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Nitrosylation of Dicyclopentadienyldi(μ-tert-butylthiolato)(μ-sulfido)-dichromium giving Tert-butylsulfanic Group. Molecular Structures of the Binuclear Complex Cp(NO)Cr(μ-$SCMe_3$)-(μ-S-$SCMe_3$)Cr(NO)Cp and Monomer $CpCr(NO)_2(ONO)$

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Reaction of CpCr(μ-$SCMe_3$)$_2$(μ-S)CrCp (I) with NO in THF led to the binuclear complex Cp(NO)Cr-(μ-$SCMe_3$)(μ-S-S-CMe_3)Cr(NO)Cp (II) and monomer $CpCr(NO)_2(ONO)$ (III). The X-ray study demonstrates that the complex II has an ordinary Cr–Cr bond (2.906(3) Å) supported by the bridged $SCMe_3$ (Cr–S 2.295(4) Å) and S–$SCMe_3$ (Cr–S 2.296(4) Å), S–S 2.076(4) Å) groups. In the monomeric complex III the Cr atom is surrounded by π-C_5H_5 (Cr–C 2.24(3) Å), two terminal NO groups (Cr–N 1.71(1) Å) and terminal ONO group (Cr–O 1.982(4) Å, ONO 115(2)°).

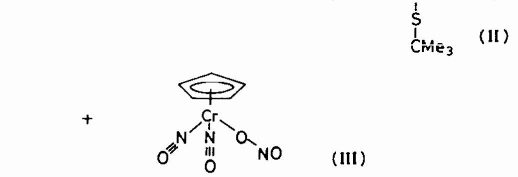
Introduction

The reactivity of the multiple metal–metal bonds is a major problem in the chemistry of the binuclear metallic complexes. In particular, an addition of small unsaturated molecules such as acetylenes, allene, CO and specifically NO to the Mo≡Mo triple bonds occurs readily [1]. In the last case the cleavage of M≡M bonds is observed *e.g.* in reactions of $(C_5Me_5)_2$-$Mo_2(CO)_4$ or $Mo_2(OR)_6$ with NO giving rise to the mononuclear $C_5Me_5Mo(CO)_2NO$ complex [2] and to the binuclear complex $(RO)_2(NO)Mo(\mu$-$OR)_2Mo$-$(NO)(OR)_2$ with the nonbonding distance Mo···Mo equal to 3.335(2) Å (R = i-Pr) [3]. On the other hand an addition of NO to $Cp_2Cr_2(\mu$-$OCMe_3)_2$ with Cr–Cr bond being of 2.650(5) Å and formally quaternary leads to the dimer $Cp_2Cr_2(NO)_2$ (μ-$OCMe_3$)$_2$ [4] which is probably similar to the known complex $Cp_2Cr_2(NO)_2(\mu$-$SPh)_2$ with the ordinary Cr–Cr bond being 2.950 Å [5].

Our intensively studied antiferromagnetic complex $(CpCrSCMe_3)_2S$ (I) contains the formally triple Cr≡Cr bond elongated to 2.689 Å probably due to the strong interligand repulsion [6]. It seemed interesting to investigate the reaction of I with NO in which one may expect an essential rearrangement of the sterically strained skeleton of I.

Results

The reaction of I with NO in THF rapidly gave the mixture of products. Its fractional recrystallization yielded the binuclear complex $(CpCrNO)_2$-$(SCMe_3)(S$-$SCMe_3)$ (II) and the mononuclear complex $CpCr(NO)_2(ONO)$ (III):



The IR spectrum of the needle–shaped crystals of II shows along with π-C_5H_5-ligand modes (826, 1021, 1450 and 3128 cm^{-1}) and CMe_3 group vibrations (1162 and 2878–2990 cm^{-1}) also a strong stretching band of the terminal NO group at 1655 cm^{-1} close

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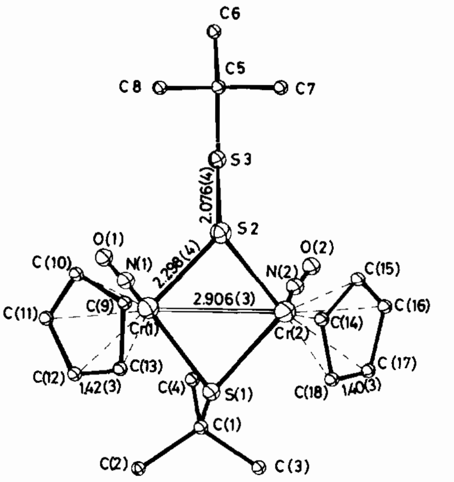

Fig. 1. Structure of (CpCrNO)$_2$(η-SCMe$_3$)(η-S-SCMe$_3$).

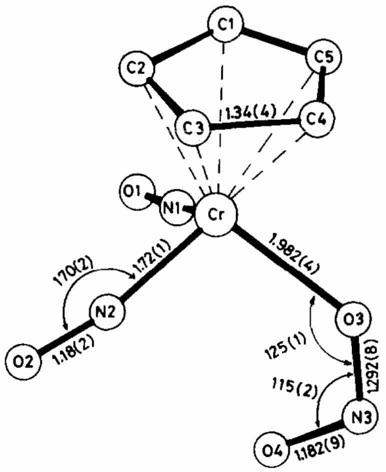

Fig. 3. Structure of CpCr(NO)$_2$NO$_2$.

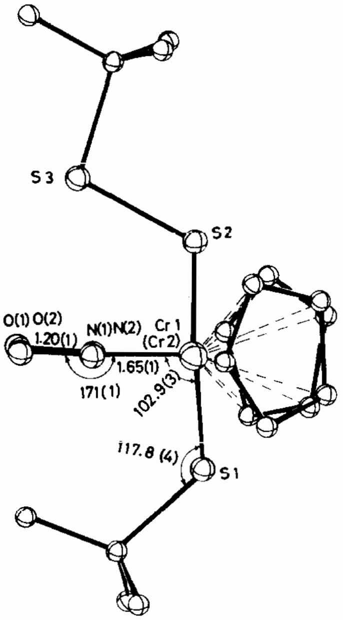

Fig. 2. Projection of the molecule II along the axis Cr–Cr.

to that observed for the above mentioned Cp$_2$Cr$_2$(NO)$_2$(μ-OCMe$_3$)$_2$ (ν_{NO} 1637 cm^{-1}) [4]. The structure of II was established by means of the total X-ray analysis (Fig. 1). The dimeric complex molecule contains two C$_5$H$_5$Cr fragments (Cr–C 2.22(2) Å mean) each of which being bound to the terminal NO group (Cr–N 1.65(1) Å, N–O 1.20(1) Å, CrNO mean 171(1)°). The chromium atoms are combined *via* the bridged tert-butylthiolate group SCMe$_3$ (Cr–S 2.295(4), S–C 1.89)1) Å, CrSC mean 118.6(1)°) as well as through tertbutylsulfanic group S-SCMe$_3$ generated upon transfer of the tert-butyl group to the bridged sulfide atom (Cr–S mean 2.297(4), S–S 2.076(4) Å CrS(2)S(3) mean 111.8(1)°, S(2)S(3)C(5) 101.7(4)°) (Fig. 2). Moreover there is an ordinary Cr–Cr bond of 2.906(3) Å close to the Cr–Cr bonds length in (CpCrNO)$_2$(SPh)$_2$ (2.950 Å) [5] and to the sum of Cr covalent atomic radii (1.46 X 2 = 2.92 Å). At the same time it is somewhat elongated with respect to the ordinary Cr–Cr bond in the recently described tetrahedral cluster Cp$_3$Cr$_3$(μ^3-S)$_4$Co(CO) (Cr–Cr mean 2.818 Å) [7]. Such an elongation of the ordinary Cr–Cr bond is probably a result of the high steric strain of complex II having the short balancing contacts S···C(C$_5$H$_5$) 3.021 ÷ 3.224 Å and S···N 2.983 ÷ 3.121 Å.

The presence of ordinary Cr–Cr in II is in agreement with diamagnetism of this complex. Actually a formal count of the number of electrons released by ligand to each Cr atom (5 from C$_5$H$_5$, 3 from NO and 3 from SCMe$_3$ or S-SCMe$_3$) for generating the Cr–Cr bond leaves one unpaired electron. In this case the spins of interacting ions are $S_a = S_b = 1/2$ and in the view of 'coupling channel model' [8] the antiferromagnetic coupling parameter (–2J) is equal to the sum of couplings (–2 J_Σ) and in the presence of M–M it would exceed essentially the values of singlet–triplet splitting (~1000 cm^{-1} at not very high temperatures).

The second product of complex I nitrosylation is complex CpCr(NO)$_2$(ONO) (III) isolated in the form of dark-green prisms. The mass spectrum of III contains the molecular ion (M$^+$ with m/e 233) and the ions of fragmentation products (m/e: CpCr(NO)(ONO)$^+$ 193, CpCr(NO)$_2^+$ 177, CpCr(NO)O$^+$ 163, CpCrNO$^+$ 147, CpCrO$^+$ 133, CpCr$^+$ 117). The data of mass spectra are in agreement with the X-ray results for III (Fig. 3). The chromium has pseudo-octahedral configuration and is combined with a tridentate π-cyclopentadienyl ligand (Cr–C mean

TABLE I. Atomic Coordinates ×10^5 for Cr and ×10^4 for O, N and C, and Anisotropic Temperature Factors in the Form T = $\exp[-\frac{1}{4}B_{11}h^2a^{*2}+\ldots+2B_{23}klb^*c^*)]$.

Atom	X	Y	Z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cr	3572(6)	13363(11)	¼	3.00(4)	3.15(4)	6.31(5)	0.06(2)	0.3(1)	0.2(1)
O(1)	−922(13)	2960(26)	4553(11)	6.9(8)	9.0(9)	6.9(5)	1.0(7)	1.2(4)	−2.9(5)
O(2)	−966(15)	2971(33)	435(16)	6.3(8)	11(1)	6.5(9)	2.0(8)	−0.7(7)	1.0(8)
O(3)	122(4)	−1689(6)	2550(24)	4.8(2)	3.5(1)	10.7(3)	−0.3(1)	−1(1)	1.8(7)
O(4)	−1443(5)	−1446(9)	2642(35)	4.5(3)	5.9(3)	19(1)	−1.4(2)	2(1)	−0.1(8)
N(1)	−444(10)	2135(26)	3743(15)	4.4(7)	6.4(7)	4.0(6)	−0.1(5)	−0.5(5)	−0.9(6)
N(2)	−458(11)	2129(22)	1250(15)	3.8(8)	4.8(6)	7.6(8)	1.1(5)	0.1(6)	−0.1(6)
N(3)	−750(5)	−2571(9)	2508(21)	5.8(3)	4.5(2)	11.9(5)	−1.5(2)	2.9(8)	−2.4(7)
C(1)	1935(12)	434(23)	2834(25)	3.4(4)	5.4(4)	14(2)	0.6(3)	−0.8(7)	2.2(8)
C(2)	1665(17)	2266(62)	3671(26)	7(1)	22(2)	5.8(9)	−8(1)	−1.8(8)	0(2)
C(3)	1482(11)	3694(15)	2875(23)	3.9(4)	4.2(3)	17(2)	−1.0(3)	1.7(7)	−4.5(7)
C(4)	1653(20)	2817(52)	1594(33)	5(1)	12(3)	16(2)	−2(2)	0(1)	6(2)
C(5)	1817(19)	980(29)	1501(27)	4.1(7)	9.2(9)	13(2)	−2.1(8)	3.2(9)	−4(1)

TABLE II. Bond Lengths d (Å) and Bond Angles in the Structure $(C_5H_5)Cr(NO)_2(NO_2)$.

Bond	d (Å)	Angle	ω (°)
Cr–O(3)	1.982(4)	O(3)CrN(1)	100.6(6)
Cr–N(1)	1.70(1)	O(3)CrN(2)	102.3(6)
Cr–N(2)	1.72(1)	CrO(3)N(3)	125(1)
Cr–C(1)	2.20(2)	N(1)CrN(2)	91.6(7)
CrC(2)	2.17(2)	CrN(1)O(1)	170(1)
Cr–C(3)	2.17(1)	CrN(2)O(2)	170(2)
Cr–C(4)	2.16(3)	O(3)N(3)O(4)	115(2)
Cr–C(5)	2.19(2)	C(2)C(1)C(5)	107(2)
O(3)–N(3)	1.292(8)	C(1)C(2)C(3)	107(2)
N(1)–O(1)	1.15(2)	C(2)C(3)C(4)	104(2)
N(2)–O(2)	1.18(2)	C(3)C(4)C(5)	120(3)
N(3)–O(4)	1.182(9)	C(1)C(5)C(4)	103(2)
C–C(C_5H_5)	1.34(4)		

2.18(2) Å), two terminal NO groups and an oxygen atom of the terminal nitrite group ONO (Cr–O 1.982(4), O(3)–N(3) 1.292(8), N(3)–O(4) 1.182(9) Å, CrO(3)N(3) 125(1), O(3)N(3)O(4) 115(2)°). The presence of the latter group in III may be explained by small amount of NO_2 present in gaseous nitrogen oxide. The nitrosyl groups and Cr atom produce the NCrN angle of 91.6(7)°, the Cr–NO being somewhat nonlinear (CrNO 170(1)°). The Cr–N bonds (1.71(1) Å mean) are essentially elongated while the N–O bonds, 1.16(2) Å (mean), are shorter than those in the complex II (1.65(1) and 1.20(1) Å respectively). Analogous distances were detected in the complex $CpCr(NO)_2Cl$ (1.717(3) and 1.158(4) Å, CrNO 169.6°) [9] described at the time of an X-ray study of complex III.

The differences in a coordination pattern of NO in III and II presented are in agreement with the IR spectral data for III having the bands of π-cyclopentadienyl ligand (840, 1024 and 3130 cm^{-1}) and a terminal nitrito group ONO (1438 and 1050 cm^{-1}) [10] as well as the two intensive bands ν_{NO} at 1730 and 1840 cm^{-1}. The analogous two bands ν_{NO} (1711 and 1816 cm^{-1}) are present in the IR spectrum of $CpCr(NO)_2Cl$ [9]. An increase of these frequencies in respect to 1655 cm^{-1} may be due to a neighbourhood of the strong electron-attractive NO and ONO groups (or NO and Cl for $CpCr(NO)_2Cl$ complex) unlike the electron-releasing $SCMe_3$ and $S\text{-}SCMe_3$ bridges. This results in the lower Cr–NO dative contribution in III with respect to II and thus in an elongation of Cr–N bond, shortening of the N–O bond and in ν_{NO} frequency growth.

The formation of complex II is somewhat unexpected: by assuming a formally triple Cr≡Cr bonding [7] one should expect its cleavage with retention of the sulfide and thiolate bridges analogously to the transformation of $Mo_2(OPr\text{-}i)_6$ into $(i\text{-}PrO)_2(NO)Mo(OPr\text{-}i)_2Mo(NO)(OPr\text{-}i)_2$ (Mo···Mo 3.335 Å) [3] noted in the Introduction. However the steric hindrance in such an adduct would be too great hence the nitrosylation is accompanied by an unusual transfer of $SCMe_3$ group to the sulfide bridge giving the tert-butylsulfanic group. Thus the complex II skeleton becomes similar to that of $Cp(NO)\overline{Cr(OCMe_3)_2Cr}(NO)Cp$, the nitrosylation product of $(CpCrOCMe_3)_2$ [4]. In the last complex with the formally quaternary Cr–Cr bond [7] an addition of three-electron-donor NO ligand to each Cr atom makes natural the cleavage of three bonds between Cr atoms [1] retaining an ordinary Cr–Cr bond. It can be noted that due to the reasons discussed above this complex should be diamagnetic and the paramagnetism observed in [4] and causing the line broadening in the PMR spectrum may be a result of impurities.

TABLE III. Atomic Coordinates × 10^4 (for Cr and S × 10^5) and Anisotropic Temperature Factors in the Form $T = \exp[-\frac{1}{4}B_{11}h^2a^{*2} + \ldots + 2B_{23}klb^*c^*)]$.

Atom	X	Y	Z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cr(1)	27883(19)	21333(10)	62575(12)	3.18(9)	4.0(1)	3.14(9)	0.08(8)	0.05(7)	0.26(8)
Cr(2)	6339(22)	29738(12)	70949(14)	4.9(1)	5.2(1)	4.7(1)	−0.66(9)	1.80(9)	−1.54(9)
S(1)	26997(33)	33983(17)	65482(19)	4.3(2)	3.8(2)	3.0(1)	−0.6(1)	0.3(1)	−0.1(1)
S(2)	9415(33)	17020(18)	69127(19)	3.9(2)	5.0(2)	3.2(1)	−0.9(1)	0.3(1)	0.1(1)
S(3)	−9462(37)	12864(19)	58907(22)	4.9(2)	5.1(2)	4.3(2)	−1.5(1)	−0.7(1)	0.8(1)
O(1)	1445(10)	1941(5)	4315(6)	7.6(6)	6.9(6)	2.7(4)	0.4(4)	0.3(4)	−0.7(4)
O(2)	−1755(12)	3268(6)	5587(9)	5.5(6)	8.7(7)	13.0(9)	1.0(6)	−0. (6)	1.6(7)
N(1)	1961(9)	2065(5)	5128(7)	4.1(5)	3.4(5)	3.1(5)	−0.1(4)	1.0(4)	0(4)
N(2)	−686(13)	3127(6)	6171(8)	4.4(6)	6.2(7)	6.7(7)	−0.2(5)	−0.1(5)	0(0.6)
C(1)	2472(16)	4055(7)	5496(8)	8.6(9)	4.1(7)	3.9(7)	−0.6(7)	0(0.7)	1.6(6)
C(2)	4059(18)	4086(10)	5240(11)	9(1)	12(1)	7.6(9)	−1.8(9)	3.4(8)	4.4(9)
C(3)	2073(21)	4814(7)	5888(11)	18(2)	3.0(7)	6.9(9)	2.3(9)	−1.2(9)	0.2(7)
C(4)	1331(17)	3798(8)	4667(9)	10(1)	5.8(8)	3.9(7)	−1.2(8)	−2.9(7)	0.1(6)
C(5)	−1282(13)	383(7)	6437(8)	4.3(7)	3.7(6)	5.3(7)	−0.1(5)	0.9(6)	0.4(5)
C(6)	−2515(13)	56(7)	5672(8)	4.4(6)	5.3(7)	5.3(6)	−2.0(6)	0.2(5)	−1.7(6)
C(7)	−1805(13)	557(7)	7377(8)	5.1(7)	7.2(9)	4.5(7)	−1.0(6)	2.5(6)	−2.2(6)
C(8)	97(15)	−129(7)	6575(9)	6.5(8)	4.7(7)	6.5(8)	2.3(6)	1.0(6)	0.2(6)
C(9)	4062(16)	1093(8)	6500(17)	3.9(8)	5.3(9)	11(1)	2.8(7)	−1.7(9)	0.3(9)
C(10)	4733(18)	1552(15)	5900(11)	3.8(9)	12(1)	6.8(9)	2.9(9)	0.8(8)	2(1)
C(11)	5242(15)	2185(11)	6394(18)	3.3(7)	8(1)	10(1)	0.2(8)	−1.2(8)	2(1)
C(12)	4919(19)	2144(11)	7318(14)	5.1(8)	8(1)	8(1)	2.6(8)	−4.6(8)	−1.5(9)
C(13)	4171(16)	1446(12)	7402(12)	4.5(8)	9(1)	5.6(9)	2.2(8)	−1.1(6)	2.9(9)
C(14)	1179(43)	2765(20)	8671(11)	11(2)	21(3)	2.4(7)	3.2(9)	1.3(9)	−3(1)
C(15)	−307(41)	2759(16)	8379(17)	16(3)	9(1)	7(1)	−2(2)	7(2)	−3(1)
C(16)	−754(29)	3430(23)	8051(18)	14(2)	13(2)	12(2)	−1(2)	8(2)	−6(2)
C(17)	393(49)	3886(14)	8072(18)	17(2)	9(2)	11(2)	−6(2)	8(2)	−7(1)
C(18)	1710(36)	3528(31)	8449(21)	11(2)	32(4)	7(1)	−13(2)	7(1)	−13(2)

TABLE IV. Bond Lengths d (A) in the Structure $(CpCr)_2(NO)_2(\mu^2\text{-SCMe}_3)(\mu^2\text{-SSCMe}_3)$.

Bond	d (A)	Bond	d (A)	Bond	d (A)
Cr(1)–Cr(2)	2.906(3)	Cr(2)–C(15)	2.19(3)	C(5)–C(6)	1.55(2)
Cr(1)–S(1)	2.295(4)	Cr(2)–C(16)	2.18(3)	C(5)–C(7)	1.54(2)
Cr(1)–S(2)	2.296(4)	Cr(2)–C(17)	2.18(3)	C(5)–C(8)	1.55(2)
Cr(1)–N(1)	1.664(9)	Cr(2)–C(18)	2.24(4)	C(9)–C(10)	1.40(3)
Cr(1)–C(9)	2.9(2)	S(1)–C(1)	1.89(1)	C(9)–C(13)	1.42(3)
Cr(1)–C(10)	2.21(2)	S(2)–S(3)	2.076(4)	C(10)–C(11)	1.37(3)
Cr(1)–C(11)	2.24(2)	S(3)–C(5)	1.86(1)	C(11)–C(12)	1.40(3)
Cr(1)–C(12)	2.26(2)	O(1)–N(1)	1.20(1)	C(12)–C(13)	1.44(3)
Cr(1)–C(13)	2.25(2)	O(2)–N(2)	1.20(2)	C(14)–C(15)	1.36(5)
Cr(2)–S(1)	2.307(4)	C(1)–C(2)	1.57(2)	C(14)–C(18)	1.50(6)
Cr(2)–S(2)	2.298(4)	C(1)–C(3)	1.53(2)	C(15)–C(16)	1.32(5)
Cr(2)–N(2)	1.65(1)	C(1)–C(4)	1.51(2)	C(16)–C(17)	1.33(5)
Cr(2)–C(14)	2.25(2)	C(5)–C(6)	1.55(2)	C(17)–C(18)	1.39(5)

Experimental

All operations were carried out under pure argon stream. The starting $(CpCrSCMe_3)_2S$ was obtained by method of [6]. THF was absolutized by distilling over Na suspension with benzophenone under argon flow. Pentane, heptane and benzene were distilled over the sodium suspension under argon counterflow. Nitrogen oxide was prepared by reaction of KNO_2 with H_2SO_4 in an argon filled Kipp generator and purified from impurities by passing through traps with NaOH solution, dry alkali and P_2O_5.

The X-ray data were obtained on 'Syntex P2_1' autodiffractometer (λMo-K_α, $2\theta \leqslant 50°$, T = 20 °C).

TABLE V. Bond Angles ω (°) in the Structure $(CpCr)_2(NO)_2(\mu^2\text{-}SCMe_3)(\mu^2\text{-}SSCMe_3)$.

Angle	ω	Angle	ω	Angle	ω
S(1)Cr(1)S(2)	101.5(1)	Cr(1)S(1)C(1)	117.8(4)	S(3)C(5)C(7)	108.4(8)
S(1)Cr(1)N(1)	102.9(3)	Cr(2)S(1)C(1)	119.3(4)	S(3)C(5)C(8)	109.3(8)
S(2)Cr(1)N(1)	96.4(3)	Cr(1)S(2)Cr(2)	78.6(1)	C(6)C(5)C(7)	112.9(9)
*Cp(1)Cr(1)Cr(2)	136.0(5)	Cr(1)S(2)S(3)	112.1(1)	C(6)C(5)C(8)	110.7(9)
Cp(1)Cr(1)S(1)	114.5(5)	Cr(2)S(2)S(3)	111.4(1)	C(7)C(5)C(8)	112.5(9)
Cp(1)Cr(1)S(2)	116.2(5)	S(2)S(3)C(5)	101.7(4)	C(10)C(9)C(13)	109(2)
Cp(1)Cr(1)N(1)	122.0(5)	Cr(1)N(1)O(1)	172.8(8)	C(9)C(10)C(11)	108(2)
S(1)Cr(2)S(2)	101.3(1)	Cr(2)N(2)O(2)	171(1)	C(10)C(11)C(12)	109(2)
S(1)Cr(2)N(2)	102.2(4)	S(1)C(1)C(2)	103.3(9)	C(11)C(12)C(13)	108(2)
S(2)Cr(2)N(2)	97.9(4)	S(1)C(1)C(3)	104.8(9)	C(9)C(12)C(13)	105(2)
Cp(2)Cr(2)Cr(1)	135.6(5)	S(1)C(1)C(4)	113.7(9)	C(15)C(14)C(18)	107(3)
Cp(2)Cr(2)S(1)	116.2(5)	C(2)C(1)C(3)	110(1)	C(14)C(15)C(16)	110(3)
Cp(2)Cr(2)S(2)	114.3(5)	C(2)C(1)C(4)	112(1)	C(15)C(16)C(17)	110(3)
Cp(2)Cr(2)N(1)	121.7(6)	C(3)C(1)C(4)	112(1)	C(16)C(17)C(18)	111(3)
Cr(1)S(1)Cr(2)	78.3(1)	S(3)C(5)C(6)	102.4(8)	C(14)C(18)C(17)	102(3)

*Cp centre of the cyclopentadienyl ring.

Complex II-1709 reflections with $F^2 \geqslant 2\sigma$, the structure was decoded by direct method using anisotropic full-matrix approximation. The atomic coordinates and their temperature factors are listed in Table III. The crystals are monoclinic: a, 9.218(6); b, 17.811(6); c, 14.286(10) Å; β, 100.03(5)°; V = 2309.7(9) Å^3; Z = 4, spatual group $P2_1/n$, R_1 = 0.084, R_w = 0.069.

Complex III-1022 reflections with $F^2 \geqslant 2\sigma$, the structure was decoded by heavy atom techniques using anisotropic full-matrix approximation. The atomic coordinates and temperature factors are listed in Table I. The crystals are rhombic, a, 13.293(7); b, 6.469(2), c, 9.824(3) Å; V = 844.8 Å^3; Z = 8; spatual group $P\,na2_1$; R_1 = 0.031, R_w = 0.034.

The IR spectra were taken in KBr pellts on UR-20 instrument. The mass-spectra were recorded on DS-50 instrument. The magnetic measurements were carried out by Faraday's method.

$(C_5H_5CrNO)_2(\mu\text{-}SCMe_3)(\mu\text{-}S\text{-}SCMe_3)$ (II)

The pure nitrogen oxide was bubbled through a solution of 0.5 g (1.13 mmol) of $(CpCrSCMe_3)_2S$ for 1 h. The obtained brown solution was filtered, evaporated to dryness and then extracted successively with pentane (Extract A) and heptane (extract B) under refluxing. The solution A was concentrated to one third of its volume and cooled to –5 °C. The precipitated red needles were separatated from the solution and dried under vacuum. Yield 35%. Found (calculated) %: N 6.32 (5.56). IR spectrum, ν cm^{-1}: 550w, 575w, 612w, 643w, 826w, 938w, 1021m, 1075m, 1162m, 1370m, 1399w, 1450m, 1655vs, 2878–2990 br,m, 3128w.

$C_5H_5Cr(NO)_2(ONO)$ (III)

The solution B was concentrated to 1/3 of its volume and cooled to –5 °C. The dark-green prisms precipitated were separated from the solution and dried under vacuum. Yield 23%. IR spectrum, ν cm^{-1}: 490m, 539m, 599s, 630w, 840s, 955w, 1024m, 1050s, 1280w, 1332m, 1395m, 1438s, 1730vs, 1840vs, 3130w.

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