

References

- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- DEDERT, P. L., THOMPSON, J. S., IBERS, J. A. & MARKS, T. J. (1982). *Inorg. Chem.* **21**, 969–977.
- FAWCETT, T. G., RUDICH, S. M., TOBY, B. H., LALANCETTE, R. A., POTENZA, J. A. & SCHUGAR, H. J. (1980). *Inorg. Chem.* **19**, 940–945.
- JOHNSON, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- KEENE, F. R., SNOW, M. R., STEPHENSON, P. J. & TIEKINK, E. R. T. (1988). *Inorg. Chem.* **27**, 2040–2045.
- KUCHARSKI, E. S., MCWHINNIE, W. R. & WHITE, A. H. (1978). *Aus. J. Chem.* **31**, 53–56; 2647–2650.
- KULASINGAM, G. C. & MCWHINNIE, W. R. (1967). *J. Chem. Soc. A*, pp. 1253–1255.
- LEWIS, D. L. & HODGSON, D. J. (1974). *Inorg. Chem.* **13**, 143–147.
- MCWHINNIE, W. R., KULASINGAM, G. C. & DRAPER, J. C. (1966). *J. Chem. Soc. A*, pp. 1199–1203.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. of York, England, and Louvain, Belgium.
- POTENZA, M. N., POTENZA, J. A. & SCHUGAR, H. J. (1988). *Acta Cryst.* **C44**, 1201–1204.
- STEWART, J. M., MACHIN, P. A., DICKINSON, C. W., AMMON, H. L., HECK, H. & FLACK, H. (1976). The XRAY76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- TASKER, P. A. & SKLAR, L. (1975). *J. Cryst. Mol. Struct.* **5**, 329–344.
- WIBAUT, J. P. & LA BASTIDE, G. L. C. (1933). *Recl Trav. Chim. Pays Bas*, **52**, 493–498.

Acta Cryst. (1992). **C48**, 1120–1121

Structure of (Benzo[c]cinnoline-*N'*)pentacarbonyltungsten

BY ERIC K. PHAM AND LISA McELWEE-WHITE

Department of Chemistry, Stanford University, Stanford, CA 94305, USA

(Received 8 April 1991; accepted 8 October 1991)

Abstract. [W(C₁₂H₈N₂)(CO)₅], *M_r* = 504.11, triclinic, *P*1̄, *a* = 7.152 (3), *b* = 10.124 (4), *c* = 12.170 (6) Å, α = 92.91 (4), β = 99.49 (3), γ = 107.62 (3)°, *V* = 823.7 (6) Å³, *Z* = 2, *D_x* = 2.03 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 71.9 cm⁻¹, *F*(000) = 476, *T* = 298 K, *R* = 0.043, *wR* = 0.049 for 3060 reflections with *I* > 3σ(*I*). The compound is a mononuclear complex of benzo[c]cinnoline involving the coordination of the W metal to the lone pair of one N atom (N1). The W⋯N2 separation is 3.01 Å. The N1–N2 distance is 1.31 (1) Å, intermediate between that of a double bond (1.25 Å) and a single bond (1.40 Å). The benzo[c]cinnoline ligand is essentially planar with a 1° twist angle between the phenyl rings.

Experimental. The title compound was obtained as an unexpected product from the reaction of the Fischer carbene complex (CO)₅W=C(OMe)(Me) with benzo[c]cinnoline which can be viewed as a rigidly fixed form of *cis*-azobenzene. Red prisms of the complex, recrystallized from a concentrated solution of CH₂Cl₂ at 253 K, had NMR data identical to an authentic sample prepared independently *via* a previously published route (Kooti & Nixon, 1976). A single crystal of dimensions 0.15 × 0.31 × 0.25 mm was mounted on a glass fiber and coated with epoxy cement. Data collection was performed on a Nicolet–

Siemens *P*2₁ diffractometer (Mo *K*α radiation, graphite monochromator). From the least-squares fit of the angular settings of 25 reflections (20 < 2θ < 25°) final unit-cell parameters were obtained. The intensities were collected at 298 K using the θ–2θ scan technique with a variable rate of 1 to 15° min⁻¹ in the range 3 ≤ 2θ ≤ 55°. The index ranges were 10 ≤ *h* ≤ 0, –19 ≤ *k* ≤ 19, –21 ≤ *l* ≤ 21. The three standard reflections measured every 97 reflections showed only 0.5% loss in intensity during the total exposure time of 255 h. Of 4095 reflections collected,

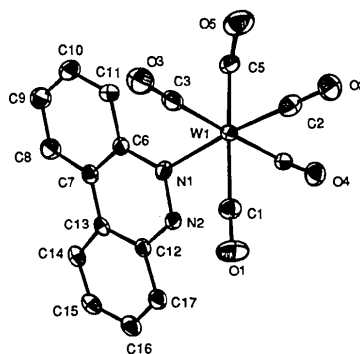


Fig. 1. ORTEP (Johnson, 1965) drawing of the title compound with the atom-numbering scheme. H atoms omitted. Ellipsoids are drawn at the 50% probability level.

Table 1. Atomic coordinates and final equivalent isotropic thermal parameters (\AA^2) with e.s.d.'s in parentheses for $[\text{W}(\text{C}_{12}\text{H}_8\text{N}_2)(\text{CO})_5]$

$$B_{\text{eq}} = (1/3) \sum_i \sum_j B_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B_{eq}
W1	0.01474 (6)	0.07529 (4)	0.22384 (3)	2.820 (6)
C1	0.155 (1)	0.004 (1)	0.3577 (8)	3.8 (2)
O1	0.227 (1)	-0.0385 (8)	0.4299 (7)	6.1 (2)
C2	-0.090 (1)	-0.116 (1)	0.1493 (8)	4.0 (2)
O2	-0.158 (1)	-0.2281 (7)	0.1032 (7)	5.9 (2)
C3	0.258 (1)	0.102 (1)	0.1505 (8)	3.8 (2)
O3	0.391 (1)	0.113 (1)	0.1080 (7)	6.2 (2)
C4	-0.234 (1)	0.0384 (9)	0.2929 (7)	3.3 (2)
O4	-0.378 (1)	0.0113 (8)	0.3266 (6)	5.3 (2)
C5	-0.137 (1)	0.146 (1)	0.0942 (8)	4.0 (2)
O5	0.225 (1)	-0.1806 (9)	-0.0217 (6)	5.9 (2)
C6	0.212 (1)	0.4207 (9)	0.2877 (7)	3.0 (2)
C7	0.273 (1)	0.5444 (9)	0.3630 (8)	3.3 (2)
C8	0.336 (2)	0.674 (1)	0.3195 (9)	4.3 (2)
C9	0.340 (2)	0.679 (1)	0.208 (1)	5.2 (3)
C10	0.283 (2)	0.558 (1)	0.1353 (9)	5.4 (3)
C11	0.221 (2)	0.431 (1)	0.1735 (8)	4.3 (2)
C12	0.201 (1)	0.3946 (9)	0.5080 (7)	3.0 (2)
C13	0.267 (1)	0.5314 (8)	0.4790 (8)	2.9 (2)
C14	0.317 (1)	0.644 (1)	0.5648 (9)	3.9 (2)
C15	0.305 (1)	0.616 (1)	0.6733 (9)	4.2 (2)
C16	0.242 (1)	0.480 (1)	0.7008 (8)	4.3 (2)
C17	0.192 (1)	0.370 (1)	0.6212 (8)	3.8 (2)
N1	0.142 (1)	0.2897 (7)	0.3254 (6)	3.1 (2)
N2	0.142 (1)	0.2781 (7)	0.4322 (6)	3.1 (2)

3485 were unique, and 3060 reflections had $I > 3\sigma(I)$. Lorentz and polarization corrections were applied. Owing to the relatively small size of the crystal no empirical absorption correction was applied. The structure was solved by Patterson methods and refined *via* standard full-matrix least squares (on F) and Fourier techniques with a Digital Equipment MicroVAX computer using locally modified Enraf-Nonius *SDP* software (Frenz, 1985). Function minimized was $\sum w(|F_o| - |F_c|)^2$, $w = 1/\sigma^2(F_o)$, $\sigma(F_o^2) = [\sigma_o^2(F_o^2) + p(F_o^2)^2]^{1/2}$; $p = 0.02$. All non-H atoms were refined with anisotropic thermal parameters. H atoms were placed according to their ideal molecular geometries (C—H = 0.95 \AA) and included in the structure-factor calculations with their positions and isotropic temperature factors fixed. The refinement of 258 parameters converged with $R = 0.043$ and $wR = 0.049$; the maximum shift/e.s.d. in the final refinement cycle was 0.01 while the final difference Fourier map at this stage was featureless; the maximum and minimum residual electron densities were 0.795 and $-0.645 \text{ e \AA}^{-3}$, respectively. Atomic scattering factors and f' , f'' were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 72–151).

Table 2. Selected interatomic distances (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses for $[\text{W}(\text{C}_{12}\text{H}_8\text{N}_2)(\text{CO})_5]$

W1—C1	2.05 (1)	C—O (av.)	1.14 (1)
W1—C2	1.959 (9)	C—C (Ph, av.)	1.39 (1)
W1—C3	2.03 (1)	C6—N1	1.40 (1)
W1—C4	2.03 (1)	C7—C13	1.43 (1)
W1—C5	2.05 (1)	C12—N2	1.37 (1)
W1—N1	2.287 (6)	N1—N2	1.31 (1)
C1—W1—C2	89.0 (4)	W1—C—O (av.)	177.6 (8)
C1—W1—C3	89.8 (4)	C—C—C (Ph, av.)	120.0 (9)
C1—W1—C4	90.3 (4)	C7—C6—N1	120.9 (8)
C1—W1—C5	177.2 (4)	C11—C6—N1	119.9 (8)
C1—W1—N1	86.2 (3)	C6—C7—C13	118.3 (8)
C2—W1—C3	88.0 (4)	C8—C7—C13	123.4 (8)
C2—W1—C4	89.0 (4)	C13—C12—N2	123.7 (9)
C2—W1—C5	91.2 (4)	C17—C12—N2	115.7 (8)
C2—W1—N1	175.0 (3)	C7—C13—C12	115.9 (7)
C3—W1—C4	177.0 (4)	C7—C13—C14	125.7 (8)
C3—W1—C5	93.0 (4)	W1—N1—C6	128.9 (6)
C3—W1—N1	93.5 (3)	W1—N1—N2	110.5 (5)
C4—W1—C5	86.9 (4)	C6—N1—N2	120.5 (6)
C4—W1—N1	89.4 (3)	C12—N2—N1	120.5 (7)
C5—W1—N1	93.5 (3)		

A drawing of the title compound with the atom-numbering scheme is shown in Fig. 1. Atomic coordinates and final equivalent isotropic thermal parameters of all non-H atoms included in the refinement are given in Table 1.* Interatomic distances and angles are listed in Table 2.

Related literature. The structure of benzo[*c*]cinnoline-bis(tricarbonyliron), $[\text{Fe}_2(\text{C}_{12}\text{H}_8\text{N}_2)(\text{CO})_6]$ (Doedens, 1970) has been reported with an N—N bond length of 1.399 (8) \AA . The corresponding N—N length in the title compound is 1.31 (1) \AA , intermediate between that of a single bond and a double bond (Power & Destana, 1991).

* Lists of structure factors, anisotropic thermal parameters, least-squares-planes data, complete bond distances and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54722 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- DOEDENS, R. J. (1970). *Inorg. Chem.* **9**, 429–436.
 FRENZ, B. A. (1985). *Enraf-Nonius Structure Determination Package*. College Station, Texas, USA.
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
 KOOTI, M. & NIXON, J. F. (1976). *J. Organomet. Chem.* **105**, 217–230.
 POWER, P. & PESTANA, D. (1991). *Inorg. Chem.* **30**, 528–535.