

Bond lengths and angles are given in Table 2, but meaningful comparisons with other oxadiazoles are precluded by the relatively high coordinate errors in the present work. The molecular disorder, which is manifested by the partial occupancies of N(1) and N(6) [and N(1') and N(6')], encompasses the N—O—N—O portion of the oxadiazole ring and the C(5)-linked NO₂ group, but cannot be readily detected in the NO₂ atoms because of the similar positions of the oxadiazole and NO₂ moieties with respect to the six-membered ring. An examination of the packing diagram (Fig. 2) reveals that the intermolecular environments of N(1)/N(6) [and N(1')/N(6')] are very similar, with nonbonded distances of 2.74 (2), 3.00 (2) Å to N(1), 2.66 (3), 3.11 (3) Å to N(6), 2.81 (2), 2.86 (2) Å to N(1') and 2.78 (3), 3.05 (2) Å to N(6'). This pattern may provide a rationale for the disorder in that the molecule has approximate C₂ symmetry about C(4)···C(7) and the intramolecular contacts do not appear to favor an oxadiazole N at one position or the other.

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2,6-Diiodo-4-methyl-1,2,6-triarsabicyclo[2.2.1]heptane

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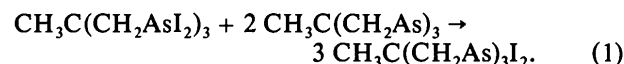
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Abstract. C₃H₉As₃I₂, *M_r* = 547.70, monoclinic, *P*2₁/*c*, *a* = 6.443 (2), *b* = 17.101 (12), *c* = 10.804 (9) Å, β = 99.62 (5)°, *V* = 1174 (2) Å³, *Z* = 4, *D_x* = 3.10 g cm⁻³, λ(Ag Kα) = 0.55936 Å, μ = 72.2 cm⁻¹, *F*(000) = 976, room temperature. Final *R* = 0.053 for 2993 unique reflections including unobserveds. The molecule consists of an I—As—As—As—I skeleton in a W-like shape and the organic group CH₃C(CH₂)₃, bridging the three As atoms. In the crystal the molecules associate into dimers by intermolecular As···As interactions [2.919 (2), 3.166 (2) Å]. These dimers form two-dimensional networks with rather short I···I distances [3.656 (2) Å].

Introduction. The structure determination of the title compound forms part of a research program on a series of cage compounds (Ellermann, Köck & Burzlaff, 1985).

Experimental. The dihalide CH₃C(CH₂As)₃I₂ was prepared according to equation (1) (Ellermann, Brehm & Moll, 1986) from CH₃C(CH₂AsI₂)₃ (Ellermann, Schössner & Lindner, 1978) and CH₃C(CH₂As)₃ (Thiele, Zoubek, Lindner & Ellermann, 1978):



Recrystallization from CH₂Cl₂ gave dark-red crystals. Spherical crystal with *r* = 0.14 ± 0.03 mm used for X-ray analysis. Intensities collected on a Philips

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PW 1100 diffractometer with Ag $K\alpha$ radiation and a graphite monochromator, ω - 2θ scans with scan width $1.0^\circ + 0.2^\circ \tan\theta$; lattice parameters from setting angles for 25 reflections ($12 \leq 2\theta \leq 15^\circ$) using refinement program *GIVER* (Krogmann, 1966); spherical absorption correction applied with $\mu R = 1.01 \pm 0.22$, absorption correction factor $A = 4.01 \pm 0.16$; index range $h \pm 9, k 0/22, l 0/14$; three standard reflections (intensity variation 0.8%); 6145 reflections measured, $\sin(\theta_{\max})/\lambda = 0.81 \text{ \AA}^{-1}$; after averaging symmetrically equivalent reflections 2993 reflections remained [92 unobserved included, $I < 3\sigma(I)$], $R_{\text{int}} = 0.064$. Structure solved by combination of direct and Patterson methods, subsequent Fourier maps revealed all C atoms, only some of the H atoms could be found in ΔF map, so all H atoms omitted from further computations; final least-squares calculations (on F values) refined scale factor, coordinates and anisotropic temperature parameters; corrections for anomalous dispersion and extinction with $r^* = 1.9 \times 10^{-4} \text{ cm}$ (Zachariasen, 1967) applied; weights $w = 1/\sigma^2(|F_o|)$; scattering factors from *International Tables for X-ray Crystallography* (1974). Final $R = 0.053$, $wR = 0.043$; $S = 1.82$; max. $\Delta/\sigma = 0.03$; $\Delta\rho$ in final difference Fourier map within $+1.3$ and -4.3 e \AA^{-3} (both close to I atoms). For all computations *CRYSTAN* system (Burzlaff, Böhme & Gomm, 1977) used and for plots *ORTEPII* (Johnson, 1977).

Discussion. Coordinates and equivalent isotropic temperature factors are listed in Table 1.†

^1H NMR, IR, Raman, and mass spectroscopy (Ellermann *et al.*, 1986) indicated the probable constitution of the title compound in solution with C_s symmetry. In the solid state the As atoms form an isosceles triangle. With the two I atoms these form an almost symmetrical W-shape. The organic group which is linked to the As atoms fixes this shape (Fig. 1). Selected bond lengths and angles are reported in Table 2; they are in agreement with the literature data (Camerman & Trotter, 1963; Fröhlich & Tebbe, 1982).

In the crystal, the molecules (Fig. 2) associate into dimeric groups by $\text{As}\cdots\text{As}$ interactions whereby the two organic parts are oriented in opposite directions. The short intermolecular $\text{As}\cdots\text{As}$ distances [2.919 (2), 3.166 (2) Å] indicate a strong interaction (Daly & Sanz, 1970). These dimers form two-dimensional networks by means of rather short $\text{I}\cdots\text{I}$ interactions [3.656 (2) Å] (see Fig. 2), which are comparable to the second-shortest distances found in the I_2 crystal (van Bolhuis, Koster & Migchelsen, 1967).

† Lists of structure amplitudes and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42721 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atom coordinates ($\times 10^4$) with e.s.d.'s in parentheses and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^2$)*

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

	x	y	z	U_{eq}
I(1)	1772 (1)	3706 (1)	2092 (1)	5.34
I(2)	1937 (2)	7872 (1)	1051 (1)	6.61
As(1)	3868 (2)	4685 (1)	952 (1)	3.80
As(2)	1534 (2)	5770 (1)	1165 (1)	4.06
As(3)	3904 (2)	6660 (1)	354 (1)	4.46
C(1)	5996 (20)	5061 (8)	2379 (12)	4.58
C(2)	3098 (22)	5931 (9)	2875 (11)	5.17
C(3)	6006 (21)	6526 (8)	1922 (13)	4.81
C(4)	5464 (21)	5882 (8)	2813 (12)	4.75
C(5)	6811 (30)	6034 (12)	4101 (16)	8.08

Table 2. *Selected bond lengths (Å) and bond angles ($^\circ$)*

E.s.d.'s are given in parentheses.

As(1)–I(1)	2.589 (2)	C–C	1.53–1.54 (2)
As(3)–I(2)	2.606 (2)	As(1)⋯As(1')	2.919 (2)*
As(1)–As(2)	2.423 (2)	As(1)⋯As(3')	3.166 (2)**
As(2)–As(3)	2.422 (2)	I(1)⋯I(2')	3.656 (2)**
As–C	1.97–2.00 (1)		
As(1)–As(2)–As(3)	90.45 (4)	As(1)–C(1)–C(4)	111.8 (7)
I(1)–As(1)–As(2)	93.91 (4)	As(2)–C(2)–C(4)	107.8 (7)
I(2)–As(3)–As(2)	91.67 (4)	As(3)–C(3)–C(4)	114.8 (8)
I(1)–As(1)–C(1)	100.7 (2)	C(1)–C(4)–C(2)	110 (1)
I(2)–As(3)–C(3)	98.0 (2)	C(1)–C(4)–C(3)	112 (1)
As(2)–As(1)–C(1)	92.2 (2)	C(2)–C(4)–C(3)	109 (1)
As(2)–As(3)–C(3)	90.0 (2)	C(1)–C(4)–C(5)	108 (1)
As(1)–As(2)–C(2)	88.2 (2)	C(2)–C(4)–C(5)	111 (1)
As(3)–As(2)–C(2)	89.8 (2)	C(3)–C(4)–C(5)	107 (1)

Symmetry codes: (i) and (ii) $1-x, 1-y, -z$; (iii) $-x, -\frac{1}{2}+y, \frac{1}{2}-z$.

* For intermolecular distances see Fig. 2.

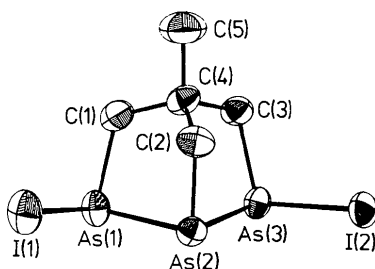


Fig. 1. The $\text{CH}_3\text{C}(\text{CH}_2\text{As})_3\text{I}_2$ molecule showing atom labeling and 50% probability thermal ellipsoids.

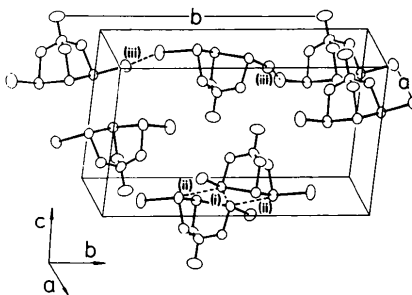


Fig. 2. Molecular packing in the crystal.

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Structure and Stereochemistry of (3*R*,5*R*,8*S*,10*R*,13*R*)-*ent*-Pimara-9(11),15-dien-3-*p*-bromobenzoate (Viguiepinol)*

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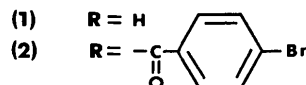
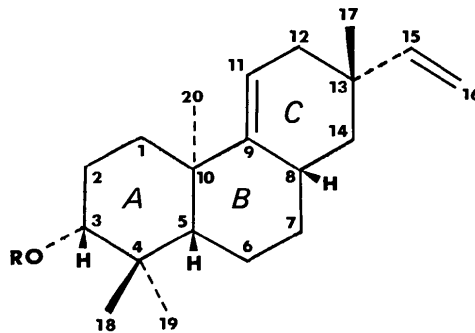
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Abstract. Systematic name: (2*R*,4*aR*,7*R*,8*aS*,10*aR*)-1-,2,3,4,5*a*,6,7,8,8*a*,9,10,10*a*-dodecahydro-1,1,4*a*,7-tetramethyl-7-vinyl-2-phenanthrenyl *p*-bromobenzoate. $C_{27}H_{35}BrO_2$, $M_r = 471.5$, orthorhombic, $P2_12_12_1$, $a = 6.220$ (2), $b = 18.773$ (7), $c = 20.708$ (7) Å, $V = 2418$ (1) Å³, $Z = 4$, $D_x = 1.29$ Mg m⁻³, Mo $K\alpha$, $\lambda = 0.7107$ Å, $\mu = 1.70$ mm⁻¹, $F(000) = 992$, $T = 293$ K, $R = 0.063$ for 1212 observed reflections. The molecular structure and absolute configuration are determined from the X-ray data (2*a*,4*a*,7*β*,8*aβ*,10*aβ*) and confirm the structure previously assigned on the basis of chemical and spectroscopic evidence. The *A/B* and *B/C* rings are *trans* fused, with chair–twist-boat and twist-boat–half-chair conformations, respectively. The molecules in the crystal are packed at normal van der Waals distances. No unusually short intermolecular contacts are noted.

Introduction. The title compound (1) is a new diterpene which was isolated from the aerial parts of *Viguiera pinnatilobata*, a Compositae widely distributed in Mexico; the sample used was collected in Oaxaca

(Estado de Oaxaca, Mexico). Chemical and spectroscopic studies led to the proposal of the chemical structure (1) (Guerrero, Nava, Quevedo, Toscano & Soriano-García, 1985), a diterpene with an *ent*-pimarane skeleton. In order to determine unambiguously the structure and absolute configuration of (1), a *p*-bromobenzoate derivative was prepared. We have undertaken the X-ray study of this compound (2).



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