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Inorganica Chimica Acta 358 (2005) 1269–1273

Inorganica Chimica Acta

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Note

A facile reaction of bibenzyl trisulfide with $[(\eta^5\text{-}C_5H_5)Cr(CO)_3]_2$: formation of a thiolato-bridged dichromium complex of $[CpCr(CO)_2(SBz)]_2$ (Bz = $PhCH_2$–)

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Received 2 June 2004; accepted 28 September 2004
Available online 28 October 2004

Abstract

The reaction of $[CpCr(CO)_3]_2$ (Cp = η^5-C_5H_5) (**1**) with an equivalent of Bz_2S_3 at ambient temperature gave $[CpCr(CO)_2]_2S$ (**3**) [L.Y. Goh, T.W. Hambley, G.B. Robertson, Organometallics 6 (1987) 1051], novel complexes of $[CpCr(CO)_2(SBz)]_2$ (**4**) and together with $[CpCr(SBz)]_2S$ (**5**) as main products. Thermolytic studies showed that **4** underwent complete decarbonylation to give $[CpCr(SBz)]_2S$ (**5**). Final thermal decomposition of 3 and 5 eventually yielded $Cp_4Cr_4S_4$ (**6**) (Goh et al., 1987) after prolonged reaction at 100 °C. However, the reaction of $[CpCr(CO)_2]_2$ ($Cr{\equiv}Cr$) (**2**) with Bz_2S_3 was much slower at ambient temperature which required 72 h to complete eventually yielding **3** and **5**. All the products have been characterized by elemental and spectral analyses. **4** has been structurally determined.

Keywords: Dicyclopentadienyldichromiumhexacarbonyl; Bibenzyl trisulfide; Thermolytic studies; Decarbonylation; Thermal decomposition; Monoclinic

1. Introduction

In the last two decades, thiolate ligands have been highlighted as versatile substituents for many organometallic complexes [1]. There have been considerable interests in their biological and catalytic properties [2a–2f]. To date, a number of published works on the reactivity of transition-metal carbonyl complexes with organic sulfides, RS^- [3], disulfides, RSSR [4a–4e] and trisulfides, RSSSR [5a–5b] have been submitted.

Our work in this report describes the reactions of $[CpCr(CO)_3]_2$ (**1**) together with its decarbonylated triply bonded $[CpCr(CO)_2]_2$($Cr{\equiv}Cr$) (**2**) with Bz_2S_3 as part of a comparative investigation involving organic trisulfides, RSSSR.

2. Experimental

2.1. General procedures

All reactions were carried out using the conventional Schlenk techniques under an inert atmosphere of argon in Vacuum Atmosphere Dribox equipped with a Model HE 493 Dri-train.

^1H and ^{13}C NMR spectra were measured on a JEOL Lambda 400 MHz spectrometer, and chemical shifts referenced to residual C_6D_6. IR spectra in Nujol mulls were measured in the range of 4000–400 cm^{-1} by means of a Perkin–Elmer 2000 FTIR instrument. Elemental analyses were performed by the analytical unit of the Research School of Chemistry, Australian National University, except for Cr which was determined as CrO_4^{2-} [13].

Solvents were distilled from sodium/benzophenone prior to use. Silica gel (Merck Kieselgel 60, 35–70 mesh)

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doi:10.1016/j.ica.2004.09.057

and Celite (Fluka AG) were activated at 120 °C for one day before chromatographic use. Bibenzyltrisulfide was purchased from Sigma. $[CpCr(CO)_3]_2$ (**1**) was synthesized as described by Manning [14] from chromium hexacarbonyl (99% purity from Merck).

2.2. Reaction of $[CpCr(CO)_3]_2$ (1) with Bz_2S_3 at ambient temperature

To a deep green solution of $[CpCr(CO)_3]_2$ (**1**) (200 mg, 0.497 mmol) in toluene (15 mL) was added Bz_2S_3 (138 mg, 0.497 mmol). The dirty green mixture was allowed to stir at ambient temperature for 2 h. The resultant yellowish brown reaction mixture was concentrated to ca. 2 mL in vacuo and then loaded onto a silica gel column (1.5 × 10 cm) prepared in *n*-hexane. The following fractions were eluted:

(i) A yellowish brown fraction in *n*-hexane–toluene (2:1) (35 mL) which when concentrated to dryness yielded deep green crystalline solids of $[CpCr(CO)_2]_2S$ (**3**) (108 mg, 0.286 mmol, 57.4% yield).

(ii) A dark brown fraction in toluene (25 mL) which when concentrated to dryness yielded dark brown crystalline solids of $[CpCr(CO)_2(SBz)]_2$ (**4**) (98 mg, 0.166 mmol, 33.3% yield). ^1H NMR (benzene-d$_6$): δ 4.16 (s, Cp). ^{13}C NMR (benzene-d$_6$): δ 89.61 (s, Cp). IR: ν (CO) at 1986 s, 1954 m, 1901 vs cm^{-1} (nujol), or 1991 vs, 1968 vs, sh, 1918 vs (toluene); other bands, 1066 vw, 1025 vw, 840 w, 722 w cm^{-1} (nujol). MS: *m/z* 149 [CpCrS], 205 $[CpCr(CO)_2S]$, 378 $[CpCr(CO)_2]_2S$, 401 $[CpCr_4S_4]$, 237 $[CpCr(CO)_2S_2]$, 261 $CpCr(CO)_4S$, 346 $[CpCr(CO)_4Bz_2S]$, 468 $[Cp_4Cr_4]$, 596 $[Cp_4Cr_4S_4]$, 560 $[Cp_2Cr_2(CO)_4Bz_2S]$, 592 $[Cp_2Cr_2(CO)_4Bz_2S_2]$. *Anal.* Calc. for $C_{28}H_{24}Cr_2O_4S_2$: C, 56.15; H, 4.35; Cr, 17.25; O, 10.37; S, 10.59. Found: C, 56.76; H, 4.05; Cr, 17.57; S, 10.81; O, 10.81%.

(iii) A purple fraction in ether (10 mL) which when concentrated to dryness yielded dark purple crystalline solids of $[CpCrSBz]_2S$ (**5**) (11 mg, 0.021 mmol, 4.3% yield). ^1H NMR (benzene-d$_6$): δ 13.89 (br, Cp, $\nu_{1/2}$ = 57 Hz). ^{13}C NMR (benzene-d$_6$): δ 101.03 (s, Cp), δ 133.14, 135.60 (s, Cp). IR: ν at 1063 vw, 1015 vw, 840 w, 732 w cm^{-1} (nujol). MS: *m/z* 149 [CpCrS], 295 $[CpCr_2S]$, 360 $[Cp_2Cr_2S]$, 371$[Cp_2Cr_2Bz_2S]$, 401 $[CpCr_4S_4]$, 480 $[Cp_2Cr_2Bz_2S_2]$, 512 $[Cp_2Cr_2Bz_2S_3]$. *Anal.* Calc. for $C_{24}H_{24}Cr_2S_3$: C, 56.25; H, 4.68; Cr, 20.31; S, 18.75. Found: C, 56.57; H, 4.15; Cr, 19.59; S, 18.31%. A greenish blue fraction remained uneluted at the top of the column.

2.3. Reaction of $[CpCr(CO)_3]_2$ (1) with Bz_2S_3 at 60 °C

To a deep green solution of **1** (200 mg, 0.498 mmol) in toluene (20 mL) was added bibenzyl trisulfide (139 mg, 0.498 mmol) and was stirred for 5 h at 60 °C. The resultant dark brown reaction mixture was concentrated to ca. 2 mL and loaded onto a silica gel column (1.5 × 8 cm) prepared in *n*-hexane. Elution gave two fractions:

(i) A dirty green solution in *n*-hexane–toluene (1:1, 45 mL) which yielded dark green crystalline solids of $[CpCr(CO)_2]_2S$ (**3**) (152 mg, 0.402 mmol, 80.8% yield).

(ii) A deep purple solution in toluene–ether (3:1, 25 mL) which yielded dark purple crystalline solids of $[CpCr(SBz)]_2S$ (**5**) (35 mg, 0.068 mmol, 13.7% yield). A greenish blue layer remained uneluted at the top rim of the column.

2.4. Reaction of $[CpCr(CO)_2]_2$ (2) with Bz_2S_3

A deep green solution of **2** (200 mg, 0.578 mmol) in toluene (20 mL) containing bibenzyl trisulfide (161 mg, 0.578 mmol) was stirred for 72 h at ambient temperature. The resultant dirty brown reaction mixture was filtered through Celite, concentrated and loaded onto a silica gel column (1.5 × 10 cm) prepared in *n*-hexane. Two fractions were eluted: (i) a dirty green solution in *n*-hexane–toluene (1:1, 45 mL) which gave dark green crystalline solids of **3** (5 mg, 0.008 mmol, 5.1% yield) and (ii) a deep purple solution in toluene–ether (3:1, 35 mL) which gave dark purple crystalline solids of **5** (135 mg, 0.264 mmol, 15.8% yield).

2.5. Thermolysis of $[CpCr(CO)_2(SBz)]_2$ (4)

A stirred dark brown solution of **4** (100 mg, 0.169 mmol) in toluene (10 mL) was maintained at 80 °C for ca. 1 h. The resultant dirty brown solution was filtered through Celite (1 cm disk), concentrated (ca. 1 mL) and loaded onto a silica gel column (1.5 × 6 cm) prepared in *n*-hexane. Two fractions were eluted: (i) a dirty green fraction in *n*-hexane–toluene (2:1, 25 mL) which gave dark green crystalline solids of **3** (39 mg, 0.103 mmol, 36.0% yield) and (ii) a deep purple solution in ether (25 mL) which gave dark purple crystalline solids of **5** (40 mg, 0.078 mmol, 27.4% yield).

2.6. Thermolysis of $[CpCr(SBz)]_2S$ (5)

A stirred deep purple solution of **5** (100 mg, 0.195 mmol) in toluene (10 mL) was maintained at 100 °C for 20 h. The resultant dirty purple solution was filtered through Celite (1 cm disk), concentrated (ca. 1 mL) and loaded onto a silica gel column (1.5 × 6 cm) prepared in *n*-hexane–toluene (1:1). Two fractions were eluted: (i) a grayish green solution in toluene (20 mL) which gave dark green crystalline solids of $\mathrm{Cp_4Cr_4S_4}$ (**6**) (25 mg, 0.042 mmol, 22.0% yield) and (ii) a deep purple solution in toluene–ether (3:1, 25 mL) which gave dark purple crystalline solids of **5** (35 mg, 0.059 mmol, 35% unreacted). A greenish blue layer remained uneluted at the top rim of the column.

2.7. NMR tube reactions

2.7.1. Reaction of [CpCr(CO)$_2$]$_2$S (3) with Bz$_2$S$_3$ at 60 °C

A dirty green mixture of **3** (20 mg, 0.05 mmol) and $\mathrm{Bz_2S_3}$ (15 mg, 0.05 mmol) in benzene-d_6 (0.6 mL) in a 5 mm NMR tube was reacted at 60 °C under an oil bath, and its proton NMR spectra were monitored at regular intervals (30, 60, 120 min and 24 h). From the spectra, **3** (74.4%), **5** (1.1%) and **6** (2.5%) were found after 24 h.

2.7.2. Reaction of [CpCr(CO)$_2$(SBz)]$_2$ (4) with Bz$_2$S$_3$ at ambient temperature

A dark brown mixture of **4** (15 mg, 0.03 mmol) and $\mathrm{Bz_2S_3}$ (7 mg, 0.03 mmol) in benzene-d_6 (0.6 mL) in a 5 mm NMR tube was clamped to a mechanical shaker, and its proton NMR spectra were monitored at regular intervals. From the spectra, **3** (27.5%) and **4** (33.4%) were found after 26 h of reaction.

Table 1
Crystal data and structure refinement for $[\mathrm{CpCr(CO)_2(SBz)}]_2$

Identification code	**4**
Empirical formula	$\mathrm{C_{28}H_{24}Cr_2O_4S_2}$
Formula weight	592.59
Temperature (K)	293(2)
Wavelength (Å)	0.71073
Crystal system	monoclinic
Space group	P_1/c
Unit cell dimensions	
a (Å)	8.6155(17)
b (Å)	24.551(5)
c (Å)	12.975(3)
α (°)	90
β (°)	107.98(3)
γ (°)	90
Volume (Å^3)	2610.5(9)
Z	4
$D_{\mathrm{Calc.}}$ (g cm^{-3})	1.508
Absorption coefficient (mm^{-1})	1.025
$F(000)$	1216
Crystal size (mm)	$0.38 \times 0.20 \times 0.16$
Theta range for data collection	1.66–27.54°
Limiting indices	$-11 \leqslant h \leqslant 11$, $-28 \leqslant k \leqslant 31$, $-15 \leqslant l \leqslant 16$
Reflections collected/unique	18368/5994 ($R_{\mathrm{int}} = 0.0304$)
Completeness to θ = 27.54	99.6%
Refinement method	full-matrix least-squares on F^2
Data/restraints/parameters	5994/0/325
Goodness-of-fit on F^2	0.832
Final R indices ($I > 2\sigma(I)$)	$R_1 = 0.0373$, $wR_2 = 0.1052$
R indices (all data)	$R_1 = 0.0477$, $wR_2 = 0.1159$
Largest different peak and hole (e Å^{-3})	0.583 and −0.205

2.8. Crystal structure determinations

Diffraction quality single crystals of **4** obtained as dark red block were grown in toluene layered with n-hexane after 3 days at −28 °C. Details of crystal

Table 2
Bond lengths (Å) and angles (°) for **4**

Bond lengths	
Cr(1)–C(15)	1.827(2)
Cr(1)–C(16)	1.829(3)
Cr(1)–S(1)	2.4163(8)
Cr(1)–S(2)	2.4230(7)
Cr(2)–S(1)	2.4046(7)
Cr(2)–S(2)	2.4192(8)
Cr(1)···Cr(2)	3.7020(7)
Cr(2)–C(17)	1.826(2)
Cr(2)–C(18)	1.828(2)
S(2)–C(8)	1.843(2)
S(2)···S(1)	2.7250(8)
S(1)–C(1)	1.848(2)
O(1)–C(15)	1.152(3)
O(2)–C(16)	1.148(3)
O(3)–C(17)	1.151(3)
O(4)–C(18)	1.152(3)
Bond angles	
C(15)–Cr(1)–C(16)	74.62(11)
C(15)–Cr(1)–S(1)	82.22(7)
C(16)–Cr(1)–S(1)	124.98(8)
C(15)–Cr(1)–S(2)	121.41(8)
C(16)–Cr(1)–S(2)	82.31(7)
S(1)–Cr(1)–S(2)	68.54(2)
C(15)–Cr(1)···Cr(2)	85.70(8)
C(16)–Cr(1)···Cr(2)	88.69(8)
S(1)–Cr(1)···Cr(2)	39.717(16)
S(2)–Cr(1)···Cr(2)	40.097(15)
C(17)–Cr(2)–C(18)	74.10(11)
C(17)–Cr(2)–S(1)	82.62(7)
C(18)–Cr(2)–S(1)	122.34(8)
C(17)–Cr(2)–S(2)	124.77(8)
C(18)–Cr(2)–S(2)	82.70(7)
S(1)–Cr(2)–S(2)	68.793(18)
C(17)–Cr(2)···Cr(1)	88.33(7)
C(18)–Cr(2)···Cr(1)	86.68(7)
S(1)–Cr(2)···Cr(1)	39.950(14)
S(2)–Cr(2)···Cr(1)	40.172(17)
C(8)–S(2)–Cr(2)	115.14(8)
C(8)–S(2)–Cr(1)	114.04(7)
Cr(2)–S(2)–Cr(1)	99.73(2)
C(8)–S(2)···S(1)	158.67(8)
Cr(2)–S(2)···S(1)	55.350(19)
Cr(1)–S(2)–S(1)	55.61(2)
Cr(2)–S(1)–Cr(1)	100.33(2)
C(1)–S(1)···S(2)	157.45(8)
Cr(2)–S(1)–S(2)	55.86(2)
Cr(1)–S(1)–S(2)	55.84(2)
O(1)–C(15)–Cr(1)	177.3(2)
O(2)–C(16)–Cr(1)	175.8(2)
O(3)–C(17)–Cr(2)	176.1(2)
O(4)–C(18)–Cr(2)	176.6(2)

Symmetry transformations used to generate equivalent atoms.

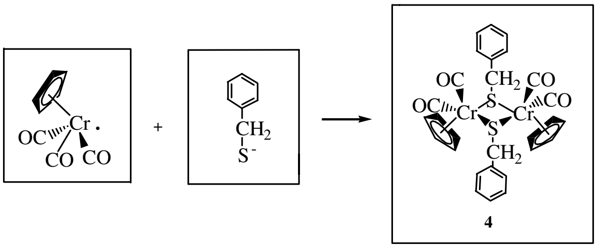
parameters, data collection, and structure refinement are given in Table 1. Its bonding parameters are given in Table 2. Tables of observed and calculated structure factors, hydrogen and non hydrogen atomic coordinates and thermal parameters, and anisotropic thermal parameters are available from the authors.

3. Results and discussion

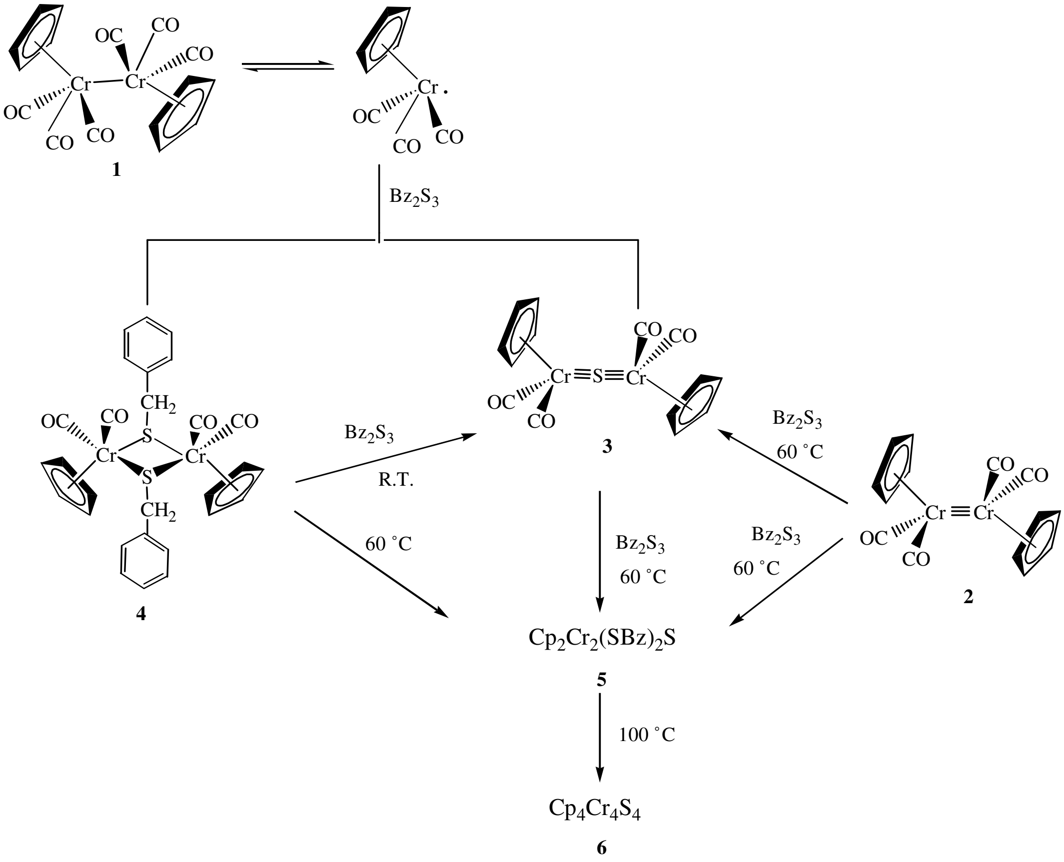
3.1. Reaction mechanistic pathway

The reaction of **1** with one mole equivalent of Bz_2S_3 at ambient temperature for 2 h has led to the isolation of $[CpCr(CO)_2]_2S$ (**3**), $[CpCr(CO)_2(SBz)]_2$ (**4**) and $[CpCr(SBz)]_2S$ (**5**) in 57.4%, 33.3% and 4.3% yield, respectively. A similar reaction at an elevated temperature of 60 °C for 5 h produced **3** and **5** in 80.8% and 13.7% yield, respectively. From NMR tube reaction study, it was shown that **4** also reacts with Bz_2S_3 at ambient temperature although at a prolonged time of 26 h to give a substantial yield of **3** (27.5%). The reaction of $[CpCr(CO)_2]_2$ (**2**), the decarbonylated congener of **1**, required a longer time of 72 h for complete reaction giving **3** (5.1%) and **5** (15.8%) at ambient temperature. Complete thermolysis of **4** after 1 h at 80 °C leads to the isolation of **3** (36%) and **5** (27.4%). Similarly, thermolysis of **5** at 100 °C for a prolonged 20 h gave **6** (22%) with unreacted **5** (35%) and a greenish blue layer which remains uneluted.

From the observed facile reactivity of **1** with Bz_2S_3, it is highly probable that such reaction proceeds via the 17-electron radical pathway as postulated in previous work carried out by Goh et al. [6a–6c] with systems such as Ph_2S_2 [4c], P_4E_3 (E = S, Se), elemental P_4 [7] and As_4 [8]. The vulnerability of the S–S linkage to cleavage processes in organic disulfides by various species is well documented [9a–9c]. Likewise, closely related organic trisulfides, such as Bz_2S_3 showed similar susceptibility toward cleavages by nucleophiles, electrophiles and radicals attack. In this case, the desulfurization process is



Scheme 1.



Scheme 2.

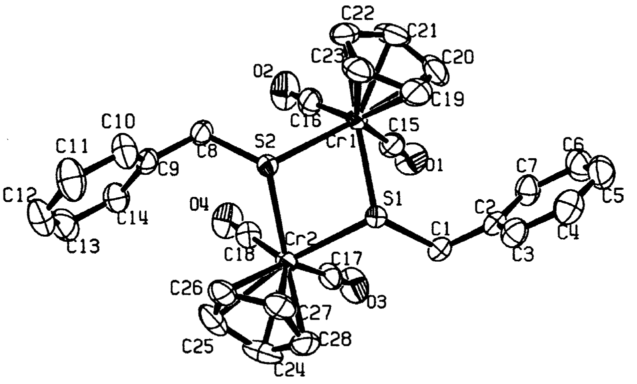


Fig. 1. Perspective view of the molecular structure of $[CpCr(CO)_2(SBz)]_2$ (**4**).

targeted towards the central sulfur of the –S–S–S– linkage [10]. It is conceivable that initial radical attack on the Bz_2S_3 leads to the primary formation of **3** by sulfur insertion between two $CpCr(CO)_2$ fragments. Formation of **4**, which is a thiolate-bridged dimer, is likely to be resulted from further radical attack on the benzyl moiety obtained after desulfurization of Bz_2S_3 as shown in Scheme 1. At elevated temperature, thermal degradation of **4** leads to total decarbonylation which converts to yield **5**. Further prolonged thermolysis ultimately resulted in the isolation of the cubane-like cluster $Cp_4Cr_4S_4$. The synthetic pathways proposed for the observed reactions as described above are illustrated in Scheme 2.

3.2. Structural study of 4

The perspective view of **4** is shown in Fig. 1 and its bonding parameters are given in Table 2. **4** was found as an isomer of *cis* arrangement of the carbonyl ligands, which is different from those of $[CpM(CO)_2(SPh)]_2$ [M = Cr [4c], Mo [4b]]. As with other related complexes mentioned earlier, **4** also maintained the same type of non-planar Cr_2S_2 fragment. The dihedral angle between Cr(1)–S(1)–Cr(2) and Cr(1)–S(2)–Cr(2) is 35.4(2)° as compared to $[CpCr(CO)_2(SPh)]_2$ [4c] (44.9(2)°). Each chromium atom is in a tetragonal–pyramidal coordination environment with the cyclopentadienyl group occupying the axial position. The two tetragonal pyramids share a basal edge [S(1)···S(2)] in an anti-relationship, the dihedral angle between the two basal planes being 75.86(7)°. The Cr(1)···Cr(2) separation with 3.702(7) Å is very close to $[CpCr(CO)_2(SPh)]_2$ [4c] (3.808(2) Å) and is much longer than the normal Cr–Cr single bonded complexes, for example, 2.950(2) Å for [CpCr(μ-SPh)(NO)]2 [11] and 2.906(3) Å for $Cp_2Cr_2(NO)_2(\mu\text{-}SCMe_3)\text{-}(\mu\text{-}S\text{–}SCMe_3)$ [12]. This indicates that the Cr(1)···Cr(2) in **4** is not bonded together.

Acknowledgements

The authors acknowledge supports from PPF Grant FP005/2003A and Vote F Grant F0164/2003C from University of Malaya.

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