(hydrogen-bond like), 3·24 (3) Å, is longer than the values observed when Gly or Leu is the third residue (Tanaka, Ashida, Shimonishi & Kakudo, 1979; Ashida *et al.*, 1977).

The proline ring adopts a conformation of type *A* (Balasubramanian, Lakshminarayanan, Sabesan, Tegoni, Venkatesan & Ramachandran, 1971). The phenylalanine side chain is in the conformation that has the highest frequency of occurrence in peptides and proteins (Benedetti, Morelli, Nemethy & Scheraga, 1983) with $\chi1 = -59$ and $\chi2 = 95°$. This conformation leads to the most sterically favorable arrangement. The conformation of the leucine side chain corresponds to that most frequently occurring with ($\chi1$, $\chi2$, 1) ~ (−60, 180°).

The crystal structure can be described as consisting of layers of tripeptide molecules stacked perpendicular to the *b* axis. Each face of the layer is wholly hydrophobic, consisting of phenyl rings and leucine side chains which stabilize the structure by van der Waals interactions. A layer can be characterized by its hydrogen bonding. The two NH groups in the molecule give one proton each. The water molecule gives its protons to the oxygen atom of the acetyl group and to the carboxyl of the Phe residue and receives one proton from the OH group. The only oxygen atom without hydrogen interaction belongs to the carboxyl of Pro.

The Leu residue, which replaces His of angiotensinogen in order to make crystallization easier, seems to play an important role only in the packing of the molecule (interactions between hydrophobic side chains of neighboring peptides) and not at the molecular conformation level. So for the 6–9 tetrapeptide of angiotensinogen such a β-turn structure might be a favorable conformation in solution as proposed by Oliveira, Juliano & Paiva (1977).

The present study shows a preferred type I β-turn conformation for a peptide containing the Pro-Phe sequence. Such a conformation may be important for renin and the angiotensinogen converting enzyme. In order to study the possible role of the β-turn in angiotensinogen, it is of great interest to investigate analogous or longer peptides in which the leucine residue is replaced by histidine. Further studies of linear oligopeptides related to angiotensinogen are in progress.

References

ASHIDA, T., TANAKA, I., SHIMONISHI, Y. & KAKUDO, M. (1977). *Acta Cryst.* B**33**, 3054–3059.

BALASUBRAMANIAN, R., LAKSHMINARAYANAN, A. V., SABESAN, M. N., TEGONI, G., VENKATESAN, K. & RAMACHANDRAN, G. N. (1971). *Int. J. Protein Res.* **3**, 25–33.

BENEDETTI, E., MORELLI, G., NEMETHY, G. & SCHERAGA, H. A. (1983). *Int. J. Pept. Protein Res.* **22**, 1–15.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

IUPAC–IUB COMMISSION ON BIOCHEMICAL NOMENCLATURE (1970). *J. Mol. Biol.* **52**, 1–17.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MERRIFIELD, R. B. (1963). *J. Am. Chem. Soc.* **85**, 2149–2154.

OLIVEIRA, M. C. F., JULIANO, L. & PAIVA, A. C. M. (1977). *Biochemistry*, **16**, 2606–2611.

SHEENAN, J. C. & NESS, G. P. (1955). *J. Am. Chem. Soc.* **77**, 1067–1068.

SKEGGS, L. T., LENTZ, K. E., KHAN, J. R. & HOCHSTRASSER, H. (1968). *J. Exp. Med.* **128**, 13–34.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

TANAKA, I., ASHIDA, T., SHIMONISHI, Y. & KAKUDO, M. (1979). *Acta Cryst.* B**35**, 110–114.

VENKATACHALAM, C. M. (1968). *Biopolymers*, **6**, 1425–1426.

Acta Cryst. (1986). C**42**, 724–727

Structure of 4-Amino-5,7-dinitrobenz[1,2-*c*][1,2,5]oxadiazole 3-Oxide

BY HERMAN L. AMMON AND DECHUN ZHANG

Department of Chemistry and Biochemistry, University of Maryland, College Park, Maryland 20742, *USA*

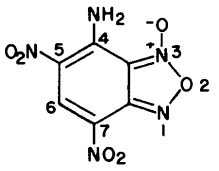
(*Received* 23 *May* 1985; *accepted* 12 *December* 1985)

Abstract. $C_6H_3N_5O_6$, $M_r = 241·1$, monoclinic, $P2_1$, $a = 11·959$ (7), $b = 9·863$ (6), $c = 7·180$ (4) Å, $\beta = 98·131$ (1)°, $V = 838·4$ (9) Å^3, $Z = 4$, $D_x = 1·910$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 15·5$ cm^{-1}, $F(000) = 488$, $T = 293$ K, final $R = 0·079$ for 1208 unique observed intensities. The space group is close to $P2_1/a$; confirmation of $P2_1$ was obtained by two statistical tests and diffraction-vector rotation scans for three reflections that appeared to violate the *a*-glide systematic absences. Both of the molecules in the asymmetric unit are disordered, resulting in a structure that appears to contain two [1,2,5]oxadiazole rings,

0108-2701/86/060724-04$01.50

namely 4-amino-8-nitrobenzo[1,2-*c*:4,5-*c'*]bis[1,2,5]-oxadiazole 3,5-dioxide. The crystal-packing environments around the two oxadiazole locations are similar, providing a rationale for the disorder.

Introduction. We are investigating the crystal structures of a series of high-density, nitro-group-containing organic compounds as part of an overall study of the relationships between molecular structure and crystal density in energetic materials. The title compound (I) is the second of the oxadiazoles to be investigated by us (Ammon & Bhattacharjee, 1982). The preparation of the compound by pyrolysis of 3-azido-2,4,6-trinitroaniline can yield two possible oxadiazoles [oxadiazole ring formation can utilize either the C(2)- or C(4)-linked nitro groups], and this study was undertaken in part to confirm the structure. The compound is an insensitive explosive, similar to TNT in impact sensitivity, with a calculated detonation velocity equal to that of *sym*-triaminotrinitrobenzene (Norris, 1984).



(I)

Table 1. *Fractional coordinates, equivalent isotropic temperature factors* (Å^2) *and e.s.d.'s in parentheses*

	x	*y*	*z*	U_{eq}*	*PP*†
C(3a)	0·1698 (7)	0·248 (1)	0·301 (1)	0·07 (2)	
C(4)	0·0897 (7)	0·129 (1)	0·300 (1)	0·04 (3)	
C(5)	0·1056 (6)	0·070 (1)	0·485 (1)	0·02 (3)	
C(6)	0·1862 (8)	0·110 (1)	0·643 (1)	0·03 (2)	
C(7)	0·2582 (6)	0·2145 (9)	0·632 (1)	0·04 (2)	
C(7a)	0·2491 (7)	0·289 (1)	0·446 (1)	0·049 (6)	
N(1)	0·303 (1)	0·387 (1)	0·393 (2)	0·02 (1)	0·62 (2)
N(3)	0·1752 (7)	0·319 (1)	0·143 (1)	0·06 (3)	
N(4)	0·0201 (5)	0·0959 (8)	0·1530 (9)	0·03 (3)	
N(5)	0·0417 (7)	−0·038 (1)	0·531 (1)	0·04 (3)	
N(6)	0·168 (2)	0·045 (3)	0·791 (3)	0·10 (2)	0·38 (2)
N(7)	0·3353 (6)	0·265 (1)	0·775 (1)	0·03 (3)	
O(2)	0·2591 (7)	0·418 (1)	0·192 (1)	0·09 (3)	
O(3)	0·1106 (6)	0·314 (1)	−0·0052 (8)	0·05 (3)	
O(4)	−0·0224 (6)	−0·104 (1)	0·412 (1)	0·03 (3)	
O(5)	0·0583 (6)	−0·0884 (9)	0·702 (1)	0·08 (3)	
O(6)	0·3372 (6)	0·205 (1)	0·933 (1)	0·04 (3)	
O(7)	0·3970 (6)	0·357 (1)	0·7620 (9)	0·04 (3)	
C(3a')	−0·3371 (6)	0·2565 (9)	0·306 (1)	0·02 (4)	
C(4')	−0·4045 (7)	0·361 (1)	0·321 (1)	0·03 (4)	
C(5')	−0·3942 (7)	0·424 (1)	0·495 (1)	0·04 (4)	
C(6')	−0·3180 (9)	0·379 (1)	0·650 (1)	0·05 (4)	
C(7')	−0·2449 (7)	0·269 (1)	0·621 (1)	0·02 (4)	
C(7a')	−0·2505 (6)	0·209 (1)	0·460 (1)	0·02 (4)	
N(1')	−0·193 (1)	0·101 (2)	0·388 (2)	0·07 (3)	0·66 (2)
N(3')	−0·3328 (6)	0·1715 (1)	0·153 (1)	0·05 (4)	
N(4')	−0·4805 (7)	0·411 (1)	0·174 (1)	0·04 (4)	
N(5')	−0·4543 (6)	0·546 (1)	0·531 (1)	0·04 (4)	
N(6')	−0·329 (2)	0·473 (2)	0·788 (3)	0·06 (4)	0·34 (2)
N(7')	−0·1558 (7)	0·245 (1)	0·796 (1)	0·03 (4)	
O(2')	−0·2474 (7)	0·081 (1)	0·184 (1)	0·04 (4)	
O(3')	−0·3834 (5)	0·172 (1)	−0·007 (1)	0·06 (4)	
O(4')	−0·5182 (6)	0·6023 (8)	0·4162 (9)	0·06 (5)	
O(5')	−0·4324 (7)	0·579 (1)	0·703 (1)	0·13 (5)	
O(6')	−0·1510 (7)	0·305 (1)	0·932 (1)	0·07 (4)	
O(7')	−0·0912 (6)	0·145 (1)	0·760 (1)	0·04 (4)	

* $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.
† *PP* is the population parameter; blank means 1·0.

Experimental. Material obtained from Drs H. Adolph and M. Chaykovsky (Naval Surface Weapons Center, Silver Spring, MD); crystallized from nitromethane, light-tan transparent plate 0·07 × 0·22 × 0·30 mm; preliminary survey with oscillation and Weissenberg photography; Picker FACS-I diffractometer, Cu radiation, graphite monochromator; unit-cell parameters from 16 reflections manually centered at $\pm 2\theta = 30{\cdot}08$–$56{\cdot}02°$, average $|2\theta_o - 2\theta_c| = 0{\cdot}007°$; $0k0$ absent for k odd, $h0l$ absent for h odd with a few low-intensity exceptions such as [*hkl*, *I*, $\sigma(I)$] (300, 6582, 120), (101, 3907, 86), (302, 3461, 76), (10$\bar{1}$, 2794, 75), (501, 1410, 44); 2θ–θ scan, 2θ scan speed 2° min^{-1}, 2θ scan width $1{\cdot}8° + 0{\cdot}3° \tan\theta$, 20 s backgrounds; h,k,l range 0–13, 0–11, $\bar{8}$–8; four standards every 100 reflections, average and maximum standard intensity variation 0·9% and 4·6%; absorption ignored; 1442 reflections measured to $2\theta_{max}$ 127°, 1346 unique data, $R_{int} = 0{\cdot}031$, 1208 with $I > 3\sigma(I)$; structure solved in $P2_1/a$ with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); structure refinement by full-matrix least squares in both $P2_1$ and $P2_1/a$ minimizing $\sum w(F_o - F_c)^2$, $w = 1/\sigma^2(F)$, anisotropic temperature factors for C, N, O, both refinements required a disordered molecular model; H atoms not included in any of the calculations; the size of the $P2_1$ problem precluded simultaneous refinement of all atoms; sets of 26 randomly selected atoms were refined in each l.s. cycle to convergence; average and maximum Δ/σ ratios in final l.s. cycle 0·12 and 0·62 for $P2_1$, 0·04 and 0·26 for $P2_1/a$; minimum and maximum values in final difference maps −0·41 and 0·45 e Å^{-3} for $P2_1$, −0·37 and 0·50 e Å^{-3} for $P2_1/a$; final *R*, *wR* and *S* values 0·079, 0·082 and 3·1 for $P2_1$, 0·101, 0·095 and 4·1 for $P2_1/a$; scattering factors from *International Tables for X-ray Crystallography* (1974).

All calculations performed on a Univac 1100/82 computer; programs: *XRAY*76 system (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976), *MULTAN*80. Atomic coordinates and equivalent isotropic temperature factors are listed in Table 1.*

Discussion. Although there were several small intensity violations of the $h0l$, h odd, systematic absences required by $P2_1/a$, the space-group assignment of $P2_1$ was by no means considered to be concrete. A few factors to be kept in mind are the possibility of multiple

* Lists of structure factors and anisotropic temperature factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42742 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

reflections producing measurable intensities for the systematic-absence violations, that the structure was solved in $P2_1/a$ having failed to yield a straightforward direct-methods solution in $P2_1$, and the similarity between the $P2_1$ and $P2_1/a$ solutions. Superimposed on top of the space-group problem was one of molecular-model disorder: both space groups required a two-site occupancy for the oxadiazole atom N(1), which could be located attached to C(7a) [see structure (I)] or to C(6). The two disorder-associated 'structures' can be envisioned as being related by a twofold rotation about C(4)···C(7). Two statistical tests, namely the Hamilton (1965) *R*-factor test and the Prince (1982) method (an example of the use of these tests can be found in Ammon & Bhattacharjee, 1984), gave clear indications that the $P2_1$ solution was correct at better than the 99·9% confidence level. Previous experience with the Hamilton and Prince methods had shown that these tests can be misleading (Ammon & Bhattacharjee, 1984), and experimental confirmation was sought by performing ψ-axis (diffraction vector) rotations on the diffractometer for the 300, 302 and 501 $P2_1/a$ systematic-absence violators. The intensities of the three reflections did not vary by more than $\pm 5\%$ with ψ rotations, confirming that the reflections indeed were not absent and the space group was $P2_1$.

The two independent molecules in the asymmetric unit are related by an approximate *a* glide (see Fig. 1). The r.m.s. deviation between the coordinates of the unprimed molecule and those of the primed molecule, transformed by the *a*-glide symmetry operation $(\frac{1}{2}+x, \frac{1}{2}-y, z)$, is 0·277 Å. This should be compared with a 0·153 Å r.m.s. deviation obtained from a best molecular fit calculated with the Nyburg (1974) program.

The average and maximum deviations of the six-membered-ring atoms from their least-squares plane are 0·010, 0·017 Å and 0·014, 0·022 Å for the unprimed and primed molecules, respectively; the corresponding values for the entire 18 (C,N,O)-atom fragments are 0·047, 0·160 Å and 0·061, 0·174 Å.

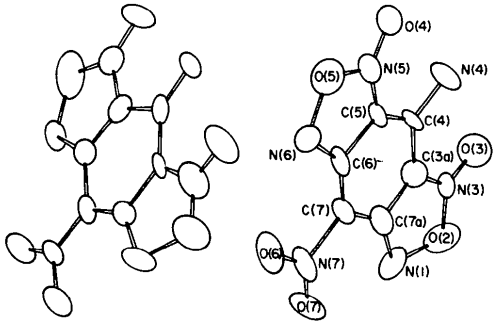


Fig. 1. An *ORTEP* drawing (Johnson, 1971) of the two molecules in the asymmetric unit. The atoms are depicted with 50% probability boundary ellipses. H atoms are not shown. The primed molecule is at the left.

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(3a)–C(7a)	1·37 (1)	C(3a')–C(7a')	1·48 (1)
C(3a)–C(4)	1·51 (1)	C(3a')–C(4')	1·32 (1)
C(3a)–N(3)	1·35 (1)	C(3a')–N(3')	1·39 (1)
C(4)–N(4)	1·29 (1)	C(4')–N(4')	1·38 (1)
C(5)–C(4)	1·44 (1)	C(5')–C(4')	1·39 (1)
C(5)–N(5)	1·38 (2)	C(5')–N(5')	1·44 (1)
C(6)–C(5)	1·44 (1)	C(6')–C(5')	1·41 (1)
C(6)–N(6)	1·29 (3)	C(6')–N(6')	1·38 (3)
C(7)–C(6)	1·36 (1)	C(7')–C(6')	1·43 (2)
C(7)–N(7)	1·38 (1)	C(7')–N(7')	1·55 (1)
C(7a)–C(7)	1·51 (1)	C(7a')–C(7')	1·29 (1)
C(7a)–N(1)	1·25 (2)	C(7a')–N(1')	1·41 (1)
N(1)–O(2)	1·50 (2)	N(1')–O(2')	1·54 (2)
N(3)–O(2)	1·41 (1)	N(3')–O(2')	1·35 (1)
N(3)–O(3)	1·22 (1)	N(3')–O(3')	1·22 (1)
N(5)–O(4)	1·24 (1)	N(5')–O(4')	1·18 (1)
N(5)–O(5)	1·31 (1)	N(5')–O(5')	1·27 (1)
N(6)–O(5)	1·90 (3)	N(6')–O(5')	1·66 (2)
N(7)–O(6)	1·28 (1)	N(7')–O(6')	1·13 (1)
N(7)–O(7)	1·18 (1)	N(7')–O(7')	1·30 (2)
C(7a)–C(3a)–C(4)	127 (1)	C(7a')–C(3a')–C(4')	124 (1)
C(7a)–C(3a)–N(3)	112 (1)	C(7a')–C(3a')–N(3')	107 (1)
C(4)–C(3a)–N(3)	121 (1)	C(4')–C(3a')–N(3')	129 (1)
C(3a)–C(4)–C(5)	108 (1)	C(3a')–C(4')–C(5')	117 (1)
C(5)–C(4)–N(4)	130 (1)	C(5')–C(4')–N(4')	119 (1)
C(3a)–C(4)–N(4)	122 (1)	C(3a')–C(4')–N(4')	124 (1)
C(4)–C(5)–C(6)	127 (1)	C(4')–C(5')–C(6')	122 (1)
C(6)–C(5)–N(5)	111 (1)	C(6')–C(5')–N(5')	114 (1)
C(4)–C(5)–N(5)	122 (1)	C(4')–C(5')–N(5')	124 (1)
C(5)–C(6)–C(7)	122 (1)	C(5')–C(6')–C(7')	118 (1)
C(7)–C(6)–N(6)	128 (1)	C(7')–C(6')–N(6')	138 (1)
C(5)–C(6)–N(6)	110 (1)	C(5')–C(6')–N(6')	103 (1)
C(6)–C(7)–C(7a)	117 (1)	C(6')–C(7')–C(7a')	121 (1)
C(7a)–C(7)–N(7)	116 (1)	C(7a')–C(7')–N(7')	127 (1)
C(6)–C(7)–N(7)	127 (1)	C(6')–C(7')–N(7')	111 (1)
C(7)–C(7a)–C(3a)	119 (1)	C(7')–C(7a')–C(3a')	118 (1)
C(3a)–C(7a)–N(1)	109 (1)	C(3a')–C(7a')–N(1')	107 (1)
C(7)–C(7a)–N(1)	132 (1)	C(7')–C(7a')–N(1')	135 (1)
C(7a)–N(1)–O(2)	109 (1)	C(7a')–N(1')–O(2')	106 (1)
C(3a)–N(3)–O(3)	128 (1)	C(3a')–N(3')–O(3')	132 (1)
C(3a)–N(3)–O(2)	106 (1)	C(3a')–N(3')–O(2')	113 (1)
O(3)–N(3)–O(2)	126 (1)	O(3')–N(3')–O(2')	115 (1)
C(5)–N(5)–O(5)	120 (1)	C(5')–N(5')–O(5')	110 (1)
C(5)–N(5)–O(4)	123 (1)	C(5')–N(5')–O(4')	124 (1)
O(5)–N(5)–O(4)	116 (1)	O(5')–N(5')–O(4')	126 (1)
C(7)–N(7)–O(7)	125 (1)	C(7')–N(7')–O(7')	108 (1)
C(7)–N(7)–O(6)	115 (1)	C(7')–N(7')–O(6')	124 (1)
O(7)–N(7)–O(6)	120 (1)	O(7')–N(7')–O(6')	128 (1)

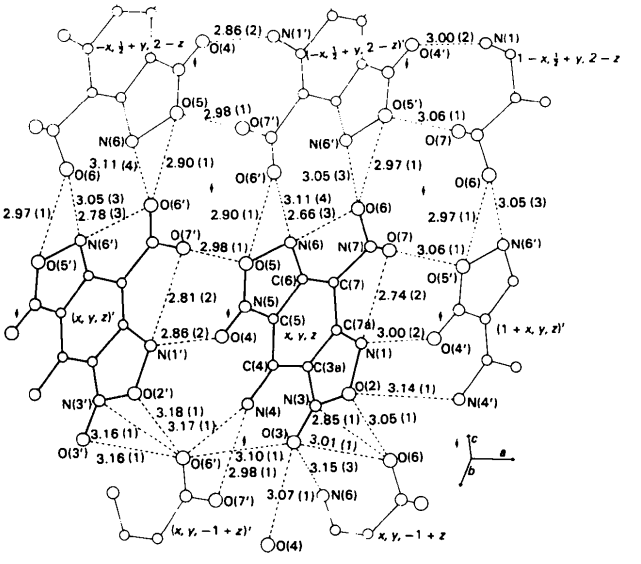


Fig. 2. Crystal-packing diagram down **b**. Some contact distances (Å) and e.s.d.'s (in parentheses) are shown.

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 NO_2 group, but cannot be readily detected in the NO_2 atoms because of the similar positions of the oxadiazole and NO_2 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 C_2 symmetry about C(4)···C(7) and the intramolecular contacts do not appear to favor an oxadiazole N at one position or the other.

This work was supported by the Naval Sea System Command, under task 61153N SR024-03 and, in part, through the facilities of the Computer Science Center of the University of Maryland.

References

AMMON, H. L. & BHATTACHARJEE, S. K. (1982). *Acta Cryst.* B**38**, 2498–2502.

AMMON, H. L. & BHATTACHARJEE, S. K. (1984). *Acta Cryst.* C**40**, 487–490.

HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.

International Tables for X-ray Crystallography (1974). Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JOHNSON, C. K. (1971). *ORTEP*II. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

NORRIS, W. P. (1984). *Gov. Rep. Announce. (US)*, **84**, 220.

NYBURG, S. C. (1974). *Acta Cryst.* B**30**, 251–253.

PRINCE, E. (1982). *Acta Cryst.* B**38**, 1099–1100.

STEWART, J. M., MACHIN, P. A., DICKINSON, C. W., AMMON, H. L., HECK, H. S. & FLACK, H. (1976). The *XRAY*76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Acta Cryst. (1986). C**42**, 727–729

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

BY JOCHEN ELLERMANN AND EBERHARD KÖCK*

Institut für Anorganische Chemie der Universität Erlangen–Nürnberg, Egerlandstrasse 1, *D*-8520 *Erlangen, Federal Republic of Germany*

AND HANS BURZLAFF

Institut für Angewandte Physik, Lehrstuhl für Kristallographie der Universität Erlangen–Nürnberg, Bismarckstrasse 10, *D*-8520 *Erlangen, Federal Republic of Germany*

(*Received* 4 *November* 1985; *accepted* 20 *December* 1985)

Abstract. $C_5H_9As_3I_2$, $M_r = 547{\cdot}70$, monoclinic, $P2_1/c$, $a = 6{\cdot}443\ (2)$, $b = 17{\cdot}101\ (12)$, $c = 10{\cdot}804\ (9)$ Å, $\beta = 99{\cdot}62\ (5)°$, $V = 1174\ (2)$ Å^3, $Z = 4$, $D_x = 3{\cdot}10$ g cm^{-3}, λ(Ag $K\alpha$) = 0·55936 Å, $\mu = 72{\cdot}2$ cm^{-1}, $F(000) = 976$, room temperature. Final $R = 0{\cdot}053$ 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 $CH_3C(CH_2)_3$, 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 $CH_3C(CH_2As)_3I_2$ was prepared according to equation (1) (Ellermann, Brehm & Moll, 1986) from $CH_3C(CH_2AsI_2)_3$ (Ellermann, Schössner & Lindner, 1978) and $CH_3C(CH_2As)_3$ (Thiele, Zoubek, Lindner & Ellermann, 1978):

$$CH_3C(CH_2AsI_2)_3 + 2\ CH_3C(CH_2As)_3 \rightarrow 3\ CH_3C(CH_2As)_3I_2. \quad (1)$$

Recrystallization from CH_2Cl_2 gave dark-red crystals. Spherical crystal with $r = 0{\cdot}14 \pm 0{\cdot}03$ mm used for X-ray analysis. Intensities collected on a Philips

* Author for correspondence.

0108-2701/86/060727-03$01.50