Refinement of the crystal structure of tris(perfluorophenyl)arsine, $C_{18}F_{15}As$, at 173 K

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Abstract

$AsC_{18}F_{15}$, triclinic, $P\bar{1}$ (No. 2), $a = 10.851(6)$ Å, $b = 11.012(5)$ Å, $c = 8.837(5)$ Å, $\alpha = 102.75(5)^\circ$, $\beta = 107.08(5)^\circ$, $\gamma = 109.81(4)^\circ$, $V = 887.4$ Å³, $Z = 2$, $R_{gt}(F) = 0.027$, $wR_{ref}(F^2) = 0.071$, $T = 173$ K.

Source of material

The compound was prepared in accord with the literature procedure [1]. Colorless crystals (mp 384 K) were obtained from the cooling of the mother liquor (benzene/hexane) to 273 K.

Discussion

New low-temperature data are presented for $(C_6F_5)_3As$ for which a room-temperature analysis has been reported previously [2]. The $d(As-C)$ range from 1.953(3) Å to 1.972(2) Å and have an average $d(As-C) = 1.965(2)$ Å. The angles subtended at trigonal pyramidal arsenic range from 96.1(1)° to 102.4(1)° and the dihedral angles between the three aromatic rings are 72.5(1)°, 73.4(2)° and 75.5(1)°, respectively, showing that the deviations from three-fold symmetry are relatively small. While the new data is in essential agreement with that reported earlier [2], the interatomic parameters have been determined to a higher level of precision in the present report.

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.15 × 0.24 × 0.27 mm
Wavelength:	Mo $K\alpha$ radiation (0.71069 Å)
μ :	20.74 cm ⁻¹
Diffractionmeter, scan mode:	Rigaku AFC7R, $\omega/2\theta$
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4286, 4074
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 3078
$N(param)_{refined}$:	307
Programs:	DIREDF92 [3], teXsan [4], SHELXL-97 [5], ORTEPII [6]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
As	2i	0.17367(3)	0.34683(3)	0.45163(3)	0.0275(1)	0.0234(1)	0.0232(1)	0.0106(1)	0.0101(1)	0.00856(9)
F(2)	2i	0.3883(2)	0.5256(2)	0.7921(2)	0.0260(8)	0.046(1)	0.0365(9)	0.0019(7)	0.0099(7)	0.0049(7)
F(3)	2i	0.3831(2)	0.5860(2)	1.1014(2)	0.047(1)	0.050(1)	0.0260(8)	0.0092(8)	0.0007(7)	−0.0020(7)
F(4)	2i	0.1361(2)	0.4599(2)	1.1388(2)	0.073(1)	0.070(1)	0.0284(8)	0.037(1)	0.0284(9)	0.0172(8)
F(5)	2i	−0.1005(2)	0.2689(2)	0.8674(2)	0.040(1)	0.079(1)	0.047(1)	0.018(1)	0.0287(9)	0.024(1)
F(6)	2i	−0.0970(2)	0.2032(2)	0.5597(2)	0.0246(8)	0.053(1)	0.0325(8)	0.0022(7)	0.0084(7)	0.0087(7)
F(8)	2i	0.1767(2)	0.1124(2)	0.6181(2)	0.0355(8)	0.0404(9)	0.0468(9)	0.0214(7)	0.0268(7)	0.0256(7)
F(9)	2i	0.3853(2)	0.0301(2)	0.7102(2)	0.049(1)	0.051(1)	0.047(1)	0.0325(9)	0.0134(8)	0.0249(8)
F(10)	2i	0.6149(2)	0.1245(2)	0.6338(2)	0.0259(9)	0.052(1)	0.064(1)	0.0228(8)	0.0045(8)	0.0016(9)
F(11)	2i	0.6321(2)	0.2934(2)	0.4508(3)	0.0310(9)	0.049(1)	0.079(1)	0.0092(8)	0.0342(9)	0.0062(9)
F(12)	2i	0.4247(2)	0.3781(2)	0.3590(2)	0.045(1)	0.0370(9)	0.0441(9)	0.0095(7)	0.0297(8)	0.0142(7)
F(14)	2i	0.0569(2)	0.0104(2)	0.2387(2)	0.0360(9)	0.0309(8)	0.0404(9)	0.0198(7)	0.0065(7)	0.0059(7)
F(15)	2i	−0.1924(2)	−0.1560(2)	−0.0204(2)	0.051(1)	0.0307(8)	0.0406(9)	0.0135(8)	0.0070(8)	−0.0016(7)

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Table 2. Continued.

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
F(16)	2i	−0.3959(2)	−0.0665(2)	−0.1206(2)	0.0333(9)	0.054(1)	0.0394(9)	0.0107(8)	−0.0031(7)	0.0043(8)
F(17)	2i	−0.3460(2)	0.1963(2)	0.0447(2)	0.038(1)	0.062(1)	0.057(1)	0.0314(9)	0.0050(8)	0.0181(9)
F(18)	2i	−0.0945(2)	0.3655(2)	0.3086(2)	0.045(1)	0.0351(9)	0.047(1)	0.0256(8)	0.0082(8)	0.0053(7)
C(1)	2i	0.1463(3)	0.3636(2)	0.6650(3)	0.026(1)	0.023(1)	0.022(1)	0.014(1)	0.0087(9)	0.0058(9)
C(2)	2i	0.2653(3)	0.4603(3)	0.8079(3)	0.025(1)	0.029(1)	0.030(1)	0.011(1)	0.010(1)	0.007(1)
C(3)	2i	0.2650(3)	0.4930(3)	0.9671(3)	0.036(1)	0.033(1)	0.022(1)	0.016(1)	0.005(1)	0.003(1)
C(4)	2i	0.1397(3)	0.4289(3)	0.9853(3)	0.049(2)	0.041(2)	0.025(1)	0.029(1)	0.019(1)	0.014(1)
C(5)	2i	0.0199(3)	0.3320(3)	0.8472(3)	0.031(1)	0.045(2)	0.035(1)	0.020(1)	0.019(1)	0.018(1)
C(6)	2i	0.0239(3)	0.3005(3)	0.6890(3)	0.025(1)	0.032(1)	0.026(1)	0.012(1)	0.009(1)	0.008(1)
C(7)	2i	0.2972(2)	0.2528(2)	0.4916(3)	0.022(1)	0.025(1)	0.023(1)	0.009(1)	0.0100(9)	0.0042(9)
C(8)	2i	0.2903(3)	0.1611(2)	0.5786(3)	0.023(1)	0.030(1)	0.026(1)	0.011(1)	0.011(1)	0.007(1)
C(9)	2i	0.3951(3)	0.1181(3)	0.6262(3)	0.030(1)	0.030(1)	0.028(1)	0.014(1)	0.005(1)	0.006(1)
C(10)	2i	0.5116(3)	0.1654(3)	0.5855(3)	0.021(1)	0.033(1)	0.038(1)	0.013(1)	0.003(1)	−0.005(1)
C(11)	2i	0.5207(3)	0.2510(3)	0.4938(3)	0.020(1)	0.033(1)	0.039(1)	0.003(1)	0.013(1)	−0.003(1)
C(12)	2i	0.4142(3)	0.2940(2)	0.4493(3)	0.030(1)	0.024(1)	0.029(1)	0.006(1)	0.015(1)	0.003(1)
C(13)	2i	−0.0092(3)	0.1958(2)	0.2839(3)	0.029(1)	0.028(1)	0.020(1)	0.013(1)	0.010(1)	0.0082(9)
C(14)	2i	−0.0394(3)	0.0614(3)	0.1962(3)	0.031(1)	0.030(1)	0.025(1)	0.015(1)	0.011(1)	0.011(1)
C(15)	2i	−0.1674(3)	−0.0261(3)	0.0609(3)	0.036(1)	0.027(1)	0.027(1)	0.011(1)	0.011(1)	0.005(1)
C(16)	2i	−0.2714(3)	0.0187(3)	0.0100(3)	0.027(1)	0.040(1)	0.025(1)	0.008(1)	0.005(1)	0.008(1)
C(17)	2i	−0.2460(3)	0.1515(3)	0.0938(3)	0.028(1)	0.045(2)	0.035(1)	0.020(1)	0.008(1)	0.018(1)
C(18)	2i	−0.1174(3)	0.2359(3)	0.2279(3)	0.035(1)	0.030(1)	0.029(1)	0.017(1)	0.011(1)	0.010(1)

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