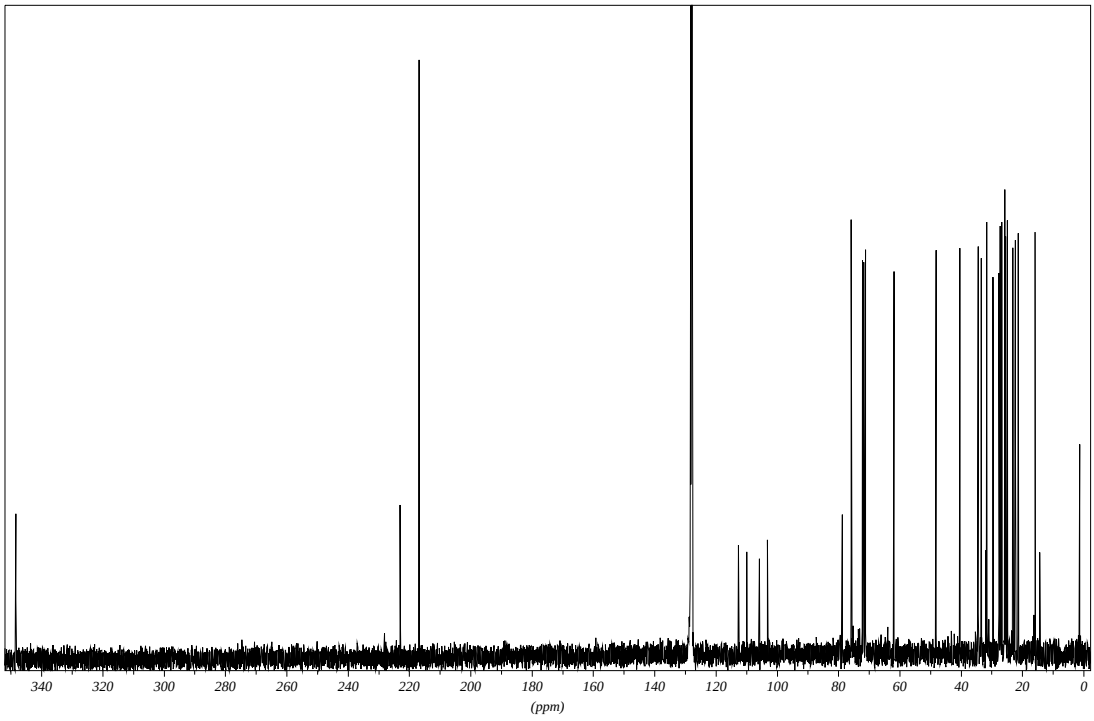


**(*1S,4R,7R,1´R,4´S,6´R,1´´´S,2´´´R,5´´´S*)-Pentacarbonyl{1-(5´´´-methyl-2´´´-methyl-ethylcyclohexyloxy)-8´,8´,2´´,2´´-tetramethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro[3.4]oct]-5-yliden}chrom(0)** ***[77]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

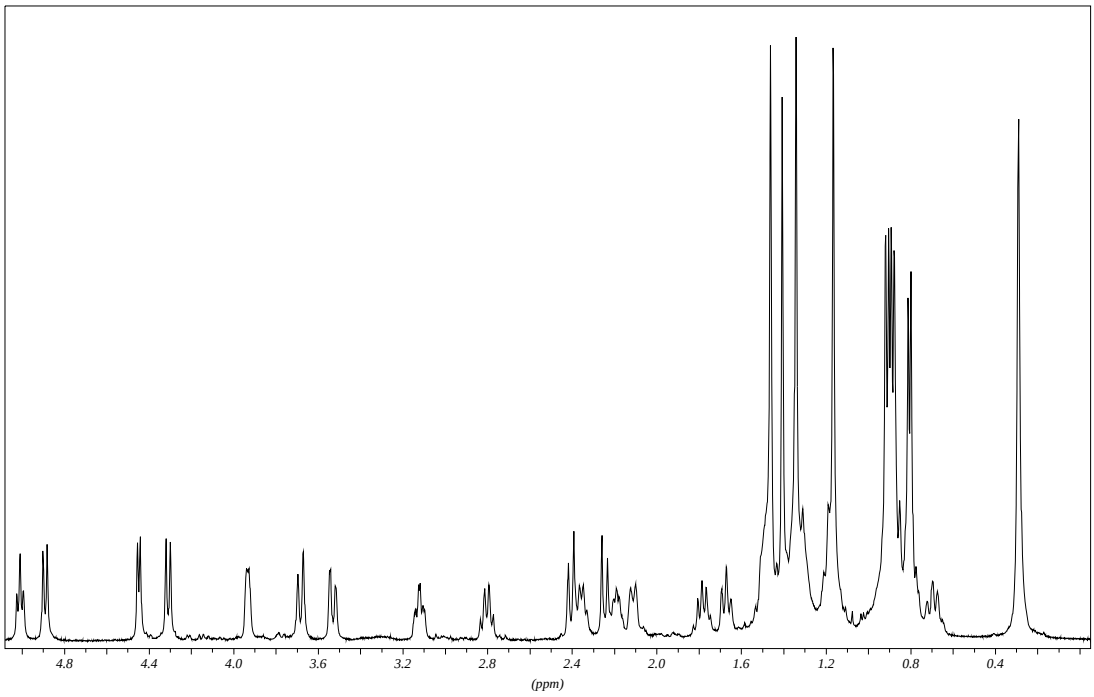



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

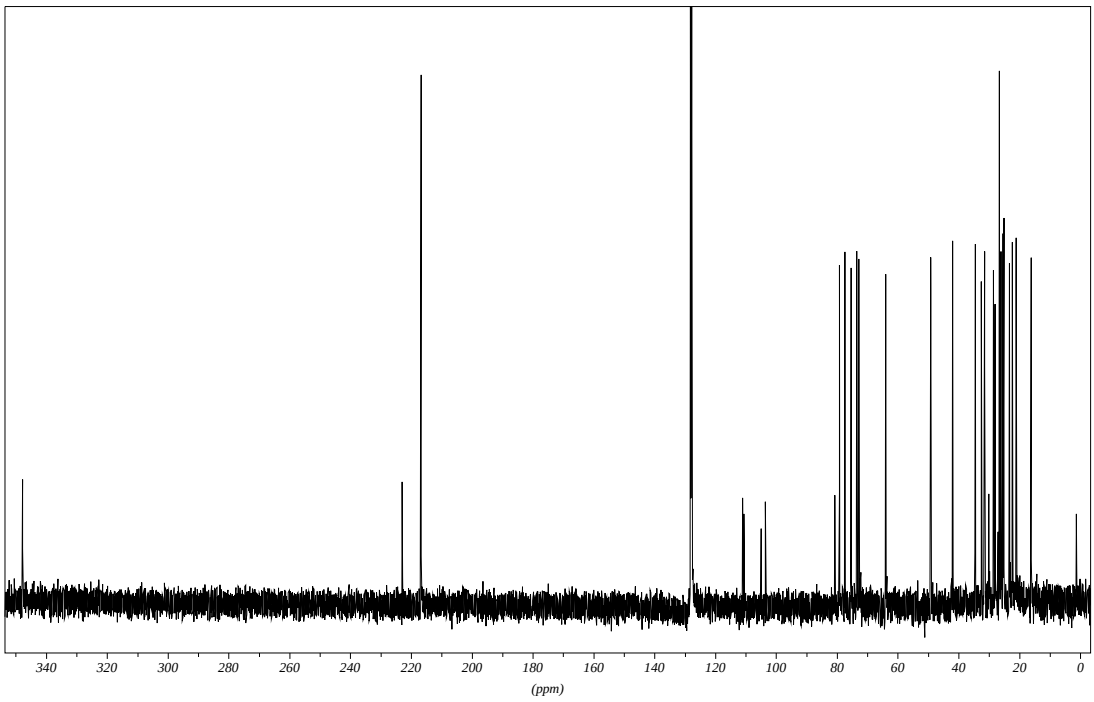


**(*1R,4S,7R,1´R,4´S,6´R,1´´´R,2´´´S,5´´´R*)-Pentacarbonyl{1-(5´´´-methyl-2´´´-methyl-ethylcyclohexyloxy)-8´,8´,2´´,2´´-tetramethyl-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro[3.4]oct]-5-yliden}chrom(0)** ***[78]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

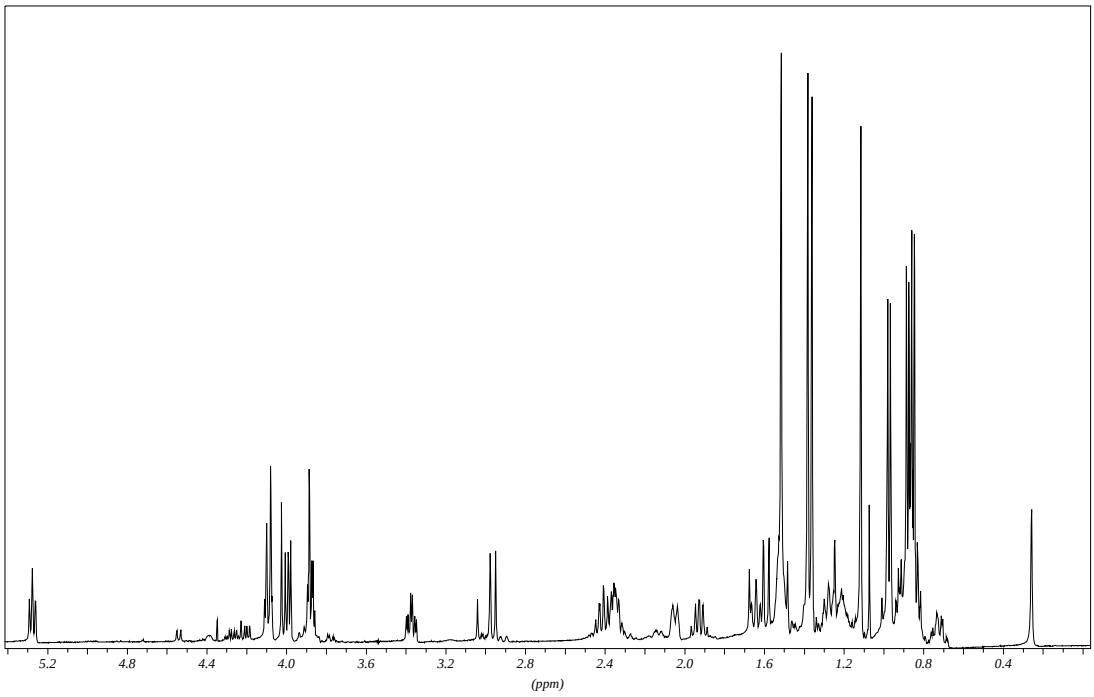



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

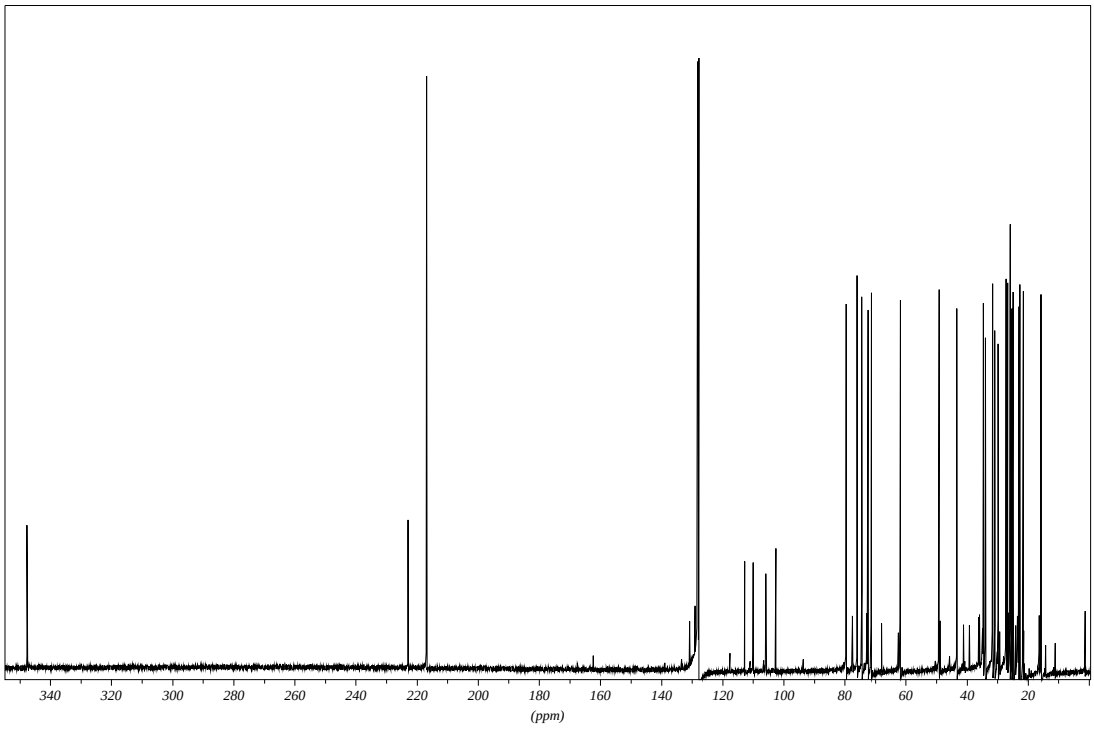


**(*1S,4R,7R,1´R,4´S,6´R,1´´´R,2´´´S,5´´´R*)-Pentacarbonyl{1-(5´´´-methyl-2´´´-methyl-ethylcyclohexyloxy)-8´,8´,2´´,2´´-tetramethyl-dispiro-[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro[3.4]oct]-5-yliden}chrom(0)** ***[78]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

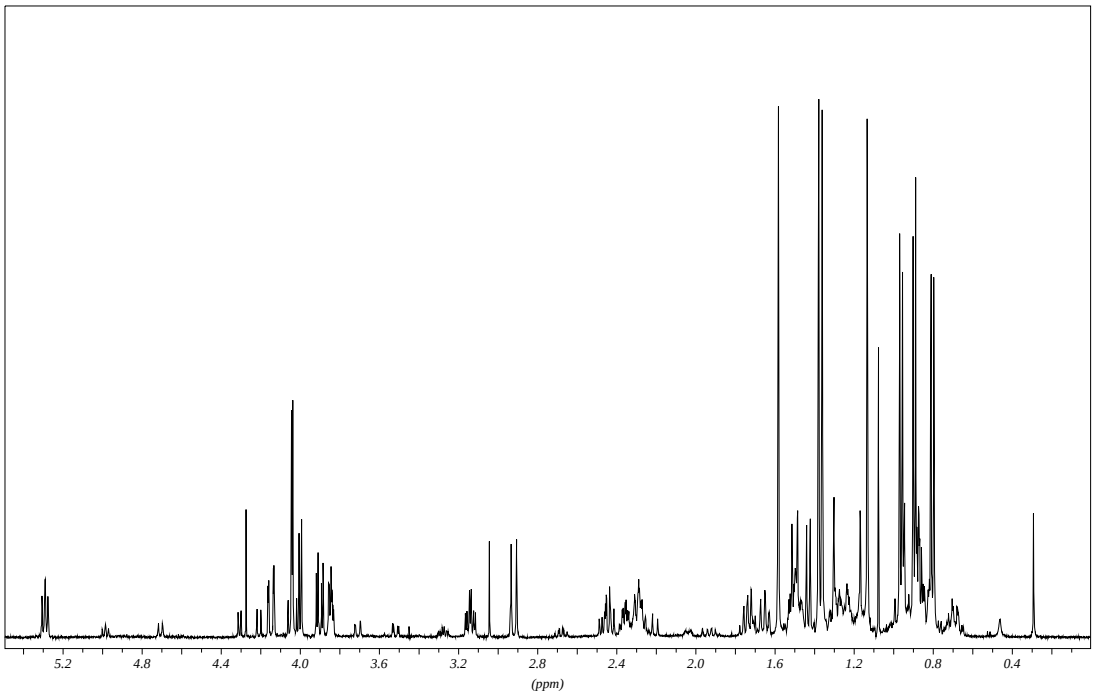



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

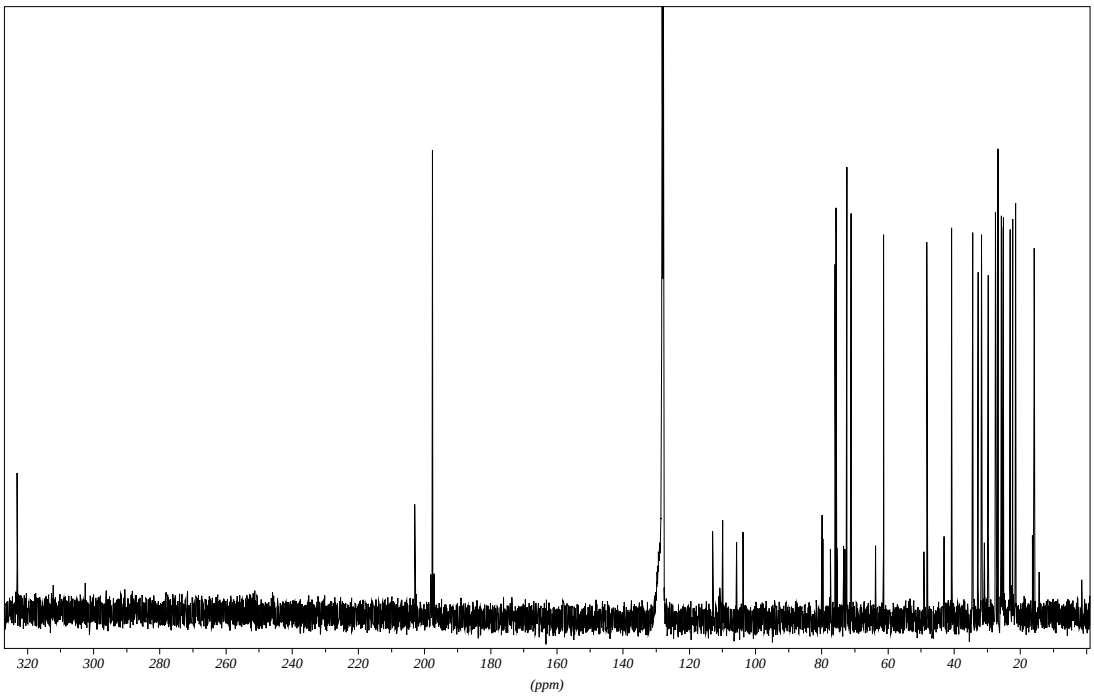


**(*1R,4S,7R,1´R,4´S,6´R,1´´´S,2´´´R,5´´´S*)-Pentacarbonyl{1-(5´´´-methyl-2´´´-methyl-ethylcyclohexyloxy)-8´,8´,2´´,2´´-tetramethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro-[3.4]oct]-5-yliden}wolfram(0) *[79]a***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

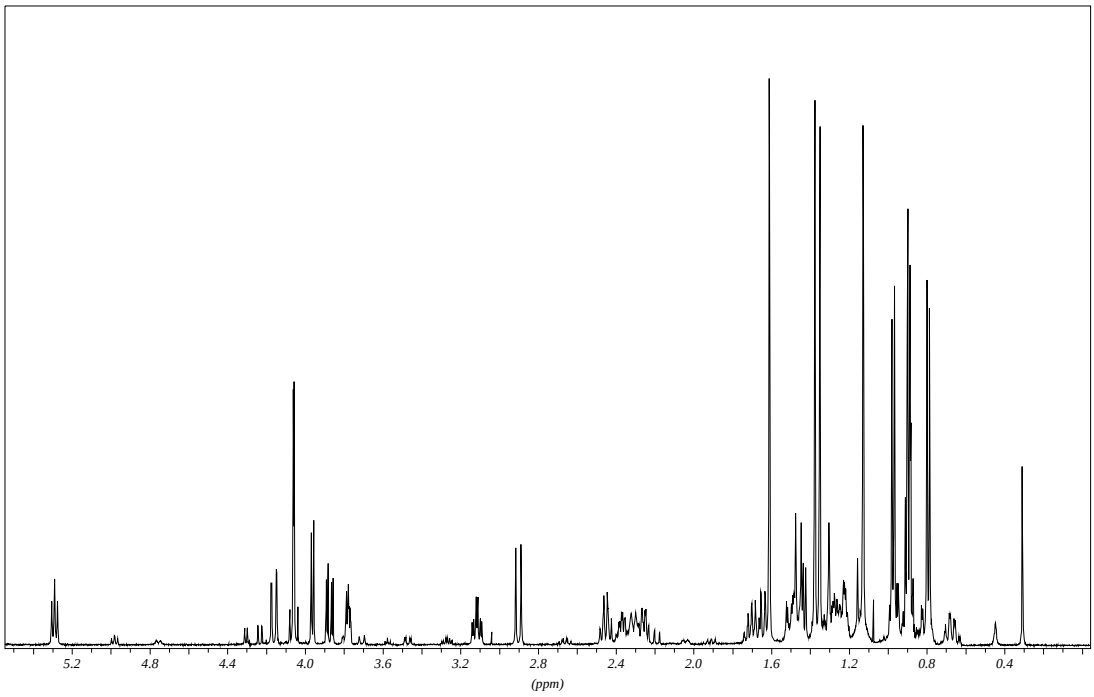



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

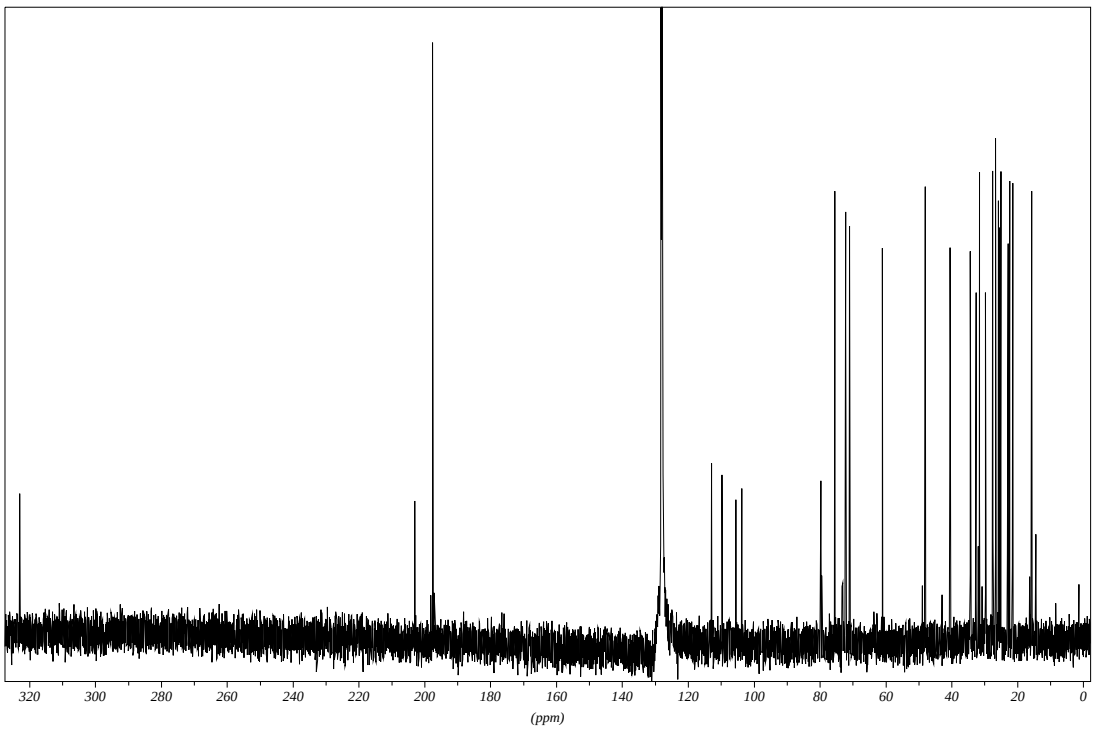


**(*1S,4R,7R,1´R,4´S,6´R,1´´´S,2´´´R,5´´´S*)-Pentacarbonyl{1-(5´´´-methyl-2´´´-methyl-ethylcyclohexyloxy)-8´,8´,2´´,2´´-tetramethyl-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,7-6-oxaspiro-[3.4]oct]-5-yliden}wolfram(0) *[79]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

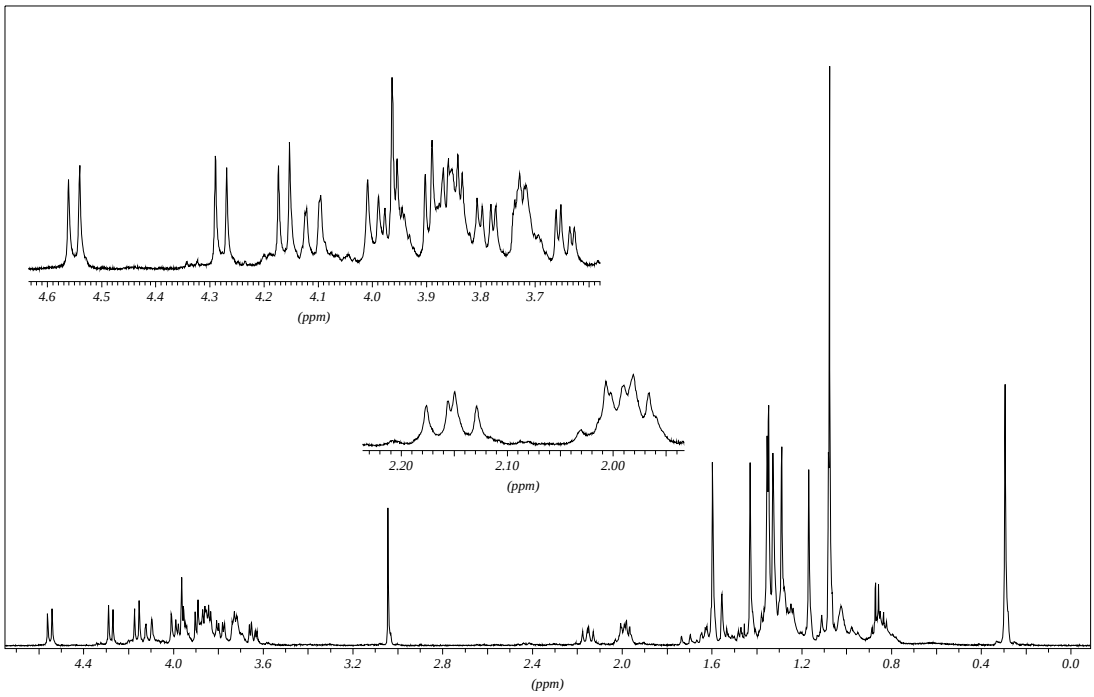



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

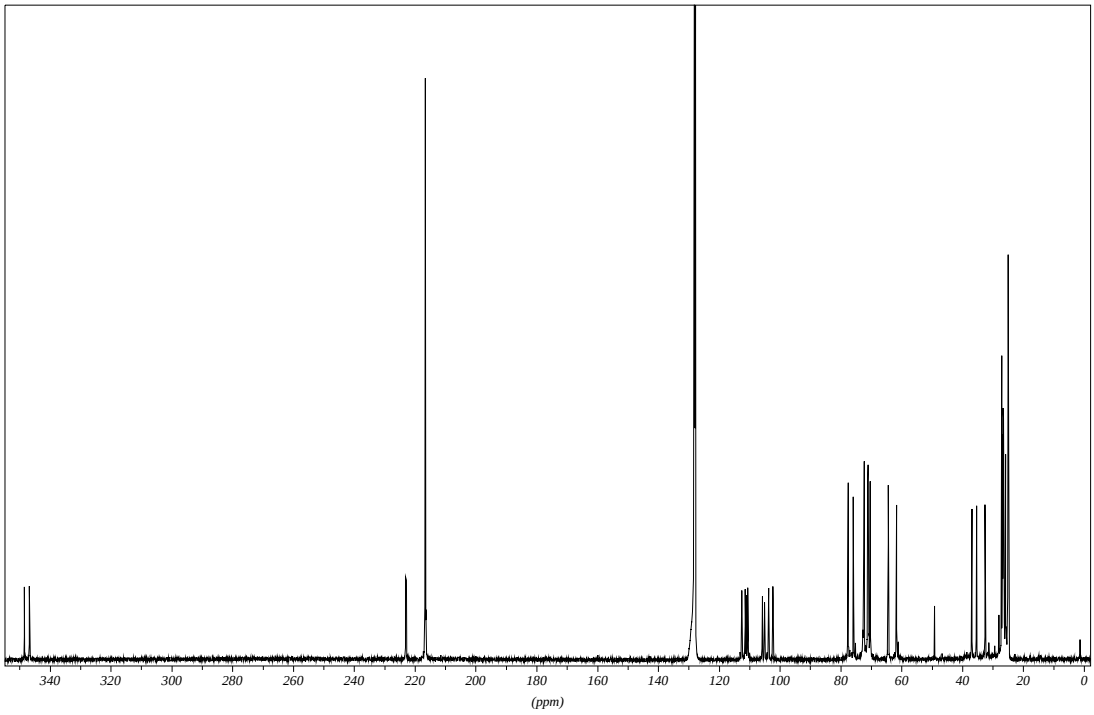


**(*1R,1S*)-Bis[pentacarbonyl{8´,8´,2´´,2´´-tetramethyl(*1´R,4´S,5´R,6´R*)-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,4-3-oxacyclopent]-2-yliden}-chrom(0)]methan *[82]b***

$^{1}$H-NMR-Spektrum (500 MHz, $C_6D_6$):

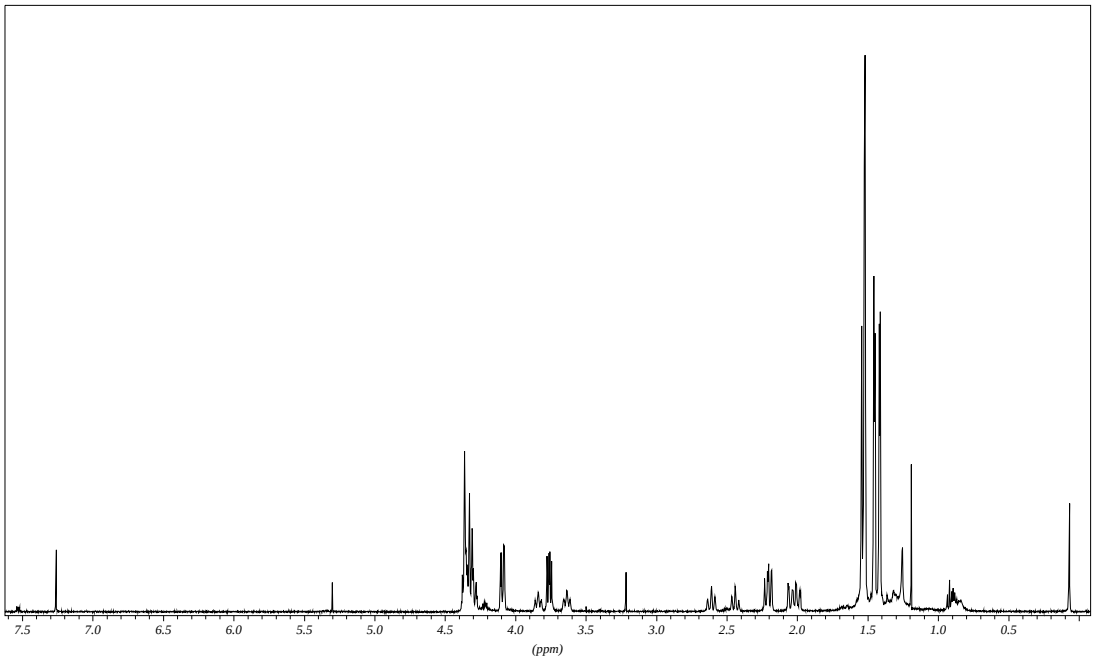



$^{13}$C-NMR-Spektrum (500 MHz, $C_6D_6$):

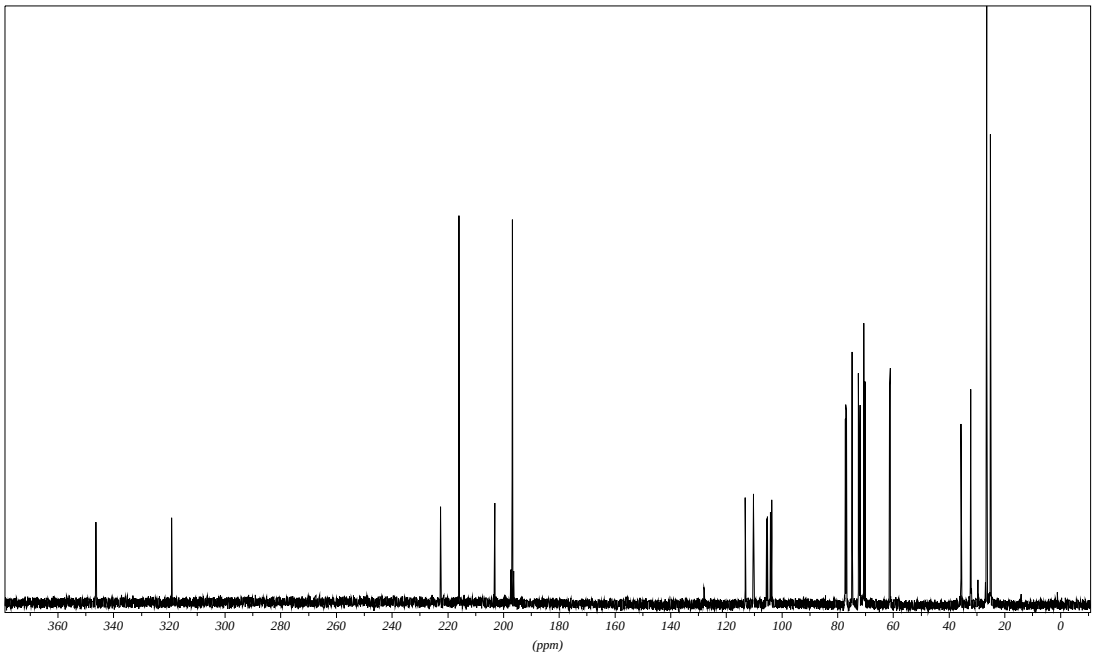


**[Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl(*1´R,4´S,5´R,6´R*)-dispiro[3,7,9-trioxabicyclo-[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,4-(*1R*)-3-oxacyclopent]-2-yliden}-chrom(0)],[Pentacarbonyl{8´,8´,2´´,2´´-tetramethyl(*1´R,4´S,5´R,6´R*)-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,4-(*1R*)-3-oxacyclopent]-2-yliden}wolfram(0)]methan *[83]***

$^{1}$H-NMR-Spektrum (500 MHz, $CDCl_3$):

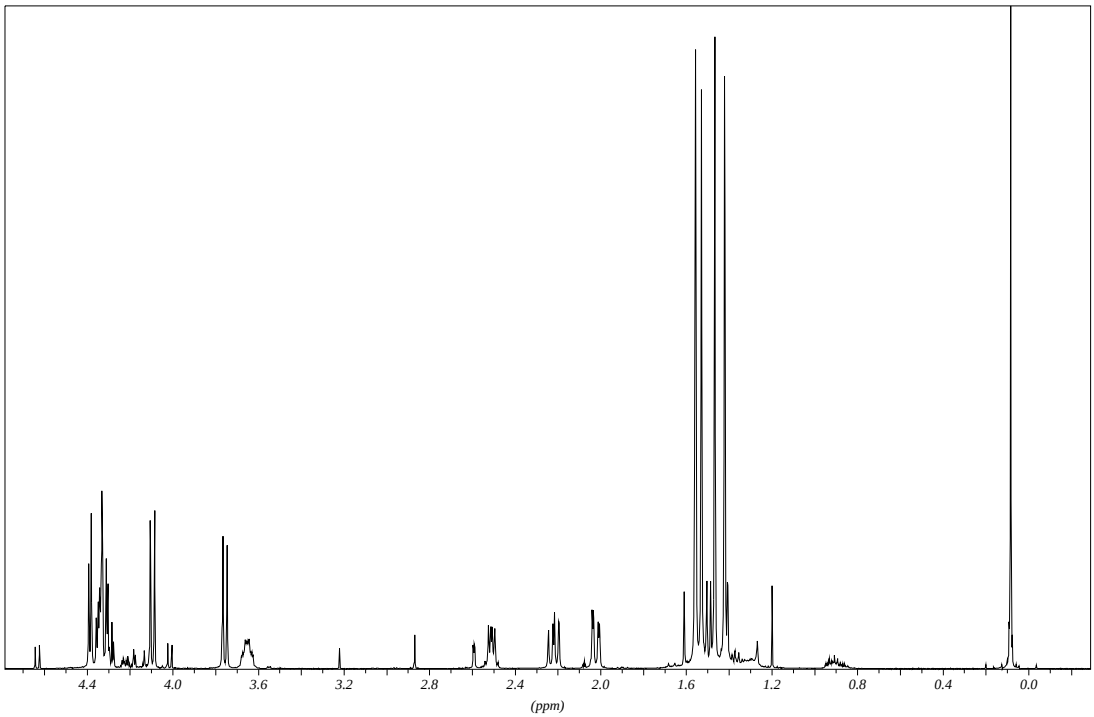



$^{13}$C-NMR-Spektrum (500 MHz, $CDCl_3$):



**(*1R,1R*)-Bis[pentacarbonyl{8´,8´,2´´,2´´-tetramethyl(*1´R,4´S,5´R,6´R*)-dispiro[3,7,9-trioxabicyclo[4.3.0]nonan-4´,4´´-1,3-dioxacyclopentan-5´,4-3-oxacyclopent]-2-yliden}-wolfram(0)]methan *[84]***

$^{1}$H-NMR-Spektrum (500 MHz, $CDCl_3$):




$^{13}$C-NMR-Spektrum (500 MHz, $CDCl_3$):