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Bicuculline and narcotine: preliminary structure data.

By E. G. Steward and R. B. Player, *Department of Physics, The City University, London E. C.* 1, *England*

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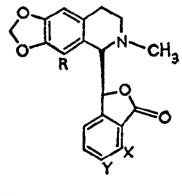
The alkaloids bicuculline and narcotine form crystals with space group $P2_12_12_1$. The cell dimensions are, respectively, $a=10·916$, $b=14·478$, $c=10·447$ Å, and $a=15·398$, $b=32·686$, $c=8·022$ Å. Other preliminary data are listed.

Bicuculline, $C_{20}H_{17}NO_6$, a phthalideisoquinoline alkaloid, is of current interest due to its possible activity as a specific antagonist of the inhibitory function of γ-aminobutyric acid in the mammalian central nervous system (Curtis, Duggan, Felix & Johnston, 1970; Steward, Player, Quilliam, Brown & Pringle, 1971). Preliminary structural data have been collected for this alkaloid and for a related phthalideisoquinoline alkaloid narcotine, $C_{22}H_{23}NO_7$. Less accurate data for narcotine have previously been reported by Lovell (1953).

Small prismatic crystals of bicuculline were obtained from ethanol/chloroform solution and larger needle crystals of narcotine from methanol.



Bicuculline
$R=H$
$X,Y= -OCH_2O-$

Narcotine
$R=X=Y=OCH_3$

Weissenberg photographs taken with Cu $K\alpha$ (1·5418 Å) radiation revealed the same systematic absences for both compounds:

$$h00,\ h=2n+1$$
$$0k0,\ k=2n+1$$
$$00l,\ l=2n+1\,,$$

defining unambiguously the space group $P2_12_12_1$. Cell dimensions were obtained from 2θ values of 44 reflexions from bicuculline and 32 reflexions from narcotine, using two axes in each case, measured with a counter diffractometer.

	Bicuculline	Narcotine
M.W.	367·34	413·41
m.p.(°C)	201	178
Crystal system	Orthorhombic	Orthorhombic
Space group	$P2_12_12_1$	$P2_12_12_1$
Cell Dimensions (Å) a	10·916 (6)	15·398 (12)
b	14·478 (12)	32·686 (36)
c (prism)	10·447 (5)	8·022 (8)
$V(\text{Å}^3)$	1651 (3)	4037 (11)
Z	4	8
$\varrho_{calc}(\text{g.cm}^{-3})$	1·478	1·360
$\varrho_{exp}(\text{g.cm}^{-3})$	1·49	1·38

Further crystallographic studies of these two alkaloids are not contemplated.

The award of an S.R.C. Grant to one of us (RBP) is gratefully acknowledged.

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Lovell, F. M. (1953). *Acta Cryst.* **6**, 869.
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The refinement of the structure of hexabromoethane.

By Gretchen Mandel and Jerry Donohue, *Department of Chemistry and Laboratory for Research on the Structure of Matter, University of Pennsylvania, Philadelphia, Pennsylvania* 19104, *U.S.A.*

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The structure of hexabromoethane (C_2Br_6) reported earlier has been confirmed and refined with a new set of three-dimensional data collected at 22° on an automated diffractometer with Cu $K\alpha$ radiation by the 2θ scan technique. The lattice parameters $a=12·043\pm0·002$, $b=10·674\pm0·002$, and $c=6·705\pm0·001$ Å were within one standard deviation of those previously reported. The measured density is 3·823 g.cm^{-3}, and, with $Z=4$, the calculated density is 3·88 g.cm^{-3}. The final R value is 6·4%. The average carbon–bromine distance is 1·944 with a bond scatter of 0·005 Å. Intramolecular distances are 3·15 Å for the two bromine atoms bonded to the same carbon atom and 3·42 Å for bromine atoms bonded to different carbon atoms. The shortest intermolecular distance is a 3·76 Å bromine–bromine contact. The average bond angle at a carbon atom is 109·6 ± 0·9°.

Introduction

The crystal structure of hexabromoethane was first reported by Snaauw & Wiebenga (1942); the space group and lattice constants had previously been reported by Yardley (1928). Since this structure contains the crystallographically unusual feature of a molecular center of symmetry which is non-coincident with a crystallographic center of symmetry, the redetermination of the structure was undertaken. Several other structures with this rather unusual feature have been reported, but on redetermination, some were found to be incorrect. The refined structure was also of interest as part of our studies of intermolecular distances in perhalogenated compounds.

Experimental

Colorless trapezoidal tablets of hexabromoethane (K & K Laboratories, Inc., Hollywood, California) were grown by slow evaporation from carbon disulfide at 6°C. Data were collected from a spherically ground crystal of *ca.* 0·17 mm diameter on a Picker FACS 1 diffractometer with Cu $K\alpha$ ($\lambda = 1{\cdot}5418$ Å) radiation. For preliminary alignment the lattice constants determined by Snaauw & Wiebenga were used; refined lattice constants of $a = 12{\cdot}043 \pm 0{\cdot}002$, $b = 10{\cdot}674 \pm 0{\cdot}002$, and $c = 6{\cdot}705 \pm 0{\cdot}001$ Å were obtained by a least-squares treatment of six observed values of θ. These values correspond to a calculated density, with four mole-

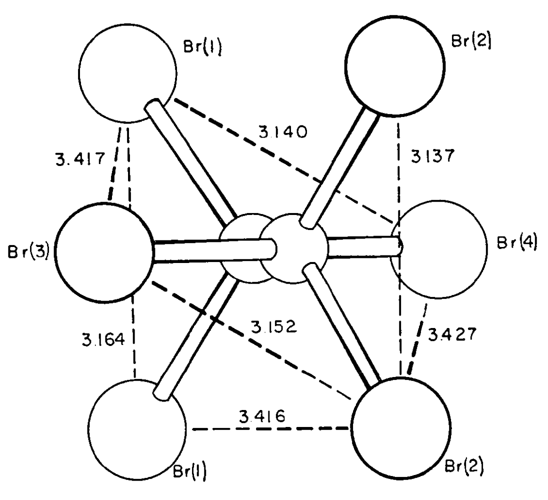

Fig. 1. Intramolecular distances.

Table 1. *Observed and calculated structure factors*

	h	k	l	F_o	F_c
	0	0	2	223.5	199.1
	0	0	4	43.0	43.9
	0	0	6	48.8	49.4
	0	1	1	90.4	65.5
	0	1	3	142.8	134.4
•	0	1	5	4.9	8.1
	0	1	7	23.9	23.6
	0	2	0	189.9	148.7
	0	2	2	58.0	45.7
	0	2	4	78.2	78.1
	0	2	6	73.2	77.5
	0	3	1	113.2	95.8
	0	3	3	286.6	306.6
	0	3	5	49.1	48.5
	0	3	7	31.2	31.0
	0	4	0	121.4	110.3
	0	4	2	19.3	14.5
	0	4	4	63.6	63.3
	0	4	6	99.3	105.6
•	0	5	1	9.0	0.2
	0	5	3	49.9	47.1
	0	5	5	16.3	14.6
	0	5	7	31.8	30.3
	0	6	0	278.2	352.2
	0	6	2	89.0	91.6
	0	6	4	20.2	15.6
	0	6	6	23.4	22.0
	0	7	1	46.4	47.4
	0	7	3	119.1	126.8
	0	7	5	16.3	14.5
	0	8	0	145.9	168.6
	0	8	2	47.3	46.6
	0	8	4	33.3	31.5
	0	9	1	23.7	25.7
	0	9	3	66.1	66.9
	0	9	5	13.2	12.0
	0	10	0	58.2	57.3
	0	10	2	13.9	14.0
	0	10	4	24.3	24.7
	0	11	1	30.8	14.1
	0	11	3	51.8	49.2
	0	12	0	42.3	38.9
	1	0	1	90.5	66.4
	1	0	2	324.0	307.2
	1	0	3	28.1	23.5
	1	0	4	67.2	68.0
	1	0	6	35.2	36.8
	1	0	5	20.5	20.0
	1	0	7	27.0	26.2
	1	1	1	79.5	59.8
	1	1	2	143.6	130.6
	1	1	3	68.9	63.7
	1	1	4	88.3	86.9
	1	1	5	27.7	28.0
	1	1	6	37.1	36.1
	1	1	7	33.8	35.1
	1	2	1	74.7	60.8
	1	2	2	56.1	48.4
	1	2	3	93.8	85.5
	1	2	4	40.7	38.3
	1	2	5	59.4	59.2
	1	2	6	28.1	28.6
•	1	2	7	7.3	6.3
	1	3	1	212.5	207.5
•	1	3	2	5.2	3.0
	1	3	3	68.8	66.4
	1	3	4	12.5	8.8
	1	3	5	99.5	102.2
	1	3	6	19.9	20.2
•	1	3	7	6.3	2.5
	1	4	1	60.1	54.2
	1	4	2	56.7	51.5
	1	4	3	106.6	105.1
	1	4	4	64.5	64.4
	1	4	5	83.9	84.5
	1	4	6	16.4	14.6
	1	4	7	23.7	21.5
	1	5	1	49.6	47.8
	1	5	2	45.1	42.2
	1	5	3	56.0	55.3
	1	5	4	25.7	25.7
	1	5	5	29.9	25.2
•	1	5	6	0.0	3.4
	1	5	7	19.1	18.6
	1	6	1	35.4	34.8
	1	6	2	126.7	131.1
	1	6	3	25.8	23.2
	1	6	4	31.5	31.9
•	1	6	5	7.4	1.1
	1	6	6	17.7	15.8
	1	7	1	55.9	57.3
	1	7	2	71.1	73.6
	1	7	3	26.3	20.8
	1	7	4	53.7	54.8
	1	7	5	36.1	34.4
	1	7	6	29.9	28.1
	1	8	1	13.3	14.4
	1	8	2	67.7	69.9
	1	8	3	14.6	13.8
•	1	8	4	7.0	5.2
•	1	8	5	0.0	2.2
	1	9	1	42.5	40.0
	1	9	2	13.6	12.5
	1	9	3	18.3	14.9
	1	9	4	14.2	14.4
	1	9	5	23.8	22.0
	1	10	1	14.5	13.9
	1	10	2	24.6	23.7
	1	10	3	34.1	32.6
	1	10	4	24.4	23.6
	1	11	1	31.6	30.1
•	1	11	2	5.1	0.8
	1	11	3	17.0	15.3
	1	12	1	10.2	10.3
	2	0	0	32.0	5.9
	2	0	1	68.0	53.6
	2	0	2	314.9	317.6
	2	0	3	99.1	93.4
	2	0	4	202.3	213.1
	2	0	5	15.8	13.8
	2	0	6	34.0	33.3
•	2	0	7	3.6	1.6
	2	1	0	167.8	142.1
	2	1	1	16.2	12.2
	2	1	2	35.3	30.7
	2	1	3	17.2	15.6
	2	1	4	17.9	14.4
	2	1	5	114.4	121.1
	2	1	6	13.7	12.1
	2	1	7	42.1	43.6
•	2	2	0	5.6	5.0
•	2	2	1	7.8	5.8
	2	2	2	26.9	24.1
	2	2	3	30.7	28.8
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	2	2	5	13.3	8.9
	2	2	6	12.6	11.8
	2	2	7	8.1	3.5
	2	3	0	89.5	83.2
	2	3	1	382.5	424.5
	2	3	2	36.8	33.4
	2	3	3	50.4	31.3
•	2	3	4	0.0	1.1
	2	3	5	14.6	11.6
	2	3	6	27.1	27.3
	2	3	7	25.2	24.2
•	2	4	0	3.9	1.8
	2	4	1	22.4	21.0
	2	4	2	129.1	126.1
	2	4	3	55.8	55.2
	2	4	4	78.5	79.4
	2	4	5	16.4	15.4
•	2	4	6	4.7	2.1
	2	4	7	9.8	6.0
	2	5	0	34.3	32.7
	2	5	1	134.1	136.5
•	2	5	2	8.6	1.8
	2	5	3	23.4	23.8
	2	5	4	10.8	5.9
	2	5	5	56.3	57.2
•	2	5	6	7.8	1.3
	2	5	7	8.3	5.0
	2	6	0	18.5	6.3
	2	6	1	23.6	23.1
	2	6	2	133.1	143.4
	2	6	3	48.3	47.8
	2	6	4	97.5	100.5
	2	6	5	10.3	6.6
	2	6	6	14.2	14.0
	2	7	0	75.6	80.3
	2	7	1	66.5	69.8
	2	7	2	23.7	22.9
•	2	7	3	2.5	6.4
	2	7	4	10.3	5.3
	2	7	5	76.8	79.0
	2	7	6	9.8	11.2
•	2	8	0	8.8	2.7
•	2	8	1	8.1	5.6
	2	8	2	51.9	54.8
	2	8	3	6.3	7.4
	2	8	4	52.4	49.0
•	2	8	5	2.8	0.9
	2	9	0	18.5	18.1
	2	9	1	86.4	91.8
•	2	9	2	9.3	7.6
•	2	9	3	5.9	2.4
•	2	9	4	7.0	1.0
•	2	9	5	3.8	2.0
•	2	10	0	3.2	4.6
	2	10	1	13.4	5.7
	2	10	2	52.1	51.2
	2	10	3	24.3	23.6
	2	10	4	36.2	35.1
•	2	11	0	5.7	6.0
	2	11	1	71.6	69.9
•	2	11	2	7.1	5.5
•	2	11	3	6.7	6.2
	2	12	0	7.6	3.9
•	2	12	1	4.2	3.7
	3	0	1	76.3	63.2
	3	0	2	217.1	218.7
	3	0	3	19.9	16.6
	3	0	4	90.3	92.1
	3	0	5	13.0	13.9
	3	0	6	62.4	64.2
	3	0	7	33.3	32.2
	3	1	1	26.0	23.2
	3	1	2	58.9	55.0
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	3	1	4	110.6	112.9
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	3	1	6	42.8	42.0
	3	1	7	36.0	37.0
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	3	2	4	46.2	45.0
	3	2	5	23.1	21.3
	3	2	6	24.5	25.4
	3	2	7	8.5	8.2
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	3	4	1	84.6	85.0
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	3	4	6	9.4	0.5
	3	4	7	21.3	21.7
	3	5	1	63.5	64.4
	3	5	2	33.5	34.0
	3	5	3	61.6	60.4
	3	5	4	40.8	40.3
	3	5	5	10.0	8.5
	3	5	6	9.6	2.7
	3	6	1	25.8	23.3
	3	6	2	92.8	96.1
	3	6	3	23.2	22.0
	3	6	4	40.3	41.1
	3	6	5	14.0	14.1
	3	6	6	31.3	29.9
	3	7	1	43.4	44.8
	3	7	2	20.6	21.2
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	3	7	4	70.5	73.1
	3	7	5	21.0	20.5
	3	7	6	33.4	32.7
•	3	8	1	4.4	0.8
	3	8	2	52.8	55.9
	3	8	3	12.9	10.3
	3	8	4	14.3	12.7
•	3	8	5	6.3	0.8
	3	9	1	42.8	42.5
	3	9	2	32.1	30.8
	3	9	3	21.1	21.3
	3	9	4	11.5	7.8
•	3	9	5	5.7	3.8
	3	10	1	30.2	28.8
	3	10	2	10.3	8.0
	3	10	3	35.0	34.2
	3	10	4	35.2	34.2
	3	11	1	37.1	35.6
	3	11	2	8.2	8.7
	3	11	3	24.8	22.2
	4	0	0	475.2	499.4
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	4	0	2	133.0	128.2
•	4	0	3	9.1	7.8
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	4	0	5	43.3	43.3
	4	0	6	32.2	31.6
	4	0	7	23.6	25.1
	4	1	0	26.6	22.5
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	4	1	2	100.9	98.4
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	4	1	4	63.8	62.9
•	4	1	5	0.0	3.9
•	4	1	6	6.8	4.7
	4	1	7	19.4	20.0
	4	2	0	90.9	84.9
	4	2	1	71.0	65.6
	4	2	2	49.9	45.6
•	4	2	3	6.8	5.3
	4	2	4	74.3	77.8
	4	2	5	16.4	16.2
	4	2	6	52.3	53.4
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	4	3	2	92.7	95.5
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	4	3	4	69.3	72.2
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	4	4	0	77.3	80.6
	4	4	1	114.8	119.0
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	4	4	3	12.5	9.5
	4	4	4	61.4	63.7
	4	4	5	23.1	23.4
	4	4	6	75.5	75.7
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•	4	5	0	0.0	2.1
	4	5	1	12.9	12.3
	4	5	2	21.7	21.6
	4	5	3	56.1	55.8
•	4	5	4	8.6	8.2
	4	5	5	11.9	5.9
•	4	5	6	3.8	0.9
	4	6	0	184.2	219.1
	4	6	1	72.2	77.2
	4	6	2	61.7	62.5
•	4	6	3	0.0	4.1
	4	6	4	27.9	27.4
	4	6	5	28.2	27.7
	4	6	6	15.1	12.6
	4	7	0	15.4	17.0
	4	7	1	24.2	22.0
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	4	7	3	71.6	75.8
	4	7	4	51.3	51.4
	4	7	5	10.1	8.6
•	4	7	6	4.9	3.0
	4	8	0	108.5	120.9
	4	8	1	14.3	14.4
	4	8	2	36.2	37.6
•	4	8	3	7.6	0.9
	4	8	4	32.9	33.1
•	4	8	5	4.2	1.4
•	4	9	0	0.0	3.1
	4	9	1	17.4	19.5
	4	9	2	26.2	25.5
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	4	9	4	20.0	19.3
	4	10	0	40.6	40.0
	4	10	1	49.1	47.7
•	4	10	2	0.0	3.2
•	4	10	3	3.8	4.8
	4	10	4	24.0	25.1
•	4	11	0	5.2	3.9
	4	11	1	12.4	12.9
	4	11	2	8.3	8.6
	5	0	1	97.8	95.4
	5	0	2	193.0	210.5
	5	0	3	41.4	39.2
	5	0	4	11.8	13.3
	5	0	5	35.8	39.0
•	5	0	6	5.5	3.5
	5	0	7	7.5	6.1
•	5	1	1	9.1	11.7
	5	1	2	126.3	130.7
	5	1	3	58.0	58.0
	5	1	4	44.0	44.2
	5	1	5	28.0	28.8
	5	1	6	15.2	14.3
	5	1	7	19.7	22.7
	5	2	1	10.4	2.9
	5	2	2	48.9	50.1
	5	2	3	50.9	52.8
	5	2	4	33.0	33.6
	5	2	5	43.4	43.5
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•	5	2	7	0.0	3.7
	5	3	1	113.7	121.9
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•	5	3	3	7.7	8.4
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	5	3	5	78.0	81.4
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	5	3	7	21.1	22.3
	5	4	1	26.8	22.7
	5	4	2	27.1	27.2
	5	4	3	48.9	52.2
	5	4	4	35.7	34.9
	5	4	5	67.7	65.3
	5	4	6	28.1	26.3
	5	5	1	48.3	51.7
	5	5	2	37.2	40.3
	5	5	3	28.6	29.2
	5	5	4	21.4	21.2
	5	5	5	26.3	25.5
	5	5	6	8.2	4.9
	5	6	1	51.1	51.7
	5	6	2	89.7	94.1
	5	6	3	25.6	26.2
	5	6	4	11.8	10.1
	5	6	5	15.4	14.6
•	5	6	6	6.1	3.5
	5	7	1	14.2	12.3
	5	7	2	79.4	83.7
	5	7	3	35.0	35.3
	5	7	4	20.6	21.8
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	5	8	4	13.3	10.3
•	5	8	5	6.5	4.2
	5	9	1	28.1	25.9
•	5	9	2	6.3	2.3
•	5	9	3	3.2	2.6
	5	9	4	27.2	27.3
	5	10	1	14.3	13.4
	5	10	2	16.0	13.7
	5	10	3	15.2	15.0
	5	11	1	24.2	23.8
•	5	11	2	6.2	1.8
	6	0	0	25.5	24.7
	6	0	1	50.1	51.0
	6	0	2	114.5	118.3
	6	0	3	120.8	132.6
	6	0	4	95.2	93.5
	6	0	5	29.2	29.2
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	6	1	7	25.3	25.0
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	6	2	4	19.3	15.6
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	6	2	6	11.4	9.7
	6	3	0	169.4	185.4
	6	3	1	169.2	197.2
	6	3	2	48.0	50.9
	6	3	3	28.7	25.2
	6	3	4	21.7	21.7
•	6	3	5	9.4	10.9
	6	3	6	41.4	41.2
	6	4	0	11.2	8.2
	6	4	1	55.3	57.8
	6	4	2	56.0	55.6
	6	4	3	105.7	116.9
	6	4	4	21.5	22.5
	6	4	5	25.1	22.5
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	6	5	1	94.2	102.3
	6	5	2	11.0	4.5
	6	5	3	15.4	17.9
•	6	5	4	5.6	2.6
	6	5	5	34.3	34.4
	6	5	6	9.5	8.1
•	6	6	0	4.9	6.9
	6	6	1	27.8	25.2
	6	6	2	50.6	52.0
	6	6	3	65.3	68.6
	6	6	4	43.0	42.8
	6	6	5	14.2	14.7
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	6	7	4	8.4	5.9
	6	7	5	47.5	47.4
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	6	8	2	32.3	31.9
•	6	8	3	7.7	7.2
	6	8	4	32.9	33.8
	6	9	0	48.8	47.6
	6	9	1	43.6	42.5
	6	9	2	17.4	10.0
•	6	9	3	4.7	4.5
	6	9	4	10.6	11.0
•	6	10	0	4.4	1.6
	6	10	1	25.6	24.3
	6	10	2	25.1	23.2
	6	10	3	51.0	50.5
	6	11	0	29.2	27.3
	6	11	1	45.4	43.9
	7	0	1	43.9	41.6
	7	0	2	63.7	67.0
	7	0	3	20.0	18.4
	7	0	4	67.7	69.2
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	7	3	3	63.6	68.7
	7	3	4	12.0	10.9
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	7	3	6	7.4	2.5
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	7	4	3	49.7	52.4
	7	4	4	46.7	46.8
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	7	5	1	40.2	43.2
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	7	5	3	40.0	41.5
	7	5	4	21.3	22.9
	7	5	5	11.0	6.9
	7	6	1	19.0	17.1
	7	6	2	29.2	29.2
	7	6	3	20.1	18.7
	7	6	4	28.5	30.1
	7	6	5	28.7	25.5
	7	7	1	17.2	17.6
	7	7	2	14.7	16.6
•	7	7	3	5.6	3.0
	7	7	4	40.3	41.6
	7	7	5	8.5	6.7
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	7	8	2	27.1	27.4
•	7	8	3	0.0	4.0
	7	8	4	21.5	21.0
	7	9	1	17.3	15.2
	7	9	2	28.1	27.5
	7	9	3	15.1	14.1
	7	10	1	37.9	31.4
	7	10	2	7.8	7.3
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	8	0	1	117.4	122.2
	8	0	2	32.6	29.3
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	8	1	5	11.0	10.7
	8	1	6	8.4	7.9
	8	2	0	97.0	102.0
	8	2	1	40.3	61.3
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•	8	2	3	0.0	5.6
	8	2	4	33.4	37.0
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	8	2	6	27.9	27.2
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	8	3	4	56.4	58.6
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	8	4	2	24.3	25.8
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	8	4	4	27.8	29.4
	8	4	5	25.5	26.0
•	8	5	0	6.7	2.9
•	8	5	1	0.0	1.1
	8	5	2	11.1	7.9
	8	5	3	31.3	34.2
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	8	5	5	8.3	5.1
	8	6	0	74.2	77.4
	8	6	1	55.8	58.3
	8	6	2	17.6	14.1
•	8	6	3	0.0	2.9
	8	6	4	20.7	19.9
	8	6	5	24.4	24.6
	8	7	0	16.9	16.6
•	8	7	1	6.5	3.3
	8	7	2	62.1	62.7
	8	7	3	15.2	14.8
	8	7	4	51.0	51.3
	8	8	0	73.3	73.9
	8	8	1	15.3	14.5
	8	8	2	16.7	16.5
•	8	8	3	0.0	0.5
•	8	9	0	0.0	1.1
•	8	9	1	3.3	3.1
	8	9	2	25.5	24.8
	8	10	0	13.7	13.0
	8	10	1	48.9	47.4
	9	0	1	68.1	68.6
	9	0	2	80.1	83.8
	9	0	3	41.1	40.6
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	9	1	1	17.0	15.3
	9	1	2	60.2	63.1
	9	1	3	41.9	42.2
•	9	1	4	0.0	1.2
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	9	2	2	38.4	41.5
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	9	3	2	17.8	17.3
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	9	3	4	40.6	42.7
	9	3	5	42.1	41.2
	9	4	1	30.2	28.1
•	9	4	2	7.1	6.8
	9	4	3	17.1	16.5
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	9	5	2	17.4	17.1
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	9	6	4	18.1	17.0
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	10	0	0	29.7	32.2
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	10	1	5	34.3	32.2
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	10	2	1	17.6	14.6
	10	2	2	18.7	20.1
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	10	3	3	22.5	21.8
	10	3	4	11.4	10.2
	10	3	5	25.9	24.9
•	10	4	0	8.7	8.0
	10	4	1	28.9	29.3
	10	4	2	9.5	8.4
	10	4	3	68.1	69.2
	10	4	4	11.0	11.5
	10	5	0	11.0	5.9
	10	5	1	48.6	48.1
•	10	5	2	5.1	1.1
	10	5	3	11.2	11.4
•	10	5	4	2.0	4.1
	10	6	0	15.4	15.2
	10	6	1	15.9	16.0
•	10	6	2	3.3	0.6
	10	7	0	78.8	79.4
	10	7	1	29.6	26.3
	10	7	2	25.7	25.6
•	10	7	3	6.2	1.9
	10	8	0	9.9	8.8
•	10	8	1	4.7	5.4
	10	8	2	17.2	16.3
	11	0	1	23.1	15.6
	11	0	2	10.8	12.9
	11	0	3	25.7	24.6
	11	0	4	19.9	17.4
	11	1	1	14.6	14.7
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	11	1	4	26.8	24.7
	11	2	1	21.1	19.8
	11	2	2	29.8	29.6
	11	2	3	19.3	17.6
•	11	2	4	4.6	2.8
	11	3	1	24.8	24.3
	11	3	2	44.2	42.7
	11	3	3	35.6	34.8
	11	3	4	8.4	5.2
	11	4	1	32.4	31.6
	11	4	2	26.6	26.6
	11	4	3	18.9	17.3
	11	4	4	19.3	17.7
	11	5	1	24.4	23.9
	11	5	2	10.3	9.6
	11	5	3	24.7	25.7
	11	6	1	9.9	8.4
•	11	6	2	0.4	5.5
•	11	7	1	0.0	4.1
	12	0	0	17.8	13.4
	12	0	1	48.6	44.5
	12	0	2	11.5	7.9
•	12	0	3	6.1	2.3
	12	1	0	8.8	6.7
•	12	1	1	7.0	7.3
	12	1	2	39.3	38.8
	12	1	3	20.9	19.6
	12	2	0	51.9	50.1
	12	2	1	22.0	19.5
	12	2	2	18.1	17.2
•	12	2	3	0.0	0.1
•	12	3	0	7.9	2.3
	12	3	1	17.5	18.1
	12	3	2	27.9	25.3
	12	3	3	8.6	6.0
	12	4	0	45.6	42.6
	12	4	1	40.8	44.7
	12	4	2	21.6	21.6
•	12	4	3	5.3	1.6
•	12	5	0	5.9	0.1
•	12	5	1	5.4	0.5
•	12	5	2	4.8	0.1
•	12	6	0	3.2	1.2
	12	6	1	20.8	19.1
	13	0	1	25.7	24.0
	13	0	2	19.0	15.5
	13	1	1	17.8	15.8
	13	1	2	23.5	22.4
	13	2	1	7.1	3.0
	13	2	2	23.0	21.5
•	13	3	1	4.5	4.1
•	13	3	2	2.7	3.9
	13	4	1	16.2	13.7

Table 2. *Final positional and thermal parameters with esd's in parentheses* (*all* × 10^4)

The form of the temperature factor is $\exp\left[-(h^2\beta_{11} + \ldots hk\beta_{12} + \ldots)\right]$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Br(1)	279 (1)	1017 (2)	7282 (2)	84 (1)	94 (2)	270 (4)	−34 (2)	−11 (3)	−53 (4)
Br(2)	2277 (1)	1030 (2)	903 (2)	106 (2)	104 (2)	259 (4)	50 (3)	−40 (4)	51 (4)
Br(3)	59 (2)	$\frac{1}{4}$*	1786 (3)	96 (2)	114 (2)	223 (5)	(0)	87 (5)	(0)
Br(4)	2474 (2)	$\frac{1}{4}$*	6369 (3)	72 (2)	119 (2)	227 (5)	(0)	48 (5)	(0)
C(5)	1415 (15)	$\frac{1}{4}$*	192 (26)	67 (13)	63 (15)	166 (37)	(0)	−9 (38)	(0)
C(6)	1126 (15)	$\frac{1}{4}$*	7973 (26)	55 (12)	77 (16)	168 (40)	(0)	19 (37)	(0)

* Fractional values are not × 10^4.

cules per unit cell, of 3·88 g.cm^{-3} as compared with the literature value of 3·823 g.cm^{-3}. The systematic absences were $hk0$, if $h=2n+1$ and $0kl$, if $k+l=2n+1$, implying two possible space groups: *Pnma*, which was previously used, or $Pn2_1a$.

The intensity data were collected at 22 °C by the moving-crystal moving-counter technique out to a maximum sin θ/λ of 0·56. The base width was 1·0°, the scan speed 2° per minute, and a 20 sec background count was used. Two standard reflections were monitored every fifty reflections; variations of 5–6 % were observed.

The data set consisted of 744 reflections, of which 137 were unobserved. All reflections having intensities less than 2·33σ (I) were taken as unobserved. Since the linear absorption coefficient was 338·1 cm^{-1}, spherical absorption corrections were made using transmission factors from Volume II of *International Tables for X-ray Crystallography* (1967).

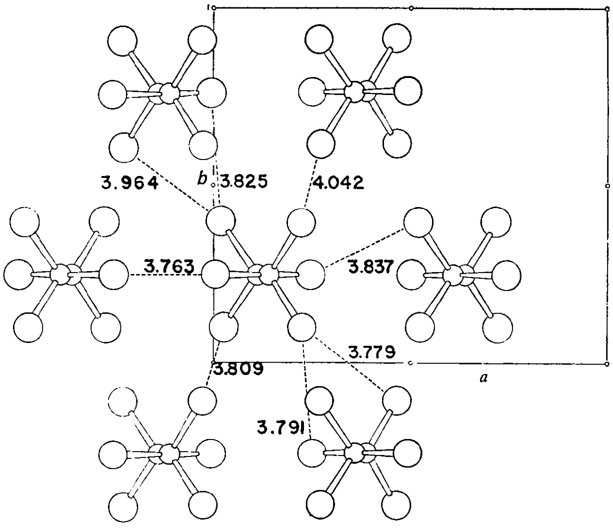

Fig. 2. Structure in projection down [001].

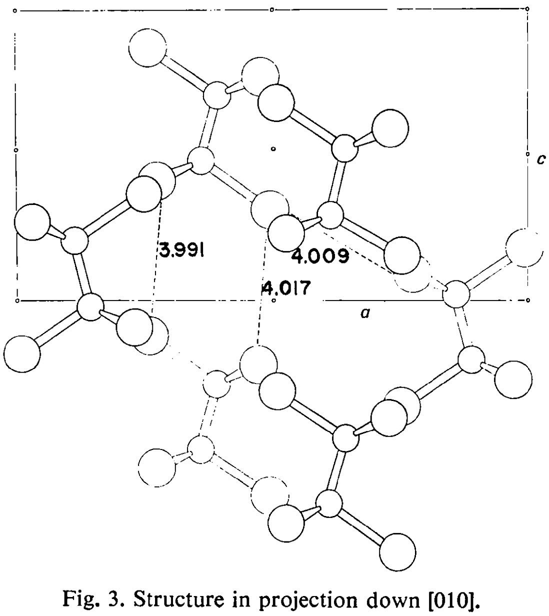

Fig. 3. Structure in projection down [010].

The refinement of the structure

The structure was first confirmed with a three-dimensional Patterson function and then refined using the UCLA full-matrix least-squares program to a final R value of 6·4 %. The atomic scattering factors were taken from Volume III of *International Tables for X-ray Crystallography* (1968). A secondary extinction correction was made with an extinction parameter of $g=2{\cdot}00\times10^{-5}$. The function minimized was the usual $\sum_h w_h[F_o(h)-(1/K)F_c(h)]^2$ where w_h is defined by $\sqrt{w_h}=1/\sigma_h=1/[(F_o^2+\sigma(I))^{1/2}-F_o]$. Observed and calculated structure factors are shown in Table 1. The final parameters and their standard deviations are shown in Table 2.

Discussion

Bond distances and angles are given in Table 3. The carbon–carbon bond length of 1·526 ± 0·024 Å agrees with carbon–carbon single bond length of 1·545 Å given by Sutton, (1965). The average bond angle is equal to the tetrahedral value at 109·6 ± 0·9° No corrections were made for the effects of thermal motion.

Table 3. *Intramolecular geometry*

Bond distances	
C(5)–C(6)	1·526 ± 0·024 Å
Br(4)–C(6)	1·945 ± 0·018
Br(1)–C(6)	1·939 ± 0·011
Br(2)–C(5)	1·940 ± 0·011
Br(3)–C(5)	1·950 ± 0·018
Bond angles	
Br(1)–C(6)–Br(1′)	109·4 ± 0·9°
Br(1)–C(6)–Br(4)	107·9 ± 0·6
Br(1)–C(6)–C(5)	110·6 ± 0·7
Br(4)–C(6)–C(5)	110·4 ± 1·2
Br(2)–C(5)–Br(2′)	107·9 ± 0·9
Br(2)–C(5)–Br(3)	108·2 ± 0·6
Br(2)–C(5)–C(6)	111·1 ± 0·7
Br(3)–C(5)–C(6)	110·1 ± 1·2
Torsion angles	
Br(1)–C(6)–C(5)–Br(3)	60·7°
Br(1)–C(6)–C(5)–Br(2)	59·2
Br(4)–C(6)–C(5)–Br(2)	60·1

The four independent carbon–bromine bond distances do not differ significantly, their average being 1·944 ± 0·005 Å, a value in good agreement with 1·93 Å given by Sutton, (1965).

Other intermolecular distances are given in Fig. 1. The average distance of approach for bromine atoms bonded to the same carbon atom is 3·148 Å, and 3·420 Å for bromine atoms bonded to different carbon atoms. These values may be compared with 3·90 Å, twice the van der Waals radius of Pauling (1960).

The shortest intermolecular bromine–bromine contacts are shown in Fig. 2 and 3. The shortest contacts are 3·763, 0·24 Å shorter than predicted by the Pauling radius.

The choice of *Pnma* in preference to $Pn2_1a$ in this work is verified since the β_{22} element is not significantly larger than the β_{11} or the β_{33} elements for each atom, after conversion to $\mathrm{\AA}^{-2}$.

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