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Bis(triphenylphosphoranediyl)ammonium Tetrachloroferrate(III)

BY PETER G. JONES, TINA SCHROEDER AND GEORGE M. SHELDRICK

Institut für Anorganische Chemie der Universität, Tammannstrasse 4, *D*-3400 *Göttingen, Federal Republic of Germany*

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Abstract. $C_{36}H_{30}NP_2^+.FeCl_4^-$, $M_r = 736{\cdot}2$, monoclinic, $P2_1/c$, $a = 17{\cdot}629\ (4)$, $b = 9{\cdot}173\ (2)$, $c = 22{\cdot}225\ (6)$ Å, $\beta = 95{\cdot}73\ (2)°$, $U = 3576$ Å^3, $Z = 4$, $D_x = 1{\cdot}37$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 8{\cdot}0$ cm^{-1}, $F(000) = 1508$, $T = 293$ K, $R = 0{\cdot}056$ for 3090 reflections. The anion is tetrahedral, with Fe—Cl 2·170–2·191 Å. The cation is bent at N; P—N—P 141·8 (3)°.

Experimental. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode (Clegg, 1981). 4785 reflections, $2\theta_{\max}$ 45°, $\pm h+k+l$. Three check reflections, no intensity change. Crystal size 0·4 × 0·3 × 0·1 mm; no absorption correction. 4642 unique reflections (R_{int} 0·020), 3090 with $F > 4\sigma(F)$ used for all calculations (program system *SHELXTL*; Sheldrick, 1983). Index range $|h| \le 18$, $k \le 9$, $l \le 23$. Cell constants refined from $\pm 2\theta$ values of 32 reflections in the range 20–23°.

Structure solution by routine direct methods (to locate P, Fe, Cl) and subsequent difference synthesis. Refinement on F to R 0·056, wR 0·050; all non-H atoms anisotropic; H atoms included using riding model with C—H 0·96 Å, $U(\text{H}) = 1{\cdot}2U_{\text{eq}}(\text{C})$; weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}0002F^2$; 397 parameters; $S = 1{\cdot}52$; max. Δ/σ 0·08; max. features in final $\Delta\rho$ map $\pm 0{\cdot}3$ e Å^{-3}. Atomic scattering factors from *SHELXTL*. Final atomic coordinates are given in

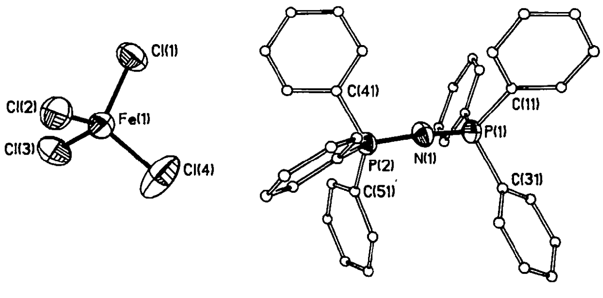

Fig. 1. Asymmetric unit of the title compound, showing the atom-numbering scheme.

Table 1. *Atomic coordinates* (×10^4) *and isotropic thermal parameters* (Å^2 ×10^3)

	x	y	z	U_{eq}*
P(1)	1696 (1)	7391 (1)	1055 (1)	38 (1)
P(2)	3359 (1)	7922 (2)	1015 (1)	37 (1)
N(1)	2497 (2)	8169 (4)	1126 (2)	42 (1)
C(11)	1172 (3)	8065 (5)	1651 (2)	41 (2)
C(12)	1552 (3)	8670 (7)	2155 (3)	66 (3)
C(13)	1158 (3)	9149 (7)	2629 (3)	89 (3)
C(14)	385 (3)	8978 (8)	2589 (3)	94 (3)
C(15)	−6 (3)	8379 (8)	2091 (3)	95 (3)
C(16)	391 (3)	7912 (7)	1624 (3)	69 (2)
C(21)	1714 (2)	5434 (5)	1107 (2)	37 (2)
C(22)	1490 (3)	4721 (5)	1612 (2)	45 (2)
C(23)	1530 (3)	3213 (6)	1650 (2)	57 (2)
C(24)	1789 (3)	2419 (6)	1191 (2)	62 (2)
C(25)	2021 (3)	3111 (6)	693 (3)	62 (2)
C(26)	1981 (3)	4615 (5)	645 (2)	50 (2)
C(31)	1160 (3)	7831 (5)	347 (2)	41 (2)
C(32)	621 (3)	6890 (7)	73 (3)	82 (3)
C(33)	194 (4)	7271 (7)	−462 (3)	99 (3)
C(34)	320 (3)	8583 (7)	−724 (3)	77 (3)
C(35)	839 (3)	9512 (6)	−463 (3)	65 (2)
C(36)	1265 (3)	9160 (6)	76 (2)	50 (2)
C(41)	3868 (3)	6855 (5)	1606 (2)	39 (2)
C(42)	3491 (3)	6230 (6)	2055 (2)	55 (2)
C(43)	3899 (4)	5458 (7)	2518 (3)	70 (3)
C(44)	4667 (4)	5295 (7)	2525 (3)	71 (3)
C(45)	5053 (3)	5927 (7)	2089 (3)	66 (3)
C(46)	4654 (3)	6707 (6)	1631 (2)	53 (2)
C(51)	3483 (2)	7086 (5)	299 (2)	39 (2)
C(52)	3844 (3)	5760 (6)	257 (3)	63 (2)
C(53)	3886 (4)	5138 (7)	−304 (3)	84 (3)
C(54)	3556 (4)	5797 (9)	−812 (3)	82 (3)
C(55)	3185 (4)	7100 (8)	−773 (3)	75 (3)
C(56)	3162 (3)	7757 (6)	−224 (2)	57 (2)
C(61)	3817 (2)	9671 (5)	1028 (2)	36 (2)
C(62)	4463 (3)	9916 (6)	733 (2)	56 (2)
C(63)	4835 (3)	11256 (7)	794 (3)	69 (3)
C(64)	4569 (4)	12327 (7)	1146 (3)	62 (3)
C(65)	3933 (3)	12081 (6)	1433 (3)	65 (2)
C(66)	3562 (3)	10760 (6)	1385 (2)	49 (2)
Fe(1)	7523 (1)	7994 (1)	1403 (1)	57 (1)
Cl(1)	7154 (1)	6676 (2)	2142 (1)	80 (1)
Cl(2)	8025 (1)	10032 (2)	1771 (1)	75 (1)
Cl(3)	8376 (1)	6747 (2)	971 (1)	82 (1)
Cl(4)	6546 (1)	8534 (2)	770 (1)	118 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

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Table 2. *Selected bond lengths* (Å) *and angles* (°)

Fe(1)–Cl(1)	2·190 (2)	Fe(1)–Cl(2)	2·191 (2)
Fe(1)–Cl(3)	2·185 (2)	Fe(1)–Cl(4)	2·170 (2)
P(1)–N(1)	1·577 (4)	P(1)–C(11)	1·798 (5)
P(1)–C(21)	1·799 (5)	P(1)–C(31)	1·797 (5)
P(2)–N(1)	1·580 (4)	P(2)–C(41)	1·803 (5)
P(2)–C(51)	1·801 (5)	P(2)–C(61)	1·795 (5)
Cl(1)–Fe(1)–Cl(3)	108·0 (1)	Cl(1)–Fe(1)–Cl(2)	109·3 (1)
Cl(1)–Fe(1)–Cl(4)	109·7 (1)	Cl(2)–Fe(1)–Cl(3)	109·9 (1)
Cl(3)–Fe(1)–Cl(4)	111·8 (1)	Cl(2)–Fe(1)–Cl(4)	108·2 (1)
N(1)–P(1)–C(11)	106·9 (2)	N(1)–P(1)–C(21)	115·8 (2)
C(11)–P(1)–C(21)	107·6 (2)	N(1)–P(1)–C(31)	112·0 (2)
C(11)–P(1)–C(31)	107·7 (2)	C(21)–P(1)–C(31)	106·6 (2)
N(1)–P(2)–C(41)	112·3 (2)	N(1)–P(2)–C(51)	113·8 (2)
C(41)–P(2)–C(51)	108·5 (2)	N(1)–P(2)–C(61)	107·9 (2)
C(41)–P(2)–C(61)	106·5 (2)	C(51)–P(2)–C(61)	107·5 (2)
P(1)–N(1)–P(2)	141·8 (3)		

Table 1, and selected bond lengths and angles in Table 2.* Fig. 1 shows the atom-numbering scheme.

Related literature. Other salts of $FeCl_4^-$: Zaslow & Rundle (1957), Kistenmacher & Stucky (1968), Richards & Gregory (1965), Müller, Conradi, Demant & Dehnicke (1984), Bottomley, Carter, Engelhardt, Lincoln, Patrick & White (1984).

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* Lists of structure factors, H-atom coordinates, bond lengths and angles, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43072 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Helenalin, a Pseudoguaianolide from *Helenium amarum*

By Frank R. Fronczek, Alfonso G. Ober and Nikolaus H. Fischer

Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803, *USA*

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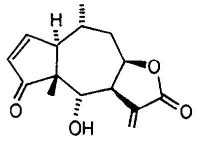
Abstract. 6α,8β-Dihydroxy-4-oxoambrosa-2,11(13)-dien-12-oic acid 12,8-lactone, $C_{15}H_{18}O_4$, $M_r = 262{\cdot}3$, monoclinic, $P2_1$, $a = 6{\cdot}1240$ (3), $b = 8{\cdot}2022$ (11), $c = 13{\cdot}8176$ (5) Å, $\beta = 98{\cdot}964$ (4)°, $V = 685{\cdot}6$ (3) Å^3, $Z = 2$, $D_x = 1{\cdot}271$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54184$ Å, $\mu = 7{\cdot}14$ cm^{-1}, $F(000) = 280$, $T = 299$ K, $R = 0{\cdot}036$ for 1394 observations (of 1528 unique data). Crystals of helenalin were isolated from *Helenium amarum* (Asteraceae) which was collected in East Baton Rouge Parish, Louisiana. The molecular structure agrees well with the previous determination of bromohelenalin [Ul-Haque & Caughlan (1969). *J. Chem. Soc. B*, pp. 956–960], including the molecular conformation. These two molecules exhibit a mean deviation of 4·1° over the 27 endocyclic torsion angles, with the largest single difference being 19·4°, for C(10)—C(1)—C(5)—C(6). The seven-membered ring in both has a distorted C_2 twist-chair conformation, the pseudodiad passing through C(10), with asymmetry parameter 13·6° for helenalin. Molecules are linked in the crystal in chains along the symmetry axis by hydrogen bonds involving the hydroxyl group and the carbonyl at C(4). The O···O distance is 2·728 (3) Å.

Experimental. The title compound (I) was obtained as colorless needles, dimensions 0·16 × 0·20 × 0·68 mm.



(I)

Space group from systematic absences 0*k*0 with *k* odd and chirality of material. Enraf–Nonius CAD-4 diffractometer with graphite monochromator and Cu $K\alpha$ radiation. Cell dimensions from setting angles of 25 reflections having $70 < 2\theta < 72°$. Data collection by ω–2θ scans designed for $I = 50\sigma(I)$, subject to 180 s maximum scan time. Scan rates varied 0·33–4·0° min^{-1}. Reflections having $2 < \theta < 75°$, $0 \le h \le 7$,

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