thione S atom. The same behaviour is found in the Pd^{II} complex (Kubiak & Głowiak, 1982).

The geometry of ttz appears to be essentially unchanged on coordination. The bond lengths and angles do not differ significantly from those of uncoordinated 1,3-thiazolidine-2-thione (Raper *et al.*, 1983), but the 4,5-dihydrothiazole ring is less planar with deviations from the least-squares plane through S(1)S(2)C(1)N being −0·340 (12) and 0·182 (12) Å for C(2) and C(3) respectively $\{\Delta C_2^{[C(2),C(3)]} = 3{\cdot}2°$ (Duax, Weeks & Rohrer, 1976)}.

One of the interesting features of this crystal structure is the intramolecular hydrogen bond between the heterocyclic N atom and equatorial $Cl(2^{iii})$ $[N\cdots Cl(2^{iii})$ 3·343 (8) Å and $N-H(1)\cdots Cl(2^{iii})$ 158°].

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Structure of *mer*-Dicarbonyl(selenocarbonyl)tris(trimethyl phosphite)chromium(0)

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Abstract. $[Cr(CO)_2(CSe)\{P(CH_3O)_3\}_3]$, $M_r = 403{\cdot}157$, orthorhombic, *Pbca*, $a = 15{\cdot}483\ (3)$, $b = 15{\cdot}213\ (5)$, $c = 18{\cdot}997\ (3)$ Å, $V = 4474{\cdot}6$ Å^3, $Z = 8$, $D_x = 2{\cdot}396$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 46{\cdot}515$ cm^{-1}, $F(000) = 272$, $T = 118$ K, $R = 0{\cdot}042$ for 3091 observed reflections. The complex has meridional octahedral stereochemistry with the two CO groups *trans* to each other. Important structural features are Cr—C—Se = 176·9 (2)°, Cr—C(Se) = 1·785 (3), C—Se = 1·750 (3), mean Cr—C(O) = 1·893 (3), Cr—P (*trans* to CSe) = 2·373 (1), mean Cr—P = 2·282 (1) Å. The variations in bond lengths observed compared with those found in similar carbonyl and/or thiocarbonyl complexes illustrate the much better electron-withdrawing capacity of CSe compared to CO and CS.

Introduction. Transition-metal carbonyls have been known since the late 19th century (Mond, Langer & Quinke, 1890), but the first examples of analogous metal thio- and selenocarbonyls were only reported quite recently (Butler, 1977). While the structural behaviour of the CS ligand is quite well understood, there have been only two X-ray studies reported for selenocarbonyls: $(\eta\text{-}C_6H_5CO_2CH_3)Cr(CO)_2(CSe)$ (Saillard & Grandjean, 1978) and $RuCl_2(CO)(CSe)[(C_6H_5)_3P]_2$ (Clark, Grundy, Harris, James & Roper, 1975). Structural information, particularly from X-ray data, is important for the CSe ligand because, unlike CO and CS, the diatomic CSe molecule has eluded isolation even at very low temperatures (Steudel, 1967).

During the course of a kinetic investigation of arene substitution by tertiary phosphines and phosphites in arene chromium chalcocarbonyl complexes (equation 1), it became important for us to know the stereochemistry (*fac* or *mer*) of the final products in these reactions in order to rationalize the mechanism.

$$(\eta\text{-Arene})Cr(CO)_2(CX) + 3L \rightarrow L_3Cr(CO)_2(CX) + \text{arene} \quad (1)$$

[arene = C_6H_6, $C_6H_5CO_2CH_3$, *etc.*; X = S, Se; L = $(C_6H_5)_3P$, $(CH_3O)_3P$, *etc.*].

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We report here the crystal and molecular structure of a typical selenocarbonyl product, $[(CH_3O)_3P]_3$-$Cr(CO)_2(CSe)$. In an earlier paper, the spectroscopic properties (IR; ^1H, ^{13}C, ^{31}P NMR) and X-ray structure of the thiocarbonyl analogue were described (Bird, Ismail & Butler, 1985).

Experimental. Orange crystals from the reaction of $(\eta$-$C_6H_6)Cr(CO)_2(CSe)$ with excess $(CH_3O)_3P$ in toluene at 338 K for 12 h were recrystallized from dichloromethane/pentane (1/2). Since very few crystals were available, a crystal of not very well defined shape had to be used; density not measured; Enraf–Nonius CAD-4 diffractometer at 118 K (low-temperature accessory: developed by J-JB and SA in 1983)* coupled to a VAX *SDP* computing system (Enraf–Nonius, 1982); lattice parameters by least-squares refinement of 25 medium-angle settings; 4539 reflections measured, $2\theta_{max} = 25°$, range of hkl: $h\,0\rightarrow16$, $k\,0\rightarrow18$, $l\,0\rightarrow22$, $\omega/2\theta$ scan, graphite-monochromated Mo $K\alpha$ radiation; four standard reflections (226, 0,0,10, 460, 800): 1% variation (72·1 h exposure); 4398 unique reflections, 3091 with $I > 3\sigma(I)$ used; intensities corrected for Lorentz–polarization, and absorption (absmin. = 0·8756, absmax. = 0·9973); structure solved by Patterson and Fourier methods using the atomic coordinates for the isostructural thiocarbonyl complex (Bird *et al.*, 1985) as starting values; full-matrix least-squares anisotropic refinements on F (254 variables) with the H atoms being included in the structure-factor calculations but not refined; $R = 0{\cdot}042$, $wR = 0{\cdot}061$, $w^2 = 1/\sigma^2$, $(\Delta/\sigma)_{max} = 0{\cdot}17$, no correction for secondary extinction, atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. The final atomic coordinates are given in Table 1,† while the structurally significant bond distances and angles are listed in Table 2. A perspective view of the structure indicating the atom-labelling scheme used is shown in Fig. 1. The coordination around the central Cr atom is essentially octahedral. The complex has meridional stereochemistry with two *trans* CO groups, two *trans* $(CH_3O)_3P$ groups and the CSe ligand *trans* to the remaining $(CH_3O)_3P$ ligand. The CSe is bonded to the Cr through the C atom and the Cr—C—Se linkage is linear [176·9 (2)°] with bond distances Cr—C = 1·785 (3), C—Se = 1·750 (3) Å. These values for the selenocarbonyl linkage are much more accurate than those in the literature from the room-temperature studies on $(\eta$-$C_6H_5CO_2CH_3)$-$Cr(CO)_2(CSe)$ [Cr—C—Se = 179·0 (7)°, Cr—C = 1·79 (1), C—Se = 1·73 (1) Å; Saillard & Grandjean, 1978] and $RuCl_2(CO)(CSe)[(C_6H_5)_3P]_2$ [Ru—C—Se = 174 (1)°, C—Se = 1·70 (2) Å; Clark *et al.*, 1975].

The only other X-ray diffraction studies reported for chromium carbonyl complexes containing trimethyl phosphite ligands are for $\{(\eta$-$C_5H_5)Cr(CO)_2$-$[(CH_3O)_3P]\}_2$ and $(\eta$-$C_5H_5)Cr(CO)_2[(CH_3O)_2PO]$-$[(CH_3O)_3P]$ (Goh, D'Aniello, Slater, Muetterties,

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Cr	0·19914 (5)	0·38146 (5)	0·38628 (4)	0·74 (1)
P(1)	0·32976 (8)	0·41673 (9)	0·43460 (7)	0·90 (2)
P(2)	0·24156 (8)	0·44238 (8)	0·27659 (7)	0·89 (2)
P(3)	0·06939 (8)	0·33459 (9)	0·34383 (7)	0·88 (2)
C(1)	0·1646 (3)	0·3461 (3)	0·4713 (3)	1·2 (1)
Se	0·12779 (4)	0·31681 (4)	0·55521 (3)	1·61 (1)
C(2)	0·2489 (3)	0·2733 (4)	0·3587 (3)	1·2 (1)
O(2)	0·2804 (3)	0·2085 (3)	0·3405 (2)	2·16 (9)
C(3)	0·1546 (3)	0·4943 (4)	0·4080 (3)	1·2 (1)
O(3)	0·1271 (3)	0·5609 (3)	0·4243 (2)	2·01 (8)
O(11)	0·3453 (2)	0·5121 (2)	0·4688 (2)	1·27 (7)
C(11)	0·2989 (4)	0·5360 (4)	0·5327 (3)	2·0 (1)
O(12)	0·4090 (2)	0·4127 (2)	0·3809 (2)	1·25 (7)
C(12)	0·4976 (3)	0·4240 (4)	0·4051 (3)	1·6 (1)
O(13)	0·3628 (2)	0·3607 (2)	0·5014 (2)	1·40 (7)
C(13)	0·3661 (4)	0·2660 (4)	0·4977 (3)	2·0 (1)
O(21)	0·2994 (2)	0·3850 (2)	0·2240 (2)	1·32 (7)
C(21)	0·2640 (4)	0·3129 (4)	0·1844 (3)	1·7 (1)
O(22)	0·3010 (2)	0·5266 (2)	0·2872 (2)	1·27 (7)
C(22)	0·3397 (4)	0·5708 (4)	0·2266 (3)	1·9 (1)
O(23)	0·1705 (2)	0·4750 (2)	0·2203 (2)	1·22 (7)
C(23)	0·1113 (4)	0·5433 (4)	0·2388 (3)	1·9 (1)
O(31)	−0·0114 (2)	0·4014 (2)	0·3434 (2)	1·35 (7)
C(31)	−0·0469 (4)	0·4317 (4)	0·4086 (3)	1·7 (1)
O(32)	0·0211 (2)	0·2542 (2)	0·3829 (2)	1·26 (7)
C(32)	0·0650 (4)	0·1732 (4)	0·3997 (3)	1·5 (1)
O(33)	0·0691 (2)	0·3051 (2)	0·2630 (2)	1·25 (7)
C(33)	−0·0079 (4)	0·2688 (4)	0·2306 (3)	1·7 (1)

Table 2. *Selected bond lengths* (Å) *and angles* (°) *for mer-*$[(CH_3O)_3P]_3Cr(CO)_2(CSe)$ *with e.s.d.'s in parentheses*

Cr—P(1)	2·285 (1)	C(1)—Se	1·750 (3)
Cr—P(2)	2·373 (1)	C(2)—O(2)	1·153 (4)
Cr—P(3)	2·279 (1)	C(3)—O(3)	1·142 (4)
Cr—C(1)	1·785 (3)	P—O	1·590–1·614 (2)
Cr—C(2)	1·891 (3)	O—C	1·430–1·461 (4)
Cr—C(3)	1·895 (3)		

C(1)—Cr—C(2)	96·4 (1)	C(3)—Cr—P(3)	92·3 (1)
C(1)—Cr—C(3)	88·1 (1)	P(1)—Cr—P(2)	90·92 (3)
C(1)—Cr—P(1)	88·4 (1)	P(1)—Cr—P(3)	174·75 (4)
C(1)—Cr—P(2)	174·5 (1)	P(2)—Cr—P(3)	93·18 (1)
C(1)—Cr—P(3)	87·8 (1)	Cr—C(1)—Se	176·9 (2)
C(2)—Cr—C(3)	175·3 (1)	Cr—C(2)—O(2)	178·8 (3)
C(2)—Cr—P(1)	87·4 (1)	Cr—C(3)—O(3)	176·7 (3)
C(2)—Cr—P(2)	89·0 (1)	Cr—P—O	111·34–120·91 (9)
C(2)—Cr—P(3)	89·4 (1)	O—P—O	96·4–103·8 (1)
C(3)—Cr—P(1)	91·3 (1)	P—O—C	119·2–122·0 (2)
C(3)—Cr—P(2)	86·5 (1)		

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* This device has been designed for routine low-temperature data collection; it uses a low dry-nitrogen flow (2 l min^{-1}), cooled in a liquid-nitrogen Dewar. The temperature is controlled by a fine low-power electronic regulator and the long-term temperature stability is better than 0·3 K.

† Complete lists of H-atom coordinates, bond lengths, bond angles, structure factors, anisotropic thermal parameters and r.m.s. amplitudes of thermal vibration have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42335 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Tavanaiepour, Chang, Fredrich & Day, 1979). In both cases, the Cr—P distance is 2·238 (1) Å, considerably shorter than the Cr—P distances in our study. However, the geometrical structures concerned are markedly different from each other.

The Cr—C(Se) distance in the present case is 0·108 (1) Å shorter than the mean Cr—C(O) distance [1·893 (3) Å] and the Cr—P distance for the $(CH_3O)_3P$ ligand *trans* to CSe is 0·091 (1) Å longer than the mean Cr—P value [2·282 (1) Å] for the other two $(CH_3O)_3P$ ligands. Similar results were obtained from the room-temperature X-ray analyses of the isomorphous thiocarbonyl complex, *mer*-$[(CH_3O)_3P]_3Cr(CO)_2(CS)$, and $(\eta\text{-}C_6H_5CO_2CH_3)Cr(CO)_2(CX)$ (X = S, Se). The pertinent bond distances are compared in Table 3. A direct comparison is not possible because no X-ray data are available for the analogous tricarbonyl complex, *mer*-$[(CH_3O)_3P]_3Cr(CO)_3$, but the observed shortenings in the Cr—C(X) bonds and lengthenings in the Cr—P bonds *trans* to CX are best attributed to the better electron-withdrawing capacities of the CS and CSe ligands compared to CO. Furthermore, the lengthening in the Cr—P bond *trans* to CSe is about 0·02 Å more than the corresponding lengthening for the thiocarbonyl complex suggesting that, if anything, CSe is slightly better than CS in terms of electron-withdrawing capacity. The Cr—(arene ring) distances in Table 3 for the arene complexes also point to the same conclusion. Our observations are in complete accord with spectroscopic studies comparing metal thiocarbonyls and selenocarbonyls, *e.g.* normal coordinate calculations (English, Plowman & Butler, 1981, 1982), absolute IR intensities of the CX stretching modes (Baibich, Butler & English, 1984), ^{13}C and ^{17}O NMR and PES measurements (Butler, 1977) and also *ab initio* molecular-orbital calculations for the free CO, CS, and CSe ligands and when coordinated in $(\eta\text{-}C_6H_5CO_2CH_3)Cr(CO)_2(CX)$ (Saillard, Grandjean, Caillet & Le Beuze, 1980). Finally, a similar very strong electron-withdrawing effect was found for CSe in $RuCl_2(CO)(CSe)[(C_6H_5)_3P]_2$ since the Ru—Cl distance *trans* to CSe is 2·480 (5) Å, while the one *trans* to CO is 2·427 (5) Å (Clark *et al.*, 1975).

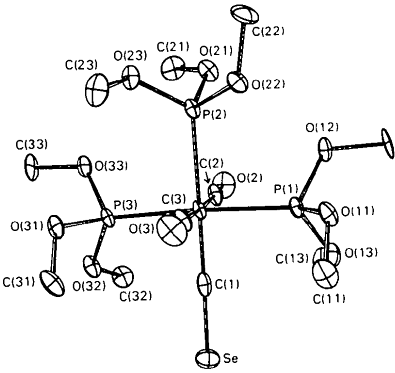

Fig. 1. Perspective view of *mer*-$[(CH_3O)_3P]_3Cr(CO)_2(CSe)$ with H atoms omitted for clarity. The H atoms are in a staggered conformation with respect to the O–C axes of the phosphite ligands.

Table 3. *Selected bond distances* (Å) *for similar chromium*(0) *complexes with e.s.d.'s in parentheses*

Bond type	*mer*-$[(CH_3O)_3P]_3$-$Cr(CO)_2(CX)$		$(\eta\text{-}C_6H_5CO_2CH_3)$-$Cr(CO)_2(CX)$	
	$X = S^a$	$X = Se^b$	$X = S^c$	$X = Se^d$
Cr—C(X)	1·782 (9)	1·785 (3)	1·797 (2)	1·786 (1)
Cr—C(O)	1·839 (9)	1·893 (3)	1·848 (3)	1·862 (12)
C—X	1·585 (9)	1·750 (3)	1·570 (2)	1·728 (10)
C—O^e	1·153 (11)	1·148 (4)	1·157 (3)	1·150 (15)
Cr—P (*trans* to CX)	2·346 (3)	2·373 (1)		
Cr—P	2·263 (3)	2·282 (1)		
Cr—(ring)^f			1·730 (1)	1·742 (1)

(*a*) Bird *et al.* (1985). (*b*) This work. (*c*) Saillard & Grandjean (1976). (*d*) Saillard & Grandjean (1978). (*e*) Mean value. (*f*) For the tricarbonyl complex, this value is 1·714 (1) Å (Saillard, Leborgne & Grandjean, 1975).

It should be mentioned in concluding that while we consider electronic effects in the chalcocarbonyl ligands to be the most important factor affecting bond lengths of groups *trans* to them, there does appear to be some steric effect since the Cr—C(O) distances in the $[(CH_3O)_3P]_3Cr(CO)_2(CX)$ complexes increase by about 0·045 Å in going from S to Se.

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Structure of Bis(triethanolamine)bis(2,4,6-trinitrophenolato)barium(II)*

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Abstract. $[\mathrm{Ba(C_6H_2N_3O_7)_2(C_6H_{15}NO_3)_2}]$, $M_r = 891\cdot9$, monoclinic, $C2/c$, $a = 21\cdot704\,(3)$, $b = 8\cdot0224\,(6)$, $c = 22\cdot650\,(4)$ Å, $\beta = 122\cdot30\,(1)^\circ$, $V = 3333\cdot5\,(9)$ Å^3, $Z = 4$, $D_x = 1\cdot777$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 11\cdot83$ cm^{-1}, $F(000) = 1800$, $T = 294$ K, $R = 0\cdot0338$ for 2837 observed reflections. Ba lies on a twofold axis and is coordinated to eight O and two N atoms which form a bicapped distorted cube. The N atom and three O atoms of triethanolamine (tea) and one O of the *p*-nitro group of the picrate (tnp) are involved in the coordination. The tea ligand is disordered over two positions which are approximately related by pseudo mirror planes through N and each of the terminal C atoms, with occupancies of 0·637 (7) and 0·363 (7). Hydrogen bonds from the three tea OH groups to two neighbouring tnp ligands link the Ba complexes in extensive sheets. Two hydrogen bonds are bifurcated and the phenolate O accepts three hydrogen bonds.

Introduction. Recently we reported the structure analyses of two complexes of Ba$^{\mathrm{II}}$: Ba(dnp)$_2$(phen)$_3$ (Kanters, Postma, Duisenberg, Venkatasubramanian & Poonia, 1983) and Ba(tnp)$_2$(phen)$_2$ (Postma, Kanters, Duisenberg, Venkatasubramanian & Poonia, 1983) (dnp = 2,4-dinitrophenolato, tnp = 2,4,6-trinitrophenolato, phen = 1,10-phenanthroline). In order to study the relative binding capacities of nitrophenolates and triethanolamine we also determined the structure of Ba(dnp)$_2$(tea)$_2$ (Kanters, Smeets, Venkatasubramanian & Poonia, 1984). In the latter compound the N and O atoms of both tea ligands are coordinated to Ba, whereas only one *o*-nitro-group O atom of one of the dnp ligands contributes to the coordination. The powerful binding capacity of triethanolamine in the presence of 2,4,6-trinitrophenolate is studied in the structure analysis of the title compound.

Experimental. Crystals obtained from the Chemistry Department, University of Indore, India. Rod-shaped, orange-reddish crystal $0\cdot6 \times 0\cdot3 \times 0\cdot2$ mm. Enraf–Nonius CAD-4 diffractometer. Cell constants from least-squares fit of positions of 25 reflections with θ range $11\cdot2$–$18\cdot6^\circ$. ω–2θ scan, $\omega = (0\cdot60 + 0\cdot35\tan\theta)^\circ$, $\sigma(I)/(I) < 1\%$ or a maximum scan time of 60 s, horizontal and vertical slit widths 3 and 4 mm respectively, Zr-filtered Mo $K\alpha$ radiation. One quadrant, h 0–26, k 0–9, l −27–23, $\theta_{\max} = 26^\circ$. 3143 unique reflections, 2837 above $2\cdot5\sigma(I)$ level. Three standard reflections, measured every hour of X-ray exposure, showed an average fluctuation of 0·9%. Intensities corrected for Lp effects, but not for absorption. Ba position from Patterson synthesis, non-H atoms of tnp, N and three terminal C atoms of tea from Fourier maps. Six maxima appeared around N, the densities of which fell into two groups with a ratio of about 2:1. The same feature was observed near the terminal C atoms where three pairs of close-lying maxima were found. This pattern was interpreted as a disorder of tea over two positions. Accordingly, the two groups of positions of the disordered C and O atoms were refined with occupancy factors p and $(1-p)$ [refined value, $p = 0\cdot637\,(7)$]. The two H atoms of tnp were located from difference maps. The H atoms of tea were placed

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* Triethanolamine is 2,2′,2″-nitrilotriethanol.

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