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Synthesis of *N*-allyl-2,3-dihydrobenzoxazol-2-ylidene complexes of chromium(0) and tungsten(0)[☆]

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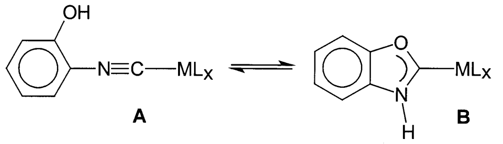
Abstract

The reaction of 2-trimethylsiloxyphenyl isocyanide (**1**) with [M(CO)$_5$(THF)] (M = Cr, W) gives the complexes [M(2-Me$_3$SiO–C$_6$H$_4$NC)(CO)$_5$] (**2**) (**2a**, M = Cr; **2b**, M = W). Hydrolysis of the Si–O bond leads to the formation of an equilibrium between complexes **3** (**3a**, M = Cr; **3b**, M = W) with a 2-hydroxyphenyl isocyanide and complexes **4** (**4a**, M = Cr; **4b**, M = W) with a 2,3-dihydrobenzoxazol-2-ylidene ligand. Deprotonation of mixtures **3**/**4** followed by alkylation with allyl bromide yields exclusively *N*-allyl-2,3-dihydrobenzoxazol-2-ylidene complexes (**5**) (**5a**, M = Cr; **5b**, M = W). Thermally induced intramolecular substitution of one *cis*-carbonyl ligand in (*N*-allyl-2,3-dihydrobenzoxazol-2-ylidene)-pentacarbonyltungsten (**5b**) results in the formation of the alkene–carbene complex **6** incorporating an η^2:η^1-(*N*-allyl-2,3-dihydrobenzoxazol-2-ylidene) ligand. The molecular structure of **5b** is reported. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Crystal structures; Chromium complexes; Tungsten complexes; Carbene complexes; Alkene complexes

1. Introduction

The use of 2-trimethylsiloxyphenyl isocyanide (**1**) [1] as a suitable starting material for the synthesis of *N*,*O*-heterocarbene complexes has been demonstrated [2,3]. A more detailed examination of this type of reaction revealed that coordination of **1** to a transition metal fragment followed by hydrolysis of the Si–O bond results in a chemical equilibrium between complexes with a 2-hydroxyphenyl isocyanide (**A**) or a 2,3-dihydrobenzoxazol-2-ylidene ligand (**B**) (Scheme 1) [4].



Scheme 1.

The position of the equilibrium depends on the 'electron-richness' of the metal fragment ML$_x$. Strong (d → π*) backbonding from relatively electron-rich metal fragments (e.g. Re(I)) can completely suppress intramolecular cyclization of the hydroxyphenyl isocyanide ligand [3,4b,4d]. On the other hand, complete ylidene formation is observed upon 2-hydroxyphenyl isocyanide coordination to relatively electron-poor metal fragments due to weaker backbonding (e.g. Re(V)) [2c,2d,4d]. Furthermore, it could be shown that even electronically stabilized isocyanide complexes of the type **A** can be converted to ylidene derivatives by deprotonation and subsequent N-alkylation. In deprotonated complexes of type **A** the phenolate oxygen atom is a strong nucleophile causing complete cyclization. N-Alkylation of the resulting monoanionic benzoxazol-2-yl complex results in the formation of complexes with *N*-alkyl-2,3-dihydrobenzoxazol-2-ylidene ligands and prevents reopening of the N,O-heterocycle.

In this contribution we like to report on the synthesis and characterization of the N-allylated complexes (*N*-allyl-2,3-dihydrobenzoxazol-2-ylidene)pentacarbonylchromium (**5a**) and -tungsten (**5b**). These compounds

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[☆] This paper is dedicated to Professor Dr B. Krebs, on the occasion of his 60th birthday.

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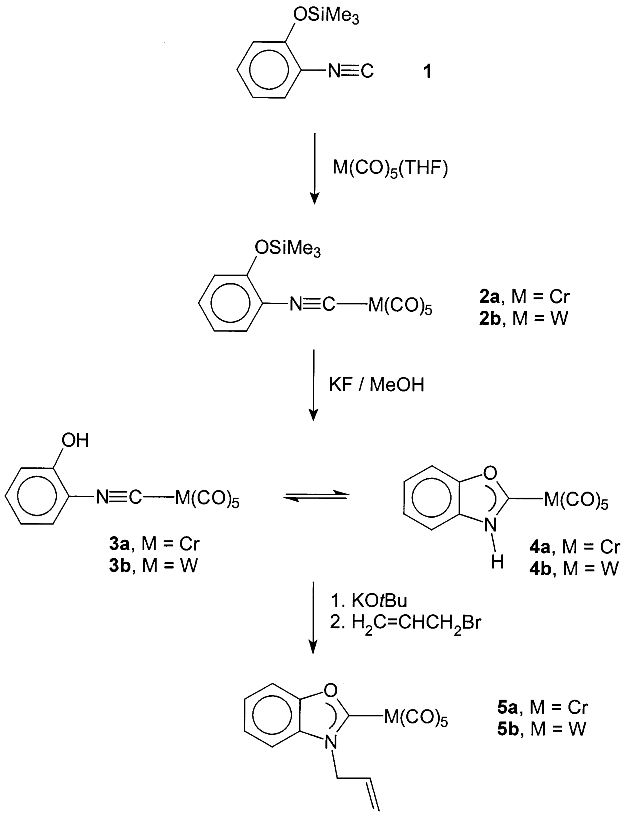
are potentially useful for the synthesis of carbene–alkene complexes [5,6], which have been proposed as intermediates in several catalytic processes such as olefin metathesis, cyclopropanation of alkenes and heterogenous Ziegler–Natta polymerization of alkenes.

2. Results and discussion

The treatment of 2-trimethylsiloxyphenyl isocyanide (**1**) with [M(CO)$_5$(THF)] gives complexes [Cr(**1**)(CO)$_5$] (**2a**) and [W(**1**)(CO)$_5$] (**2b**) as colorless viscous oils in good yields (Scheme 2). Hydrolysis of the Si–O bond is best achieved by stirring complexes **2** in methanolic solution with a catalytic amount of KF for 2 days [2,4]. After chromatography on Al_2O_3 (4% H_2O) mixtures of the desilylated complexes **3** and **4** are isolated as off-white solids.

The isocyanide complexes **3** can be easily identified in these mixtures by the strong IR absorptions due to the C≡N stretching mode (2132 cm^{-1} for **3a**; 2141 cm^{-1} for **3b**), while the carbene compounds **4** show a characteristic IR absorption for the N–H bond (3436 cm^{-1} for **4a**; 3433 cm^{-1} for **4b**). The ^{1}H NMR spectra in CD_2Cl_2 exhibit broad singlets for the O*H* resonances at about 5.6 ppm as well as for the N*H* resonances at about 10.4 ppm. Integration of these resonances allowed us to determine the ratio of complexes **3**/**4** showing that the chromium complexes **3a**/**4a** form a 29:71 mixture, whereas the tungsten derivatives **3b**/**4b** form a 15:85 mixture [4b]. As the position of the equilibrium for both complexes resides more strongly on the right, resulting in an excess of the carbene component, all ^{13}C NMR resonances can be unambiguously assigned to complexes **3** or **4**, respectively (Table 1).



Scheme 2.

The CO carbon resonances of **4a** and **4b** are observed at a lower field than those of **3a** and **3b**, indicating that the carbene ligand in **4** is a weaker π-acceptor than the isocyanide ligand in **3** [7]. Consequently, the ^{13}C NMR spectra of complexes **4** exhibit larger Δδ differences between the *trans*- and *cis*-CO resonances than those of complexes **3**, as the *trans*-carbonyl ligand directly competes with the ylidene or isocyanide ligand for electron-density from the metal center.

After treatment of mixtures **3**/**4** with KO^{t}Bu in DMF followed by addition of allyl bromide the stable N-allylated carbene complexes **5a** and **5b** are exclusively isolated, which cannot rearrange to the 'open' isocyanide form (Scheme 2). No 2-allyloxyphenyl isocyanide derivatives can be isolated from the reaction mixture. In deprotonated **3**, the negatively charged phenolate oxygen atom is a strong nucleophile which rapidly attacks the isocyanide carbon atom under formation of anionic benzoxazol-2-yl complexes, which are subsequently allylated at the nitrogen atom. Naturally, the spectroscopic data of the carbene derivatives **4** and **5** are very similar.

The molecular structure of **5b** was established by X-ray diffraction analysis, and Fig. 1 shows an ORTEP diagram. The bond distances and angles compare well with equivalent parameters in related tungsten complexes with 2,3-dihydrobenzoxazol-2-ylidene ligands [2b]. The O–C–N angle at the carbene carbon atom

Table 1
Selected ^{13}C NMR data[a]

Compound	M–C	*trans*-CO	*cis*-CO	Δδ[b]
2a	173.0	216.9	214.6	2.3
2b	153.6	196.5	194.1	2.4
3a	174.0	218.1	215.6	2.5
3b	[c]	197.5	194.9	2.6
4a	232.0	222.2	217.7	4.5
4b	211.6	202.3	197.8	4.5
5a	233.8	221.1	216.5	4.6
5b	215.5	201.8	197.3	4.5

[a] δ (ppm), 62.90 MHz; **2** in $CDCl_3$, **3** and **4** in d^6-acetone, **5** in CD_2Cl_2.

[b] δ(*trans*-CO)–δ(*cis*-CO).

[c] Not observed.

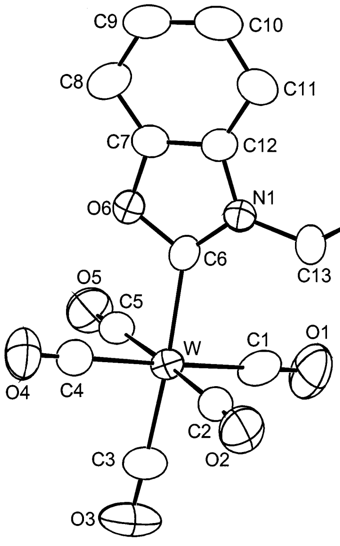


Fig. 1. ORTEP drawing of **5b**. Selected bond lengths (Å) and angles (°): W–C1 2.041(6), W–C2 2.030(5), W–C3 1.993(5), W–C4 2.051(5), W–C5 2.048(5), W–C6 2.198(5), $O–C_{carb}$ 1.120(6)–1.142(6), O6–C6 1.368(5), O6–C7 1.374(5), N1–C6 1.349(6), N1–C12 1.403(5), N1–C13 1.457(5), C7–C12 1.361(6), C13–C14 1.487(6), C14–C15 1.293(7); C6–O6–C7 109.5(3), C6–N1–C12 110.2(3), C6–N1–C13 125.1(4), C12–N1–C13 124.6(4), $W–C_{carb}–O$ 176.7(4)–180.0(5), W–C6–O6 118.9(3), W–C6–N1 134.9(3), O6–C6–N1 106.2(4).

(106.2(4)°) is typical for coordinated cyclic heterocarbenes [8].

The carbene ligand in **5a** and **5b** is potentially bidentate, as coordination of the alkene moiety of the allyl substituent can lead to the formation of alkene–carbene complexes. Several such complexes with acyclic [6] and cyclic [9] carbene ligands have been reported, and their role as intermediates in several catalytic processes such as olefin metathesis, cyclopropanation of alkenes and heterogenous Ziegler–Natta polymerization of alkenes has been outlined extensively [5,6]. In order to show whether intramolecular alkene coordination is possible for complexes of the type **5**, the tungsten derivative **5b** was heated in toluene under reflux for 3 days. After purification by chromatography, complex **6** containing an η^2:η^1-(*N*-allyl-2,3-dihydrobenzoxazol-2-ylidene) ligand was isolated as a yellow solid in a relatively low yield (14%, Scheme 3).

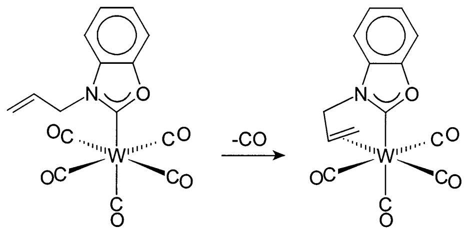
The intramolecular olefin coordination is easily detected by ^{1}H NMR spectroscopy, and the resonances and coupling constants for the protons of the allyl group are summarized in Table 2.

The ^{1}H NMR spectra of **5b** and **6** showing only the allyl resonances are depicted in Fig. 2. All resonances for the allyl protons are observed at higher field upon coordination of the alkene moiety with the resonances of *cis*- and *trans*-H3 being most strongly shifted by ca. 2 ppm. In **6**, two resonances are observed for the diastereotopic allylic protons H1 with a ^2J coupling constant of 12.5 Hz. Thereby, the resonance of proton H2 changes from a ddt in **5b** to a dddd in **6**.

The use of *N*-allyl-2,3-dihydrobenzoxazol-2-ylidene complexes for the synthesis of alkene–carbene complexes could be demonstrated. This has prompted us to synthesize related 2,3-dihydro-1*H*-benzimidazol-2-ylidene complexes, which can be doubly allylated at the nitrogen atoms [10]. Currently, the suitability of these ligands for the synthesis of carbene-bis(alkene) complexes or even homoleptic bis(carbene)–tetrakis(alkene) complexes is being investigated.

3. Experimental

All operations were performed in an atmosphere of dry argon by using Schlenk and vacuum techniques.



5b **6**

Scheme 3.

Table 2
Selected ^{1}H NMR data of **5b** and **6**[a,b,c]

Compound	H1		H2	*trans*-H3	*cis*-H3
5b	5.12 (ddd) $^3J = 5$ $^4J \approx 2, 1$		6.02 (ddd) $^3J = 17, 10, 5$	5.40 (dt) $^3J = 10$ $^4J \approx 1$	5.28 (dt) $^3J = 17$ $^4J \approx 2$
6	4.62 (dd) $^2J = 12.5$ $^3J = 5$	4.43 (dd) $^2J = 12.5$ $^3J = 5$	4.88 (dddd) $^3J = 12, 8, 5, 3$	3.46 (d) $^3J = 8$	3.26 (d) $^3J = 12$

[a] δ/(ppm), 250 MHz.

[b] Coupling constants in Hz.

[c] In $CDCl_3$.

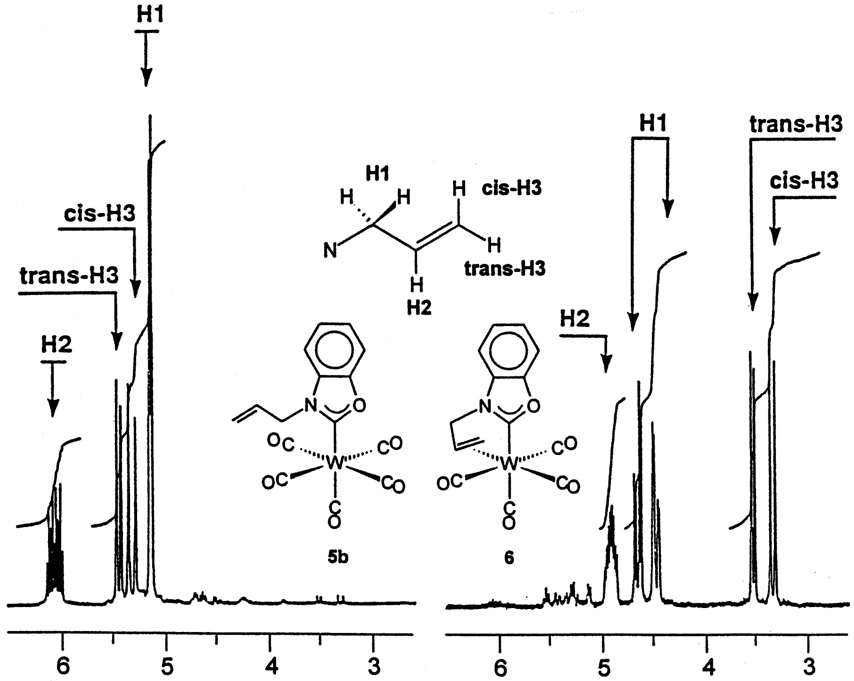


Fig. 2. ^{1}H NMR spectra (allylic resonances only) of **5b** and **6** (250 MHz, $CDCl_3$, δ in ppm).

Solvents were dried by standard methods and distilled prior to use. NMR spectra were recorded on a Bruker AM 250 (250 MHz) instrument. IR spectra were taken on a Perkin-Elmer 983 instrument in KBr. Elemental analyses (C, H, N) were performed at the Freie Universität Berlin on a Heraeus CHN-Rapid elemental analyzer. Mass spectra (EI, 70 eV) were recorded on a Varian MAT 711 instrument. Ligand **1** was prepared as described in Ref. [1].

3.1. *Pentacarbonyl(2-trimethylsiloxyphenyl isocyanide)-chromium(0) (**2a**)*

$[Cr(CO)_6]$ (5 g, 23 mmol) dissolved in 180 ml of THF was irradiated in a photoreactor (high pressure mercury vapour lamp) for 6 h. To the solution of $[Cr(CO)_5(THF)]$ was added **1** (4.35 g, 23 mmol) and the reaction mixture was stirred overnight. The solvent was removed in vacuo and the residue was used directly for subsequent reactions. ^{1}H NMR ($CDCl_3$, 250 MHz): δ 7.29 (m, 2H, Ar*H*); 6.98 (m, 2H, Ar*H*); 0.40 (s, 9H, $SiCH_3$) ppm. ^{13}C NMR ($CDCl_3$, 62.90 MHz): δ 216.9 (*trans*-*C*O); 214.6 (*cis*-*C*O); 173.0 (Ar–N*C*); 151.6 (*C*OSi); 130.0, 127.2, 121.6, 120.2 (Ar*C*); 120.7 (*C*–NC); 0.1 (Si*C*H_3) ppm.

3.2. *Pentacarbonyl(2-trimethylsiloxy)phenyl isocyanide)-tungsten(0) (**2b**)*

Complex **2b** was prepared analogously to **2a** from $[W(CO)_6]$ (2.56 g, 7 mmol) and **1** (1.39 g, 7 mmol). ^{1}H NMR ($CDCl_3$, 250 MHz): δ 7.46–6.91 (m, 4H, Ar*H*); 0.38 (s, 9H, $SiCH_3$) ppm. ^{13}C NMR ($CDCl_3$, 62.90 MHz): δ 196.5 (*trans*-*C*O); 194.1 (*cis*-*C*O, $^1J(^{183}W-^{13}C) = 125$ Hz); 153.6 (Ar–N*C*); 151.8 (*C*OSi); 130.3, 127.3, 121.7, 120.3 (Ar–*C*); 119.8 (*C*–NC); 0.1 (Si*C*H_3) ppm. IR (KBr): ν 2148 (CN), 2062, 1980, 1963 (CO) cm^{-1}.

3.3. *Mixture of pentacarbonyl(2-hydroxyphenyl isocyanide)chromium(0) (**3a**) and pentacarbonyl-(2,3-dihydrobenzoxazol-2-ylidene)chromium(0) (**4a**)*

Complex **2a** (from 5 g of $[Cr(CO)_6]$ and 4.35 g of **1**) was dissolved in 100 ml of methanol. After addition of a catalytic amount of KF (50 mg) the reaction mixture was stirred for 12 h. The solvent was evaporated in vacuo and the residue was purified by chromatography on Al_2O_3 (4% H_2O). Mixture **3a**/**4a** was eluted with $MeOH/Et_2O$ (1:10). Evaporation of the solvents afforded an off-white solid, 4.16 g (60%). ^{1}H NMR (CD_2Cl_2, 250 MHz): δ 10.44 (s br, N*H*, **4a**); 7.67–6.93 (m, Ar*H*, **3a** + **4a**); 5.58 (s br, O*H*, **3a**). For selected ^{13}C NMR data, see Table 1. *Anal.* Calc. for $C_{12}H_5CrNO_6$ ($M = 311.17$): C, 46.32; H, 1.62; N, 4.50. Found: C, 46.12; H, 1.94; N, 4.63%. IR (KBr): ν 3436 (NH); 2132 (CN) cm^{-1}. MS (EI, 70 eV): *m/z* (%) 311 (14) [M^+].

3.4. *Mixture of pentacarbonyl(2-hydroxyphenyl isocyanide)tungsten(0) (**3b**) and pentacarbonyl-(2,3-dihydrobenzoxazol-2-ylidene)tungsten(0) (**4b**)*

Complex **2b** (from 2.56 g of $[W(CO)_5]$) was used, and the preparation and isolation followed the procedure described under Section 3.3. Complexes **3b**/**4b** were obtained as an off-white solid, 1.67 g (52%). ^{1}H NMR (CD_2Cl_2, 250 MHz): δ 10.43 (s br, N*H*, **4b**); 7.66–6.95 (m, C*H*, **3b** + **4b**); 5.63 (s br, O*H*, **3b**). For selected ^{13}C NMR data, see Table 1. *Anal.* Calc. for $C_{12}H_5NO_6W$ ($M = 443.02$): C, 32.53; H, 1.14; N, 3.16. Found: C, 32.84; H, 1.29; N, 3.30%. IR (KBr): ν 3433 (NH); 2141 (CN) cm^{-1}. MS (EI, 70 eV): m/z (%) 443 (48) [M^+].

3.5. (N-Allyl-2,3-dihydrobenzoxazol-2-ylidene) pentacarbonylchromium(0) (5a)

A solution of **3a**/**4a** (805 mg, 2.6 mmol) in 20 ml of DMF was treated with KO^{t}Bu (337 mg, 3.0 mmol) at −40°C. After stirring for 3 h at room temperature allyl bromide (363 mg, 3 mmol) was added and stirring continued overnight. The solvent was evaporated in vacuo and the residue was purified by chromatography on Al_2O_3 (4% H_2O) with CH_2Cl_2/hexane. Recrystallization from dichloromethane afforded **5a** as colorless crystals, 745 mg (82%). ^{1}H NMR ($CDCl_3$, 250 MHz): δ 7.68–7.26 (m, 4H, Ar*H*); 6.08 (ddt, 1H, C*H*=CH_2); 5.44 (dt, 1H, *trans*-CH*H*); 5.32 (dt, 1H, *cis*-CH=C*H*H); 5.18 (dt, 2H, NCH_2) ppm. ^{13}C NMR (CD_2Cl_2, 62.90 MHz): δ 233.8 (N*C*O); 221.1 (*trans*-*C*O); 216.5 (*cis*-*C*O); 152.9, 131.4 (Ar*C*); 130.3 (H*C*=CH_2); 125.0, 124.8 (Ar*C*); 119.7 (HC=*C*H_2); 111.0, 110.9 (Ar*C*); 50.1 (N*C*H_2) ppm. *Anal.* Calc. for $C_{15}H_9CrNO_6$ ($M =$ 351.24): C, 351.29; H, 2.58; N, 3.99. Found: C, 50.91; H, 2.80; N, 4.08%. IR (KBr): ν 2063, 1978, 1926, 1892 (CO) cm^{-1}. MS (EI, 70 eV): m/z (%) 351 (11) [M^+].

3.6. (N-Allyl-2,3-dihydrobenzoxazol-2-ylidene) pentacarbonyltungsten(0) (5b)

Complex **5b** was prepared analogously to **5a** from a mixture of **3b**/**4b** (368 mg, 0.8 mmol), KO^{t}Bu (104 mg, 0.9 mmol) and allyl bromide (112 mg, 0.9 mmol) in 8 ml of DMF. Recrystallization from dichloromethane afforded **5b** as yellowish crystals, 205 mg (53%). ^{1}H NMR ($CDCl_3$, 250 MHz): δ 7.67–7.28 (m, 4H, Ar*H*); 6.02 (ddt, 1H, C*H*=CH_2); 5.40 (dt, 1H, *trans*-CH*H*); 5.28 (dt, 1H, *cis*-CH=C*H*H); 5.12 (dt, 2H, NCH_2) ppm. ^{13}C NMR (CD_2Cl_2, 62.90 MHz): δ 215.5 (N*C*O); 201.8 (*trans*-*C*O); 197.3 (*cis*-*C*O, $^1J(^{183}W-^{13}C) = 125$ Hz); 153.3, 131.1 (Ar*C*); 130.7 (H*C*=CH_2); 125.8, 125.6 (Ar*C*); 119.8 (HC=*C*H_2); 111.9, 111.6 (Ar*C*); 51.7 (N*C*H_2) ppm. *Anal.* Calc. for $C_{15}H_9NO_6W$ ($M =$ 483.09): C, 37.29; H, 1.88; N, 2.90. Found: C, 37.35; H, 2.24; N, 3.14%. IR (KBr): ν 2069, 1974, 1925, 1896 (CO) cm^{-1}. MS (EI, 70 eV): m/z (%) 483 (51) [M^+].

3.7. X-ray crystal structure determination of 5b

Crystals of **5b** were grown from a CH_2Cl_2 solution at −10°C. Selected crystallographic details: size of data crystal 0.34 × 0.25 × 0.20 mm, formula $C_{15}H_9NO_6W$, $M = 483.09$ amu, monoclinic, space group $P2_1/c$, $a =$ 7.230(2), $b = 15.815(3)$, $c = 13.703(2)$ Å, $\beta = 95.49(3)°$, $V = 1559.6(10)$ Å^3, $Z = 4$, $\rho_{exp} = 2.02$, $D_{calc} = 2.057$ g cm^{-3}, Mo Kα radiation ($\lambda = 0.71073$ Å, graphite monochromator), μ(Mo Kα) = 75.93 cm^{-1}. In total 4541 symmetry independent diffraction data were measured at 20(2)°C in the 2θ range of 2–60°. Structure solution with Patterson and refinement with Fourier methods, refinement of positional parameters of all non-hydrogen atoms with anisotropic thermal parameters. Hydrogen atoms on calculated positions (d(C–H) = 0.95 Å, [11]) with $B_{eq(H)} = 1.3B_{eq(C)}$. $R = 0.026$, $R_w = 0.032$ for 3135 absorption corrected (five Ψ-scans) structure factors $F_o^2 \geq 3\sigma(F_o^2)$ and 208 refined parameters. Neutral atomic scattering factors were used and all scattering factors were corrected for anomalous dispersion [12]. All calculations were carried out with the MOLEN program package [13].

4. Supplementary material

Further details of the crystal structure investigation may be obtained from the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen, Germany on quoting the depository number CSD-59458 and the journal citation.

Acknowledgements

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