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Key indicators

Single-crystal X-ray study
 $T = 173$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.032
 wR factor = 0.077
 Data-to-parameter ratio = 17.1

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

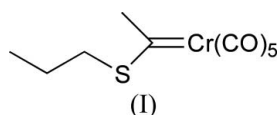
Pentacarbonyl[methyl(*n*-propylsulfanyl)-carbene]chromium(0)

The title compound, $[\text{Cr}(\text{C}_5\text{H}_{10}\text{S})(\text{CO})_5]$, comprises a methyl(*n*-propylsulfanyl)carbene ligand coordinated to a pentacarbonylchromium fragment, with the Cr atom in an octahedral coordination.

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Comment

In our ongoing study of the chemistry of Fischer-type carbene complexes, we have focused on the utilization of simple derivatives as organometallic synthons (Raubenheimer *et al.*, 2002, 2005). In a recent report (Raubenheimer *et al.*, 2006), we discussed the reaction of deprotonated thiocarbene complexes with the electrophile Ph_3PAu^+ to afford mercaptovinyl complexes of Au that are S-coordinated to the $M(\text{CO})_5$ ($M = \text{Cr}, \text{W}$) unit of the original Fischer carbene complex. Since structural information on the starting thiocarbene complexes is limited, the structure of the title compound, (I), is reported here.



The molecular structure of (I) (Fig. 1) comprises a methyl(*n*-propylsulfanyl)carbene ligand coordinated to a central Cr atom. Five carbonyl ligands complete the octahedral coordination around chromium. The thiocarbene is staggered with respect to the equatorial CO ligands, with S–C–Cr–C torsion angles of 39.70 (12), 129.69 (11), –140.28 (11) and –49.06 (11)°. The two S–C bond lengths (Table 1) differ significantly, suggesting that a certain amount of π -donation from the heteroatom to the carbene C atom occurs, thus shortening the C11–S11 bond. This is similar to the structure of pentacarbonyl[methyl(phenylthio)carbene]chromium (Hoare & Mills, 1972), where the S–C(carbene) and S–C(phenyl) distances are 1.690 (3) and 1.781 (3) Å, respectively.

Experimental

Compound (I) was prepared according to a somewhat modified version of a literature procedure (Aumann & Schroeder, 1990). Dropwise addition of 1-propanethiol (0.28 ml, 3.09 mmol) to a yellow methanolic solution (20 ml) of pentacarbonyl[methoxy(methyl)carbene]chromium(0) (0.76 g, 3.04 mmol) and Na_2CO_3 (0.32 g, 3.02 mmol), cooled to 268 K, afforded a dark-red solution. After 15 min at 273 K, extraction with a mixture of *n*-hexane/water (200/10 ml) and drying of the organic layer over Na_2SO_4 , followed by final

stripping of solvent, yielded a microcrystalline product. Recrystallization from *n*-hexane at 195 K provided dark-red needles (0.55 g, 62%; m.p. 316 K). IR [*n*-hexane, $\nu(\text{CO}) \text{ cm}^{-1}$]: 2059 (A_1), 1988 (B_1), 1957 (E); $^1\text{H NMR}$ (CD_3COCD_3): δ 3.71 (*s*, 3 H, CH_3), 3.40 (*t*, 2 H, $J = 7.5 \text{ Hz}$, SCH_2), 1.77 (*m*, 2 H, $J = 7.5 \text{ Hz}$, $\text{SCH}_2\text{-CH}_2$), 1.04 [*t*, 3 H, $J = 7.5 \text{ Hz}$, $\text{S}(\text{CH}_2)_2\text{-CH}_3$]; $^{13}\text{C NMR}$ (CD_3COCD_3): δ 369.8 (C_{carb}), 228.5 (CO_{trans}), 217.9 (CO_{cis}), 46.9 (CH_3), 45.5 (SCH_2), 21.3 ($\text{SCH}_2\text{-CH}_2$), 13.3 [$\text{S}(\text{CH}_2)_2\text{-CH}_3$]; EI-MS (m/z): 294 [M] $^+$, 238 [$M - 2\text{CO}$] $^+$, 182 [$M - 4\text{CO}$] $^+$, 154 [$M - 5\text{CO}$] $^+$, 112 [$M - \text{C}_3\text{H}_7$] $^+$.

Crystal data

$[\text{Cr}(\text{C}_5\text{H}_{10}\text{S})(\text{CO})_5]$	$V = 632.17 (12) \text{ \AA}^3$
$M_r = 294.24$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.546 \text{ Mg m}^{-3}$
$a = 6.1320 (7) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 8.4685 (9) \text{ \AA}$	$\mu = 1.07 \text{ mm}^{-1}$
$c = 12.6187 (14) \text{ \AA}$	$T = 173 (2) \text{ K}$
$\alpha = 77.264 (2)^\circ$	Needle, dark red
$\beta = 81.530 (2)^\circ$	$0.30 \times 0.10 \times 0.10 \text{ mm}$
$\gamma = 87.878 (2)^\circ$	

Data collection

Bruker APEX CCD area-detector diffractometer	6077 measured reflections
ω scans	2661 independent reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2001)	2434 reflections with $I > 2\sigma(I)$
$T_{\text{min}} = 0.764$, $T_{\text{max}} = 0.900$	$R_{\text{int}} = 0.017$
	$\theta_{\text{max}} = 26.8^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0352P)^2 + 0.4906P]$
$R[F^2 > 2\sigma(F^2)] = 0.032$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.077$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.42 \text{ e \AA}^{-3}$
2661 reflections	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
156 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (\AA , $^\circ$).

Cr1—C11	2.0442 (19)	S11—C13	1.8229 (19)
S11—C11	1.6802 (19)	C11—C12	1.499 (3)
C11—S11—C13	112.20 (9)	Cr1—C11—S11	117.27 (10)

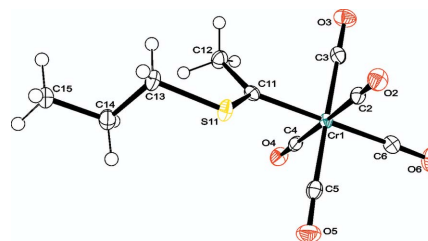


Figure 1

The molecular structure and atom-labelling scheme for (I), showing 50% probability displacement ellipsoids.

The H atoms were included in the riding-model approximation, with methylene C—H = 0.99 \AA and methyl C—H = 0.98 \AA , and with $U_{\text{iso}}(\text{H}) = 1.2$ and $1.5U_{\text{eq}}(\text{C})$ for methylene and methyl H atoms, respectively.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP* for Windows (Farrugia, 1997); software used to prepare material for publication: *X-SEED* (Barbour, 2001).

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