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Bis(η^5-cyclopentadienyl)bis(thiocyanato-κN)-vanadium(IV)

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.029
wR factor = 0.064
Data-to-parameter ratio = 18.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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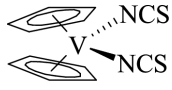
In the crystal structure of the title vanadocene complex, [V(η^5-$C_5H_5)_2(NCS)_2$], the V atom has distorted tetrahedral coordination with two η^5-bonded cyclopentadienyl rings and two N-bonded thiocyanate ligands.

Comment

In the title compound, (I) (Fig. 1), the molecule has the typical bent metallocene structure, in which two η^5-bonded cyclopentadienyl rings and two N atoms of thiocyanate ligands occupy pseudo-tetrahedral coordination sites around the V^{IV} atom [$Cg1—V—Cg2$ = 133.86 (4)° and N—V—N = 86.06 (7)°; $Cg1$ and $Cg2$ are the centroids of the cyclopentadienyl rings C1–C5 and C6–C10, respectively]. The cyclopentadienyl rings have a staggered conformation. The $Cg1—V$ and $Cg2—V$ bond lengths are 1.9614 (9) and 1.9647 (9) Å, respectively. The V—N distances [2.0381 (15) and 2.0358 (15) Å] are comparable to corresponding distances in [V(η^5-$C_5H_4CH_3)_2(NCO)_2$] [2.034 (2) and 2.036 (2) Å; Honzíček *et al.*, 2004] and are shorter than those in both [V(η^5-$C_5H_5)_2$(bpy)](OTf)$_2$ [bpy is 2,2′-bipyridine and OTf is trifluoromethanesulfonate; 2.128 (2) and 2.129 (2) Å; Ghosh *et al.*, 1999] and [V(η^5-$C_5H_5)_2$(phen)](OTf)$_2$ [phen is 1,10-phenanthroline; 2.1344 (18) and 2.1386 (19) Å; Ghosh *et al.*, 1999]. In the title compound, the coordinated thiocyanate groups are almost linear: N1—C11—S1 = 179.79 (18)° and N2—C12—S2 = 179.9 (18)°.



(I)

Experimental

The title compound was prepared according to the literature procedure of Doyle & Tobias (1968) with some modifications. [V(η^5-$C_5H_5)_2Cl_2$] (0.5 g, 2.0 mmol) was dissolved in water (20 ml). To this solution, potassium thiocyanate (0.48 g, 5.0 mmol) was added with vigorous stirring. The bright green precipitate which formed was filtered off, washed twice with water and vacuum dried (yield: 0.54 g, 91%). Dark green crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetone solution. EPR (acetone solution): g_{iso} = 1.9844, A_{iso} = 68.48 × 10^{-4} cm^{-1}; IR (Nujol mull, cm^{-1}): 3092 (*m*, ν C—H, Cp), 2089 (*vs*, ν_aC≡N), 2069 (*vs*, ν_sC≡N), 1449 (*s*, ν_aC—C, Cp), 1435 (*s*, ν_aC—C, Cp), 1378 (*m*, ν_aC—C, Cp), 1181 (*m*), 1142 (*m*), 1129 (*m*), 1075 (*m*, δ C—H, Cp), 1026 (*m*, δ C—H, Cp), 1010 (*m*), 959 (*m*) 885 (m) 843 (*vs*, γ C—H, Cp), 416 (*m*); Raman (quartz capillary, cm^{-1}): 3109 (*m*, ν C—H, Cp), 3093 (*sh*, ν C—H, Cp), 2083 (*vs*, ν_aC≡N), 2069 (*vs*, ν_sC≡N), 1128 (*vs*, ν_sC—C, Cp)(*vs*), 831 (*m*, γ C—H, Cp), 436 (*m*), 411 (*s*), 384 (*s*), 287 (*vs*, κ Cp); positive-ion MS (ESI): m/z 239 [Cp_2VNCS]$^+$ (100%); positive-ion MS/MS of 239:

m/*z* 199 $[Cp_2V + H_2O]^+$, 181 $[Cp_2V]^+$ (100%); negative-ion MS: *m*/*z* 629 $[2M + Cl]^-$, 332 $[M + Cl]^-$ (100%); negative-ion MS/MS of 332: *m*/*z* 267 $[M + Cl - Cp]^-$ (100%).

Crystal data

$[V(C_5H_5)_2(NCS)_2]$	D_x = 1.605 Mg m^{-3}
M_r = 297.28	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 2982 reflections
a = 9.6500 (3) Å	θ = 1–27.5°
b = 9.6740 (2) Å	μ = 1.12 mm^{-1}
c = 13.6410 (4) Å	T = 150 (2) K
β = 104.9680 (14)°	Block, green
V = 1230.24 (6) Å^3	0.20 × 0.20 × 0.15 mm
Z = 4	

Data collection

Nonius KappaCCD area-detector diffractometer	2480 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.035
Absorption correction: none	θ_{max} = 27.5°
19398 measured reflections	$h = -12 \rightarrow 12$
2827 independent reflections	$k = -12 \rightarrow 12$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0188P)^2 + 0.9617P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.029	
$wR(F^2)$ = 0.064	
S = 1.08	$(\Delta/\sigma)_{max}$ = 0.001
2827 reflections	$\Delta\rho_{max}$ = 0.33 e Å^{-3}
154 parameters	$\Delta\rho_{min}$ = −0.28 e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å,°)..

Cg_1—V1	1.9614(9)	N1—C11	1.163(2)
Cg_2—V1	1.9647(9)	N2—C12	1.161(2)
V1—N1	2.0381 (15)	C11—S1	1.6241(19)
V1—N2	2.0358 (15)	C12—S2	1.6295(18)
Cg_1—V1—Cg_2	133.86(4)	N1—V1—N2	86.06(6)
V1—N1—C11	176.43(15)	V1—N2—C12	177.99(15)
N1—C11—S1	179.79(18)	N2—C12—S2	179.9(2)

Notes: Cg_1 is the centroid of ring C1–C5 and Cg_2 is the centroid of ring C6–C10.

Data collection: *COLLECT* (Hooft, 1998) and *DENZO* (Otwinowski & Minor, 1997); cell refinement: *COLLECT* and *DENZO*; data reduction: *COLLECT* and *DENZO*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97.

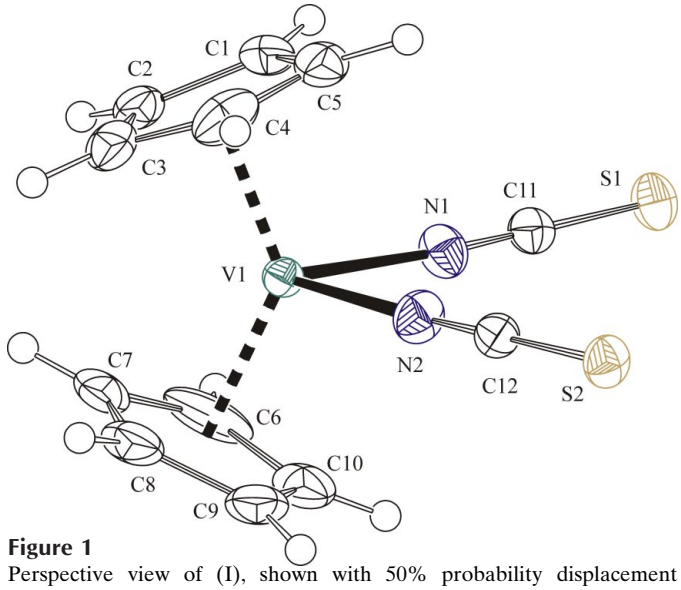


Figure 1
Perspective view of (I), shown with 50% probability displacement ellipsoids.

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