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## Structure Reports

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**(Acetonitrile- $\kappa$ N)pentacarbonyl-tungsten(0)****Christoph E. Strasser,\* Stephanie Cronje and Helgard G. Raubenheimer**

Department of Chemistry and Polymer Science, University of Stellenbosch, Private Bag X1, Matieland 7602, South Africa

Correspondence e-mail: ce.strasser@gmx.net

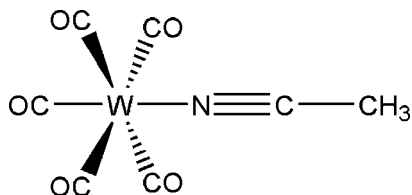
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å;  $R$  factor = 0.022;  $wR$  factor = 0.055; data-to-parameter ratio = 15.4.

The acetonitrile ligand in the title compound,  $[\text{W}(\text{CH}_3\text{CN})(\text{CO})_5]$ , is coordinated end-on to a pentacarbonyltungsten(0) fragment with a  $\text{W}-\text{N}$  bond length of 2.186 (4) Å, completing an octahedral coordination environment around the  $\text{W}$  atom.

**Related literature**

For other structures with an (alkyl nitrile- $\kappa$ N)pentacarbonyl-tungsten(0) fragment, see: Darensbourg *et al.* (1992); Reibenspies *et al.* (1994); Jefford *et al.* (1996). For structures with conjugated nitriles, see: Fischer *et al.* (1993); Helten *et al.* (2010) and for structures with nitriles that are part of an organometallic complex, see: Busetto *et al.* (1992); Duclos *et al.* (1999); Tang *et al.* (1999); Trylus *et al.* (1999); Cordiner *et al.* (2006). For the preparation, see: Strasser *et al.* (2010).

**Experimental***Crystal data*

$[\text{W}(\text{C}_2\text{H}_3\text{N})(\text{CO})_5]$   
 $M_r = 364.95$   
 Monoclinic,  $P2_1/n$   
 $a = 5.6485$  (6) Å

$b = 13.6231$  (15) Å  
 $c = 12.8642$  (15) Å  
 $\beta = 101.883$  (2)°  
 $V = 968.69$  (19) Å<sup>3</sup>

$Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 11.92$  mm<sup>-1</sup>

$T = 100$  K  
 $0.17 \times 0.07 \times 0.05$  mm

*Data collection*

Bruker APEX CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2002)  
 $T_{\min} = 0.337$ ,  $T_{\max} = 0.549$

5297 measured reflections  
 1973 independent reflections  
 1694 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.021$

*Refinement*

$R[F^2 > 2\sigma(F^2)] = 0.022$   
 $wR(F^2) = 0.055$   
 $S = 1.08$   
 1973 reflections

128 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 1.69$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -1.08$  e Å<sup>-3</sup>

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2003); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: X-SEED (Atwood & Barbour, 2003; Barbour, 2001); software used to prepare material for publication: X-SEED.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2110).

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## supporting information

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**(Acetonitrile- $\kappa N$ )pentacarbonyltungsten(0)**

**Christoph E. Strasser, Stephanie Cronje and Helgard G. Raubenheimer**

**S1. Comment**

The title compound is an important starting material in the synthesis of complexes containing the pentacarbonyltungsten(0) fragment. The nitrile ligand is coordinated end-on to  $W(CO)_5$ , completing an octahedral coordination geometry at the tungsten atom.

The *trans*-influence of the acetonitrile ligand is echoed by the comparatively short  $W1-C11$  bond [1.975 (5) Å] compared to the *cis*-carbonyls [ $W-C$  bond lengths 2.023 (5) Å to 2.056 (5) Å].

When the present molecular structure of  $[W(CH_3CN)(CO)_5]$  is compared to other literature examples of (nitrile- $\kappa N$ )pentacarbonyltungsten(0) compounds, surprisingly no significant variations in bond lengths can be observed within the metal–ligand bonds despite different electronic and steric properties of the nitriles used. Related crystal structures reported include the  $W(CO)_5$  adducts of alkylnitriles (Reibenspies *et al.*, 1994; Jefford *et al.*, 1996), conjugated nitriles (Fischer *et al.*, 1993; Tang *et al.*, 1999; Cordiner *et al.*, 2006; Helten *et al.*, 2010),  $\alpha$ -metal-substituted alkyl nitriles (Busetto *et al.*, 1992; Trylus *et al.*, 1999) as well as deprotonated 2,3,4-tricyanopent-2-enedinitrile (Duclos *et al.*, 1999).

**S2. Experimental**

The compound was obtained as a side product after chromatography of a mixture obtained by treating pentacarbonyl[(4-methyl-1,3-thiazol-2-yl)carbonyl]tungsten(0) with bis(trichloromethyl)carbonate (Strasser *et al.*, 2010). Decomposition of the initial product followed by reaction of the pentacarbonyltungsten fragment with acetonitrile in the mobile phase resulted in formation of the title compound. Crystals of the title compound were formed by layering a dichloromethane solution with hexane.

**S3. Refinement**

All H atoms were positioned geometrically ( $C-H = 0.98$  Å) and constrained to ride on their parent atoms; the  $U_{iso}(H)$  values were set at 1.5 times  $U_{eq}(C)$ .

The maximum residual electron density of  $1.69 e \text{ \AA}^{-3}$  is located  $0.85$  Å next to  $W1$ .

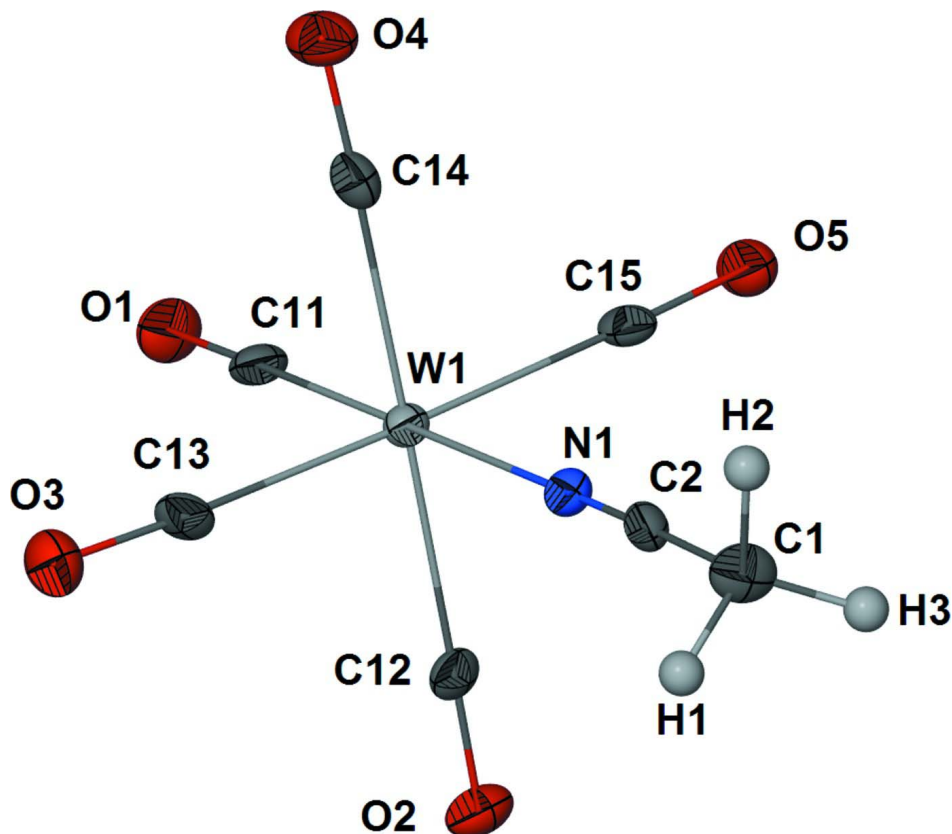


Figure 1

The asymmetric unit of the title compound, ellipsoids are drawn at the 50% probability level.

**(Acetonitrile- $\kappa$ N)pentacarbonyltungsten(0)**

*Crystal data*

[W(C<sub>2</sub>H<sub>3</sub>N)(CO)<sub>5</sub>]

$M_r = 364.95$

Monoclinic,  $P2_1/n$

Hall symbol: -P 2yn

$a = 5.6485$  (6) Å

$b = 13.6231$  (15) Å

$c = 12.8642$  (15) Å

$\beta = 101.883$  (2)°

$V = 968.69$  (19) Å<sup>3</sup>

$Z = 4$

$F(000) = 664$

$D_x = 2.502$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 3152 reflections

$\theta = 2.2$ – $26.4$ °

$\mu = 11.92$  mm<sup>-1</sup>

$T = 100$  K

Prism, light yellow

$0.17 \times 0.07 \times 0.05$  mm

*Data collection*

Bruker APEX CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2002)

$T_{\min} = 0.337$ ,  $T_{\max} = 0.549$

5297 measured reflections

1973 independent reflections

1694 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 26.4$ °,  $\theta_{\min} = 2.2$ °

$h = -7 \rightarrow 7$

$k = -17 \rightarrow 16$

$l = -16 \rightarrow 10$

Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.022$	H-atom parameters constrained
$wR(F^2) = 0.055$	$w = 1/[\sigma^2(F_o^2) + (0.0282P)^2 + 0.8554P]$
$S = 1.08$	where $P = (F_o^2 + 2F_c^2)/3$
1973 reflections	$(\Delta/\sigma)_{\max} = 0.002$
128 parameters	$\Delta\rho_{\max} = 1.69 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -1.08 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > 2\sigma(F^2)$  is used only for calculating  $R$ -factors(gt) etc. and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
W1	0.12706 (3)	0.623964 (12)	0.793281 (13)	0.01429 (8)
O1	0.3950 (6)	0.4656 (3)	0.6833 (3)	0.0283 (8)
O2	-0.1535 (6)	0.4537 (2)	0.8890 (3)	0.0258 (8)
O3	-0.2754 (7)	0.6294 (2)	0.5820 (3)	0.0296 (8)
O4	0.4077 (6)	0.7892 (3)	0.6922 (3)	0.0304 (9)
O5	0.5558 (7)	0.6182 (2)	0.9985 (3)	0.0263 (8)
N1	-0.0645 (7)	0.7352 (3)	0.8667 (3)	0.0172 (8)
C1	-0.3065 (9)	0.8698 (3)	0.9416 (4)	0.0247 (11)
H1	-0.4783	0.8629	0.9087	0.037*
H2	-0.2493	0.9347	0.9252	0.037*
H3	-0.2848	0.8623	1.0188	0.037*
C2	-0.1690 (8)	0.7948 (3)	0.9004 (4)	0.0171 (9)
C11	0.2982 (8)	0.5241 (3)	0.7252 (4)	0.0199 (10)
C12	-0.0548 (8)	0.5153 (3)	0.8553 (4)	0.0183 (9)
C13	-0.1353 (8)	0.6289 (3)	0.6600 (4)	0.0202 (10)
C14	0.3031 (9)	0.7309 (3)	0.7303 (4)	0.0205 (10)
C15	0.4008 (8)	0.6203 (3)	0.9262 (4)	0.0191 (10)

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
W1	0.01444 (11)	0.01441 (11)	0.01459 (11)	-0.00045 (6)	0.00428 (8)	-0.00013 (7)
O1	0.029 (2)	0.0272 (19)	0.032 (2)	0.0021 (15)	0.0142 (17)	-0.0088 (16)
O2	0.0288 (19)	0.0226 (17)	0.030 (2)	-0.0014 (14)	0.0161 (17)	0.0021 (15)
O3	0.0275 (19)	0.036 (2)	0.0238 (19)	-0.0018 (16)	0.0005 (17)	-0.0008 (16)

O4	0.0265 (19)	0.033 (2)	0.031 (2)	-0.0066 (16)	0.0033 (17)	0.0104 (16)
O5	0.0228 (18)	0.032 (2)	0.0234 (19)	-0.0028 (14)	0.0031 (16)	0.0037 (15)
N1	0.0175 (19)	0.0197 (19)	0.0147 (19)	-0.0014 (15)	0.0040 (16)	0.0010 (16)
C1	0.022 (2)	0.022 (2)	0.033 (3)	0.0035 (19)	0.011 (2)	-0.004 (2)
C2	0.018 (2)	0.018 (2)	0.014 (2)	-0.0032 (18)	0.0008 (19)	-0.0012 (18)
C11	0.017 (2)	0.022 (2)	0.024 (3)	-0.0038 (19)	0.011 (2)	-0.001 (2)
C12	0.020 (2)	0.019 (2)	0.017 (2)	0.0008 (18)	0.0091 (19)	-0.0029 (19)
C13	0.010 (2)	0.025 (2)	0.025 (3)	0.0002 (18)	0.0033 (19)	-0.002 (2)
C14	0.018 (2)	0.022 (2)	0.019 (2)	0.000 (2)	-0.001 (2)	-0.002 (2)
C15	0.018 (2)	0.019 (2)	0.022 (2)	-0.0039 (18)	0.007 (2)	0.0045 (19)

*Geometric parameters (Å, °)*

W1—N1	2.186 (4)	C1—H1	0.9800
W1—C11	1.975 (5)	C1—H2	0.9800
W1—C12	2.054 (5)	C1—H3	0.9800
W1—C13	2.023 (5)	C11—O1	1.160 (6)
W1—C14	2.024 (5)	C12—O2	1.141 (6)
W1—C15	2.056 (5)	C13—O3	1.143 (6)
N1—C2	1.140 (6)	C14—O4	1.157 (6)
C1—C2	1.448 (6)	C15—O5	1.139 (6)
O1—C11—W1	178.6 (5)	C11—W1—C14	89.6 (2)
O2—C12—W1	178.8 (4)	C11—W1—C15	90.0 (2)
O3—C13—W1	176.5 (4)	C12—W1—C15	90.58 (18)
O4—C14—W1	177.3 (5)	C13—W1—C12	90.86 (19)
O5—C15—W1	178.6 (4)	C13—W1—C14	88.38 (19)
N1—C2—C1	178.7 (5)	C13—W1—C15	178.36 (16)
C2—N1—W1	176.9 (4)	C14—W1—C12	179.23 (19)
C11—W1—N1	179.26 (18)	C14—W1—C15	90.18 (18)
C12—W1—N1	90.02 (16)	C2—C1—H1	109.5
C13—W1—N1	90.09 (16)	C2—C1—H2	109.5
C14—W1—N1	90.09 (18)	C2—C1—H3	109.5
C15—W1—N1	90.70 (16)	H1—C1—H2	109.5
C11—W1—C13	89.2 (2)	H1—C1—H3	109.5
C11—W1—C12	90.32 (19)	H2—C1—H3	109.5