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Chemical assembly and molecular structure of the octanuclear heterometallic cluster $(MeC_5H_4)_2Cr_2(\mu_3\text{-}S)_4Co_2$-$[(\mu_3\text{-}S)_2Fe_2(CO)_6]$. $[(\mu_3\text{-}S)Fe_2(CO)_6]$ having a "triangular metal ornament" core

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Abstract

The octanuclear cluster $(MeC_5H_4)_2Cr_2(\mu_3\text{-}S)_4Co_2[(\mu_3\text{-}S)_2Fe_2(CO)_6][(\mu_3\text{-}S)Fe_2(CO)_6]$ was obtained from the photochemical reaction of $S_2Fe_2(CO)_6$ with $(MeC_5H_4)_2Cr_2S_3Co_2(CO)_4$, which has a "metallic butterfly" core. The X-ray diffraction study (space group $P2_1/n$, $a = 12.111(2)$, $b = 16.567(3)$, $c = 19.049(3)$ Å, $\beta = 106.82(1)°$, $Z = 4$, $R = 0.063$) shows the molecule to contain a pseudocubane fragment $Cr_2Co_2S_4$ with Cr–Cr and Cr–Co bonds of 2.772(3) and 2.704(2)–2.739(2) Å, respectively and a non-bonding Co $\cdots$ Co separation of 3.160(1) Å. One of the Co atoms is asymmetrically bonded to the binuclear fragment $S_2Fe_2(CO)_6$ (Co–Fe 2.645(2) and 3.366(2) Å) and symmetrically bonded to the $Fe_2S(CO)_6$ fragment (Co–Fe 2.584(2) and 2.571(2) Å).

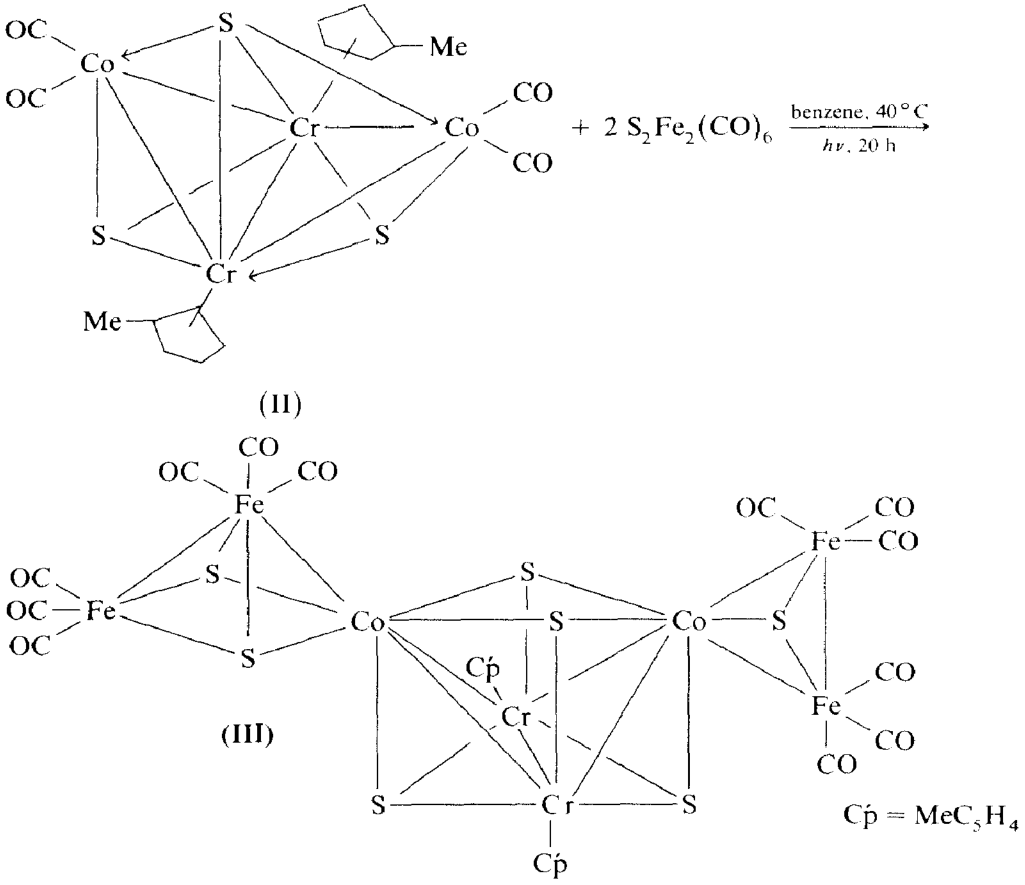
Introduction

The idea of "cluster design", viz. the use of metal complexes as ligands at the central metal atom of other complexes is extensively used for the purposeful synthesis of heterometallic clusters [1–6]. It was recently shown that the combination of the binuclear metal-containing ligands $Cp_2Cr_2(SCMe_3)(S)_2$ (Q) and $S_2Fe_2(CO)_6$ with the complexes, which act as the sources of Fe, Co or Rh atoms affords heterometallospirane clusters having Cr_2MFe_2 cores. The geometry, particularly the Fe–Fe and M–Fe distances vary considerably, and depend on the nature of the central M atom in the cluster [5].

These clusters, as well as the heterometallospirane clusters Q_2M with the Cr_2MCr_2 (M = Cr, Mn, Fe, Co [1]) cores we studied previously, consist of two metal triangles that have a common vertex. On the other hand, clusters possessing a "heterometallic butterfly" core have two triangles joined by a common edge [1,4,7]. Both types of clusters may be regarded as fragments of heterometallic polymers, consisting of triangles (peculiar "triangular metal ornaments"). Thus it was of interest to build up the "metallic butterfly" by addition of the two binuclear fragments to form an octanuclear "metal ornament". For this purpose we chose a reaction of $Fe_2S_2(CO)_6$ (I) with the "heterometallic butterfly" cluster, $CP_2'Cr_2(\mu_3\text{-}S)_2(\mu_4\text{-}S)Co_2(CO)_4$ (II) ($Cp' = MeC_5H_4$) since the characteristics of the $Co(CO)_2$ groups in II are the same as of those in the triangular cluster $Q'Co(CO)_2$ ($Q' = (MeC_5H_4)_2Cr_2(SCMe_3)(S)_2$), which readily reacts with I [1].

Results and discussion

The octanuclear cluster $[Cp_2'Cr_2(\mu_3\text{-}S)_4Co_2][(S)_2Fe_2(CO)_6][SFe_2(CO)_6]$ (III) is formed as a result of the interaction of I and II taken in the molar ratio 1 : 2 under the UV-irradiation in benzene at 40 °C for 20 hours:



(II)

(III)

Cp = MeC_5H_4

In the IR spectrum of III the bands present correspond to the stretching vibrations of terminal CO groups (1950, 1970, 1990, 2005, 2030, 2040 cm^{-1}). The X-ray diffraction study shows (Fig. 1) that the central fragment of III is a distorted cube

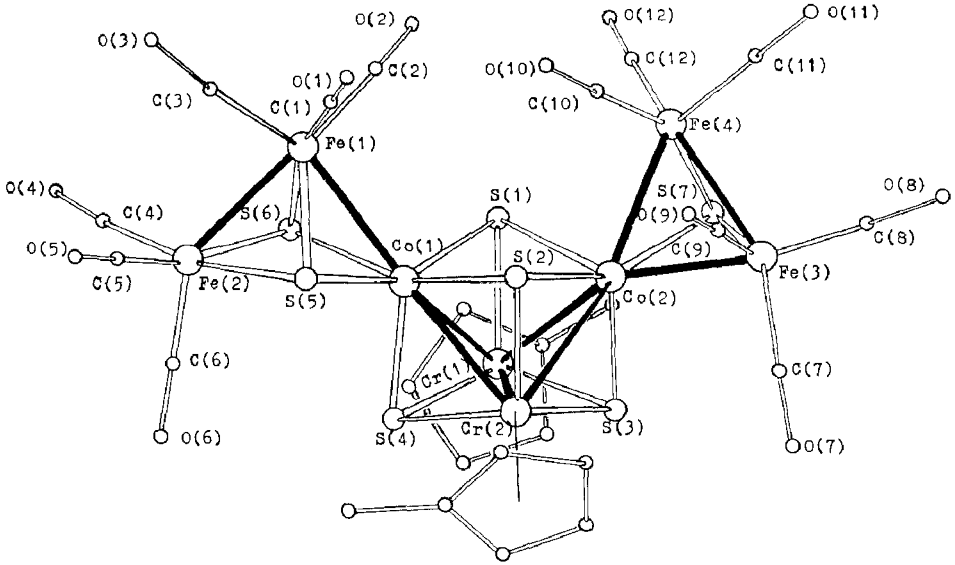


Fig. 1. The structure of the cluster $(MeC_5H_4)_2Cr_2(\mu_3\text{-}S)_4Co_2[(\mu_3\text{-}S)_2Fe_2(CO)_6][(\mu_3\text{-}S)Fe_2(CO)_6]$.

$Cr_2Co_2(\mu_3\text{-}S)_4$ (Co–S 2.161(3), 2.179(3), 2.208(4), 2.233(3), 2.245(4) and 2.256(4) Å; Cr–S 2.217(3)–2.240(4) Å), its Cr_2Co_2 metal core has the structure of a butterfly with one Cr–Cr bond (2.772(3) Å, four Cr–Co (2.704(2)–2.739(2) Å) bonds and a non-bonded Co · · · Co (3.160(1) Å) separation which is shorter than the corresponding distance 4.052 Å in the initial butterfly cluster II [1,2,7]. As a result, the dihedral angle between the Cr_2Co wings changes drastically, viz. from 124.8° in II [7] to 85.1° in III, because of the increased number of bridging sulfur atoms in III.

The geometry of the central "butterfly" core $Cp_2Cr_2(\mu_3\text{-}S)_4Co_2$ is similar to that found recently for $Cp_2Mo_2(\mu_3\text{-}S)_4Ni_2(CO)_2$ [8] (Ni · · · Ni 2.96 Å, Mo–Mo 2.829, Mo–Ni 2.720–2.724, Mo–S (between the wings) 2.252 and 2.262, Mo–S (face) 2.328, 2.329, 2.325, 2.332 Å; Ni–S (wings) 2.288, 2.286, 2.278, 2.282; Ni–S (face) 2.158, 2.162 Å). The Ni(CO) groups, and the Co-containing fragments together

Table 1

Crystal data for the cluster $(MeC_5H_4)_2Cr_2(\mu_3\text{-}S)_4Co_2[(\mu_3\text{-}S)_2Fe_2(CO)_6][(\mu_3\text{-}S)Fe_2(CO)_6]$

System	monoclinic
Space group	$P2_1/n$
a (Å)	12.111(2)
b (Å)	16.567(3)
c (Å)	19.049(3)
β (°)	106.82(2)
V(Å^3)	3658.45
Z	4
Number of reflections measured	5711
Number of reflections with $I > 3\sigma(I)$ used in the refinement	2594
R_1	0.063
R_w	0.075

Table 2

Positional parameters and their estimated standard deviations.

Atom	x	y	z
Co(1)	0.7349(2)	0.0615(1)	0.2746(1)
Co(2)	0.9134(2)	0.0838(1)	0.1836(1)
Fe(1)	0.5082(2)	0.0557(2)	0.2185(1)
Fe(2)	0.5239(2)	0.0364(2)	0.3552(2)
Fe(3)	0.9891(2)	0.1746(2)	0.0973(2)
Fe(4)	0.8160(2)	0.0831(2)	0.0444(1)
Cr(1)	0.9397(2)	−0.0161(2)	0.3004(2)
Cr(2)	0.9343(2)	0.1501(2)	0.3165(2)
S(1)	0.7976(3)	−0.0145(3)	0.1964(2)
S(2)	0.7897(3)	0.1655(3)	0.2159(2)
S(3)	1.0624(3)	0.0757(3)	0.2804(3)
S(4)	0.8694(3)	0.0547(3)	0.3777(2)
S(5)	0.6079(3)	0.1358(3)	0.3100(3)
S(6)	0.6197(3)	−0.0368(3)	0.2928(3)
S(7)	0.9902(4)	0.0432(3)	0.0986(3)
O(1)	0.413(1)	0.2022(8)	0.1358(9)
O(2)	0.520(1)	−0.0316(9)	0.0893(7)
O(3)	0.276(1)	0.001(1)	0.2094(9)
O(4)	0.394(1)	−0.108(1)	0.3734(9)
O(5)	0.366(1)	0.152(1)	0.3903(8)
O(6)	0.691(1)	0.023(1)	0.5011(9)
O(7)	1.200(1)	0.222(1)	0.216(1)
O(8)	1.079(1)	0.215(1)	−0.025(1)
O(9)	0.823(1)	0.3053(9)	0.0826(8)
O(10)	0.5986(9)	0.1629(9)	0.0473(7)
O(11)	0.799(1)	0.140(1)	−0.1031(7)
O(12)	0.717(1)	−0.0764(9)	−0.004(1)
C(1)	0.455(1)	0.148(1)	0.165(1)
C(2)	0.517(1)	0.005(1)	0.1396(8)
C(3)	0.368(1)	0.021(1)	0.216(1)
C(4)	0.442(1)	−0.053(1)	0.368(1)
C(5)	0.425(1)	0.108(1)	0.375(1)
C(6)	0.623(2)	0.029(1)	0.440(1)
C(7)	1.124(2)	0.204(1)	0.172(1)
C(8)	1.040(2)	0.197(1)	0.024(1)
C(9)	0.887(1)	0.252(1)	0.091(1)
C(10)	0.686(2)	0.133(1)	0.0491(9)
C(11)	0.808(2)	0.118(1)	−0.046(1)
C(12)	0.754(2)	−0.015(1)	0.019(1)
C(13)	1.019(2)	−0.131(1)	0.278(1)
C(14)	1.098(2)	−0.093(1)	0.341(1)
C(15)	1.044(2)	−0.088(1)	0.395(1)
C(16)	0.930(2)	−0.123(1)	0.372(1)
C(17)	0.915(1)	−0.149(1)	0.298(1)
C(18)	1.042(2)	−0.150(1)	0.213(1)
C(19)	0.923(1)	0.240(1)	0.405(1)
C(20)	0.906(1)	0.280(1)	0.341(1)
C(21)	1.003(2)	0.274(1)	0.313(1)
C(22)	1.084(1)	0.232(1)	0.361(1)
C(23)	1.042(2)	0.205(1)	0.420(1)
C(24)	0.840(2)	0.236(2)	0.451(1)

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $(4/3)[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab(\cos\gamma)B_{12} + ac(\cos\beta)B_{13} + bc(\cos\alpha)B_{23}]$.

donate 24e$^-$, to give a total number of electrons of 62, as in the heterometallic butterfly cluster II.

On the other hand, III differs from the cubane core Cr_2Co_2 in cluster $Cp_2Cr_2Co_2(\mu_3\text{-}S)_4(P(OEt)_3)_2$ [9] by the absence of the Co–Co bond, since the two $P(OEt)_3$ ligands donate only 4e$^-$; non-equivalently bonded iron-sulfur groups contribute 6e$^-$. One Co atom is bonded to the $(S)_2Fe_2(CO)_6$ fragment (Fe–Fe 2.575(2) Å) via two Co–S σ-bonds (2.222(3) and 2.236(3) Å) and one Fe(1)→Co(1) bond 2.645(2) Å, which evidently has a donor–acceptor character (the second Fe–Co bond is absent; the Fe(2) ··· Co(1) separation 3.366(1) Å). In this case $(S)_2Fe_2(CO)_6$ contributes 4 electrons to the cluster. The second Co atom is bonded to the monosulfide $SFe_2(CO)_6$ fragment by the S→Co (2.194(4) Å) and two Co→Fe (2.571(2) and 2.584(2) Å) dative bonds, thus accepting only 2 electrons. Along with the geometrical similarity of the $Cr_2Co_2S_4$ moiety in III and $Mo_2Ni_2S_4$ in IV, the geometry of the $CoFe_2S(CO)_6$ and $CoFe_2S_2(CO)_6$ groups is very much the same as in the previously studied $Co_2FeS(CO)_9$ [12] and $Fe_3S_2(CO)_9$ [13] clusters. This similarity suggests that the structures of complex triangular heterometallic ornaments may be described (and perhaps even predicted) on the basis of the geometry of known smaller clusters.

Experimental

Syntheses were carried out in a stream of pure argon in dry solvents. The initial complexes $S_2Fe_2(CO)_6$ and II were synthesized by published procedures [10,7]. The IR-spectra were recorded with a Specord 75IR instrument in KBr pellets. The X-ray diffraction data were obtained with a four-circle diffractometer CAD-4 (λ Mo-K_α, θ–2θ scan, Table 1). The structure was solved by direct methods and refined anisotropically for all non-hydrogen atoms by full-matrix least squares to $R_1 = 0.063$, $R_w = 0.075$. Atomic coordinates, bond lengths and bond angles are listed in Tables 2–4. All calculations were performed with the SDP program package [11] on a PDP 11/23 Plus computer.

Table 3

Bond distances (Å)

Co(1)–Fe(1)	2.645(2)	Fe(1)–Fe(2)	2.575(3)
Co(1)–Cr(1)	2.708(2)	Fe(1)–S(5)	2.246(4)
Co(1)–Cr(2)	2.739(2)	Fe(1)–S(6)	2.254(4)
Co(1)–S(1)	2.245(4)	Fe(2)–S(5)	2.235(4)
Co(1)–S(2)	2.256(4)	Fe(2)–S(6)	2.242(4)
Co(1)–S(4)	2.161(3)	Fe(3)–Fe(4)	2.546(3)
Co(1)–S(5)	2.222(3)	Fe(3)–S(7)	2.176(5)
Co(1)–S(6)	2.236(3)	Fe(4)–S(7)	2.166(4)
Co(2)–Fe(3)	2.584(2)	Cr(1)–Cr(2)	2.772(3)
Co(2)–Fe(4)	2.571(2)	Cr(1)–S(1)	2.217(3)
Co(2)–Cr(1)	2.718(3)	Cr(1)–S(3)	2.236(4)
Co(2)–Cr(2)	2.704(3)	Cr(1)–S(4)	2.234(4)
Co(2)–S(1)	2.208(4)	Cr(2)–S(2)	2.205(3)
Co(2)–S(2)	2.233(3)	Cr(2)–S(3)	2.240(4)
Co(2)–S(3)	2.179(3)	Cr(2)–S(4)	2.237(4)
Co(2)–S(7)	2.194(4)		

Table 4

Bond angles in degrees

Fe(1)Co(1)Cr(1)	147.66(9)	Fe(3)Co(2)Fe(4)	59.19(8)
Fe(1)Co(1)Cr(2)	149.5(1)	Fe(3)Co(2)Cr(1)	153.71(8)
Fe(1)Co(1)S(1)	102.8(2)	Fe(3)Co(2)Cr(2)	113.90(8)
Fe(1)Co(1)S(2)	104.3(2)	Fe(3)Co(2)S(1)	148.5(1)
Fe(1)Co(1)S(4)	141.9(1)	Fe(3)Co(2)S(2)	102.3(2)
Fe(1)Co(1)S(5)	54.11(9)	Fe(3)Co(2)S(3)	102.4(1)
Fe(1)Co(1)S(6)	54.16(9)	Fe(3)Co(2)S(7)	53.4(2)
Cr(1)Co(1)Cr(2)	61.19(6)	Fe(4)Co(2)Cr(1)	138.77(9)
Cr(1)Co(1)S(1)	52.17(9)	Fe(4)Co(2)Cr(2)	149.11(9)
Cr(1)Co(1)S(2)	93.9(1)	Fe(4)Co(2)S(1)	89.7(2)
Cr(1)Co(1)S(4)	53.2(1)	Fe(4)Co(2)S(2)	98.2(2)
Cr(1)Co(1)S(5)	152.4(1)	Fe(4)Co(2)S(3)	153.2(1)
Cr(1)Co(1)S(6)	101.9(2)	Fe(4)Co(2)S(7)	53.4(2)
Cr(2)Co(1)S(1)	93.2(1)	Cr(1)Co(2)Cr(2)	61.52(7)
Cr(2)Co(1)S(2)	51.28(9)	Cr(1)Co(2)S(1)	52.27(9)
Cr(2)Co(1)S(4)	52.7(1)	Cr(1)Co(2)S(2)	94.1(2)
Cr(2)Co(1)S(5)	104.1(2)	Cr(1)Co(2)S(3)	53.0(2)
Cr(2)Co(1)S(6)	151.9(1)	Cr(1)Co(2)S(7)	116.4(1)
S(1)Co(1)S(2)	83.9(1)	Cr(2)Co(2)S(1)	95.0(1)
S(1)Co(1)S(4)	105.3(1)	Cr(2)Co(2)S(2)	51.99(9)
S(1)Co(1)S(5)	155.4(1)	Cr(2)Co(2)S(3)	53.3(2)
S(1)Co(1)S(6)	92.3(1)	Cr(2)Co(2)S(7)	150.6(1)
S(2)Co(1)S(4)	103.8(1)	S(1)Co(2)S(2)	85.3(1)
S(2)Co(1)S(5)	93.2(1)	S(1)Co(2)S(3)	105.0(1)
S(2)Co(1)S(6)	156.8(1)	S(1)Co(2)S(7)	105.9(1)
S(4)Co(1)S(5)	99.2(1)	S(2)Co(2)S(3)	105.1(1)
S(4)Co(1)S(6)	99.3(1)	S(2)Co(2)S(7)	148.3(1)
S(5)Co(1)S(6)	80.7(1)	S(3)Co(2)S(7)	100.5(1)
Co(1)Fe(1)Fe(2)	80.30(7)	S(1)Cr(1)S(4)	103.8(1)
Co(1)Fe(1)S(5)	53.28(9)	S(3)Cr(1)S(4)	99.0(1)
Co(1)Fe(1)S(6)	53.62(9)	Co(1)Cr(2)Co(2)	70.98(7)
Fe(2)Fe(1)S(5)	54.7(2)	Co(1)Cr(2)Cr(1)	58.86(6)
Fe(2)Fe(1)S(6)	54.9(2)	Co(1)Cr(2)S(2)	53.0(1)
S(5)Fe(1)S(6)	79.8(1)	Co(1)Cr(2)S(3)	104.3(1)
Fe(1)Fe(2)S(5)	55.1(2)	Co(1)Cr(2)S(4)	50.24(9)
Fe(1)Fe(2)S(6)	55.2(2)	Co(2)Cr(2)Cr(1)	59.49(7)
S(5)Fe(2)S(6)	80.3(1)	Co(2)Cr(2)S(2)	52.9(1)
Co(2)Fe(3)Fe(4)	60.15(7)	Co(2)Cr(2)S(3)	51.3(1)
Co(2)Fe(3)S(7)	54.1(2)	Co(2)Cr(2)S(4)	104.5(1)
Fe(4)Fe(3)S(7)	53.9(2)	Cr(1)Cr(2)S(2)	93.3(2)
Co(2)Fe(4)Fe(3)	60.66(7)	Cr(1)Cr(2)S(3)	51.7(2)
Co(2)Fe(4)S(7)	54.4(2)	Cr(1)Cr(2)S(4)	51.6(2)
Fe(3)Fe(4)S(7)	54.3(1)	S(2)Cr(2)S(3)	104.1(1)
Co(1)Cr(1)Co(2)	71.24(7)	S(2)Cr(2)S(4)	102.9(1)
Co(1)Cr(1)Cr(2)	59.96(6)	S(3)Cr(2)S(4)	98.7(1)
Co(1)Cr(1)S(1)	53.09(9)	Co(1)S(1)Co(2)	90.5(1)
Co(1)Cr(1)S(3)	105.4(1)	Co(1)S(1)Cr(1)	74.7(1)
Co(1)Cr(1)S(4)	50.74(9)	Co(2)S(1)Cr(1)	75.8(1)
Co(2)Cr(1)Cr(2)	59.00(7)	Co(1)S(2)Co(2)	89.5(1)
Co(2)Cr(1)S(1)	52.0(1)	Co(1)S(2)Cr(2)	75.8(1)
Co(2)Cr(1)S(3)	51.1(1)	Co(2)S(2)Cr(2)	75.1(1)
Co(2)Cr(1)S(4)	104.1(1)	Co(2)S(3)Cr(1)	76.0(1)
Cr(2)Cr(1)S(1)	92.9(2)	Co(2)S(3)Cr(2)	75.5(1)
Cr(2)Cr(1)S(3)	51.8(1)	Cr(1)S(3)Cr(2)	76.6(1)

Table 4 (continued)

Cr(2)Cr(1)S(4)	51.7(2)	Co(1)S(4)Cr(1)	76.1(1)
S(1)Cr(1)S(3)	102.8(1)	Co(1)S(4)Cr(2)	77.0(1)
Cr(1)S(4)Cr(2)	76.6(1)	Co(1)S(6)Fe(2)	97.5(1)
Co(1)S(5)Fe(1)	72.6(1)	Fe(1)S(6)Fe(2)	69.9(1)
Co(1)S(5)Fe(2)	98.1(1)	Co(2)S(7)Fe(3)	72.5(1)
Fe(1)S(5)Fe(2)	70.2(1)	Co(2)S(7)Fe(4)	72.3(1)
Co(1)S(6)Fe(1)	72.2(1)	Fe(3)S(7)Fe(4)	71.8(1)

$(MeC_5H_4)_2Cr_2(\mu_3\text{-}S)_4Co_2[(\mu_3\text{-}S)_2Fe_2(CO)_6][(\mu_3\text{-}S)Fe_2(CO)_6]$ (III)

A mixture of $(MeC_5H_4)_2Cr_2S_3Co_2(CO)_4$ (II) (0.20 g; 0.34 mmol) and $S_2Fe_2(CO)_6$ (0.25 g; 0.73 mmol) in benzene solution (35 ml) was irradiated with UV light at 40 °C for 20 h. Heptane (15 ml) was added to the mixture, it was concentrated at 40 °C/0.1 torr to a volume of 15 ml, and left to stand at −18 °C. The solution was separated from the crystals of unchanged II by decantation, concentrated to 7 ml, and chromatographed on Al_2O_3 (25 × 2.5 cm). The unchanged II was washed with hexane, and the brown zone was then eluted with benzene. This benzene solution was evaporated to dryness at 40 °C/0.1 torr and the solid residue was extracted with hexane. The hexane solution was concentrated to 3 ml and left to stand at −18 °C. The precipitated brown prisms that separated were washed with cold pentane and dried under vaccuum at 0.1 torr. Yield 0.03 g (7.5%).

The IR spectrum (ν, cm^{-1}): 500 m., 560 m., 580 m.sh., 630 w., 740 w., 760 w., 820 m., 875 w., 1020 w., 1120 w., 1400 m., 1430 m., 1950 s., sh., 1970 v.s., 1990 v.s., 2005 v.s.sh., 2030 v.s., 2040 v.s., 3040 w.

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