15·198 (2) Å, $\alpha = 92{\cdot}14$ (2), $\beta = 95{\cdot}04$ (1), $\gamma = 98{\cdot}78$ (2)°. The structure is very similar to the one discussed above with a Cu^{2+} pentacoordinated by four N and one O donor, but there is no water molecule in this compound. No further work on this form is planned.

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Structure of Bis(η-pentamethylcyclopentadienyl)chromium(III) Iodide, $[Cr(C_5Me_5)_2]I$

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Abstract. $C_{20}H_{30}Cr^+.I^-$, $M_r = 449{\cdot}36$, triclinic, $P\bar{1}$, $a = 10{\cdot}414$ (3), $b = 11{\cdot}721$ (3), $c = 8{\cdot}710$ (3) Å, $\alpha = 100{\cdot}96$ (2), $\beta = 98{\cdot}99$ (2), $\gamma = 80{\cdot}89$ (2)°, $V = 1021{\cdot}8$ Å^3, $Z = 2$, $D_x = 1{\cdot}46$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 2{\cdot}04$ mm^{-1}, $F(000) = 454$, $T = 294$ K, final $R = 0{\cdot}036$ for 2136 reflections. The structure consists of a cationic moiety, in which a Cr atom is sandwiched between two C_5Me_5 rings, and an iodide counter ion. The rings are planar to within 0·05 Å and are slightly rotated from an ideal eclipsed position by 6·6°. The Cr—ring-centroid distances are 1·863 (5) and 1·858 (5) Å.

Introduction. The reaction of chromocene with halides and alkyl halides produces the chromocenium cation (Fischer, Ulm & Kuzel, 1963; Kohler, de Cao, Ackermann & Seldlmair, 1983). The cationic species have so far only been characterized by elemental analysis and by paramagnetic ^1H and ^{13}C NMR spectroscopy. Based on these analyses, the structure of the chromocenium cation in these salts is assumed to have the sandwiched structure characteristic of metallocenes.

In our effort to generate and isolate R_3Si^+ we reacted $(C_5Me_5)_2Cr$ and R_3SiX (where R = Me, *i*-Pr and X = Cl, Br, I) with the hope that the decamethylchromocene would abstract the halide to form $[(C_5Me_5)_2CrI]^-$ and R_3Si^+. The solid products obtained, though ionic, had no silicon present as evidenced by ^{29}Si NMR and the reactions appeared to have followed the course of Fischer's reaction described above. An X-ray structural analysis of the iodide analogue was carried out to determine the structure of the decamethylchromocene cation. The results of our study are reported herein.

Experimental. Bis(pentamethylcyclopentadienyl)-chromium(III) iodide was prepared by the reaction of $(C_5Me_5)_2Cr$ and degassed Me_3SiI in tetrahydrofuran under an argon atmopshere. Crystals suitable for X-ray analysis were obtained by slow diffusion of hexane into a CH_2Cl_2 solution of the compound (caution: the product decomposes in chlorinated solvents over a period of one week). The highly air- and moisture-sensitive crystals were mounted in glass capillaries by using a glovebox with an inert N_2 atmosphere.

Orange cut plate, 0·14 × 0·32 × 0·43 mm; Enraf–Nonius CAD-4F diffractometer, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters

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from least-squares refinement of 25 reflections with $17{\cdot}5 \leq \theta \leq 21{\cdot}0°$; space group determined from examination of intensities and successful solution and refinement of the structure; 3198 unique reflections collected, 2136 considered observed at the $3\sigma(I)$ level [$\sigma(I)$ from counting statistics]; $\theta_{\max} = 24°$, $\omega/2\theta$ scans, scan range $1{\cdot}5\ (0{\cdot}80 + 0{\cdot}347\tan\theta)°$, scan speed 1–4° $\min^{-1}$; three standard reflections ($\bar{3}14$, $\bar{2}60$, $53\bar{1}$) measured every 2400 s of X-ray exposure time, max. variation 0·5%; data collected: $\pm h$, $+k$, $\pm l$ to max. indices of 11,13,9; data corrected for background and Lp; corrected for absorption using empirical method (*DIFABS*; Walker & Stuart, 1983), min./max. correction = 0·76/1·30. Structure solved by Patterson method (I^- located) and difference Fourier techniques, refined by full-matrix least squares based on F, minimizing the function $\sum w(|F_o| - |F_c|)^2$; w defined as $[\sigma^2(F_o) + (0{\cdot}04F_o)^2]^{-1}$; *SDP*/VAX system of programs (Frenz, 1978); H atoms included in calculated positions (based on those found on a difference Fourier map) with isotropic thermal parameters set to 1·2 times $B_{\rm eq}$ of the bonded atom; H-atom parameters were not refined; all non-H were refined with anisotropic thermal parameters. When I^- was treated as a single anisotropic atom: $R = 0{\cdot}087$, $wR = 0{\cdot}082$ and peaks over 3·0 e Å^{-3} were observed on a difference Fourier map. In final cycles I^- was treated as being disordered over three positions with occupancy factors of 0·55, 0·30 and 0·15. The model converged with 2136 reflections and 218 variables, $R = 0{\cdot}036$, $wR = 0{\cdot}045$, max. $(\Delta/\sigma) = 5{\cdot}75$ (for disordered I^-, max. 0·3 for all other atoms), $S = 1{\cdot}46$, isotropic extinction parameter $2{\cdot}8\ (6) \times 10^{-7}$, max. residual electron density 0·34 (7) e Å^{-3} associated with I^-. $R_{\rm int}(I) = 0{\cdot}018$ for 262 observations. Scattering factors were those of Cromer & Waber (1974). Anomalous-dispersion corrections were included for non-H atoms (Cromer, 1974). Atomic parameters may be found in Table 1 and bond lengths and angles are given in Table 2.* An *ORTEP* (Johnson, 1965) drawing is presented in Fig. 1.

Table 1. *Positional and thermal parameters, with e.s.d.'s in parentheses*

$$B_{\rm eq} = (8/3)\pi^2(a^{*2}U_{11}^2 + b^{*2}U_{22}^2 + c^{*2}U_{33}^2 + 2a^*b^*\cos\gamma U_{12} + 2a^*c^*\cos\beta^* U_{13} + 2b^*c^*\cos\alpha^* U_{23}).$$

	x	y	z	$B_{\rm eq}$(Å^2)	Occ.
I1	0·20738 (8)	0·73293 (7)	0·1561 (1)	6·07 (2)	0·55
I2	0·2137 (1)	0·7105 (1)	0·1152 (2)	5·00 (3)	0·30
I3	0·1888 (3)	0·7670 (3)	0·2229 (5)	6·38 (8)	0·15
Cr	0·27226 (7)	0·25155 (7)	0·2666 (1)	2·75 (2)	
C1	0·2901 (5)	0·2987 (5)	0·5250 (6)	3·7 (1)	
C2	0·2038 (5)	0·2126 (5)	0·4760 (6)	3·6 (1)	
C3	0·0938 (5)	0·2611 (5)	0·3789 (6)	3·4 (1)	
C4	0·1123 (5)	0·3756 (5)	0·3657 (6)	3·1 (1)	
C5	0·2337 (5)	0·3987 (5)	0·4571 (6)	3·4 (1)	
C6	0·4201 (6)	0·2870 (6)	0·6315 (7)	5·1 (2)	
C7	0·2220 (7)	0·0943 (5)	0·5240 (7)	5·8 (2)	
C8	−0·0246 (6)	0·2007 (6)	0·3102 (8)	5·3 (2)	
C9	0·0189 (6)	0·4595 (5)	0·2764 (7)	4·4 (1)	
C10	0·2913 (6)	0·5130 (5)	0·4857 (8)	5·1 (2)	
C11	0·3938 (5)	0·2870 (4)	0·0986 (6)	3·1 (1)	
C12	0·4614 (5)	0·1931 (4)	0·1729 (6)	3·3 (1)	
C13	0·3856 (5)	0·0993 (4)	0·1368 (6)	3·2 (1)	
C14	0·2685 (5)	0·1350 (4)	0·0385 (6)	3·1 (1)	
C15	0·2751 (5)	0·2516 (4)	0·0155 (6)	3·0 (1)	
C16	0·4442 (6)	0·3999 (5)	0·1008 (7)	4·8 (1)	
C17	0·5974 (5)	0·1922 (5)	0·2686 (8)	4·9 (2)	
C18	0·4244 (6)	−0·0176 (5)	0·1897 (7)	4·6 (1)	
C19	0·1603 (6)	0·0613 (5)	−0·0315 (7)	4·3 (1)	
C20	0·1728 (6)	0·3232 (5)	−0·0812 (6)	4·4 (1)	

Table 2. *Bond distances* (Å) *and angles* (°), *with e.s.d.'s in parentheses*

Cr—C1	2·198 (5)	C3—C4	1·413 (8)
Cr—C2	2·207 (6)	C3—C8	1·505 (8)
Cr—C3	2·210 (5)	C4—C5	1·421 (7)
Cr—C4	2·201 (5)	C4—C9	1·507 (7)
Cr—C5	2·189 (5)	C5—C10	1·516 (8)
Cr—C11	2·212 (6)	C11—C12	1·417 (7)
Cr—C12	2·209 (6)	C11—C15	1·411 (7)
Cr—C13	2·200 (5)	C11—C16	1·494 (8)
Cr—C14	2·185 (5)	C12—C13	1·413 (7)
Cr—C15	2·193 (6)	C12—C17	1·527 (7)
C1—C2	1·418 (8)	C13—C14	1·429 (7)
C1—C5	1·417 (8)	C13—C18	1·503 (8)
C1—C6	1·520 (7)	C14—C15	1·433 (8)
C2—C3	1·423 (7)	C14—C19	1·511 (8)
C2—C7	1·498 (9)	C15—C20	1·501 (7)

C2—C1—C5	107·9 (4)	C1—C5—C4	108·6 (5)
C2—C1—C6	126·6 (5)	C1—C5—C10	125·1 (5)
C5—C1—C6	125·5 (5)	C4—C5—C10	126·3 (5)
C1—C2—C3	107·6 (5)	C12—C11—C15	107·8 (5)
C1—C2—C7	126·2 (5)	C12—C11—C16	125·5 (4)
C3—C2—C7	126·2 (6)	C15—C11—C16	126·6 (5)
C2—C3—C4	108·8 (4)	C11—C12—C13	109·0 (5)
C2—C3—C8	124·5 (5)	C11—C12—C17	125·0 (5)
C4—C3—C8	126·6 (5)	C13—C12—C17	126·0 (5)
C3—C4—C5	107·2 (4)	C12—C13—C14	107·7 (4)
C3—C4—C9	126·7 (4)	C12—C13—C18	125·5 (4)
C5—C4—C9	126·2 (5)	C14—C13—C18	126·8 (5)
C13—C14—C19	125·9 (5)	C13—C14—C15	107·4 (4)
C11—C15—C14	108·3 (4)	C15—C14—C19	126·6 (4)
C14—C15—C20	125·6 (4)	C11—C15—C20	126·2 (5)

Discussion. The cation consists of a Cr atom sandwiched between two C_5Me_5 rings. The sandwich structure is similar to those of other metallocenes. Both C_5Me_5 rings, including the carbons of the methyl substituents, are essentially planar to within 0·05 Å. The C—C and C—Me distances do not vary significantly and average 1·419 (5) and 1·508 (2) Å, respectively. Similarly, the C—C—C angles [average 108·0 (3)°] are internally consistent. The rings are parallel to within 1° but are rotated from an ideal eclipsed position by 6·6°. The Cr atom is found to lie equidistant, within experimental error, between the planes formed by the two rings [1·858 (5),

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52459 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

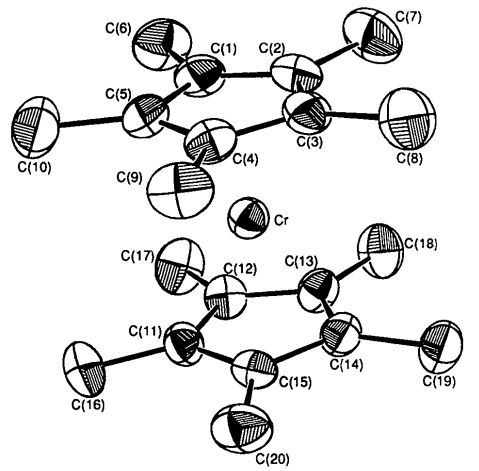

Fig. 1. *ORTEP* plot (Johnson, 1965) of $[Cr(C_5Me_5)_2]^+.I^-$. Thermal ellipsoids are drawn at the 50% probability level.

1·863 (5) Å]. The average metal—ring-centroid distance for this formally Cr^{3+} complex is slightly shorter than the distance found for $(C_5Me_5)_2Cr_2O_4$ [1·931 (6) Å] (Heberhold, Kremnitz, Razavi, Schollhorn & Thewalt, 1985) but slightly longer than that found for $(C_5Ph_4H)_2Cr$ [1·832 (5) Å] (Castellani, Geib, Rheingold & Trogler, 1987) which have formal oxidation states of + 5 and + 2 respectively. Metal—ring-C-atom averages are more consistent with values of 2·200 (4), 2·200 (6) and 2·197 (5) Å for complexes with oxidation states of + 5, + 3 and + 2 respectively.

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Structural Chirality of [Bis(desferrithiocin)]copper(II)

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Abstract. [2-(3-Hydroxy-2-pyridyl)-4-methyl-4,5-dihydro-4-thiazolecarboxylato(2−)][2-(3-hydroxy-2-pyridyl)-4-methyl-4,5-dihydro-4-thiazolecarboxylic acid]-copper(II), $[Cu(C_{10}H_8N_2O_3S)(C_{10}H_{10}N_2O_3S)]$, M_r = 538·06, monoclinic, $P2_1$, a = 10·412 (1), b = 6·656 (4), c = 16·788 (1) Å, β = 98·59 (3)°, V = 1150·5 Å^3, Z = 2, D_x = 1·553 Mg m^{-3}, Cu $K\alpha$, λ = 1·54184 Å, μ = 3·3739 mm^{-1}, $F(000)$ = 550, T = 293 K. The final R value is 0·039 for 3465 significant [$I > 3\cdot\sigma(I)$] reflections. The Cu atom has a square-pyramidal coordination sphere and is bound to two O and two N atoms, each in a *trans* position, and to one N atom in an axial position. The last bond exhibits a pronounced elongation [2·340 (4) Å] due to Jahn–Teller distortion.

Introduction. Desferrithiocin ($dftH_2$) is known to be a naturally occurring chelator and was isolated as the

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