Preparation of Trimeric and Tetrameric Bis(trifluoromethyl)arsazene; *X*-Ray Study of $[(CF_3)_2AsN]_4$ †

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Two new aminoarsines, $(CF_3)_2AsN(SiMe_3)_2$ (1) and $CF_3As[N(SiMe_3)_2]_2$ (2), are formed by reaction of $(CF_3)_2AsCl$ and $(CF_3)AsCl_2$ with $LiN(SiMe_3)_2$. Chlorination of (1) yields $[(CF_3)_2As(Cl)N(SiMe_3)]_2$ (3). On refluxing (3) in n-hexane or n-heptane, the synthesis of trimeric and tetrameric bis(trifluoromethyl)-arsazenes, $[(CF_3)_2AsN]_3$ (4) and $[(CF_3)_2AsN]_4$ (5), is accomplished. Pyrolysis of (3) also produced (4) and (5). Crystals of (5) are triclinic, space group $P\bar{1}$, with a = 991.2(5), b = 1 012.9(6), c = 1 432.8(7) pm, α = 108.21(4), β = 96.03(4), γ = 109.44(4)°, and Z = 2. The structure was solved by direct methods and refined to R 0.069 for 2 183 unique observed diffractometer data. The molecular symmetry approximates to $\bar{4}$ (S_4), with a slight alternation in As–N distances [171.6(7) and 173.2(9) pm].

The chemistry of trimeric and tetrameric phosphazenes, $(NPX_2)_n$ (n = 3 or 4, and X is a halogen or an organic substituent) is well established.[1] Analogous compounds with arsenic are rare. To our knowledge only two phenyl-substituted arsazenes, $(NAsPh_2)_n$ (n = 3 or 4), involving six- and eight-membered As–N rings, have been investigated by *X*-ray analysis.[2, 3] We were interested in the preparation and characterisation of compounds which contain electron-withdrawing groups attached to arsenic, because it is known that phosphazenes with similar groups exhibit unusual properties.[4] We report here the isolation and characterisation of the pure trimer and tetramer of $(CF_3)_2AsN$.

Experimental

All manipulations were conducted under dry nitrogen using standard techniques; i.r. spectra were recorded on a Perkin-Elmer model 180 spectrometer and are accurate to ±1 cm^{-1}. Bruker WP 80SY and 60E spectrometers were used to obtain ^{19}F n.m.r. spectra. The iodoarsenes $(CF_3)_2AsI$ and CF_3AsI_2 were made by the literature methods [5,6] and converted to $(CF_3)_2AsCl$ and CF_3AsCl_2 by repeated treatment with $HgCl_2$.[7] The compound $LiN(SiMe_3)_2$ was prepared by the usual method.[8]

Preparation of $(CF_3)_2AsN(SiMe_3)_2$ (1).—To a mixture of $(CF_3)_{3-n}AsCl_n$ (n = 0—2; 22 g) in n-hexane (40 cm^3) at −20 °C was added a n-hexane solution of $LiN(SiMe_3)_2$ (10 g, 40 cm^3). The mixture was allowed to warm up slowly and stirred for about 4 h. LiCl was filtered off, and solvent and unreacted $(CF_3)_3As$ removed under vacuum. From the remaining residue, $(CF_3)_2AsN(SiMe_3)_2$ (1) distilled out at 23 °C (0.1 mmHg) as a colourless liquid (0.8 g) [Found: C, 26.5; H, 5.2; F, 30.6; N, 3.8. Calc. for $(CF_3)_2AsN(SiMe_3)_2$: C, 25.75; H, 4.8; F, 30.55; N, 3.75%]. I.r. (neat) 2 965s, 2 910m, 2 860w, 1 434w, 1 405m, 1 272vs, 1 258vs, 1 178vs, 1 132vs, 1 118vs, 1 096vs, 890vs, 865vs, 848vs, 828vs, 805 (sh), 760m, 734w, 722s, and 676m cm^{-1}. ^{19}F N.m.r. (CH_2Cl_2): $\delta(CF_3)$ −51.5 p.p.m.

Preparation of $CF_3As[N(SiMe_3)_2]_2$ (2).—In the above reaction, (2) is also formed and was isolated by further distillation of the residue at 75 °C (0.1 mmHg) as a yellow-brown liquid (1.5 g) (Found: C, 33.8; H, 8.0; N, 5.9. Calc. for $CF_3As[N(SiMe_3)_2]_2$: C, 33.6; H, 7.75; N, 6.05%). I.r. (neat) 2 958s, 2 905m, 1 435w, 1 405w, 1 256vs, 1 180m, 1 152vs, 1 108vs, 1 078s, 1 016w, 965 (sh), 922vs, 902vs, 868 (sh), 848vs, 792s, 762s, 732w, 701s, and 672s cm^{-1}. ^{19}F N.m.r. (CH_2Cl_2): $\delta(CF_3)$ −54.2 p.p.m.

Preparation of $[(CF_3)_2As(Cl)N(SiMe_3)]_2$ (3).—Chlorine [1.37 g, 19.5 mmol (5% excess)] was condensed onto a solution of (1) (6.95 g, 18.6 mmol) in CH_2Cl_2 (20 cm^3) cooled to liquid-air temperature and the mixture was allowed to warm up slowly and stirred at room temperature for 2 h. The progress of the reaction was checked by ^{19}F n.m.r. A strong signal at $\delta(CF_3)$ −54.2 p.p.m. was initially observed which disappeared completely within 24 h with increase of another signal at $\delta(CF_3)$ −58.1 p.p.m., indicating initial formation of $(CF_3)_2As(Cl){=}N(SiMe_3)$ and its complete conversion to the dimer (3). When the mixture was kept at −30 °C for one day, the dimer separated out as a white crystalline solid (3.9 g, 56%). Compound (3) can be recrystallised from n-hexane or CH_2Cl_2. It can also be sublimed without change at 40 °C and 1 mmHg (Found: C, 17.5; H, 2.8; Cl, 10.7; N, 4.7. Calc. for $[(CF_3)_2As(Cl)N(SiMe_3)]_2$: C, 17.9; H, 2.7; Cl, 10.55; N, 4.15%). I.r. (Nujol) 2 960vs, 2 930vs, 2 875 (sh), 2 860vs, 1 460s, 1 408w, 1 376m, 1 258vs, 1 206vs, 1 188vs, 1 166vs, 1 104s, 1 085 (sh), 978w, 882vs, 852vs, 828vs, 810 (sh), 768w, 742vs, and 678w cm^{-1}. ^{19}F N.m.r. (CH_2Cl_2): $\delta(CF_3)$ −58.1 p.p.m. M.p. 116 °C (sealed capillary).

Preparation of $[(CF_3)_2AsN]_3$ (4) *and* $[(CF_3)_2AsN]_4$ (5).—A mixture of arsazenes (4) and (5) was obtained when (3) (3 g) was refluxed (bath temperature 120 °C) in n-hexane (or n-heptane) (20 cm^3) for about 8 h. The progress of the reaction was followed by ^{19}F n.m.r. The signal at $\delta(CF_3)$ −58.1 p.p.m. disappeared and two signals at $\delta(CF_3)$ −60.8 and −62.8 p.p.m. were observed. The solvent and $SiMe_3Cl$ were removed from

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† Cyclotetra[bis(trifluoromethyl)-λ^5-arsazene].

Supplementary data available (No. SUP 23536; 15 pp.): structure factors, thermal parameters. See Notices to Authors No. 7, *J. Chem. Soc., Dalton Trans.*, 1981, Index issue.

Non-S.I. unit employed: 1 mmHg = (101 325/760) Pa.

the mixture under reduced pressure. Vacuum sublimation of the residue yielded (4) (23 °C, 1 mmHg; 0.75 g, 25%) and (5) (38 °C, 1 mmHg; 0.9 g, 30%) as crystalline solids. Compound (4) was recrystallised from CH_2Cl_2–CCl_4 (*ca.* 1 : 1) (Found: C, 10.7; N, 6.1. Calc. for $[(CF_3)_2AsN]_3$: C, 10.55; N, 6.15%). I.r. (Nujol) 2 956vs, 2 915vs, 2 865 (sh), 2 856vs, 1 455s, 1 378m, 1 276m, 1 180vs, br, 1 162vs, 1 118vs, 978vs, and 742m cm^{-1}. ^{19}F N.m.r. (CH_2Cl_2): $\delta(CF_3)$ −60.8 p.p.m. M.p. 40 °C (sealed capillary). Compound (5) was crystallised from n-hexane (Found: C, 10.3; N, 6.1. Calc. for $[(CF_3)_2AsN]_4$: C, 10.55; N, 6.15%). I.r. (Nujol) 2 955vs, 2 926vs, 2 875 (sh), 2 858vs, 1 454m, 1 402w, 1 370w, 1 184s, 1 165s, 1 124s, 986m, and 745w cm^{-1}. ^{19}F N.m.r. (n-hexane): $\delta(CF_3)$ −62.8 p.p.m. M.p. 65 °C (sealed capillary).

When (3) was pyrolysed under vacuum (150 °C; 0.01 mmHg) in a system which was directly connected to the mass spectrometer, formation of only the two products (4) and (5) was observed.

Mass spectra. Field ionisation mass spectra were obtained on a Varian CH5 at room temperature. They showed the high stability of the molecular ions; no fragmentation occurred. The electronic ionisation mass spectra, using a direct-probe inlet system, were recorded with a Varian MAT 731 with data system at 70 eV ionisation voltage and 100 °C source temperature. Accurate mass measurements were made by peak-matching. Measured masses are generally accurate to better than 5 p.p.m. Table 1 shows the most intense ions (characteristic peaks) of $[(CF_3)_2AsN]_3$ (4) and $[(CF_3)_2AsN]_4$ (5).

Crystal Structure Determination.—A colourless prismatic crystal of (5) (0.25 × 0.02 × 0.01 mm) prepared as described above was sealed in a Lindemann glass capillary. Intensities were measured by profile analysis [9] on a Stoe-Siemens four-circle diffractometer with graphite-monochromated Mo-K_α radiation.

Crystal data. $C_8As_4F_{24}N_4$, $M = 907.76$, Triclinic, space group $P\bar{1}$, $a = 991.2(5)$, $b = 1\,012.9(6)$, $c = 1\,432.8(7)$ pm, $\alpha = 108.21(4)$, $\beta = 96.03(4)$, $\gamma = 109.44(4)°$, $U = 1.2530$ nm^3, $Z = 2$, $D_c = 2.406$ Mg m^{-3}, $F(000) = 848$, Mo-K_α radiation, $\lambda = 71.069$ pm, $\mu = 5.46$ mm^{-1}.

3 267 Unique data were collected in the range $7 < 2\theta < 50°$, of which 2 183 with $F > 4\sigma(F)$ were used for all calculations. Lorentz, polarisation, and semi-empirical absorption corrections were applied. The structure was solved by multi-solution sigma-2 refinement and refined with all atoms anisotropic, complex neutral-atom scattering factors, and weights $w^{-1} = [\sigma^2(F) + 0.0012\ F^2]$ to $R' = \Sigma w^{\frac{1}{2}}\Delta/\Sigma w^{\frac{1}{2}}|F_o| = 0.068$, $R = \Sigma\Delta/\Sigma|F_o| = 0.069$. The final atomic co-ordinates are given in Table 2, bond lengths and angles in Table 3.

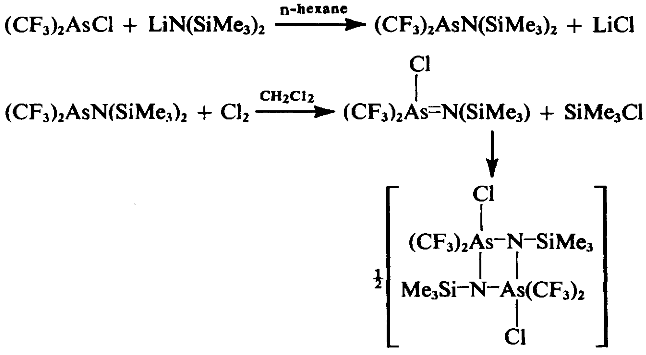
Crystallographic calculations were performed using an Eclipse S-250 mini-computer and programs written by G. M. S. Data collection programs were written by Dr. W. Clegg.

Results and Discussion

In a recent study [10] on As–N systems in which electron-withdrawing CF_3 groups are attached to arsenic we were able to synthesise and characterise compound (3) (Scheme 1). The formation of the intermediate monomer $(CF_3)_2As(Cl)=N(SiMe_3)$ was indicated by ^{19}F n.m.r. spectroscopy; however, it could not be isolated. This suggests that overlap of the 2*p* nitrogen orbitals with the 4*d* arsenic orbitals is ineffective even though electron-withdrawing groups are attached to arsenic. Compound (3) is a white crystalline solid, characterised by *X*-ray crystallography.[10] When it is refluxed in inert solvents or pyrolysed under vacuum, (4) and (5) are formed (Scheme 2). A number of experiments have indicated that (4) and (5), which are white crystalline solids, can be obtained pure by repeated vacuum sublimation.

Table 1. Mass spectral data for $[(CF_3)_2AsN]_3$ and $[(CF_3)_2AsN]_4$

Mass	$[(CF_3)_2AsN]_3$ Relative intensity (%)	$[(CF_3)_2AsN]_4$ Relative intensity (%)
908	—	10.81 M^+
839	—	100 $(M - CF_3)^+$
789	—	5.8 $(M - C_2F_5)^+$
681	3 M^+	—
612	100 $(M - CF_3)^+$	25.4
562	7 $(M - C_2F_5)^+$	11.7
474	25	13.2
429	16.9	30.3
385	51.4	32.9
340	20.3	17.7
202	37.7	51.4
183	29.6	42.2
164	26.6	42.6
158	23.5	88.9
113	43	82.9
69	85.4	96.3

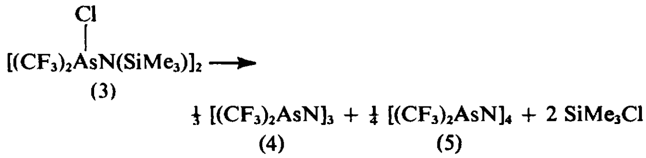


$$(CF_3)_2AsCl + LiN(SiMe_3)_2 \xrightarrow{\text{n-hexane}} (CF_3)_2AsN(SiMe_3)_2 + LiCl$$

$$(CF_3)_2AsN(SiMe_3)_2 + Cl_2 \xrightarrow{CH_2Cl_2} (CF_3)_2As(Cl){=}N(SiMe_3) + SiMe_3Cl \longrightarrow \tfrac{1}{2}[(CF_3)_2As(Cl)\text{–}N(SiMe_3)]_2$$

(3)

Scheme 1.



$$[(CF_3)_2As(Cl)N(SiMe_3)]_2 \longrightarrow \tfrac{1}{3}[(CF_3)_2AsN]_3 + \tfrac{1}{4}[(CF_3)_2AsN]_4 + 2\ SiMe_3Cl$$

(3) (4) (5)

Scheme 2.

Although the molecule of (5) possesses no crystallographic symmetry, it approximates fairly accurately to $\bar{4}$ (S_4) local molecular symmetry. The Figure shows the view down the pseudo-$\bar{4}$ axis. The As and N atoms lie alternatively at ±38 and ±63 pm respectively from the mean plane through the eight ring atoms. There is a barely significant alternation of the As–N bond lengths, bonds in which both atoms are on the same side of the mean plane being slightly shorter [mean 171.6(7) pm] than those which cross the mean plane [mean 173.2(9) pm]. A much more pronounced bond-length alternation [167(3) and 179(3) pm] was observed [2] in $[Ph_2AsN]_4$. The trimer $[Ph_2AsN]_3$ exhibited no alternation, but a longer mean As–N distance of 175.8(4) pm.[3] The ring conformation

Table 2. Fractional atomic co-ordinates ($\times 10^4$) with estimated standard deviations in parentheses

Atom	*x*	*y*	*z*	Atom	*x*	*y*	*z*
As(1)	19 959(2)	2 383(1)	3 183(1)	F(15)	20 873(17)	4 284(13)	2 187(11)
As(2)	16 718(2)	1 725(2)	2 494(1)	F(16)	22 587(13)	4 166(14)	3 023(16)
As(3)	16 152(2)	−1 459(2)	2 401(1)	F(21)	17 481(20)	1 871(18)	759(11)
As(4)	19 202(2)	−933(2)	2 003(1)	F(22)	17 197(18)	3 822(13)	1 604(12)
N(1)	18 380(13)	2 642(12)	3 386(10)	F(23)	15 443(15)	1 860(18)	777(11)
N(2)	15 810(13)	−205(12)	1 928(9)	F(24)	14 112(11)	2 006(15)	2 701(13)
N(3)	17 809(12)	−1 648(12)	2 571(9)	F(25)	15 409(17)	2 396(21)	4 043(12)
N(4)	20 067(13)	937(11)	2 197(8)	F(26)	15 873(13)	4 024(12)	3 427(14)
C(11)	20 855(22)	2 536(23)	4 519(16)	F(31)	14 311(14)	−1 379(18)	3 691(9)
C(12)	21 287(22)	4 179(20)	3 029(15)	F(32)	15 626(16)	−2 528(17)	3 920(9)
C(21)	16 747(21)	2 395(20)	1 320(15)	F(33)	16 559(15)	−223(15)	4 433(8)
C(22)	15 395(18)	2 606(18)	3 158(15)	F(34)	13 320(11)	−3 456(12)	1 538(11)
C(31)	15 654(21)	−1 332(21)	3 739(14)	F(35)	14 745(13)	−4 526(10)	1 646(9)
C(32)	14 595(18)	−3 397(16)	1 460(13)	F(36)	14 734(14)	−3 628(11)	559(8)
C(41)	18 721(18)	−1 924(19)	515(15)	F(41)	17 730(15)	−1 596(16)	112(8)
C(42)	20 669(20)	−1 709(19)	2 398(18)	F(42)	18 142(17)	−3 395(12)	244(10)
F(11)	20 936(15)	3 716(15)	5 251(9)	F(43)	19 827(13)	−1 565(13)	131(9)
F(12)	20 279(16)	1 370(16)	4 709(9)	F(44)	21 210(19)	−1 106(22)	3 346(12)
F(13)	22 309(14)	2 787(17)	4 591(10)	F(45)	20 162(15)	−3 100(13)	2 119(16)
F(14)	21 452(16)	5 390(13)	3 696(13)	F(46)	21 807(14)	−1 289(18)	2 010(13)

Table 3. Bond distances (pm) and angles (°) with estimated standard deviations in parentheses

(*a*) Distances

As(1)–N(1)	171.0(15)	C(12)–F(15)	128.1(29)	As(1)–N(4)	173.1(12)	C(12)–F(16)	129.3(28)
As(1)–C(11)	196.1(24)	C(21)–F(21)	125.4(30)	As(1)–C(12)	195.2(21)	C(21)–F(22)	127.2(23)
As(2)–N(1)	173.4(12)	C(21)–F(23)	127.6(23)	As(2)–N(2)	172.5(11)	C(22)–F(24)	121.6(19)
As(2)–C(21)	200.0(24)	C(22)–F(25)	135.0(30)	As(2)–C(22)	198.2(21)	C(22)–F(26)	126.4(20)
As(3)–N(2)	172.1(16)	C(31)–F(31)	131.0(27)	As(3)–N(3)	171.7(14)	C(31)–F(32)	130.7(30)
As(3)–C(31)	200.6(22)	C(31)–F(33)	122.4(18)	As(3)–C(32)	198.6(14)	C(32)–F(34)	126.3(22)
As(4)–N(3)	174.2(14)	C(32)–F(35)	130.2(23)	As(4)–N(4)	171.4(12)	C(32)–F(36)	127.0(23)
As(4)–C(41)	197.7(20)	C(41)–F(41)	127.5(28)	As(4)–C(42)	198.0(24)	C(41)–F(42)	131.2(21)
C(11)–F(11)	129.3(25)	C(41)–F(43)	127.4(24)	C(11)–F(12)	125.7(29)	C(42)–F(44)	127.1(28)
C(11)–F(13)	136.4(26)	C(42)–F(45)	123.7(21)	C(12)–F(14)	124.3(23)	C(42)–F(46)	131.3(28)

(*b*) Angles

N(1)–As(1)–N(4)	125.6(5)	As(3)–C(31)–F(31)	109.2(14)	As(1)–N(4)–As(4)	123.5(8)
N(4)–As(1)–C(11)	113.5(9)	F(31)–C(31)–F(32)	105.1(17)	As(1)–C(11)–F(12)	113.8(11)
N(4)–As(1)–C(12)	102.6(8)	F(31)–C(31)–F(33)	112.7(19)	As(1)–C(11)–F(13)	109.9(16)
N(1)–As(2)–N(2)	124.8(7)	As(3)–C(32)–F(34)	112.1(11)	F(12)–C(11)–F(13)	106.9(22)
N(2)–As(2)–C(21)	103.1(7)	F(34)–C(32)–F(35)	107.3(15)	As(1)–C(12)–F(15)	112.8(11)
N(2)–As(2)–C(22)	109.4(6)	F(34)–C(32)–F(36)	111.5(17)	As(1)–C(12)–F(16)	111.7(17)
N(2)–As(3)–N(3)	124.9(6)	As(4)–C(41)–F(41)	110.4(14)	F(15)–C(12)–F(16)	104.4(21)
N(3)–As(3)–C(31)	101.4(8)	F(41)–C(41)–F(42)	105.0(14)	As(2)–C(21)–F(22)	111.8(14)
N(3)–As(3)–C(32)	110.1(7)	F(41)–C(41)–F(43)	108.2(20)	As(2)–C(21)–F(23)	109.6(15)
N(3)–As(4)–N(4)	124.8(6)	As(4)–C(42)–F(44)	111.0(17)	F(22)–C(21)–F(23)	106.5(21)
N(4)–As(4)–C(41)	103.0(7)	F(44)–C(42)–F(45)	110.5(25)	As(2)–C(22)–F(25)	107.1(15)
N(4)–As(4)–C(42)	109.6(7)	F(44)–C(42)–F(46)	103.8(16)	As(2)–C(22)–F(26)	113.2(14)
As(1)–N(1)–As(2)	123.8(7)	N(1)–As(1)–C(11)	101.6(9)	F(25)–C(22)–F(26)	103.2(17)
As(3)–N(3)–As(4)	122.9(9)	N(1)–As(1)–C(12)	108.2(8)	As(3)–C(31)–F(32)	109.9(14)
As(1)–C(11)–F(11)	113.9(18)	C(11)–As(1)–C(12)	103.6(9)	As(3)–C(31)–F(33)	111.8(15)
F(11)–C(11)–F(12)	110.4(20)	N(1)–As(2)–C(21)	112.9(7)	F(32)–C(31)–F(33)	107.9(19)
F(11)–C(11)–F(13)	100.9(14)	N(1)–As(2)–C(22)	103.8(7)	As(3)–C(32)–F(35)	110.6(12)
As(1)–C(12)–F(14)	114.6(17)	C(21)–As(2)–C(22)	100.3(10)	As(3)–C(32)–F(36)	110.4(11)
F(14)–C(12)–F(15)	105.9(20)	N(2)–As(3)–C(31)	114.8(8)	F(35)–C(32)–F(36)	104.6(15)
F(14)–C(12)–F(16)	106.6(15)	N(2)–As(3)–C(32)	101.2(7)	As(4)–C(41)–F(42)	110.4(16)
As(2)–C(21)–F(21)	110.5(18)	C(31)–As(3)–C(32)	102.5(8)	As(4)–C(41)–F(43)	113.3(10)
F(21)–C(21)–F(22)	112.2(19)	N(3)–As(4)–C(41)	114.4(6)	F(42)–C(41)–F(43)	109.0(17)
F(21)–C(21)–F(23)	105.9(16)	N(3)–As(4)–C(42)	101.7(9)	As(4)–C(42)–F(45)	112.7(13)
As(2)–C(22)–F(24)	114.9(13)	C(41)–As(4)–C(42)	100.9(9)	As(4)–C(42)–F(46)	109.9(18)
F(24)–C(22)–F(25)	105.5(18)	As(2)–N(2)–As(3)	123.9(7)	F(45)–C(42)–F(46)	108.5(19)
F(24)–C(22)–F(26)	111.9(19)				

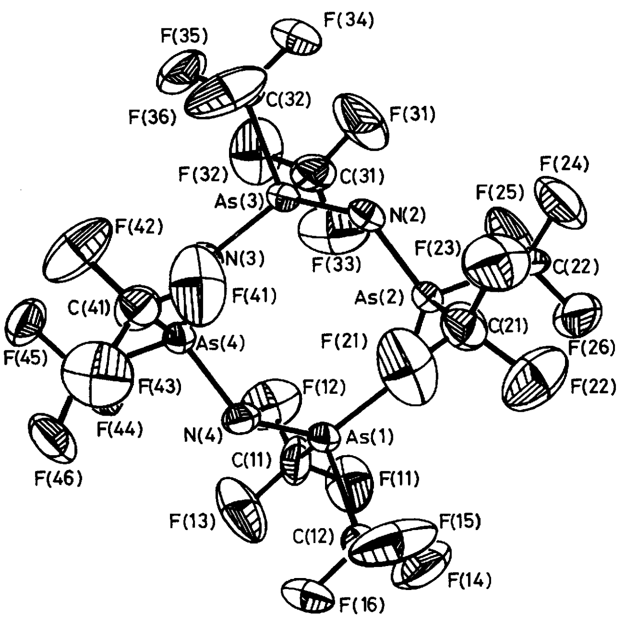


Figure. The molecule of $[(CF_3)_2AsN]_4$ (5) viewed down the pseudo-inverse tetrad axis, showing 30% probability thermal motion ellipsoids

results in a clear differentiation of the CF_3 group environments, four being axial and four equatorial. As frequently observed, the CF_3 groups exhibit appreciable thermal motion about the As–C bond directions, and so 30% probability ellipsoids are shown in the Figure (instead of the usual 50%).

Acknowledgements

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