SHORT-FORMAT PAPERS

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Structure of Difluorotris(pentafluorophenyl)arsenic(V)

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Abstract. $[As(C_6F_5)_3F_2]$, $M_r = 614{\cdot}10$, orthorhombic, *Pbcn*, $a = 8{\cdot}959\ (6)$, $b = 11{\cdot}214\ (9)$, $c = 18{\cdot}343\ (9)$ Å, $V = 1843\ (2)$ Å^3, $Z = 4$, $D_x = 2{\cdot}213$ Mg m^{-3}, $\lambda(\text{Ag } K\alpha) = 0{\cdot}5608$ Å, $\mu = 1{\cdot}1$ mm^{-1}, $F(000) = 1176$, $T = 291\ (1)$ K, final $R = 0{\cdot}027$ for 1142 unique observed diffractometer data and 165 variables. The molecule has crystallographic symmetry 2. The atoms (two F, three C) bound to As form a trigonal bipyramid with the F atoms in the apical positions. Mean values of the bond distances: As–F 1·781 (2), As–C 1·915 (4) Å; average C–F 1·332 (5), C–C 1·372 (5) Å.

Experimental. The new compound was prepared by low-temperature liquid-phase direct fluorination of $(C_6F_5)_3As$ in 95% yield. Colourless single crystals obtained by slow evaporation of a hexane/chloroform solution at room temperature. Crystal size ~0·30 × 0·15 × 0·13 mm, $\omega/2\theta$ scan, scan speed 0·95–2·2° min^{-1} in θ, Nonius CAD-4 diffractometer, graphite-monochromated Ag $K\alpha$; lattice parameters from least-squares fit with 25 reflections up to $2\theta = 24{\cdot}0°$; four standard reflections recorded every 2·5 h, only random deviations; 3948 reflections measured; $1 \le \theta \le 20°$, $-10 \le h \le 10$, $0 \le k \le 13$, $0 \le l \le 22$; after averaging ($R_{int} = 0{\cdot}020$): 2031 unique reflections, 1142 with $I > 1{\cdot}96\sigma(I)$; Lorentz–polarization correction and absorption correction *via* ψ scans (transmission factors from 1·00 to 0·95; systematic absences $(0kl)$ $k = 2n + 1$, $(h0l)$ $l = 2n + 1$, $(hk0)$ $h + k = 2n + 1$, space group *Pbcn*; structure solution *via* direct methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all atoms; refinement on F with 1142 reflections and 165 refined parameters; $w =$

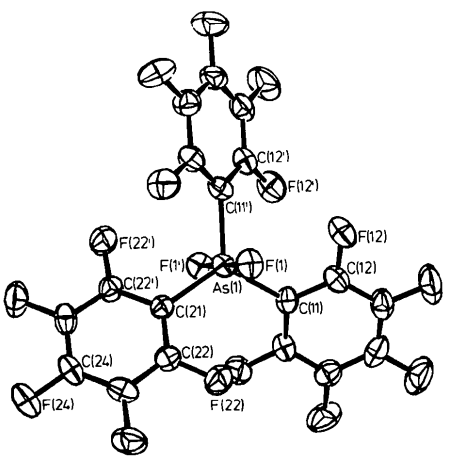

Fig. 1. General view of the molecule [symmetry code: (i)–x, y, $1{\cdot}5 - z$].

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å$^2 \times 10^3$)

$U_{eq} = (1/6\pi^2) \sum_i \sum_j \beta_{ij} \mathbf{a}_i . \mathbf{a}_j$.

	x	y	z	U_{eq}
As(1)	0·000	0·36909 (4)	0·750	32
F(1)	0·1882 (2)	0·3698 (2)	0·7187 (1)	42
F(12)	0·1176 (2)	0·6055 (2)	0·6876 (1)	56
F(13)	0·0407 (3)	0·7210 (2)	0·5654 (2)	73
F(14)	−0·1775 (4)	0·6344 (2)	0·4792 (1)	80
F(15)	−0·3140 (3)	0·4267 (3)	0·5131 (1)	74
F(16)	−0·2372 (3)	0·3091 (2)	0·6346 (1)	55
F(22)	−0·0576 (3)	0·1929 (2)	0·8755 (1)	55
F(23)	−0·0565 (3)	−0·0453 (2)	0·8746 (1)	60
F(24)	0·000	−0·1657 (2)	0·750	56
C(11)	−0·0587 (4)	0·4549 (3)	0·6644 (2)	35
C(12)	0·0105 (4)	0·5599 (3)	0·6454 (2)	41
C(13)	−0·0290 (4)	0·6206 (3)	0·5834 (2)	50
C(14)	−0·1401 (5)	0·5765 (3)	0·5399 (2)	50
C(15)	−0·2110 (4)	0·4711 (4)	0·5574 (2)	49
C(16)	−0·1698 (4)	0·4130 (3)	0·6195 (2)	40
C(21)	0·000	0·1983 (4)	0·750	33
C(22)	−0·0298 (4)	0·1360 (3)	0·8131 (2)	39
C(23)	−0·0289 (4)	0·0129 (3)	0·8123 (2)	42
C(24)	0·000	−0·0483 (4)	0·750	41

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$4F_o^2/[\sigma^2(F_o^2) + (0{\cdot}040F_o^2)^2]$; $S = 1{\cdot}00$, $R = 0{\cdot}027$, $wR = 0{\cdot}033$; $(\Delta/\sigma)_{max} = 0{\cdot}12$; no extinction correction; largest peak in final ΔF map $\pm 0{\cdot}2$ (1) e Å^{-3}; complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); programs: Enraf–Nonius *SDP* (Frenz, 1981), *ORTEP*II (Johnson, 1976), *MULTAN*80 (Main *et al.*, 1980). The structure of the title compound is shown in Fig. 1, positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1,* bond distances and angles are listed in Table 2.

Table 2. *Bond distances* (Å) *and angles* (°)

As(1)–F(1)	1·781 (2)	F(24)–C(24)	1·317 (5)
As(1)–C(21)	1·915 (4)	C(11)–C(12)	1·375 (5)
As(1)–C(11)	1·915 (3)	C(11)–C(16)	1·375 (5)
F(12)–C(12)	1·336 (4)	C(12)–C(13)	1·371 (5)
F(13)–C(13)	1·330 (4)	C(13)–C(14)	1·369 (6)
F(14)–C(14)	1·332 (5)	C(14)–C(15)	1·379 (6)
F(15)–C(15)	1·327 (5)	C(15)–C(16)	1·363 (5)
F(16)–C(16)	1·341 (4)	C(21)–C(22)	1·378 (4)
F(22)–C(22)	1·334 (4)	C(22)–C(23)	1·380 (5)
F(23)–C(23)	1·339 (4)	C(23)–C(24)	1·358 (4)
F(1)–As(1)–F(i)	180·0 (2)	C(13)–C(14)–C(15)	120·6 (4)
F(1)–As(1)–C(21)	90·26 (7)	F(15)–C(15)–C(16)	121·4 (3)
F(1)–As(1)–C(11)	89·6 (1)	F(15)–C(15)–C(14)	120·0 (4)
F(1)–As(1)–C(11i)	90·1 (1)	C(16)–C(15)–C(14)	118·7 (4)
C(21)–As(1)–C(11)	120·2 (1)	F(16)–C(16)–C(15)	117·8 (3)
C(11)–As(1)–C(11i)	119·7 (2)	F(16)–C(16)–C(11)	120·0 (3)
C(16)–C(11)–C(12)	117·9 (3)	C(15)–C(16)–C(11)	122·2 (3)
C(16)–C(11)–As(1)	121·2 (3)	C(22)–C(21)–C(22i)	119·0 (4)
C(12)–C(11)–As(1)	120·9 (3)	C(22)–C(21)–As(1)	120·5 (2)
F(12)–C(12)–C(13)	118·4 (3)	F(22)–C(22)–C(21)	120·9 (3)
F(12)–C(12)–C(11)	120·4 (3)	F(22)–C(22)–C(23)	119·2 (3)
C(13)–C(12)–C(11)	121·3 (3)	C(21)–C(22)–C(23)	119·9 (3)
F(13)–C(13)–C(14)	120·2 (4)	F(23)–C(23)–C(24)	120·5 (3)
F(13)–C(13)–C(12)	120·4 (4)	F(23)–C(23)–C(22)	118·5 (3)
C(14)–C(13)–C(12)	119·4 (3)	C(24)–C(23)–C(22)	121·0 (4)
F(14)–C(14)–C(13)	119·6 (4)	F(24)–C(24)–C(23)	120·4 (2)
F(14)–C(14)–C(15)	119·8 (4)	C(23)–C(24)–C(23i)	119·3 (4)

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43204 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Room-Temperature and Low-Temperature Structure of Triphenyltin Chloride*

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Abstract. $C_{18}H_{15}ClSn$, $M_r = 385{\cdot}5$, $P2_1/a$, $Z = 8$, $\lambda(\text{Mo } K\alpha_1) = 0{\cdot}70930$ Å, $F(000) = 1520$. At room temperature, $a = 18{\cdot}664$ (4), $b = 9{\cdot}721$ (4), $c = 18{\cdot}983$ (5) Å, $\beta = 105{\cdot}601$ (20)°, $V = 3317$ (3) Å^3, $D_x = 1{\cdot}544$ Mg m^{-3}, $\mu = 1{\cdot}70$ mm^{-1}, $R = 0{\cdot}033$ for 5060 unique reflections. At 110 K, $a = 18{\cdot}410$ (4), $b = 9{\cdot}5593$ (19), $c = 18{\cdot}704$ (5) Å, $\beta = 105{\cdot}201$ (19)°, $V = 3176$ (1) Å^3, $D_x = 1{\cdot}612$ Mg m^{-3}, $\mu = 1{\cdot}76$ mm^{-1}, $R = 0{\cdot}031$ for 5342 unique reflections. Our room-temperature results substantiate the results of an earlier report. There are eight molecules per unit cell with two crystallographically distinct molecules. Unlike its trimethyltin chloride analogue, the triphenyltin chloride molecules are loosely packed and unassociated in the crystalline state. The stereochemistry around the tin atoms is a slightly distorted tetrahedron. The average C–Sn–Cl and C–Sn–C valence angles are 105·21 (16) and 113·38 (20)° respectively. The shortest intermolecular Cl···Sn distance of 5·847 (2) Å is much longer than the sum of their van der Waals radii and precludes any reasonable intermolecular interaction. Cooling the crystal to 110 K did not lead to a phase transition as speculated earlier. The molecules remain discrete with the closest Cl···Sn distance shortened to 5·644 (2) Å. Inspection of the crystal packing diagram revealed no new or unusual feature compared with that of the room-temperature structure.

* NRCC contribution No. 25700.

Experimental. The title compound obtained from Alfa Products was recrystallized from a benzene solution. Crystal size 0·6 × 0·6 × 0·6 mm. Cell dimensions were obtained by least-squares refinement of 25 θ values measured on a Picker diffractometer. Intensity measurements were made using the θ–2θ mode up to