Selenolato Chromium Complexes. Synthesis, Thermolytic Degradation and Crystal Structure of [Cr(η-C_5H_5)$(CO)_3$(SePh)]†

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The instantaneous reaction of [{Cr(cp)$(CO)_3$}$_2$] **1** (cp = η^5-C_5H_5) with 1 molar equivalent of Ph_2Se_2 at ambient temperature led to the isolation of [Cr(cp)$(CO)_3$(SePh)] **2** and [{Cr(cp)$(CO)_2$(SePh)}$_2$] **3** in 43.6% and *ca.* 7%, yields respectively. At elevated temperatures, compound **2** underwent decarbonylation with concomitant coupling to yield **3**, [{Cr(cp)(CO)(SePh)}$_2$] **4** and finally [{Cr(cp)(SePh)}$_2$Se] **5** in 45.6% yield. The latter was also obtained in 49.8% yield from the reaction of [{Cr(cp)$(CO)_2$}$_2$] with Ph_2Se_2 at 60 °C. The structure of **2** was established by X-ray diffraction analysis: space group $P2_1/c$ (no. 14), a = 10.401(3), b = 8.701(2), c = 15.604(4) Å, β = 99.51(2)° and Z = 4.

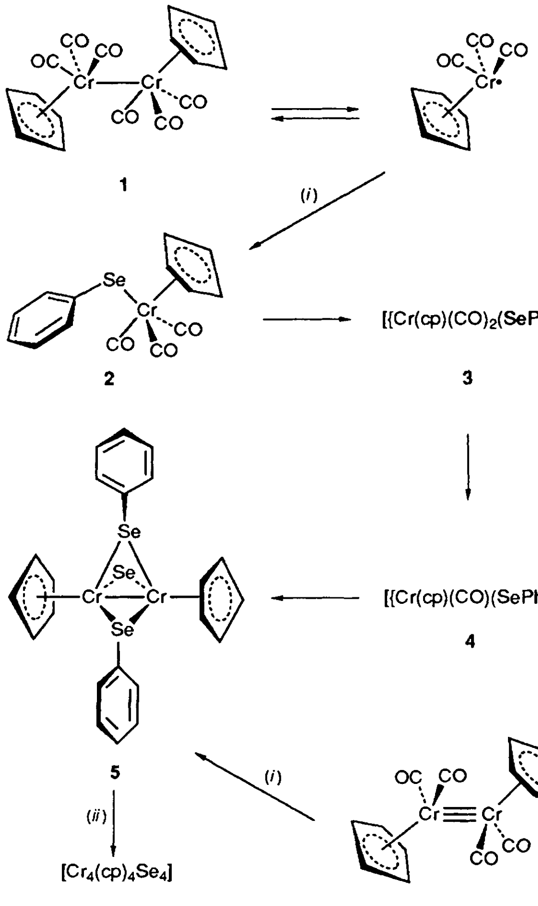
While transition-metal complexes containing thiolate ligands have attracted and sustained much interest owing to their relevance to biological[1] and catalytic[2] processes, very little attention has been directed to the study of the analogous selenium complexes. The few reports available have described the reaction of Ph_2Se_2 with [{Mo(cp)$(CO)_3$}$_2$][3] (cp = η^5-C_5H_5) and [{Mo(η-C_5H_4Me)$(CO)_3$}$_2$],[4] and more recently with the half-sandwich compounds [V(cp)$(CO)_4$] and [V(η-C_5Me_5)$(CO)_4$][5] and the cluster [$Ru_3(CO)_{12}$].[6] Having completed a study on the reaction of [{Cr(cp)$(CO)_3$}$_2$] and its related Cr≡Cr derivative [{Cr(cp)$(CO)_2$}$_2$] with Ph_2S_2,[7] we have extended the investigation to Ph_2Se_2 and herein report our findings.

Results and Discussion

Synthetic Studies.—A deep green suspension of [{Cr(cp)$(CO)_3$}$_2$] **1** in toluene reacted instantaneously with 1 molar equivalent of Ph_2Se_2 at ambient temperature to give a brown homogeneous solution from which was isolated the pure complex [Cr(cp)$(CO)_3$(SePh)] **2** as brown crystals in 43.6% yield and [{Cr(cp)$(CO)_2$(SePh)}$_2$] **3** as impure brown crystals in *ca.* 7% yield. It has not been possible to separate **3** from trace amounts of a cp-containing complex with δ 5.15. Its characterisation was based on the exact match of its CO stretching frequencies and their relative intensities with those of [{Cr(cp)$(CO)_2$(SPh)}$_2$].[7] An identical reaction mixture after 3 h at 50 °C yielded **2**, [{Cr(cp)(CO)(SePh)}$_2$] **4** and [{Cr(cp)(SePh)}$_2$Se] **5** in 31.1, 30.0 and 11.3% yields, respectively. At 60 °C only **5** was isolated (45.6% yield).

The Cr≡Cr triply bonded complex [{Cr(cp)$(CO)_2$}$_2$] also reacted with Ph_2Se_2, though much less readily, and only at elevated temperatures. At 60 °C the half-life was approximately 4.5 h, producing only **5** in 49.8% yield.

Thermolytic Degradations.—Complex **2** in toluene and **4** in tetrahydrofuran (thf) were completely converted into [{Cr(cp)(SePh)}$_2$Se] **5** after 1.5–2 h at 50–60 °C. In the thermolysis of **2** the formation of trace amounts of **3** (δ 4.56) was detected. A more exhaustive thermolysis for *ca.* 20 h at 110 °C degraded **5** totally to the cubane complex [{Cr(cp)Se}$_4$], the ultimate decomposition product of all cyclopentadienyl chromium selenium complexes studied to date.[8,9]



Scheme 1 (*i*) Ph_2Se_2; (*ii*) 110°C

Reaction Pathways.—The variation of produts with reaction conditions and the results of thermolysis experiments, as described above, suggest reaction pathways illustrated in Scheme 1. As proposed for the corresponding reactions with

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† *Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1992, Issue 1, pp. xx–xxv.

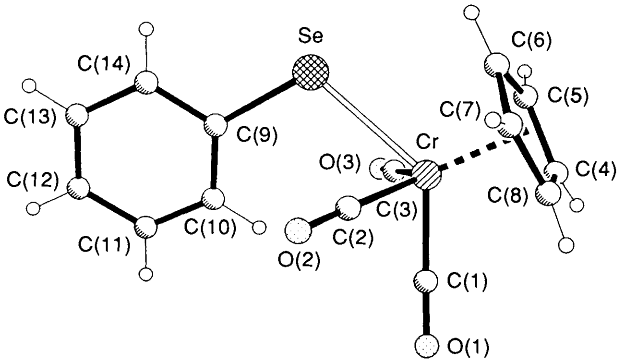

Fig. 1 Molecular structure of $[Cr(cp)(CO)_3(SePh)]$ **2**

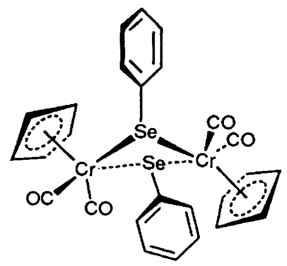

Fig. 2 Proposed molecular structure of $[\{Cr(cp)(CO)_2(SePh)\}_2]$ **3**

Se_8[8] and Ph_2S_2,[7] the facile nature of the reaction must originate from the presence of the 17e $[Cr(cp)(CO)_3]$ radical species in solution[10] and the susceptibility of the Se–Se bond in Ph_2Se_2 to radical-induced cleavage. Indeed, where dissociation of the dimeric complex does not occur, as in the case of $[\{Mo(L)(CO)_3\}_2]$, the reaction required 2 h at 10 °C for L = η^5-C_5H_5[3] or UV irradiation for L = η^5-C_5H_4Me.[4] The isolation of $[Cr(cp)(CO)_3(SePh)]$ **2** as the predominant product under mild reaction conditions, and $[\{Cr(cp)(CO)(SePh)\}_2]$ **4** and $[\{Cr(cp)(SePh)\}_2Se]$ **5** under more rigorous thermolytic conditions, indicates that **2** is the primary product. Evidence for this comes from independent thermolysis studies which show that **2** and **4** readily decarbonylate at moderate temperatures, resulting in the formation of **5**. An NMR spectral study of the thermolysis of **2** at 60 °C showed the formation of $[\{Cr(cp)(CO)_2(SePh)\}_2]$ **3** as a transient species. The Mo analogues of this tetracarbonyl species, $[\{Mo(L)(CO)_2(SePh)\}_2]$ (L = η^5-C_5H_5[3] or η^5-C_5H_4Me[4]), had been detected in the product solution from the reaction of $[\{Mo(L)(CO)_3\}_2]$ with Ph_2Se_2 in refluxing benzene, and under UV irradiation, respectively. In contrast to the present finding, it is noted that the mononuclear complex $[Cr(cp)(CO)_3(SPh)]$, the sulfur analogue of **2**, has not been isolated or detected in the reaction with Ph_2S_2, which produced $[\{Cr(cp)(CO)_2(SPh)\}_2]$ as the principal product.[7] Presumably, the higher electronegativity of S, compared to Se, will result in a large δ^+ change at the metal centre, and hence less π back bonding to the CO ligands, which therefore become less strongly bonded to the metal. Hence the ease of loss of CO from $[Cr(cp)(CO)_3(EPh)]$ with concomitant dimerisation to form $[\{Cr(cp)(CO)_2(EPh)\}_2]$ will be higher for E = S than for E = Se, as was observed.

The extremely low yield of $[\{Cr(cp)(CO)_2(SePh)\}_2]$ even at ambient temperature indicates its high tendency to decarbonylate further to **4**, a process which only required mild thermolysis *in vacuo* for the analogous molybdenum complexes.[4] Total decarbonylation leading to the formation of $[\{Cr(cp)(SePh)\}_2Se]$ **5** containing a Cr_2Se_3 core, as shown in Scheme 1, is reminiscent of the formation of $[\{Cr(cp)(SPh)\}_2S]$ from similar thermolysis reactions.[7] The structure of **5** is similar to that of the analogous sulfur complex and will be presented elsewhere. Complete decarbonylation of the molybdenum complexes has been reported to generate $[\{Mo(cp)(SePh)_2\}_n]$ as a highly insoluble solid.[3]

Table 1 Bond lengths (Å) and angles (°) of compound **2***

(*i*) Co-ordination geometry about Se atom			
Se–Cr	2.588(1)	Se–C(9)	1.911(4)
Cr–Se–C(9)	109.6(1)		
(*ii*) Co-ordination geometry about Cr atom			
Cr–C(1)	1.867(5)	Cr–C(2)	1.865(5)
Cr–C(3)	1.869(5)	Cr–C(4)	2.164(5)
Cr–C(5)	2.197(6)	Cr–C(6)	2.212(5)
Cr–C(7)	2.201(5)	Cr–C(8)	2.178(4)
Cr–Cp	1.837(5)	O(1)–C(1)	1.123(6)
O(2)–C(2)	1.147(7)	O(3)–C(3)	1.131(6)
Se–Cr–C(1)	130.9(2)	Se–Cr–C(2)	73.8(1)
C(1)–Cr–C(2)	80.7(2)	Se–Cr–C(3)	74.1(2)
C(1)–Cr–C(3)	82.3(2)	C(2)–Cr–C(3)	118.0(2)
Se–Cr–Cp	113.0(2)	C(1)–Cr–Cp	116.1(2)
C(2)–Cr–Cp	121.0(2)	C(3)–Cr–Cp	120.1(2)
Cr–C(1)–O(1)	178.7(4)	Cr–C(2)–O(2)	177.1(4)
Cr–C(3)–O(3)	176.4(5)		
(*iii*) Cyclopentadienyl group			
C(4)–C(5)	1.413(7)	C(4)–C(8)	1.405(7)
C(5)–C(6)	1.392(7)	C(6)–C(7)	1.409(8)
C(7)–C(8)	1.396(7)		
C(5)–C(4)–C(8)	108.0(4)	C(4)–C(5)–C(6)	107.5(4)
C(5)–C(6)–C(7)	108.6(4)	C(6)–C(7)–C(8)	107.8(4)
C(4)–C(8)–C(7)	108.0(4)		
(*iv*) Phenyl group			
C(9)–C(10)	1.391(6)	C(9)–C(14)	1.387(6)
C(10)–C(11)	1.330(7)	C(11)–C(12)	1.373(7)
C(12)–C(13)	1.382(8)	C(13)–C(14)	1.396(7)
Se–C(9)–C(10)	124.9(3)	Se–C(9)–C(14)	116.3(3)
C(10)–C(9)–C(14)	118.8(4)	C(9)–C(10)–C(11)	120.5(4)
C(10)–C(11)–C(12)	120.7(5)	C(11)–C(12)–C(13)	119.7(5)
C(12)–C(13)–C(14)	119.9(5)	C(9)–C(14)–C(13)	120.4(5)

* Cp represents centroid of cyclopentadienyl ring.

The high thermal lability of compounds **2** and **4**, as discussed above, strongly suggests that together with **3** they are the probable intermediates in the formation of $[\{Cr(cp)(SePh)\}_2Se]$ **5**, the sole product isolated from the reaction of $[\{Cr(cp)(CO)_2\}_2]$ with Ph_2Se_2 at elevated temperatures.

Structures.—The molybdenum analogue of complex **2** has been elementally and spectrally characterised.[3] Molecular structures of selenium complexes remain scarce. Hence we have determined that of **2** which is shown in Fig. 1. Bond lengths and angles are presented in Table 1. The Cr atom assumes a four-legged piano-stool geometry and achieves an 18-electron configuration. The Cr–Se distance (2.588 Å) is longer than the bridging Cr–Se (selenolate) distances in $[\{Cr(cp)(SePh)\}_2Se]$ (2.371–2.500 Å) and in $[\{Cr(cp)(SePh)\}_2Se\{Cr(CO)_5\}]$ (2.439–2.485 Å).[11] These Cr–Se bond distances are shorter than the sum of the single-bond radii of Cr (1.48 Å)[12] and Se (1.17 Å in α-monoclinic selenium[13] or 1.145 Å which is half the Se–Se bonded distance in Ph_2Se_2[14]).

In view of the exact match of the CO stretching frequencies of compound **3** with those of its sulfur analogue in the IR spectrum, it is very likely to possess a similar structure, as depicted in Fig. 2.[7] Since **4** is formed by partial decarbonylation of **3**, it would appear that it also possesses the same structural characteristics with two selenolate bridges, especially since these same bridges still persist in the thermolytic derivative **5**. However, the possible existence of bridging carbonyl ligands, or of metal–metal bonding, can only be ascertained *via* a single-crystal structural analysis, which has not been feasible so far.

Experimental

General Procedures and Physical Measurements.—Inert-atmosphere manipulation techniques, sources and preparation of reagents and solvents were as previously described.[15] Proton and ^{13}C NMR spectra were measured on a JEOL FX100 MHz spectrometer, and chemical shifts are relative to residual C_6H_6 in C_6D_6, $CHCl_3$ in $CDCl_3$ or to $SiMe_4$. Infrared spectra were measured on a Perkin-Elmer 1330 instrument. Elemental analyses were made by the Analytical Unit of the Research School of Chemistry, Australian National University, except for Cr which we analysed as CrO_4^{2-}.[16]

Reaction of $[\{Cr(cp)(CO)_3\}_2]$ **1** *with* Ph_2Se_2.—*At ambient temperature.* Diphenyl diselenide (155.3 mg, 0.50 mmol) was added to a stirred deep green suspension of compound **1** (200 mg, 0.50 mmol) in toluene (20 cm^3) at ambient temperature. Instantaneous reaction was indicated by an immediate change to a brown homogeneous solution. After 20 min the product solution was filtered through a Celite disk (*ca.* 1 cm thick). Concentration of the filtrate to *ca.* 3 cm^3, followed by addition of hexane and cooling at −78 °C for 30 min yielded brown crystals (*ca.* 270 mg), consisting mainly of **2** (described below) in admixture with small amounts of $[\{Cr(cp)(CO)_2(SePh)\}_2]$ **3** and another cp-containing species (δ 5.15).

The crystals were dissolved in toluene (2 cm^3) and chromatographed on a silica gel column (10 × 1.5 cm) prepared in hexane. Two fractions were eluted: (*i*) a brown eluate in hexane–toluene (9:1, 25 cm^3) which yielded brown crystals (25 mg, *ca.* 7% yield), of a 4:1 molar mixture of **3** [^1H NMR (C_6D_6) δ(cp) 4.56; IR (Nujol) ν(CO) at 1945vs, 1920vs, 1875vs and 1855s cm^{-1}] and an unidentified cp-containing compound (δ 5.15); and (*ii*) a brown eluate in hexane–toluene (1:1, 35 cm^3) which gave brown crystals (195 mg) of $[Cr(cp)(CO)_3(SePh)]$ **2**, slightly contaminated by **3** and the species with δ(cp) 5.15. Recrystallisation of this in toluene–hexane yielded pure brown crystals of **2** (155 mg, 0.43 mmol, 43.6% yield) {Found: C, 47.25; H, 2.75; Cr, 14.25; Se, 22.30. $[Cr(cp)(CO)_3(SePh)]$ requires C, 47.10; H, 2.85; Cr, 14.55; Se, 22.10%}. NMR: ^1H (C_6D_6), δ(cp) 4.04 and δ (C_6H_5) 6.97 and 7.84 (partially obscured by solvent peaks); ^{13}C ($CDCl_3$), δ(cp) 91.16, δ(C_6H_5) 136.00, 129.45, 128.44, 126.72 and δ(CO) 247.82, 236.43 (br). IR (Nujol): ν(CO) 2000vs, 1940vs and 1920vs; ν(cp) 850s cm^{-1}. Electron impact mass spectrum (for ^{80}Se): *m*/*z* 274, $[Cr(cp)(SePh)]$; 201, $[Cr(cp)(CO)_3]$; 173, $[Cr(cp)(CO)_2]$; 157, SePh; 145, $[Cr(cp)(CO)]$; and 117, Cr(cp).

At 50 °C. A deep green suspension of compound **1** (200 mg, 0.50 mmol) in toluene (20 cm^3) containing Ph_2Se_2 (155.3 mg, 0.50 mmol) was stirred for 3 h at 50 °C. The resultant brown suspension was filtered through Celite to collect the fine brown product, which was dissolved in thf and recrystallised with addition of hexane to yield fine brown crystals of $[\{Cr(cp)(CO)(SePh)\}_2]$ **4** (90 mg, 0.15 mmol, 30.0% yield) (Found: C, 45.75; H, 3.45; Cr, 17.30; Se, 25.40. $[\{Cr(cp)(CO)(SePh)\}_2]$ requires C, 47.85; H, 3.35; Cr, 17.25; Se, 26.25%). ^1H NMR ($CDCl_3$): δ(cp) 10.12 ($\nu_{\frac{1}{2}}$ 20 Hz) and δ(C_6H_5) 7.55, 7.52 (partially obscured by solvent peaks). IR (Nujol): ν(CO) 1890vs and 1760vs (br) cm^{-1}.

The mother-liquor was concentrated to *ca.* 1 cm^3 and loaded on to a silica gel column (7 × 1.5 cm) prepared in hexane. Elution gave three fractions: (*i*) a brown eluate in hexane–toluene (2:1, 15 cm^3) which yielded 32 mg of a 2:1 molar mixture of compounds **2** and **3** in admixture with *ca.* 10% of the species possessing δ(cp) 5.15; (*ii*) a brown fraction in hexane–toluene (1:1, 20 cm^3) which gave brown crystals of $[Cr(cp)(CO)_3(SePh)]$ **2** (102 mg, 0.29 mmol, 31.1%); and (*iii*) a bluish purple fraction in diethyl ether (15 cm^3) which yielded purple crystals of $[\{Cr(cp)(SePh)\}_2Se]$ **5** (35 mg, 0.05 mmol, 11.3% yield).

At 60 °C. A brown product solution resulting from the instantaneous reaction of compound **1** (200 mg, 0.50 mmol) with Ph_2Se_2 (155.3 mg, 0.50 mmol) in toluene (20 cm^3) was stirred at 60 °C for 2.5 h. The dark purple product solution was then filtered through a 1 cm thick Celite disk, concentrated to *ca.* 1 cm^3 and loaded on to a silica gel column (7 × 1.5 cm) prepared in hexane. Elution gave three fractions: (*i*) a solution in hexane (20 cm^3) which gave a green oil (13 mg) of unknown composition; (*ii*) a bluish purple solution in ether (15 cm^3) which gave purple crystals of $[\{Cr(cp)(SePh)\}_2Se]$ **5** (142 mg, 0.22 mmol, 45.6%) (Found: C, 43.05; H, 3.30; Cr, 16.60; Se, 38.05. $[\{Cr(cp)(SePh)\}_2Se]$ requires C, 42.25; H, 3.25; Cr, 16.65; Se, 37.90%) and (*iii*) a purple solution in diethyl ether–thf (1:1, 6 cm^3) which gave a purplish black oily uncharacterisable residue (20 mg). NMR: ^1H (C_6D_6) δ(cp) 15.91 ($\nu_{\frac{1}{2}}$ 66 Hz); ^{13}C (CD_2Cl_2), δ(cp) 99.22. IR (Nujol): ν(cp) 800m cm^{-1}; other bands at 750m, 720m, 690w and 665vw cm^{-1}. Fast atom-bombardment mass spectrum: *m*/*z* 628, $[\{Cr(cp)(SePh)\}_2Se]$; 471, $[\{Cr_2(cp)_2(SePh)\}Se]$; 314, Se_2Ph_2; 234, $[Cr_2(cp)_2]$; 157, SePh; and 117, Cr(cp).

Thermolysis Reactions.—$[Cr(cp)(CO)_3(SePh)]$ **2**. A stirred brown solution of compound **2** (100 mg, 0.28 mmol) in toluene (5 cm^3) was maintained at 60 °C for 1.5 h. The resultant dark purple solution was filtered through Celite, loaded on to a silica gel column (6 × 1 cm) and eluted with ether (10 cm^3). From the bluish purple eluate were obtained fine purple crystals of $[\{Cr(cp)(SePh)\}_2Se]$ **5** (50 mg, 0.08 mmol, 57.1%). In a separate experiment the reaction was followed in an NMR tube to detect the formation of intermediate species.

$[\{Cr(cp)(CO)(SePh)\}_2]$ **4**. A reddish brown saturated solution of compound **4** in thf in a 5 mm tube was maintained at 50 °C for 2 h. Examination of the resultant purple solution by TLC on a silica gel plate showed only the presence of **5** (R_f = 0.53 with toluene as eluent).

$[\{Cr(cp)(SePh)\}_2Se]$ **5**. A bluish purple saturated solution of compound **5** in [$^2H_8$]toluene in a 5 mm NMR tube was maintained at 110 °C. The reaction which took *ca.* 20 h was monitored by the disappearance of its cp resonance. This was accompanied by the growth of the cp resonance (δ 5.13)[17] of $[\{Cr(cp)Se\}_4]$, as well as the precipitation of this product on the walls and bottom of the tube. The precipitate was also identified *via* its mass spectrum.[17]

Reaction of $[\{Cr(cp)(CO)_2\}_2]$ *with* Ph_2Se_2.—No reaction was observed at ambient temperature. At 60 °C a deep green solution of $[\{Cr(cp)(CO)_2\}_2]$ (100 mg, 0.29 mmol) in toluene (8 cm^3) containing Ph_2Se_2 (90.17 mg, 0.29 mmol) turned to blackish green after 4.5 h. The product solution was filtered through a Celite disk and concentrated to *ca.* 1 cm^3. Chromatography on a silica gel column (6 × 1.5 cm) gave two fractions: (*i*) a green solution in hexane–toluene (1:1, 40 cm^3) which yielded green crystals of unreacted $[\{Cr(cp)CO)_2\}_2]$ (47 mg, 47%); and (*ii*) a bluish purple solution in ether (30 cm^3) which yielded purple crystals of compound **4** (90 mg, 0.14 mmol, 49.8%).

Crystal Structure Determination.—Diffraction-quality dark brown chunky cuboidal crystals of compound **2** were obtained from toluene–hexane, after 3 h at −30 °C. Details of the crystal parameters, data collection, and structure refinement are given in Table 2. Raw intensities collected on a Nicolet R3m/V four-circle diffractometer at room temperature (294 K) were processed with the profile-fitting procedure of Diamond[18] and corrected for absorption using ψ-scan data.[19] Patterson superposition yielded the positions of the chromium and selenium atoms. The coordinates of the other non-hydrogen atoms were derived from successive Fourier difference syntheses. The non-hydrogen atoms were subjected to anisotropic refinement and the H atoms were generated geometrically (C–H bonds fixed at 0.96 Å) and assigned the same isotropic thermal parameter of U = 0.08 Å^2. Computations were performed using the SHELXTL-PLUS program package[20] on a DEC MicroVAX-II computer. Analytic

Table 2 Data collection and processing parameters

M	357.20
Colour and habit	Dark brown plate
Crystal dimensions/mm	0.24 × 0.42 × 0.44
Crystal system	Monoclinic
Space group	$P2_1/c$ (no. 14)
a/Å	10.401(3)
b/Å	8.701(2)
c/Å	15.604(4)
β/°	99.51(2)
U/Å^3	1392.7(5)
Z	4
F(000)	704
D_c/g cm^{-3}	1.70
Radiation	Graphite-monochromatized Mo-Kα (λ = 0.710 73 Å)
Standard reflections	(231), (313)
Intensity variation	±2%
R_{int} (from merging of equivalent reflections)	0.031
μ/cm^{-1}	33.88
Mean μr	0.621
Transmission factors	0.154–0.308
Scan type, rate/° min^{-1}	ω, 3.01–15.63
Scan range	0.60° below $K\alpha_1$ to 0.70° above $K\alpha_2$
Background counting	Stationary counts for one-fifth of scan time at each end of scan range
Collection range	$h,k,\pm l$; $2\theta_{max} = 55°$
Unique data measured	3173
Obs. data with $\lvert F_o\rvert \geqslant 6\sigma(\lvert F_o\rvert)$, n	2037
No. of variables, p	172
$R_F = \Sigma\lVert F_o - \lvert F_c\rVert/\Sigma\lvert F_o\rvert$	0.042
Weighting scheme	$[\sigma^2(F_o) + 0.0005\lvert F_o\rvert^2]^{-1}$
$R_G = [\Sigma w(\lvert F_o\rvert - \lvert F_c\rvert)^2/\Sigma w\lvert F_o\rvert^2]^{\frac{1}{2}}$	0.050
$S = [\Sigma w(\lvert F_o\rvert - \lvert F_c\rvert)^2/(n - p)]^{\frac{1}{2}}$	1.492
Residual extrema in final difference map/e Å^{-3}	+0.30 to −0.61

Table 3 Atomic coordinates (×10^5 for Se and Cr, ×10^4 for other atoms) for compound **2**

Atom	x	y	z
Se	10 685(4)	− 627(5)	37 435(4)
Cr	23 262(6)	−25 034(7)	43 463(4)
O(1)	5 126(4)	−3 387(5)	4 450(3)
O(2)	2 517(4)	−2 424(4)	2 442(2)
O(3)	3 475(5)	40(4)	5 526(3)
C(1)	4 078(5)	−3 037(6)	4 412(3)
C(2)	2 452(5)	−2 414(5)	3 169(3)
C(3)	3 041(5)	−888(6)	5 059(3)
C(4)	2 153(5)	−4 187(6)	5 342(3)
C(5)	1 150(5)	−3 092(6)	5 347(3)
C(6)	342(5)	−3 165(6)	4 542(3)
C(7)	828(5)	−4 295(6)	4 034(3)
C(8)	1 941(5)	−4 931(5)	4 531(3)
C(9)	2 211(4)	1 279(5)	3 257(3)
C(10)	3 564(5)	1 174(5)	3 407(3)
C(11)	4 308(5)	2 236(6)	3 050(3)
C(12)	3 727(6)	3 392(6)	2 525(3)
C(13)	2 384(6)	3 515(6)	2 366(3)
C(14)	1 624(5)	2 451(5)	2 729(3)

expressions of atomic scattering factors were employed, and anomalous dispersion corrections were incorporated.[21] Atomic coordinates of non-hydrogen atoms are listed in Table 3.

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom coordinates and thermal parameters.

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