Carbon–Tellurium Bond Cleavage in Tellurolatochromium Complexes. Synthesis and Single-crystal Structures of $[Cr_4(cp)_4Te_nO_{4-n}]$ (cp = η^5-C_5H_5, n = 1–3)†

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The exhaustive thermolysis of $[Cr(cp)(CO)_3(TePh)]$ (cp = η^5-C_5H_5) in toluene for 48 h at 80 °C led to the isolation of $[Cr_4(cp)_4Te_7]$ (*ca.* 24%), $[Cr_4(cp)_4Te_3O]$ (14.4%), $[Cr_4(cp)_4Te_2O_2]$ (37.2%) and $[Cr_4(cp)_4TeO_3]$ in minute amounts. Thin-layer chromatography and proton NMR spectral monitoring showed the formation of $[Cr_4(cp)_4Te_3O]$ and $[Cr_4(cp)_4Te_2O_2]$ from the intermediate $[\{Cr(cp)(TePh)\}_2Te]$. Under thermolytic conditions, $[Cr_4(cp)_4Te_3O]$ and $[Cr_4(cp)_4Te_2O_2]$ undergo reversible transformation with simultaneous formation of $[Cr_4(cp)_4TeO_3]$. The structures of three of the complexes were established by X-ray diffraction analysis.

Pyrolytic decarbonylation of transition-metal carbonyl complexes, accompanied by condensation and transformations, often generates clusters of unexpected geometries and structures.[1–7] In particular, the presence of sulfide ligand with its multi-co-ordination and donor capacity has facilitated the designed assembly of smaller complexes to give higher-nuclearity cluster compounds with complex and unusual structural features.[8,9] Our own studies have shown that self-condensation of some chalcogenido complexes under thermolytic conditions generates cubane-like complexes $[Cr_4(cp)_4E_2(CO)_2]$, $[Cr_4(cp)_4E_4]$ (E = S[10–12] or Se[13,14]) and $[Cr_4(cp)_4Se_2O_2]$ (cp = η^5-C_5H_5).[14] Thermolytic degradations of dichromium carbonyl complexes containing Group 15 atoms have led to the isolation of triple-decker $[Cr_2(cp)_2E_5]$ (E = P[15] and As[16]) sandwich compounds, as was also implicated in Scherer's cothermolysis of $[\{Cr(\eta^5\text{-}C_5Me_5)(CO)_2\}_2]$ with P_4[17] and As_4[18b] and $[\{Cr(\eta^5\text{-}C_5H_4R)(CO)_3\}_2]$ (R = H or Me) with As_4.[18a] In a recent study on the thiolato- and selenolato-bridged dichromium complexes $[\{Cr(cp)(CO)_2(EPh)\}_2]$ (E = S or Se) we observed that controlled thermolysis yielded the chalcogenido- and chalcogenolato-bridged complexes $[\{Cr(cp)(EPh)\}_2E]$ (E = S or Se), while exhaustive thermolysis gave the cubanes $[Cr_4(cp)_4E_4]$ (E = S[19] or Se[20,21]). Here we describe the thermolytic conversion of $[Cr(cp)(CO)_3(TePh)]$ *via* $[\{Cr(cp)(TePh)\}_2Te]$ into a mixture of complexes $[Cr_4(cp)_4Te_nO_{4-n}]$ (n = 1–3) together with their crystal structures.

Results and Discussion

Thermolytic Synthesis.—A brownish green solution of $[Cr(cp)(CO)_3(TePh)]$ **2**, prepared *in situ* from the instantaneous reaction of $[\{Cr(cp)(CO)_3\}_2]$ **1** with Ph_2Te_2, was transformed after 8 h at 80 °C to a dark green solution containing mainly $[\{Cr(cp)(TePh)\}_2Te]$ **4**. During this interval the presence of $[\{Cr(cp)(CO)_2(TePh)\}_2]$ **3** was detected, owing to the interconversion between **2** and **3** under thermolytic conditions.[22] After 40 h more at 80 °C a dark coloured suspension was produced. Filtration removed an insoluble grey-black solid (24% yield, based on chromium analysis), the elemental analysis of which conforms to $[Cr_4(cp)_4Te_7]$ **5**. The proton NMR spectrum of the mother-liquor showed **6** and **7** as the major components. Chromatographic work-up separated some unreacted Ph_2Te_2, and trace amounts of **2**–**4** from $[Cr_4(cp)_4Te_3O]$ **6** and $[Cr_4(cp)_4Te_2O_2]$ **7** which were both isolated as dark green crystals in 14.4 and 37.2% yields, respectively, and $[Cr_4(cp)_4TeO_3]$ **8** which was isolated as a few dark blue crystals.

Reaction Pathways.—The thermolytic transformations described above are illustrated in Scheme 1. The formation of the Cr_2Te_3 framework in **4** and the oxotelluride cubanes **6**–**8** indicates the occurrence of C–Te bond cleavage in the tellurolate ligands, a phenomenon as yet unprecedented as far as we are aware. In contrast, metal-induced cleavage of C–S bonds in thiolate ligands is well documented, *e.g.* in osmium complexes,[23] metallic hydrogensulfides,[24] in the reaction of $[Co_2(CO)_8]$ with $PhCH_2SH$[25] and of chromocenes with Me_3CSH.[26] The incidence of C–Se bond cleavage remains scarce.[27] It is noted that the sulfur and selenium analogues of the monooxo cubane **6**, dioxo cubane **7**, and trioxo cubane **8** have not been obtained from thermolysis of the analogous systems.[19–21] The formation of the oxo complexes **6**–**8** from the 'non-oxygen' compound **4** has precedents in the formation of $[Cr_4(cp)_4Se_2(\mu_3\text{-}O)_2]$ from $[Cr_2(cp)_2(CO)_4Se]$,[14] $[Mo_3(cp)_3(\mu_3\text{-}O)]$-type complexes from $[\{Mo(cp)(CO)_3\}_2]$[28] and $[Nb_3(\eta\text{-}C_5H_4Bu^t)_3S_{10}(\mu_3\text{-}O)]$ from $[Nb_2(\eta\text{-}C_5H_4Bu^t)_4S_n]$ (n = 8 or 9).[29] In these reactions, as well as in the transformation of **6** to a mixture of **7** and **8**, and of **7** to a mixture of **6** and **8** (observed in the NMR-tube reactions), one can only speculate on the extraneous source of oxygen, possibly the glass walls of the reaction vessel.

Although $[\{Cr(cp)(EPh)\}_2E]$ (E = S or Se) readily yields $[Cr_4(cp)_4E_4]$ as the sole product on exhaustive thermolysis,[19–21] the formation of the analogous Te_4 complex cannot be confirmed, despite mass-spectral indication of its presence in the product **5**. To date Te_4 cubane complexes are, indeed, rare despite the large numbers of soluble tellurides containing the Te_4 unit.[30] Only three cases have been reported, namely $[Fe_4Te_4(CO)_{12}]$[31] which was 'assumed cubic' by analogy to the isoelectronic complexes of Sb and Bi,[32] followed by $[Fe_4Te_4(TePh)_4]^{3-}$, the first telluridotellurolato complex[33] and $[Ga_4Bu^t_4Te_4]$.[34]

Properties and Spectral Characteristics.—Both complexes **6** and **7** exist as dark green crystals, which dissolve in organic solvents to give dirty green solutions. Complex **8** forms dark crystals soluble in MeOH and sparingly soluble in other organic

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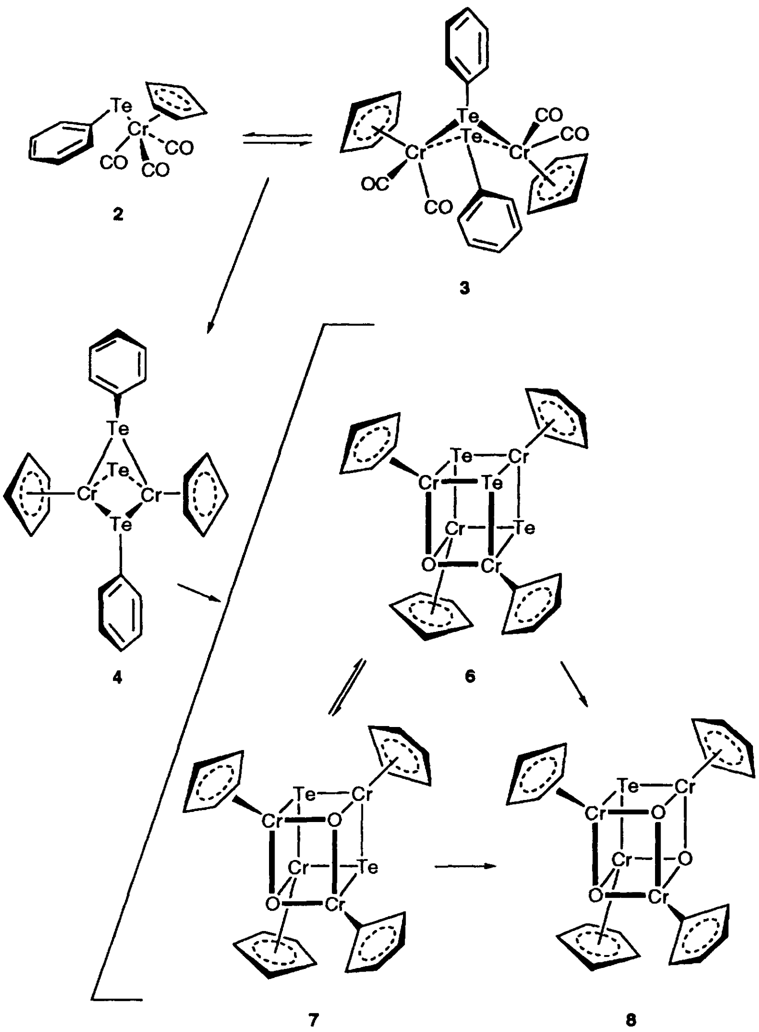
† *Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1994, Issue 1, pp. xxiii–xxviii.



Scheme 1

solvents to give blue solutions. Spectral data are collected in Table 1. The infrared spectra show bands due to $[Cr(\mu_3\text{-}O)]_4$ within the expected range.[35] The ^1H NMR spectrum in C_6D_6 exhibits two broad cp resonances indicative of inequivalent rings. These cp resonances of **7** are of equal intensities, but those of **6** and **8** possess relative intensities 3 : 1. In contrast to **7**, the analogous $[Cr_4(cp)_4Se_2O_2]$ only possesses one broad cp signal at δ 2.44 ($\nu_{\frac{1}{2}}$ 70 Hz) in its ^1H NMR spectrum.[14] The broad linewidth of the cp resonances of these complexes suggests some degree of paramagnetism, as was found for the antiferromagnetic distorted cubane $[Cr_4(cp)_4O_4]$ and its derivative $[Cr_4(cp)_4O_3(\eta^3\text{-}C_5H_4)]$[36] which gave no signals or very broad featureless signals, respectively, in their NMR spectra. Electron-impact (EI) mass spectral data for **5** together with the positive-ion FAB mass spectral data for **6**–**8** are given in Table 1. The EI mass spectrum of **5** shows a mass-fragmentation pattern characteristic of $[Cr_4(cp)_4E_4]$ (E = S[11] or Se[14]). The highest peak, clustered at *m/z* 979, possesses the isotopic distribution pattern of $[Cr_4(cp)_4Te_4]$ (Fig. 1). It is not possible to ascertain whether this is a component species of **5** or is formed from the fragmentation of **5**. The fragmentation patterns of **6**–**8** and the isotopic distribution patterns of their respective molecular ions are consistent with their formulations as $[Cr_4(cp)_4Te_nO_{4-n}]$ (n = 3, 2 and 1, respectively). Like similar chromium chalcogen cubanes, *viz.* $[Cr_4(cp)_4S_4]$,[11] $[Cr_4(cp)_4Se_4]$ and $[Cr_4(cp)_4Se_2O_2]$,[14] the Cr–Te complexes **6** and **7** undergo stepwise successive cleavage of their cp rings to yield the Cr_4Te_3O and $Cr_4Te_2O_2$ core units, respectively, attesting to the high stability of these cubane-like frameworks. Bottomley *et al.*[37,38] had also observed the loss of all four cp rings in the FAB mass spectrum of the $[Cr_4(cp)_4O_4]$ analogue and had ascribed instability of the cp rings to 'the lack of electrons for π-backbonding to cp'. A comparative study of the analogous C_5Me_5 complex showed that the ease of loss of the cp rings is determined by electronic factors.[38] The fragmentation pattern of $[Cr_4(cp)_4TeO_3]$ **8** is anomalous, only the loss of one cp ring being observed.

Molecular Structures.—A perspective view of the molecular structure of complex **6** is shown in Fig. 2. The molecule contains a mirror plane cutting through two chromium, one tellurium, one oxygen and two cyclopentadienyl carbon atoms. In compound **7**, there are two crystallographically independent molecules as illustrated in Fig. 3. For compound **8** there are eight molecules per unit cell with a three-fold axis passing through a Te and a Cr on the body diagonal of the cubane moiety. One of the four cp rings is located on the three-fold axis giving rise to a six-membered ring. Fig. 4 shows a perspective view of **8** through its three-fold symmetry axis. Atomic coordinates of **6**–**8** are listed in Tables 2–4, selected bond lengths and angles in Tables 5–7.

The structures belong to a class of cubane-like clusters of chromium with Group 16 main group atoms[11,12,14] in which the Cr atoms occupy alternate corners of a distorted cube. Complexes **6**–**8** contain three, two and one μ_3-Te groups and one, two and three μ_3-O groups, respectively. The dimensions of the cubane cores and of the chromium tetrahedra are given in

Table 1 Spectral data

	NMR (δ) (C_6D_6)		IR[a]/cm^{-1}		Mass spectra
Complex	^1H	^{13}C	ν[Cr(μ_3-O)]	Other bands	*m*/*z*[b]
5	33.8 (cp, $\nu_{\frac{1}{2}}$ *ca.* 100 Hz)	*c*	—	[3422m (vbr), 3095vw, 2954vw, 1620vw (vbr), 1429m, 1208s (sh), 1115vs (br), 1014s (sh), 803vs, 614m][d]	979.4 [$Cr_4(cp)_4Te_4$] 914.4 [$Cr_4(cp)_3Te_4$] 849.5 [$Cr_4(cp)_2Te_4$] 784.4 [$Cr_4(cp)Te_4$] 719.3 (Cr_4Te_4)
6	34.5 (3 cp, $\nu_{\frac{1}{2}}$ *ca.* 100 Hz) 22.5 (1 cp, $\nu_{\frac{1}{2}}$ *ca.* 60 Hz)	*c*	362	3094m, 2920m, 2851m, 1828vw, 1733m, 1061w, 1529w, 1503w, 1429s, 1352w, 1262w, 1176w, 1101w, 1058w, 1019m, 1004s, 909w, 841s, 818vs, 800vs, 701vw, 676vw, 599m, 568m	867.4 [$Cr_4(cp)_4Te_3O$] 802.5 [$Cr_4(cp)_3Te_3O$] 737.2 [$Cr_4(cp)_2Te_3O$] 672.4 [$Cr_4(cp)Te_3O$] 607.4 (Cr_4Te_3O)
7	37.4 (2 cp, $\nu_{\frac{1}{2}}$ *ca.* 160 Hz) 20.6 (2 cp, $\nu_{\frac{1}{2}}$ *ca.* 60 Hz)	117.9[e]	351	3439m (vbr), 3084m, 2922m, 2851m, 1798 (vbr) (1699, 1679, 1607),[f] 1431s, 1353m, 1262w, 1116w, 1081w, 1008s, 797vs, 542vs (br)	754.7 [$Cr_4(cp)_4Te_2O_2$] 690.5 [$Cr_4(cp)_3Te_2O_2$] 625.5 [$Cr_4(cp)_2Te_2O_2$] 562.4 [$Cr_4(cp)Te_2O_2$] 495.5 ($Cr_4Te_2O_2$)
8	28.43 (3 cp, $\nu_{\frac{1}{2}}$ *ca.* 100 Hz) 37.44 (1 cp, $\nu_{\frac{1}{2}}$ *ca.* 200 Hz)	*c*	(450, 318)[g]	[3450m (vbr), 3077vw, 2926m, 2853w, 1627w (br), 1583w, 1434m, 1355vw, 1262w, 1242w, 1060s, 1000vs, 791m, 748m, 69m][g]	644.9 [$Cr_4(cp)_4TeO_3$] 578.7 [$Cr_4(cp)_3TeO_3$]

[a] As CsI discs, unless otherwise indicated. [b] The most intense peak in the 'isotopic clusters' as illustrated in Fig. 1. Unidentified low-intensity peaks are not listed. All positive-ion FAB spectra except for the EI spectrum of complex **5**. [c] Resonances not observed, probably being obscured by solvent peaks. [d] As KBr disc. [e] Other expected resonances obscured by solvent peaks. [f] Unresolved multiplet. [g] Polythene disc.

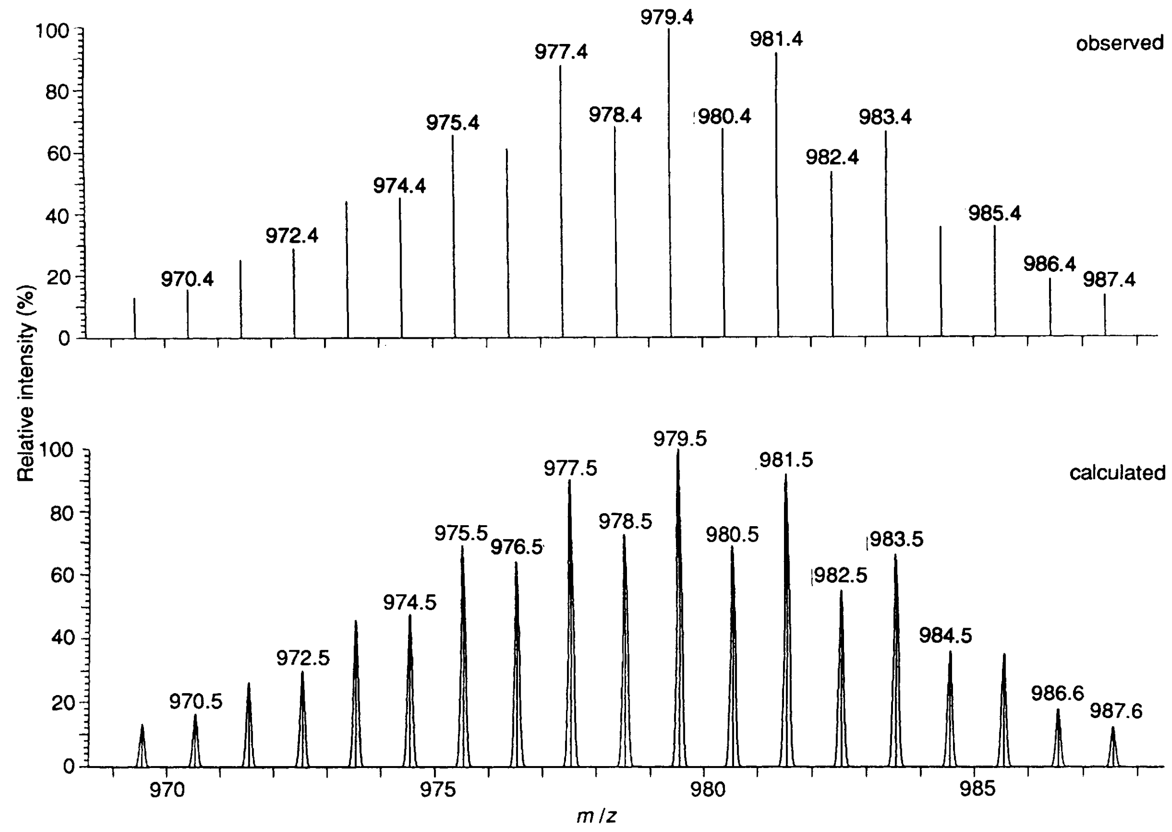


Fig. 1 Isotopic distribution pattern for a molecular ion of complex **5**

Table 8, which also lists those of [$Cr_4(cp)_4O_4$] **9**[39] for comparison. It will be noted that the Cr–Te bond distances are all comparable. The Cr–O distances vary from 1.892 Å for **6** to 1.94 Å in **8** and **9**. The O···O, Te···O and Te···Te distances in **6**, **7** and **8** (2.618–2.627, 3.269–3.454 and 3.941–3.961 Å, respectively) are all shorter than the sum of their van der Waals radii, *i.e.* 2.80, 3.60 and 4.40 Å.[40] The bond angles of the cubanes are dictated by the symmetry elements and the types of atoms forming the bonds. All angles about the Te atoms are acute while those at the oxygen atoms are obtuse except for one such angle of **9**. The angles at the chromium atoms in each of the three compounds may be acute or obtuse depending on the types of adjoining atoms.

The Cr_4 tetrahedra all suffer from distortion, the extent of which is influenced by the environment about each Cr atom. There are two types of environment for the chromium atoms in each cubane. In **6** Cr(1) is bonded to three Te atoms, while Cr(2) and Cr(3) are each bonded to two Te atoms, giving rise to two

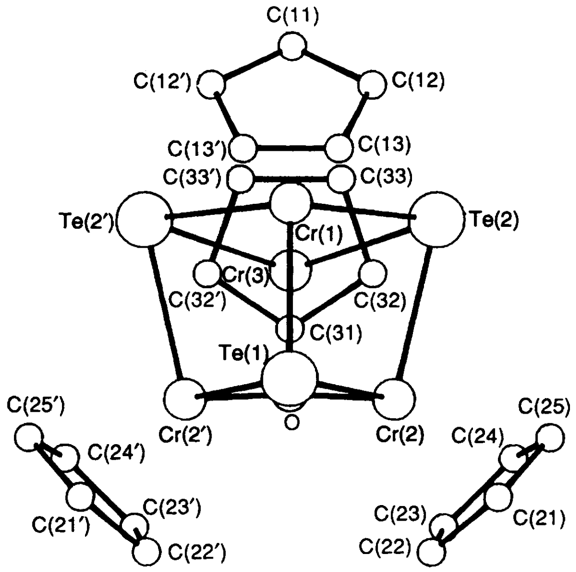


Fig. 2 Perspective view of complex **6**

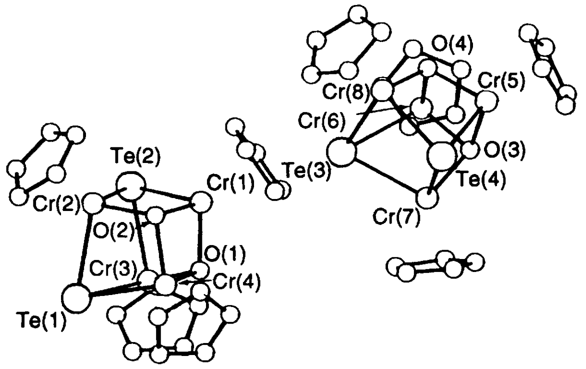


Fig. 3 Perspective view of the crystallographically independent molecules of complex **7**

sets of Cr···Cr distances [average 2.973(3) Å for Cr(1)···Cr(2) and Cr(1)···Cr(3) and 3.415(3) Å for Cr(2)···Cr(2′) and Cr(2)···Cr(3)]. The angles at Cr(1) are significantly smaller than those at Cr(2) and Cr(3) [50.1(1)–52.4(1) *vs.* 58.3(1)–65.7(1)°]. In each of the two crystallographically independent molecules of **7** two chromium atoms [Cr(2), Cr(3) and Cr(7), Cr(8)] are each bonded to two Te atoms and the other two Cr atoms [Cr(1), Cr(4) and Cr(5), Cr(6)] are bonded to one Te atom. This results in three sets of Cr···Cr distances [average 2.780(4), 3.013(4), 3.410(4) Å]. In **8** Cr(1) is bonded to one Te atom while Cr(2) is not linked to any Te, giving two sets of Cr···Cr distances [2.776(4) and 3.025(4) Å]. The minimum Cr···Cr distance of *ca.* 2.78 Å observed in **7** and **8** agrees with reported values for [$Cr_4(cp)_4O_4$] **9** and similar structures.[38,39] It is also evident that the higher degree of distortion in **6**–**8**, when compared with **9**, is reflected in the much higher discrepancies in the Cr···Cr distances in each tetrahedron.

Table 2 Atomic coordinates of complex **6**

Atom	*x*	*y*	*z*
Te(1)	−0.015 15(5)	0.25	0.505 46(4)
Te(2)	0.192 36(3)	0.108 14(2)	0.288 26(3)
Cr(1)	0.269 2(1)	0.25	0.471 78(9)
Cr(2)	−0.113 29(8)	0.148 55(5)	0.265 69(7)
Cr(3)	0.060 6(1)	0.25	0.109 44(9)
O	−0.138 0(4)	0.25	0.139 7(4)
C(11)	0.548 1(9)	0.25	0.563 7(9)
C(12)	0.501 0(7)	0.174 0(4)	0.618 2(7)
C(13)	0.420 6(7)	0.203 4(4)	0.698 0(6)
C(21)	−0.254 6(7)	0.046 2(4)	0.338 7(6)
C(22)	−0.370 7(6)	0.111 2(4)	0.242 3(6)
C(23)	−0.366 2(6)	0.098 6(4)	0.107 1(7)
C(24)	−0.250 3(7)	0.031 4(4)	0.120 8(6)
C(25)	−0.180 2(7)	−0.002 4(4)	0.260 9(6)
C(31)	−0.098 8(9)	0.25	−0.129 4(7)
C(32)	0.002 2(6)	0.171 8(4)	−0.095 0(5)
C(33)	0.169 1(6)	0.202 2(4)	−0.044 3(4)

Table 3 Atomic coordinates of complex **7**

Atom	*x*	*y*	*z*	Atom	*x*	*y*	*z*
Te(1)	0.532 02(5)	−0.394 14(5)	0.665 30(5)	C(33)	0.678 7(7)	−0.375 8(7)	0.490 0(7)
Te(2)	0.630 60(5)	−0.228 11(5)	0.590 22(5)	C(34)	0.604 4(7)	−0.418 4(7)	0.478 0(6)
Te(3)	1.015 46(5)	−0.177 28(5)	1.134 68(5)	C(35)	0.634 1(8)	−0.472 4(7)	0.540 9(7)
Te(4)	1.197 65(5)	−0.183 37(5)	1.388 55(5)	C(41)	0.812 4(8)	−0.417 3(8)	0.923 0(8)
Cr(1)	0.758 0(1)	−0.251 55(9)	0.756 5(1)	C(42)	0.766(1)	−0.473 7(7)	0.872 3(9)
Cr(2)	0.561 2(1)	−0.254 2(1)	0.704 2(1)	C(43)	0.682(1)	−0.477(1)	0.871(1)
Cr(3)	0.662 7(1)	−0.368 6(1)	0.617 0(1)	C(44)	0.680 6(9)	−0.421 4(8)	0.923 0(8)
Cr(4)	0.693 9(1)	−0.374 4(1)	0.805 5(1)	C(45)	0.757 8(9)	−0.384 0(8)	0.954 5(8)
Cr(5)	1.279 2(1)	−0.102 60(9)	1.312 8(1)	C(51)	1.385 7(8)	−0.024 2(8)	1.318 7(8)
Cr(6)	1.159 7(1)	−0.113 30(9)	1.131 7(1)	C(52)	1.424 7(8)	−0.092 0(8)	1.344 5(8)
Cr(7)	1.170 9(1)	−0.244 6(1)	1.232 9(1)	C(53)	1.422 6(7)	−0.108 9(8)	1.421 8(8)
Cr(8)	1.088 4(1)	−0.087 6(1)	1.274 5(1)	C(54)	1.380 1(8)	−0.054 3(7)	1.445 0(7)
O(1)	0.756 6(4)	−0.353 0(4)	0.735 2(4)	C(55)	1.355 0(8)	−0.003 9(7)	1.381 8(9)
O(2)	0.674 9(4)	−0.271 8(4)	0.805 6(4)	C(61)	1.137 4(9)	−0.139 9(8)	0.992 4(7)
O(3)	1.249 4(4)	−0.174 0(3)	1.219 9(4)	C(62)	1.089 1(9)	−0.080 8(9)	0.989 5(7)
O(4)	1.171 1(4)	−0.054 3(4)	1.230 6(4)	C(63)	1.145(1)	−0.026 6(7)	1.033 3(8)
C(11)	0.802 2(9)	−0.141 4(7)	0.788(1)	C(64)	1.232 7(9)	−0.056 4(9)	1.066 2(7)
C(12)	0.840 6(9)	−0.174 6(8)	0.866(1)	C(65)	1.221 9(8)	−0.123(1)	1.036 4(8)
C(13)	0.893(1)	−0.235 3(8)	0.872(1)	C(71)	1.258 7(9)	−0.339 6(7)	1.248(1)
C(14)	0.904 8(9)	−0.233 1(9)	0.797(1)	C(72)	1.204(1)	−0.335 3(8)	1.167(1)
C(15)	0.858(1)	−0.179 8(9)	0.742(1)	C(73)	1.114(1)	−0.346 5(8)	1.156(2)
C(21)	0.494 3(7)	−0.211 0(7)	0.785 3(7)	C(74)	1.133(1)	−0.355 5(8)	1.245(1)
C(22)	0.425 9(7)	−0.244 1(7)	0.709 8(7)	C(75)	1.220(1)	−0.349 6(8)	1.300(1)
C(23)	0.421 6(7)	−0.203 9(7)	0.634 3(7)	C(81)	0.969 0(8)	−0.014 7(7)	1.229 7(8)
C(24)	0.484 1(7)	−0.149 8(6)	0.665 3(7)	C(82)	1.044 5(8)	0.022 5(7)	1.281(1)
C(25)	0.530 0(7)	−0.153 9(6)	0.757 8(7)	C(83)	1.087(1)	−0.002 3(9)	1.367(1)
C(31)	0.727 9(7)	−0.465 3(7)	0.592 0(6)	C(84)	1.027 2(8)	−0.058 2(9)	1.367 3(8)
C(32)	0.754 7(7)	−0.404 6(7)	0.560 5(6)	C(85)	0.959 2(7)	−0.064 2(7)	1.285 6(7)

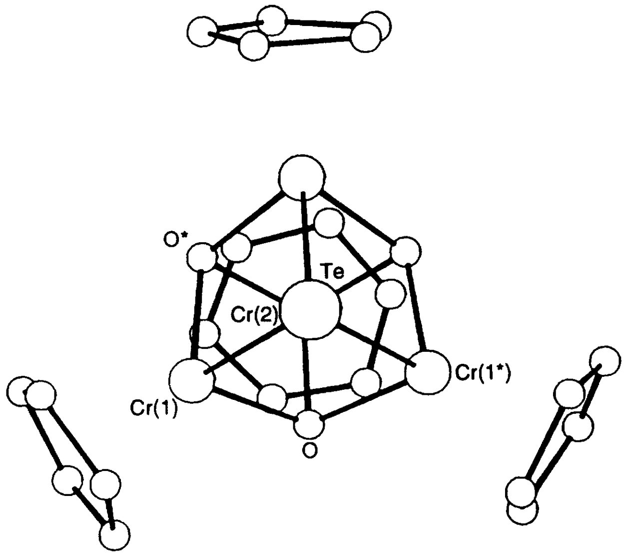


Fig. 4 Perspective view of complex **8** through its three-fold symmetry axis

Table 4 Atomic coordinates of complex **8**

Atom	*x*	*y*	*z*
Te	0.2120(2)	0.212	0.212
Cr(1)	0.3014(2)	0.3488(2)	0.2003(2)
Cr(2)	0.3602(2)	0.360	0.360
O	0.3911(6)	0.3042(6)	0.2619(6)
C(1)	0.367(1)	0.450(1)	0.130(1)
C(2)	0.288(1)	0.468(1)	0.135(1)
C(3)	0.242(1)	0.410(1)	0.092(1)
C(4)	0.300(1)	0.349(1)	0.062(1)
C(5)	0.375(1)	0.381(1)	0.088(1)
C(6)	0.477(2)	0.433(2)	0.371(2)
C(7)	0.483(2)	0.378(2)	0.422(2)

Table 5 Selected bond lengths (Å) and angles (°) of complex **6**

Te(1)–Cr(1)	2.659(1)	Cr(3)–O	1.893(4)
Te(1)–Cr(2)	2.6485(8)	Cr(1)–Cp 1 *	1.908(7)
Te(2)–Cr(1)	2.6509(7)	Cr(2)–Cp 2 *	1.901(6)
Te(2)–Cr(2)	2.6627(8)	Cr(3)–Cp 3 *	1.893(6)
Te(2)–Cr(3)	2.6519(7)	⟨C–C⟩$_{cp}$	1.382(9)
Cr(2)–O	1.891(3)		
Cr(1)–Te(1)–Cr(2)	81.11(3)	Cr(2)–Te(1)–Cr(2′)	66.9(1)
Cr(1)–Te(2)–Cr(2)	81.00(3)	Cr(1)–Te(2)–Cr(3)	78.20(3)
Cr(2)–Te(2)–Cr(3)	68.69(3)	Te(1)–Cr(1)–Te(2)	96.46(3)
Te(2)–Cr(1)–Te(2′)	100.8(1)	Te(1)–Cr(2)–Te(2)	96.44(3)
Te(2)–Cr(2)–O	93.1(1)	Te(1)–Cr(2)–O	95.4(1)
Te(2)–Cr(3)–O	93.34(8)	Te(2)–Cr(3)–Te(2′)	100.7(1)
Cr(2)–O–Cr(3)	104.8(2)	Cr(2)–O–Cr(2′)	101.1(1)
⟨C–C–C⟩$_{cp}$	108.0(6)		

* Cyclopentadienyl ring centre.

Table 6 Selected bond lengths (Å) and angles (°) of complex **7**

Te(1)–Cr(2)	2.675(2)	Cr(7)–O(3)	1.921(7)
Te(1)–Cr(3)	2.649(2)	Cr(3)–O(1)	1.904(7)
Te(1)–Cr(4)	2.668(2)	Cr(4)–O(1)	1.914(7)
Te(2)–Cr(1)	2.658(2)	Cr(4)–O(2)	1.935(8)
Te(2)–Cr(2)	2.657(2)	Cr(5)–O(3)	1.928(7)
Te(2)–Cr(3)	2.664(2)	Cr(5)–O(4)	1.914(7)
Te(3)–Cr(6)	2.672(2)	Cr(8)–O(4)	1.910(7)
Te(3)–Cr(7)	2.653(2)	Cr(1)–Cp 1	1.90(2)
Te(3)–Cr(8)	2.673(2)	Cr(2)–Cp 2	1.91(1)
Te(4)–Cr(5)	2.673(2)	Cr(3)–Cp 3	1.90(1)
Te(4)–Cr(7)	2.680(2)	Cr(4)–Cp 4	1.90(2)
Te(4)–Cr(8)	2.639(2)	Cr(5)–Cp 5	1.91(1)
Cr(1)–O(1)	1.919(7)	Cr(6)–Cp 6	1.91(1)
Cr(1)–O(2)	1.914(8)	Cr(7)–Cp 7	1.91(2)
Cr(2)–O(2)	1.907(7)	Cr(8)–Cp 8	1.90(1)
Cr(6)–O(3)	1.917(7)	⟨C–C⟩$_{cp}$	1.36(2)
Cr(6)–O(4)	1.916(7)		
Cr(2)–Te(1)–Cr(3)	79.42(6)	Cr(3)–O(1)–Cr(4)	101.0(3)
Cr(2)–Te(1)–Cr(4)	69.71(6)	Cr(1)–O(1)–Cr(3)	106.2(3)
Cr(3)–Te(1)–Cr(4)	67.28(6)	Cr(1)–O(1)–Cr(4)	93.3(3)
Cr(1)–Te(2)–Cr(2)	67.45(6)	Cr(2)–O(2)–Cr(4)	105.3(4)
Cr(1)–Te(2)–Cr(3)	70.08(6)	Cr(1)–O(2)–Cr(2)	101.1(3)
Cr(2)–Te(2)–Cr(3)	79.48(6)	Cr(1)–O(2)–Cr(4)	92.8(3)
Cr(6)–Te(3)–Cr(7)	66.66(6)	Cr(6)–O(3)–Cr(7)	99.4(3)
Cr(6)–Te(3)–Cr(8)	71.37(6)	Cr(5)–O(3)–Cr(7)	108.9(3)
Cr(7)–Te(3)–Cr(8)	79.83(7)	Cr(5)–O(3)–Cr(6)	92.4(3)
Cr(5)–Te(4)–Cr(7)	71.59(6)	Cr(6)–O(4)–Cr(8)	109.1(3)
Cr(5)–Te(4)–Cr(8)	66.68(6)	Cr(5)–O(4)–Cr(6)	92.8(3)
Cr(7)–Te(4)–Cr(8)	79.95(6)	Cr(5)–O(4)–Cr(8)	99.6(3)
Te(2)–Cr(1)–O(1)	91.7(2)	Te(1)–Cr(2)–O(2)	92.6(2)
Te(2)–Cr(1)–O(2)	95.5(2)	Te(1)–Cr(2)–Te(2)	95.32(6)
O(1)–Cr(1)–O(2)	86.2(3)	Te(2)–Cr(2)–O(2)	95.7(2)
Te(1)–Cr(3)–O(1)	96.2(2)	O(1)–Cr(4)–O(2)	85.7(3)
Te(2)–Cr(3)–O(1)	91.9(2)	Te(1)–Cr(4)–O(1)	95.3(2)
Te(1)–Cr(3)–Te(2)	95.75(7)	Te(1)–Cr(4)–O(2)	92.2(2)
Te(4)–Cr(5)–O(3)	89.4(2)	Te(3)–Cr(6)–O(3)	96.3(2)
Te(4)–Cr(5)–O(4)	95.8(2)	Te(3)–Cr(6)–O(4)	89.3(2)
O(3)–Cr(5)–O(4)	85.9(3)	O(3)–Cr(6)–O(4)	86.1(3)
Te(3)–Cr(7)–Te(4)	95.35(7)	Te(3)–Cr(8)–Te(4)	95.86(7)
Te(3)–Cr(7)–O(3)	96.8(2)	Te(4)–Cr(8)–O(4)	97.0(2)
Te(4)–Cr(7)–O(3)	89.4(2)	Te(3)–Cr(8)–O(4)	89.4(2)
⟨C–C–C⟩$_{cp}$	108(2)		

Table 7 Bond lengths (Å) and angles (°) of complex **8**

Te–Cr(1)	2.669(4)	Cr(1)–Cp 1	1.92(2)
Cr(1)–O	1.91(1)	Cr(2)–Cp 2	1.90(3)
Cr(1)–O^{I}	1.94(1)	⟨C–C⟩$_{cp}$	1.37(3)
Cr(2)–O	1.91(1)	⟨C–C⟩ *	1.22(2)
Cr(1)–Te–Cr(1^{I})	69.1(1)	Cr(1)–O–Cr(1^{II})	103.5(6)
O–Cr(1)–O^{I}	86.0(7)	Cr(1)–O–Cr(2)	93.3(5)
Te–Cr(1)–O	93.6(4)	Cr(1^{II})–O–Cr(2)	92.5(5)
Te–Cr(1)–O^{I}	93.1(4)	⟨C–C–C⟩$_{cp}$	108(3)
O–Cr(2)–O^{I}	87.0(5)	⟨C–C–C⟩ *	120(4)

Symmetry transformations: I *z*, *x*, *y*; II *y*, *z*, *x*.

* Disordered six-membered ring.

The oxotelluride cubane complexes [$Cr_4(cp)_4Te_3O$] **6**, [$Cr_4(cp)_4Te_2O_2$] **7** and [$Cr_4(cp)_4TeO_3$] **8** obtained here add to the family of M(μ_3-O) clusters, of which many examples are now known and have been covered in a recent exhaustive review on organometallic oxo compounds.[41] The μ_3-O ligand possesses a co-ordination mode common for O atoms bound to surfaces, hence the interest in M(μ_3-O) complexes stems from their useful role as models for catalytically active metal oxides.

Experimental

General.—All reactions were carried out either by use of conventional Schlenk techniques under nitrogen or in an argon atmosphere in a Vacuum Atmospheres Dribox equipped with a model HE493 Dri-Train. Proton and ^{13}C NMR spectra were measured on a JEOL FX100 100 MHz spectrometer, and chemical shifts referenced to residual C_6H_6 in C_6D_6 or to $SiMe_4$. Infrared spectra were measured in the range 4000–200 cm^{-1} by means of a JASCO IR Report-100 instrument. Carbon and hydrogen analyses were performed by the Analytical Unit of the Research School of Chemistry, Australian National University, and chromium analyses as [CrO_4]$^{2-}$ by ourselves.[42]

Table 8 Comparison of selected bond parameters (lengths in Å, angles in °)

Chromium tetrahedra^{a}					
	$[Cr_4(cp)_4Te_3O]$ **6**	$[Cr_4(cp)_4Te_2O_2]$ **7**		$[Cr_4(cp)_4TeO_3]$ **8**	$[Cr_4(cp)_4O_4]$ **9**^{39}
	Cr(1)···Cr(2) 3.451(3) × 2	Cr(1)···Cr(2) 2.951(4)	Cr(5)···Cr(8) 2.921(4)	Cr(1)···Cr(2) 2.776(4) × 3	Cr(1)···Cr(2) 2.900(6)
	Cr(1)···Cr(3) 3.344(3)	Cr(1)···Cr(3) 3.057(4)	Cr(6)···Cr(8) 3.119(4)	Cr(1)···Cr(1^{II}) 3.025(4) × 3	Cr(3)···Cr(4) 2.897(5)
	Cr(2)···Cr(3) 2.999(3) × 2	Cr(2)···Cr(4) 3.053(4)	Cr(5)···Cr(7) 3.132(4)		Cr(1)···Cr(3) 2.702(6)
	Cr(2)···Cr(2^{I}) 2.921(3)	Cr(3)···Cr(4) 2.946(4)	Cr(6)···Cr(7) 2.926(4)		Cr(2)···Cr(4) 2.712(2)
		Cr(1)···Cr(4) 2.786(3)	Cr(5)···Cr(6) 2.775(3)		Cr(1)···Cr(4) 2.811(6)
		Cr(2)···Cr(3) 3.402(4)	Cr(7)···Cr(8) 3.419(4)		Cr(2)···Cr(3) 2.841(4)
Cubane core^{b}					
Te–Cr	2.6546(8)	2.663(2)		2.669(4)	—
Cr–O	1.892(4)	1.917(7)		1.91(1), 1.91(1), 1.94(1)	1.936(2)
Cr–Te–Cr	[66.9(1), 81.11(3), 81.11(3)]^{c} 68.69(3), 78.20(3), 81.00(3)	67.02(6), 70.69(6), 79.67(6)		69.1(1)^{d}	—
Te–Cr–Te	[96.46(3), 96.46(3), 100.8(1), 100.7(1)]^{c} 96.44(3)	95.57(7)		—	—
Te–Cr–O	[93.34(8), 93.34(8)]^{c} 93.1(1), 95.4(1)	90.7(2), 95.7(2) 90.8(2), 96.4(2)		93.1(4), 93.6(4)	—
O–Cr–O	—	86.03(3)		86.0(7), 87.0(5)^{d}	83.24(7), 86.30(7), 90.15(7)
Cr–O–Cr	[101.1(1), 104.8(2), 104.8(2)]^{c}	92.8(3), 100.3(3), 107.4(3)		92.5(5), 93.3(5), 103.5(6)	88.79(7), 93.65(7), 96.64(7)

Symmetry transformations: I $x, \frac{1}{2} - y, z$; II z, x, y.

[a] Braces denote sets of bond distances. [b] Average values for complexes **7** and **9**. [c] Central atom on a mirror plane. [d] Central atom on a three-fold axis.

Table 9 Data collection and processing parameters for complexes **6**–**8***

	6	**7**	**8**
M_r	867.17	755.57	643.96
Crystal colour and habit	Dark green plates	Dark green plates	Dark blue plates
Crystal size (mm)	0.07 × 0.15 × 0.2	0.25 × 0.25 × 0.12	0.3 × 0.08 × 0.2
a/Å	8.7871(3)	16.4285(8)	16.282(2)
b/Å	14.3953(7)	18.6080(8)	16.282(2)
c/Å	10.1224(3)	16.639(1)	16.282(2)
α/°	90	90	90
β/°	114.751(3)	116.460(5)	90
γ/°	90	90	90
U/Å^3	1163(2)	4553.6(9)	4316.5
Z	2	8	8
D_c/Mg m^{-3}	2.477	2.204	1.979
Crystal system	Monoclinic	Monoclinic	Cubic
Space group	$P2_1/m$	$P2_1/c$	$Pa\bar{3}$
μ/mm^{-1}	5.49	4.35	3.26
Absorption correction (T_{min}, T_{max})	75.84, 99.91	84.68, 99.93	77.08, 99.98
No. of reflections measured	1880	8550	3207
No. of independent reflections	1767	7966	949
θ_{max}/°	25	25	22.5
No. of observed reflections [$I > 3\sigma(I)$]	1475	4010	311
Variation of standards (% h^{-1})	-2.38×10^{-2}	-5.02×10^{-2}	−2.41
hkl Ranges	−8 to 8, 0–13, 0–19	−18 to 0, 0–22, −17 to 19	0–17, 0–17, −17 to 0
R	0.020	0.039	0.042
$R'\{w = [\sigma(F)^2]^{-1}\}$	0.026	0.042	0.045
No. of parameters refined	136	505	53
No. of reflections used in refinement	1475	4010	311
S	0.981	1.142	1.026
$(\Delta/\sigma)_{max}$	0.01	0.84	0.02
$(\Delta\rho)_{max}$/e Å^{-3}	0.391	0.618	0.568

* Details in common: Mo-Kα radiation (λ 0.710 73 Å); 27 °C; ω–2θ scan; three standard reflections every 400.

The complex [{Cr(cp)(CO)$_3$}$_2$] was synthesized from [Cr(CO)$_6$] (Strem Chemicals) by the method of Manning and co-workers.[43] The compound Ph$_2$Te$_2$ was prepared using a published procedure.[44] Silica gel (Merck Kieselgel 60, 35–70 mesh) was dried at 140 °C overnight before chromatographic use. All solvents used were distilled from sodium–benzophenone prior to use.

Thermolytic Reactions.—A brownish green solution of [Cr(cp)(CO)$_3$(TePh)] **2** from the instantaneous reaction of [{Cr(cp)(CO)$_3$}$_2$] **1** (128 mg, 0.32 mmol) with Ph$_2$Te$_2$ (130 mg, 0.32 mol) in toluene (22 cm^3)[22] was maintained with stirring at 80 °C. The solution was periodically monitored, *via* its proton NMR spectrum and its TLC characteristics on Merck silica-gel plates with toluene–hexane (1 : 1) as eluent, for the presence of complexes **2** [δ 3.98, R_f = 0.58 (dirty green)], [{Cr(cp)(CO)$_2$(TePh)}$_2$] **3** [δ 4.41, R_f = 0.62 (brown)] and [{Cr(cp)(TePh)}$_2$Te] **4** [δ 15.80 ($\nu_{\frac{1}{2}}$ 36) and 19.80 (20 Hz); R_f = 0.21 (green) and 0.15 (greyish green)]. After 8 h **4** was found to be the major component in the reaction mixture. Finally after 48 h the proton NMR spectrum only showed new broad resonances at δ 20.62, 34.50, 37.57 with very weak peaks at δ 22.73 and 28.9. Thin-layer chromatography on Merck silica-gel plates with toluene–hexane (1 : 1) followed by tetrahydrofuran (thf)–toluene (1 : 1) gave two spots with R_f = 0.48 (green) for compound **6** and R_f = 0.36 (green) for **7**, described below. The resulting dark suspension was then filtered to remove a grey-black precipitate (52 mg, 0.038 mol, 24% yield), the elemental analysis of which indicated [Cr$_4$(cp)$_4$Te$_7$] (Found: C, 16.65; H, 1.85; Cr, 15.45, 15.20. Calc.: C, 17.65; H, 1.50; Cr, 15.30%). The filtrate was loaded onto a silica gel column (16 × 120 mm), prepared in toluene. Elution gave: (*i*) a yellow fraction (5 cm^3), followed by a dirty green fraction (30 cm^3) in toluene, which gave greasy residues (16 and 37 mg, respectively) consisting mainly of Ph$_2$Te$_2$, in admixture with trace amounts of **2** and **3**; (*ii*) a turquoise-blue fraction in toluene–diethyl ether (1 : 1, 25 cm^3) which yielded **4** (*ca.* 1 mg); (*iii*) a dirty green fraction in toluene–diethyl ether (1 : 9, 30 cm^3) which yielded fine dark green crystals [Cr$_4$(cp)$_4$Te$_3$O] **6** (Found: C, 28.65; H, 2.45; Cr, 23.48. Calc.: C, 27.70; H, 2.30; Cr, 24.00%) (20 mg, 0.023 mmol, 14.4% yield); (*iv*) a dirty green fraction in thf (30 cm^3) which gave fine dark green crystals of [Cr$_4$(cp)$_4$Te$_2$O$_2$] **7** (45 mg, 0.060 mmol, 37.2% yield) (Found: C, 32.65; H, 2.75; Cr, 27.30. Calc.: C, 31.80; H, 2.65; Cr, 27.55%); and (*v*) a blue fraction in MeOH (10 cm^3), which gave a few crystals of [Cr$_4$(cp)$_4$TeO$_3$] **8**.

Of complexes **6** *and* **7**: *an NMR study*. Dilute solutions of complexes **6** and **7** in [^{2}H$_8$]toluene in 5 mm NMR tubes were maintained at 90 °C and their proton NMR spectra monitored at intervals. After 20 h the solution of **6** only showed the presence of **7** and **8** in an approximately equimolar proportion. After 20 h more only **8** was observed in the solution together with slight precipitation of a dark insoluble solid. A similar study with **7** showed conversion into an approximately 1 : 2 molar mixture of **6** and **8** after 20 h. After 20 h more, slight precipitation had occurred and no signal was observed in the spectrum.

Structure Analysis.—Diffraction-quality crystals of complex **6** were obtained from a saturated solution in C$_6$H$_6$ after 2 weeks at ambient temperature. Those of **7** were obtained from a saturated solution in toluene–hexane after 6 weeks at ambient temperature, and those of **8** were obtained by slow evaporation of a methanol solution of the total product over a couple of weeks at ambient temperature.

The crystals were coated with epoxy glue to prevent decomposition in air and were mounted on a CAD4 diffractometer. Twenty-five strong reflections were used for the accurate determination of the unit-cell parameters. Details of the crystal parameters, data collection and structure refinement are given in Table 9. Raw intensities were processed for Lorentz-polarization effects, decay and corrected for absorption.[45] The structures of complexes **6** and **8** were solved by Patterson syntheses and that of **7** by the direct method MULTAN.[46] All non-hydrogen atoms were subjected to anisotropic refinement, except the C atoms in **8**. The hydrogen atoms were generated geometrically with C–H 0.95 Å and allowed to ride with B = 1.3 times the value of their respective parent C atom. Each of the six carbon atoms on the disordered cp ring was given a fixed multiplicity of 0.83 in the refinement. All carbon atoms in this structure were refined isotropically.

Computations were performed using the MolEN[47] package on a DEC MicroVAX-II computer. Analytic expressions of atomic scattering factors were employed and anomalous dispersion corrections were incorporated.[48]

Additional material available from the Cambridge Crystallographic Data Centre comprises thermal parameters and remaining bond lengths and angles.

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