

References

- AZAROFF, L. V. (1955). *Acta Cryst.* **8**, 701–704.
- CANDELORO DE SANCTIS, S., COIRO, V. M., GIGLIO, E., PAGLIUCA, S., PAVEL, N. V. & QUAGLIATA, C. (1978). *Acta Cryst.* **B34**, 1928–1933.
- CANDELORO DE SANCTIS, S. & GIGLIO, E. (1979). *Acta Cryst.* **B35**, 2650–2655.
- CRAVEN, B. M. & DETITTA, G. T. (1972). *Chem. Commun.* pp. 530–531.
- DOMENICANO, A., SPAGNA, R. & VACIAGO, A. (1969). *Atti Accad. Naz. Lincei Cl. Sci. Fis. Mat. Nat. Rend.* **47**, 331–336.
- GIACOMELLO, G. & BIANCHI, E. (1943). *Gazz. Chim. Ital.* **73**, 3–13.
- International Tables for X-ray Crystallography* (1974). Vol. IV, pp. 99–101. Birmingham: Kynoch Press.
- PAVEL, N. V., QUAGLIATA, C. & SCARCELLI, N. (1976). *Z. Kristallogr.* **144**, 64–75.
- RHEINBOLDT, H., FLUME, E. & KÖNIG, O. (1929). *Z. Physiol. Chem.* **180**, 180–186.
- RHEINBOLDT, H., PIEPER, H. & ZERVAS, P. (1927). *Ann. Chem.* **451**, 256–261.
- SOBOTKA, H. & GOLDBERG, A. (1932). *Biochem. J.* **26**, 555–568.
- TANG, C. P. (1979). PhD Thesis. The Feinberg Graduate School, The Weizmann Institute of Science, Rehovot, Israel.
- WIELAND, H. & SORGE, H. (1916). *Z. Physiol. Chem.* **97**, 1–27.

Acta Cryst. (1980). **B36**, 852–857

Crystals Exhibiting Disorder – The Monoclinic Polymorph of 9-Bromo-10-methylantracene

BY RAYMOND D. G. JONES AND T. R. WELBERRY

Research School of Chemistry, Australian National University, PO Box 4, Canberra, ACT, Australia 2600

(Received 25 September 1979; accepted 5 November 1979)

Abstract

The crystal structure of C₁₅H₁₁Br has been determined from X-ray (Mo K α radiation) and neutron ($\lambda = 0.988$ Å) diffraction data. The compound crystallizes in the monoclinic space group *P2₁/c* with $a = 8.307$ (1), $b = 9.844$ (1), $c = 13.815$ (2) Å, $\beta = 95.96$ (1)°, $Z = 4$. Refinement led to *R* and *wR* values of 0.101 and 0.045 for 2197 X-ray reflexions (0.047 and 0.042 for reflexions with $I > 2.3\sigma_I$) and 0.167 and 0.056 for 2299 neutron reflexions (0.059 and 0.046). The 9,10-substituents show disorder of Br and CH₃, the 9-substituent occupied on average by 0.643 Br and 0.357 C and *vice versa* for the 10-substituent. Additionally, the neutron data show two orientations for the H atoms of the CH₃.

Introduction

For some time in this laboratory, models for describing the way in which disorder may be introduced into binary mixed crystals at growth have been studied from a theoretical aspect (Welberry, 1977*a,b*; Welberry & Miller, 1977, 1978; Miller & Welberry, 1979). As a parallel study, we decided to examine some molecular crystal systems which might exhibit growth-disorder.

To reduce the problem to one of site occupancy at one or two atomic positions, the compounds selected had to be reasonably rigid and, once incorporated in the crystal lattice, not undergo large molecular rotations or translations, *i.e.* the molecules would not rearrange to some equilibrium configuration. We wished to determine the average structure by conventional Bragg scattering. Details of disorder would be studied by X-ray diffuse scattering. To ensure pronounced diffuse-scattering effects in the scattering patterns, the components of the binary solid should have a large difference in scattering power.

Polycyclic aromatic ring systems were considered to be suitable basic units. Because of their ease of synthesis, anthracene derivatives were used. Kitaigorodsky (1973) has deduced that the volumes occupied by CH₃, Cl, Br, NO₂, COOH and NH₂ groups in a crystal are similar. On volume considerations alone, one might conclude that binary mixed crystals could be crystallized from similarly substituted anthracenes containing the above groups. Others in our group are undertaking thermodynamic calculations of lattice energies. To enable use of the best published parameters for these calculations, we did not consider substituents containing oxygen or which could participate in hydrogen bonding. Finally, we decided that the properties of a binary mixed solid

could be modelled with a 9,10-disubstituted anthracene where the substituents were different. We postulated that, if the 9-substituent was a halogen and the 10-substituent a CH_3 group, the crystal formed would have halogen and CH_3 averaged over the 9- and 10-positions.

Preparative methods and cell parameters had been recorded for 9-bromo-10-methylanthracene (BMA) and the chloro analogue (Heller & Schmidt, 1971). Each compound has polymorphic forms, one monoclinic, the other orthorhombic. A two-dimensional X-ray study has been reported for the orthorhombic form of BMA in which disorder of the 9,10-substituents was found (Prat, 1961).

We have undertaken the three-dimensional crystal structure determination of the monoclinic BMA. The components of the binary solid are Br and CH_3 which satisfy the criteria of a large difference in scattering power. For X-ray diffraction, the derived occupancy of Br in the Br/ CH_3 site will be dominated by the relatively large scattering power of Br. As a check against possible bias due to the domination of the scattering by Br, the structure determination was repeated by neutron diffraction. In this case, Br and C have similar scattering lengths and so the site occupancy will be determined by the methyl H atoms which are readily located by neutron diffraction methods. In addition, the H positions do not overlap with other atomic positions.

Experimental

BMA was prepared by the method of Heller & Schmidt (1971). Crystals were grown at room temperature by evaporation of a solution in petroleum ether (b.p. 333–353 K). Yellow crystals of both polymorphs formed but were nearly exclusively the monoclinic form. The crystals of the monoclinic form are rhomb-shaped with developed faces of forms $\{011\}$ and $\{100\}$.

Crystal data

$\text{C}_{15}\text{H}_{11}\text{Br}$, $M_r = 271.16$, monoclinic, space group $P2_1/c$, $a = 8.307$ (1), $b = 9.844$ (1), $c = 13.815$ (2) Å, $\beta = 95.96$ (1)°, $U = 1123.6$ Å³, $d_m = 1.60$, $d_c = 1.60$ Mg m⁻³ for $Z = 4$, $F(000) = 544$; Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu(\text{Mo } K\alpha) = 3.836$ mm⁻¹; neutron wavelength 0.988 Å, $\mu(\text{neutrons}) = 0.177$ mm⁻¹.

Data collection

X-ray

A crystal $0.45 \times 0.24 \times 0.18$ mm was used to determine the cell dimensions and measure the intensities. The crystal was mounted approximately along b.

Cell dimensions were obtained by least squares from the 2θ values of 12 high-angle reflexions. Intensities were collected by the θ - 2θ continuous scan method with graphite-monochromated Mo $K\alpha$ radiation. The scan width was 1.8°, extended for the α_1, α_2 splitting, and stationary background counts were taken for 10 s at the scan limits. Three standard reflexions were periodically monitored. The mean deviation of any standard from its mean value was 1.4%. One quadrant of data was collected up to $2\theta = 55^\circ$.

Intensities were corrected for background, absorption and the Lorentz-polarization factor. For each reflexion, the variance, σ_i^2 , was determined from $\sigma_i^2 = [\sigma_c^2 + \sigma_A^2 + (0.014I)^2]$ where σ_c is due to counting statistics, σ_A is due to absorption (Elcombe, Cox, Pryor & Moore, 1971) and I is the net intensity. If $I < 0$, it was set to zero and given zero weight. 2197 unique reflexions were measured of which 1533 had $I > 2.3\sigma_i$.

Neutron

The crystal measured $5.44 \times 3.38 \times 0.94$ mm. It was mounted along $[011]$. Data were collected on the Australian Institute of Nuclear Science and Engineering four-circle diffractometer, 2TAN A, at the Australian Atomic Energy Commission Research Reactor, HIFAR. A θ - 2θ step-scan technique was used (Elcombe, Cox, Pryor & Moore, 1971), each step being 0.01° in 2θ . The scan range was 2.2° in 2θ . All unique reflexions were scanned up to $2\theta = 80^\circ$. A standard reflexion was periodically monitored. Its mean deviation from the mean value was 2.8%.

Intensities were corrected for background, absorption and the Lorentz factor. σ_i^2 was determined as before. 2299 unique reflexions were measured, of which 1190 had $I > 2.3\sigma_i$.

Structure solution and refinement

X-ray

The structure was solved with *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1974). All non-hydrogen atoms were located in the E map using phases from the starting set which gave the highest figure of merit and lowest residual. A Patterson synthesis suggested that the 9,10-substituents were disordered. A structure factor calculation with the subset of 1533 reflexions gave $R = \sum(|F_o| - k|F_c|) / \sum|F_o| = 0.33$. 50/50 disorder at 9,10-substituents was assumed. An electron density map was consistent with 60/40 substitution in favour of Br at the 9-substituent site and *vice versa* at 10. With this change, R was 0.23. Refinement of positional and isotropic thermal parameters led to an R of 0.133. Unit weights were used and the function minimized was

$\sum w(|F_o| - k|F_c|)^2$. If the occupancy factor of Br was varied, R reduced to 0.124. Refinement of anisotropic thermal parameters for Br and C and the introduction of ring H atoms (found in a difference map) but with only their coordinates refined resulted in an R of 0.048 at convergence. During these refinements, the C parameters at the disordered positions were fixed at the Br values and the occupancy at $1 - x$, x being the Br occupancy. Final refinement was carried out with all reflexions (Moore, 1972), experimental weights $w = \sigma_{F_o}^{-2}$ and anomalous-dispersion corrections for Br (Cromer & Liberman, 1970). At convergence R was 0.101 and wR [$\sum w(|F_o| - k|F_c|)^2 / \sum w|F_o|^2$] was 0.045. The error of fit was 1.24. For the subset of 1533 reflexions, R was 0.047 (0.042). $w\Delta^2$ was reasonably constant for ranges of F_o and $(\sin \theta)/\lambda$. A final difference map showed no significant features. The refined value for the occupancy of Br at the 9-substituent was 0.641 (2). At no stage did we find evidence of split Br/C(methyl) positions. In addition, there was no evidence for disorder in the anthracene ring. Scattering factors were the analytical forms tabulated in *International Tables for X-ray Crystallography* (1974).

Neutron

Structure factors were calculated with Br and C positions determined by X-ray data and with average thermal parameters. The subsequent difference synthesis showed all ring H atoms. After recalculation of structure factors, a fine-grid difference synthesis had six possible positions for H atoms near each of the 9,10-substituents ranging in height from $\frac{1}{3}$ to $\frac{1}{2}$ that of a ring H atom. Calculated positions for H atoms were determined with respect to a C atom placed 1.51 Å from the relevant ring C. Six positions thus calculated were near those in the difference map and were nearly symmetrically distributed around the calculated C position. We treated these positions as two sets of half-occupancy methyl H atoms. Refinement, with unit weight, of positional and isotropic thermal parameters of all atoms except methyl H atoms and the assumption of 50/50 disorder at 9,10-substituents resulted in an R of 0.127 for the subset of 1190 reflexions. When methyl H thermal parameters were varied, those near C(15) were $\sim 11 \text{ \AA}^2$, near Br $\sim 20 \text{ \AA}^2$. When occupancies were based on the X-ray results, values of ~ 7 and $\sim 11 \text{ \AA}^2$ were obtained; R was 0.097. Further refinement used all reflexion data and $w = \sigma_{F_o}^{-2}$. Anisotropic thermal parameters were used for all atoms except methyl H. Isotropic thermal motion described the latter and, in addition, their occupancy as a group at each partially occupied C was varied. Although refinement converged, H(165) and H(166) were giving unsatisfactory positions. When all parameters were varied except those of these atoms and then the process reversed, reasonable positions were obtained. There

Table 1. *Final fractional coordinates* ($\times 10^4$ for non-hydrogen atoms, $\times 10^3$ for X-ray hydrogen coordinates, $\times 10^4$ for neutron ring hydrogen coordinates and $\times 10^3$ for neutron methyl hydrogen coordinates)

Neutron coordinates are given first.			
	x	y	z
Br	2405 (5)	5697 (5)	8283 (3)
	2470.6 (.6)	5731.4 (.5)	8263.5 (.3)
C(1)	2764 (4)	3694 (3)	9979 (2)
	2763 (4)	3684 (3)	9978 (2)
C(2)	3065 (4)	2841 (4)	10744 (3)
	3062 (4)	2837 (4)	10743 (2)
C(3)	1846 (4)	1968 (4)	11019 (3)
	1857 (5)	1966 (3)	11012 (2)
C(4)	355 (4)	1970 (3)	10507 (2)
	364 (4)	1970 (3)	10506 (2)
C(5)	-3472 (4)	3820 (3)	7838 (2)
	-3470 (3)	3829 (3)	7833 (2)
C(6)	-3783 (4)	4688 (4)	7070 (3)
	-3764 (4)	4686 (3)	7070 (2)
C(7)	-2552 (4)	5557 (4)	6792 (3)
	-2549 (4)	5552 (3)	6798 (2)
C(8)	-1054 (4)	5545 (3)	7292 (2)
	-1062 (4)	5537 (3)	7294 (2)
C(9)	867 (3)	4605 (3)	8633 (2)
	863 (3)	4610 (3)	8628 (2)
C(10)	-1575 (3)	2874 (3)	9174 (2)
	-1573 (3)	2875 (3)	9178 (2)
C(11)	1215 (3)	3734 (3)	9427 (2)
	1211 (3)	3734 (3)	9426 (2)
C(12)	-26 (3)	2851 (3)	9699 (2)
	-28 (3)	2851 (2)	9702 (2)
C(13)	-673 (3)	4651 (3)	8102 (2)
	-669 (3)	4651 (3)	8102 (2)
C(14)	-1920 (3)	3760 (3)	8384 (2)
	-1910 (3)	3763 (3)	8383 (2)
C(15)	-2982 (5)	1859 (4)	9494 (3)
	-3125 (1)	1793 (1)	9520 (1)
H(1)	3726 (8)	4374 (8)	9781 (6)
	350 (4)	423 (3)	979 (2)
H(2)	4258 (8)	2825 (9)	11163 (6)
	407 (4)	282 (3)	1106 (2)
H(3)	2111 (10)	1292 (9)	11641 (6)
	208 (4)	138 (3)	1159 (2)
H(4)	-582 (9)	1294 (8)	10735 (6)
	-49 (4)	139 (3)	1068 (2)
H(5)	-4434 (8)	3169 (8)	8040 (6)
	-428 (4)	329 (3)	805 (2)
H(6)	-4973 (9)	4704 (9)	6661 (6)
	-477 (4)	472 (3)	676 (2)
H(7)	-2805 (9)	6239 (8)	6171 (6)
	-274 (4)	622 (3)	621 (2)
H(8)	-114 (9)	6209 (8)	7078 (6)
	-25 (4)	612 (3)	709 (2)
H(151) ^(a)	-406 (3)	193 (2)	906 (2)
H(152)	-258 (2)	84 (2)	946 (2)
H(153)	-308 (3)	202 (2)	1025 (2)
H(154)	-328 (3)	122 (3)	887 (2)
H(155)	-253 (3)	130 (3)	1010 (2)
H(156)	-394 (3)	250 (3)	963 (2)
H(161) ^(b)	304 (5)	597 (4)	890 (3)
H(162)	272 (6)	535 (6)	777 (4)
H(163)	191 (5)	656 (5)	815 (3)
H(164)	337 (5)	507 (4)	829 (3)
H(165)	193 (4)	586 (4)	750 (3)
H(166)	215 (5)	662 (5)	875 (3)

(a) Hydrogen atoms associated with 10-substituted carbon.

(b) Hydrogen atoms associated with 9-substituted carbon.

was evidence for extinction, especially for the reflexion 100 which was given zero weight in refinement. At convergence R was 0.167 and wR 0.056. The error of fit was 1.3. For the subset of reflexions, R was 0.059, wR 0.046. The occupancy of Br at the 9-substituent and C at the 10-substituent and its attached H was 0.644 (8). $w\Delta^2$ was reasonably constant for ranges of F_o and $(\sin \theta)/\lambda$. A final difference synthesis showed no significant features. Scattering lengths of the atoms were as tabulated by Bacon (1972). Again, the data did not allow resolution of the Br and C at the disordered sites.*

Computer programs used were from the ANUCRYS structure determination package (P. O. Whimp, D. Taylor, G. M. McLaughlin and D. A. Kelly).

Results

Final atomic coordinates are listed in Table 1. Interatomic distances and angles for the X-ray structure are shown in Fig. 1; those for the neutron structure in Figs. 2 and 3. E.s.d.'s were derived from the

* Lists of structure factors and anisotropic thermal parameters for both determinations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34972 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

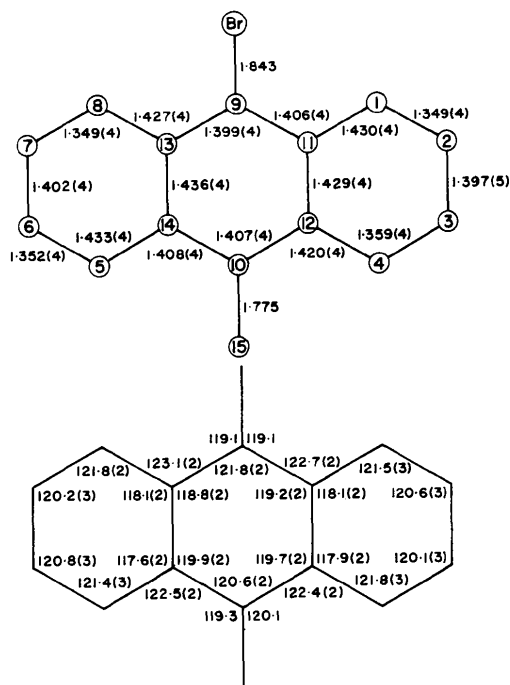


Fig. 1. Interatomic distances (Å) and angles ($^{\circ}$) derived from the X-ray data. In this figure, and subsequent similar figures, the e.s.d.'s are shown in parentheses.

least-squares variance-covariance matrix. Table 2 gives the equation to the plane of least-squares fit to the molecule and the deviations from the plane. The two sets of coordinates of the ring C atoms were analysed by the half-normal probability method (Abrahams &

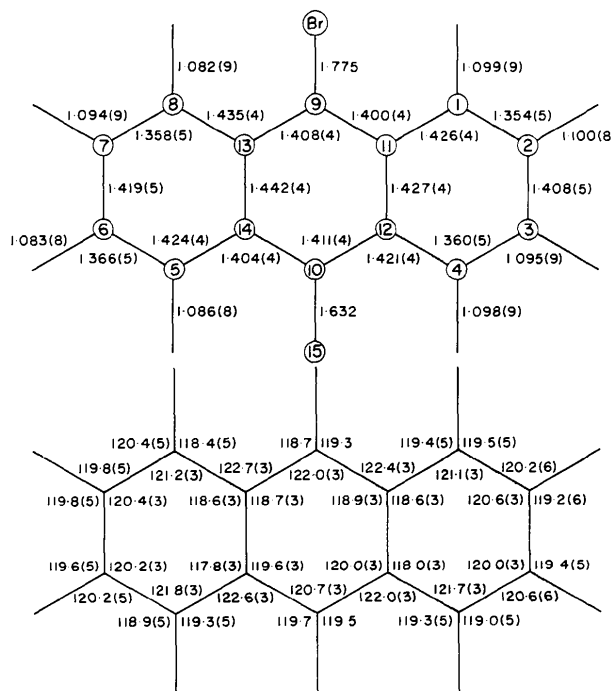


Fig. 2. Interatomic distances (Å) and angles ($^{\circ}$) derived from the neutron data.

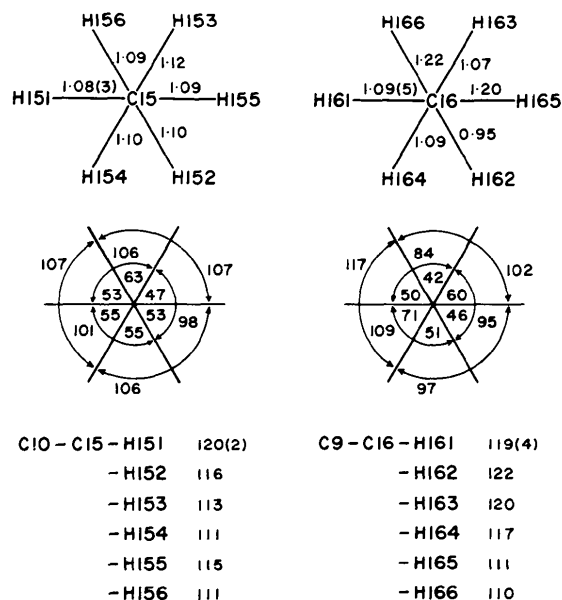


Fig. 3. Interatomic distances (Å) and angles ($^{\circ}$) involving methyl H atoms located in the neutron structure determination.

Table 2. A least-squares plane and deviations (Å) of atoms from it

The equation to the plane of best fit for the anthracene moiety is $0.3532X - 0.7042Y - 0.6160Z + 10.690 = 0$, where X , Y and Z (in Å) are along a , b and c^* .

C(1)	-0.004 (3)	C(9)	0.009 (3)
C(2)	-0.015 (3)	C(10)	0.003 (3)
C(3)	-0.006 (3)	C(11)	0.003 (3)
C(4)	0.007 (3)	C(12)	0.002 (3)
C(5)	-0.008 (3)	C(13)	0.003 (3)
C(6)	-0.004 (3)	C(14)	0.002 (3)
C(7)	-0.004 (3)	C(15)†	-0.009 (1)
C(8)	-0.002 (3)	Br†	0.030 (1)

† Atoms not included in the plane calculation.

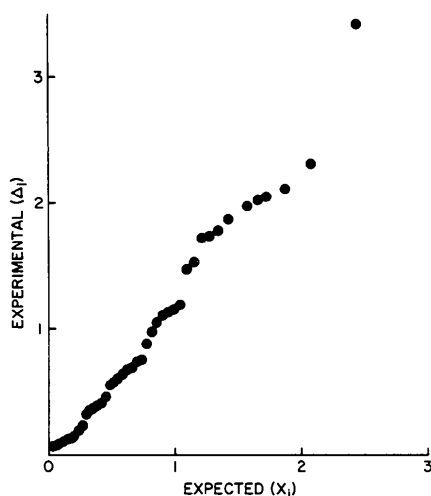


Fig. 4. Half-normal probability plot for atomic coordinates derived from X-ray and neutron data. Only ring C atoms were considered.

Keve, 1971). The 9,10-substituents were not included since, because of the different scattering processes involved and the relative scattering of Br and C in X-ray and neutron diffraction, one would not expect the derived average positions to agree for the two techniques. The half-normal probability plot is shown in Fig. 4. Although the points on the plot tend to segregate in batches, those points with experimental $|\Delta| < 2$ give a good fit to a straight line. This line has slope 1.37 and intercept -0.11 which suggests acceptable agreement between the coordinate sets and a reasonable weighting scheme.

Discussion

That the structure is disordered confirms our ideas on substitutional-disorder properties of Br and CH_3 ,

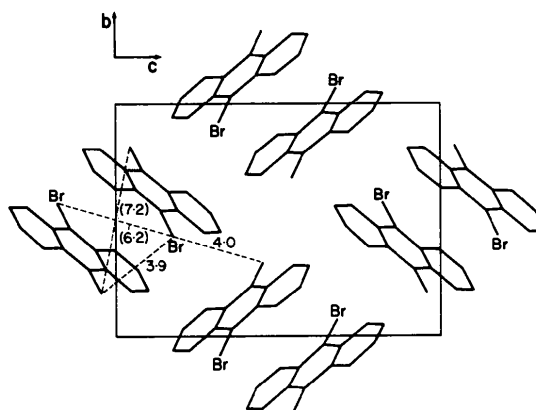


Fig. 5. Projection of the structure down b showing contacts involving 9,10-substituents.

groups. The perhaps unexpected result is that disorder is not 50/50 for the 9,10-substituents but 64.3/35.7. This has been confirmed by two independent methods and is not an artifact of the refinement procedure.

The X-ray diffuse scattering studies to be reported later (Welberry, 1980) show that, in addition to the preferred site occupancy determined here, the orientations of neighbouring molecules related by the statistical centre of symmetry are additionally correlated. In this way, we find that the Br/Me sites which are 6.2 Å apart (see Fig. 5) are both occupied by Br most frequently, the sites which are 7.2 Å apart are both occupied by Br rather less frequently, while the sites which are only 3.9 Å apart are rarely occupied by Br simultaneously.

Disorder is only apparent at the 9,10-substituents. The data do not allow atomic resolution of the Br and methyl C at these positions. Consequently, $\text{Br}-\text{C}(9)$, 1.843 Å, and $\text{C}(15)-\text{C}(10)$, 1.775 Å, lie between 1.897 Å for $\text{Br}-\text{C}(\text{aromatic})$ (James & Williams, 1973) and 1.51 Å for $\text{C}(\text{methyl})-\text{C}(\text{aromatic})$ as deduced from the values reported for some 9- and 10-methyl-substituted anthracenes (Dellaca, Penfold & Robinson, 1969; Chomyn, Glusker, Berman & Carrell, 1972; Glusker & Zacharias, 1972; Iball & Low, 1974; Cox & Sim, 1979). Disorder has been observed, or from the interatomic distances quoted there would appear to be disorder, in other 9,10-disubstituted anthracenes. 9-Bromo-10-chloroanthracene (Hospital, 1961) has been reported to have 50/50 random disorder of the Cl and Br atoms. This conclusion was reached because the molecule was placed at the centre of symmetry of $P1$. $P1$ was apparently not considered. Few details of the structure were published and perhaps $P1$ might have led to different conclusions. For 9-chloromethyl-10-methylantracene (Glusker, Carrell, Berman, Gallen & Peck, 1977), the Cl occupancies at the 9- and 10-positions are 0.737 and 0.241. The authors suggest that 'molecules are flipped over in

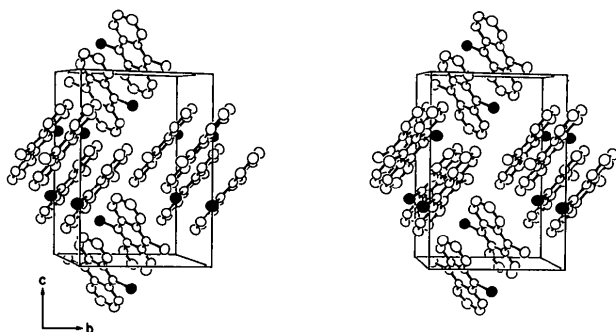


Fig. 6. Stereoscopic view of the structure down a^* .

domains in the crystal since a random packing of CH_3 and CH_2Cl groups would not fit (the Cl atoms would be too close to each other). Kitaigorodsky (1973) gives the volume of CH_3 as 23.5 \AA^3 and, with his method of calculation, the volume of CH_2Cl is 34.6 \AA^3 . Although the latter is a 50% increase over the former, the shape of CH_2Cl is very anisotropic. It seems possible that molecules in alternate orientations could be closest neighbours especially if the Cl atoms tended to be furthest apart. This would not require large mosaic blocks but could be accommodated with relaxation of the lattice after only a small number of ordered molecules. This suggestion could possibly be checked by analysis of diffuse scattering.

Interatomic distances and angles in BMA parallel those found in anthracene (Mason, 1964; Lehmann & Pawley, 1972) and the anthracene derivatives referred to earlier. This is consistent with our not detecting any disorder in the anthracene ring. The major difference from anthracene is the opening up of the exocyclic angle at the ring junctions and a decrease in angles of type $\text{C}(1)-\text{C}(11)-\text{C}(12)$.

Heller & Schmidt (1971) have reported that BMA does not photodimerize in solution or the solid state. As discussed previously, diffuse-scattering measurements show that a pair of molecules related by the statistical centre of symmetry do largely have the head-to-tail configuration. However, the central rings do not overlap, but a central ring overlaps one of the end rings (Fig. 6). Intermolecular contacts are $\text{C}(9)\cdots\text{C}(10')$ (the prime refers to the molecule at $\bar{x}, \bar{y}, \bar{z}$) and $\text{C}(10)\cdots\text{C}(9')$ of 3.91 \AA and $\text{C}(9)\cdots\text{C}(4')$ and $\text{C}(10)\cdots\text{C}(1')$ of 3.75 \AA . Whereas the former distances are probably too large for dimerization in the solid state, the latter are not.

Neutron diffraction measurements were made possible by a grant from the Australian Institute of Nuclear Science and Engineering.

References

- ABRAHAMSON, S. C. & KEVE, E. T. (1971). *Acta Cryst.* **A27**, 157–165.
- BACON, G. E. (1972). *Acta Cryst.* **A28**, 357–358.
- CHOMYN, A., GLUSKER, J., BERMAN, H. M. & CARRELL, H. L. (1972). *Acta Cryst.* **B28**, 3512–3517.
- COX, P. J. & SIM, G. A. (1979). *Acta Cryst.* **B35**, 404–410.
- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
- DELLACA, R. J., PENFOLD, B. R. & ROBINSON, W. T. (1969). *Acta Cryst.* **B25**, 1589–1594.
- ELCOMBE, M. M., COX, G. W., PRYOR, A. W. & MOORE, F. H. (1971). *Programs for the Management and Processing of Neutron Diffraction Data*. Australian Atomic Energy Commission Report AAEC/TM578.
- GLUSKER, J., CARRELL, H. L., BERMAN, H. M., GALLEN, B. & PECK, R. M. (1977). *J. Am. Chem. Soc.* **99**, 595–601.
- GLUSKER, J. & ZACHARIAS, D. E. (1972). *Acta Cryst.* **B28**, 3518–3525.
- HELLER, E. & SCHMIDT, G. M. J. (1971). *Isr. J. Chem.* **9**, 449–462.
- HOSPITAL, M. (1961). *Acta Cryst.* **14**, 76.
- IBALL, J. & LOW, J. N. (1974). *Acta Cryst.* **B30**, 2203–2205.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press.
- JAMES, M. N. G. & WILLIAMS, G. J. B. (1973). *Acta Cryst.* **B29**, 1172–1174.
- KITAIGORODSKY, A. I. (1973). *Molecular Crystals and Molecules*. New York: Academic Press.
- LEHMANN, M. S. & PAWLEY, G. S. (1972). *Acta Chem. Scand.* **26**, 1996–2004.
- MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J. P. (1974). *MULTAN 74. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MASON, R. (1964). *Acta Cryst.* **17**, 547–555.
- MILLER, G. H. & WELBERRY, T. R. (1979). *Acta Cryst.* **A35**, 391–400.
- MOORE, F. H. (1972). *Acta Cryst.* **A28**, S256.
- PRAT, M.-T. (1961). *Acta Cryst.* **14**, 110–112.
- WELBERRY, T. R. (1977a). *Proc. R. Soc. London Ser. A*, **353**, 363–376.
- WELBERRY, T. R. (1977b). *J. Appl. Cryst.* **10**, 344–348.
- WELBERRY, T. R. (1980). *J. Appl. Cryst.* To be published.
- WELBERRY, T. R. & MILLER, G. H. (1977). *J. Appl. Prob.* **14**, 862–868.
- WELBERRY, T. R. & MILLER, G. H. (1978). *Acta Cryst.* **A34**, 120–123.