1045 reflections
88 parameters
Only coordinates of H atoms refined

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{eq} = (1/3)\Sigma_i\Sigma_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
C1	0.4443 (3)	0.6395 (1)	0.2319 (1)	2.88 (2)
C2	0.6375 (3)	0.8022 (1)	0.3514 (1)	3.16 (2)
C3	0.2590 (3)	0.8025 (1)	0.1111 (1)	2.78 (2)
C4	−0.0579 (5)	3/4	−0.1172 (2)	4.05 (4)
C5	0.0579 (3)	0.8467 (1)	−0.0250 (1)	3.46 (3)
C6	0.6012 (3)	0.4770 (1)	0.3259 (2)	3.73 (3)
N1	0.4286 (3)	0.53800 (9)	0.2224 (1)	3.61 (2)
N2	0.2648 (4)	0.5851 (1)	0.5979 (2)	5.19 (3)

Table 2. *Selected geometric parameters* (Å, °)

C1—N1	1.307 (2)	C3···C5′	2.365 (2)
C1···C1′	2.839 (2)	C3···C1′	2.434 (2)
C1···C5	2.637 (2)	C3···C4	2.390 (2)
C1···C6	2.310 (2)	C3′···C4	2.390 (2)
C2—C2′	1.340 (2)	C3···N1	2.346 (2)
C2—C1	1.456 (2)	C5—C4	1.538 (2)
C2···C1′	2.434 (2)	C5′—C4	1.538 (2)
C2···C3	2.491 (2)	C5···C5′	2.483 (2)
C2···C3′	2.831 (2)	C5···N1	2.932 (2)
C2···C6	2.847 (2)	N1—C6	1.344 (2)
C2···N1	2.455 (2)	N2—C6	1.142 (2)
C3—C3′	1.347 (2)	N2···N1	2.478 (2)
C3—C5	1.499 (2)		
C2—C1—N1	125.3 (1)	C1—C3—C5	126.8 (1)
C2′—C2—C1	121.0 (1)	C3′—C3—C1	121.0 (1)
C3′—C3—C5	112.2 (1)	C5—C4—C5′	107.7 (1)
N2—C6—N1	171.2 (1)	C1—N1—C6	121.3 (1)

Data collection: Enraf-Nonius CAD-4 computer controlled diffractometer. Cell constants and the orientation matrix for data collection were obtained from least-squares refinement, using setting angles measured by a computer controlled diagonal slit method of centering. All calculations were performed on a VAX computer using *SDP/VAX* (Frenz, 1978). Refinement was by full-matrix least squares.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71550 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SE1017]

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A Cubic Modification of (Triphenylarsine)diiodine

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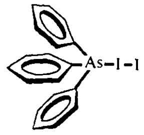
Abstract

Cubic packing has allowed (triphenylarsine)diiodine, $(C_6H_5)_3AsI_2$, to adopt strict C_3 point symmetry with the three phenyl rings arranged in the form of a propeller around the linear As—I—I chain [I—As—C—C 54 (2)°]. Donation of the arsenic lone pair to iodine [As—I 2.616 (3) Å] considerably weakens the I—I bond [I—I 3.014 (3) Å].

Comment

The cubic form of Ph_3AsI_2 (I) is a further example of the four-coordinate 'spoke' structure established for other crystallographic modifications of this compound (Beagley *et al.*, 1988) and for Ph_3PXX [X = Br (Bricklebank, Godfrey, Mackie, McAuliffe & Pritchard, 1992), I

(Godfrey *et al.*, 1991)]. These charge-transfer adducts contrast with compounds such as Ph_3AsF_2 (Augustine, Ferguson & March, 1975) and Ph_3PF_2 (Weller, Nuszhar, Dehnicke, Gingl & Strahle, 1991), which have trigonal-bipyramidal geometry.



(I)

Although cubic packing has not led to an unusual molecular conformation, the As—I bond length of 2.616 (3) Å is short by comparison with the above modifications [2.638 (2)-2.653 (2) Å] and the I—I distance is long [3.014 (3) *cf.* 3.002 (2)-3.005 (2) Å]. This suggests that non-bonding interactions of the type seen in I_3^- (Belanger & Beauchamp, 1993) are also able to influence the degree of polarization of this molecular compound.

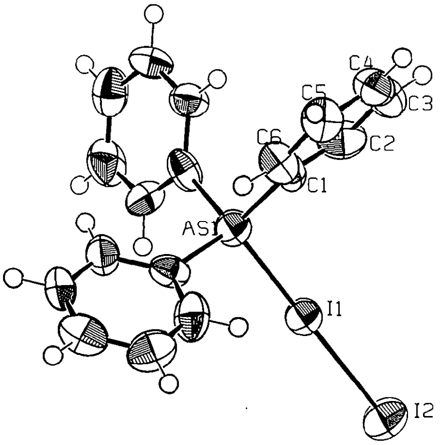


Fig. 1. An *ORTEP*II (Johnson, 1976) plot of the title molecule, showing the atomic-numbering scheme. Thermal ellipsoids are plotted at the 50% probability level.

Experimental

Ph_3AsI_2 was prepared directly from triphenylarsine and diiodine according to the published method (Godfrey, 1993). The bright yellow compound was recrystallized from diethyl ether to produce orange crystals suitable for single-crystal X-ray analysis.

Crystal data

$C_{18}H_{15}AsI_2$
$M_r = 560.05$
Cubic
$Pa\bar{3}$
$a = 15.448$ (3) Å
$V = 3686.9$ (7) Å^3
$Z = 8$
$D_x = 2.018$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 23 reflections
$\theta = 6.41$-$12.43°$
$\mu = 5.1465$ mm^{-1}
$T = 296$ K
Needle
0.30 × 0.20 × 0.20 mm
Orange

Data collection

AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
$T_{\min} = 0.64$, $T_{\max} = 1.0$
1918 measured reflections
1221 independent reflections
439 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int} = 0.062$

$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 18$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 18$
3 standard reflections monitored every 150 reflections intensity variation: −1.30%

Refinement

Refinement on *F*
$R = 0.041$
$wR = 0.042$
$S = 1.28$
439 reflections
65 parameters
H-atom parameters not refined
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{\max} = 0.006$

$\Delta\rho_{\max} = 0.61$ e Å^{-3}
$\Delta\rho_{\min} = -0.53$ e Å^{-3}
Extinction correction: Zachariasen (1967) type II Gaussian, isotropic
Extinction coefficient: 7 (2) × 10^{-8}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$U_{\rm eq}$
I(1)	0.78896 (6)	x	x	0.0554
I(2)	0.67634 (7)	x	x	0.0782
As(1)	0.88671 (10)	x	x	0.0508
C(1)	0.8215 (10)	0.980 (1)	0.9347 (9)	0.0526
C(2)	0.749 (1)	0.963 (1)	0.9781 (10)	0.0683
C(3)	0.697 (1)	1.024 (1)	1.016 (1)	0.0736
C(4)	0.724 (1)	1.109 (1)	1.009 (1)	0.0714
C(5)	0.797 (1)	1.1280 (10)	0.9648 (10)	0.0602
C(6)	0.8477 (10)	1.064 (1)	0.926 (1)	0.0563

Table 2. *Selected geometric parameters* (Å, °)

I(1)—I(2)	3.014 (3)	C(2)—C(3)	1.38 (2)
I(1)—As(1)	2.616 (3)	C(3)—C(4)	1.39 (2)
As(1)—C(1)	1.91 (1)	C(4)—C(5)	1.35 (2)
C(1)—C(2)	1.33 (2)	C(5)—C(6)	1.39 (2)
C(1)—C(6)	1.37 (2)		
I(2)—I(1)—As(1)	180.00	C(1)—C(2)—C(3)	124 (2)
I(1)—As(1)—C(1)	110.9 (5)	C(2)—C(3)—C(4)	116 (2)
C(1)—As(1)—C(1)	108.0 (5)	C(3)—C(4)—C(5)	120 (2)
As(1)—C(1)—C(2)	119 (1)	C(4)—C(5)—C(6)	122 (1)
As(1)—C(1)—C(6)	122 (1)	C(1)—C(6)—C(5)	118 (1)
C(2)—C(1)—C(6)	119 (1)		

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN FINISH*. Literature survey: *CSSR* (1984).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71814 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1065]

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Acta Cryst. (1994). C**50**, 719–721

(2*Z*,4*E*,6*E*)-8-Ethyl-1,1,1-trifluoro-5-trifluoromethyl-3,4,8-triazadeca-2,4,6-triene-2-amine: Formation of Hydrogen-Bonded Pairs *via* an Amine–Diazine Interaction

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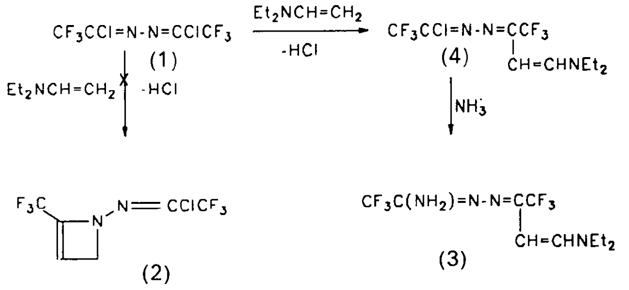
Abstract

An approximately planar conjugated region of the triazadecatriene chain in the title compound, $C_{10}H_{14}F_6N_4$, which begins at the diethylamine N atom, is disrupted by a twist between the diazo N atoms [N—N 1.405 (5) Å, C=N—N=C −144.4 (4)°]. This deformation facilitates hydrogen bonding between molecules related by a crystallographic twofold axis *via* a diazo N atom and an amine H atom [N···N 3.067 (7), H···N 2.02 (7) Å; N—H···N 157 (4)°].

Comment

The structure determination reported herein was part of an investigation of the reaction of 2,5-dichloro-1,1,1,6,6,6-hexafluoro-3,4-diazahexa-2,4-diene (1) with enamines (Benomar, 1989; Abdul-Ghani, 1992). It has been reported (Benomar, O'Reilly & Tipping, 1991) that exposure of a mixture of azine (1) and triethylamine (1:2 molar ratio) to daylight gave an orange oil (40%), considered to be the Δ^2-azetine (2) on the evidence available, which was formed *via* the intermediacy of the enamine $Et_2NCH{=}CH_2$. In the present work, a solid derivative of the product was prepared by the reaction of the supposed Δ^2-azetine (2) with ammonia in order to characterize this product more fully. The ammonia derivative was subjected to an X-ray crystal structure determination and shown to be the open-chain triene (3), thus establishing that the orange oil was the chlorotriene (4) and not the Δ^2-azetine (2).



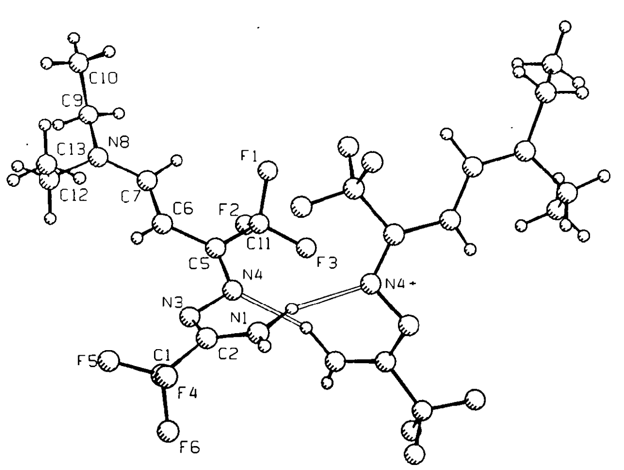


Fig. 1. The title hydrogen-bonded dimer, including atomic numbering scheme, drawn using *PLUTO* (Motherwell & Clegg, 1978).