1976). These are usually associated with the onset of disorder of the cations in the structure (Kind *et al.*, 1979). A DTA scan of this salt revealed no evidence of a similar transition. Apparently the multiple hydrogen-bonding possibilities for the $-NH_3^+$ moieties allow disorder of the organic chain, but the hydrogen bonding to the lattice chloride is so restrictive that it prohibits such motion.

The $MnCl_6$ octahedra are tipped away from the normal to the layer by 7·5°. This tilt has been associated with the weak ferromagnetic behavior of these salts, as evidenced by the existence of a small residual magnetic moment in the magnetically ordered state and the onset of hysteresis behavior at the magnetic phase-transition temperature, $T_N$ (Groenendijk, van Duyneveldt & Willett, 1979). Preliminary a.c. susceptibility measurements were made on a Hartshorn bridge at Iowa State University over the temperature range 30–50 K. This is the region anticipated for the onset of three-dimensional ordering. The existence of the sharp peak in the susceptibility at 43 K is characteristic of a weak ferromagnetic system. Thus, the existence of spin canting is confirmed in this salt, consistent with the small canting of the spin moment in the direction of the tilt of the terminal Mn—Cl bond away from the normal to the layer.

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# *o,o'*-Dibromodibenzyl Ether

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**Abstract.** $C_{14}H_{12}Br_2O$, $M_r$ = 356·06, monoclinic, $P2_1/c$, $a$ = 12·222 (2), $b$ = 10·157 (3), $c$ = 13·183 (2) Å, $\beta$ = 125·86 (1)°; $Z$ = 4, $D_c$ = 1·783 Mg m$^{-3}$. The structure was solved by heavy-atom techniques and refined by least squares, using 936 observed data and anisotropic temperature factors, to $R$ = 0·044. The two halves of the molecule are not related by symmetry. Molecules of *o,o'*-dibromodibenzyl ether are approximately planar with both Br atoms on the same side of the molecule.

**Introduction.** Many tricyclic compounds show psychotropic activity (Chang, 1979). Useful precursors to these potential drugs contain *ortho*-bromo substituted phenyl rings separated by one or more atoms (Chang, 1979). These latter materials provide a vehicle for the study of the effects of intramolecular interactions on the configuration about the central atoms. The two-atom separation case has been the study of several investigations (Corey, 1979 and references therein). To extend these studies to the three-atom separation case the structure of the title compound was investigated.

Crystals were prepared by V. Chang (Chang, 1979). An approximately spherical crystal with a diameter of 0·22 mm was mounted on a Syntex $P2_1$ diffractometer and a total of 1865 intensities were measured using Mo

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$K\alpha$ (graphite monochromator) radiation ($\lambda$ = 0·71069 Å). All reflections with $2\theta < 45°$ were measured with a $\theta$–$2\theta$ scan speed of 2·0° min$^{-1}$. Backgrounds were measured at each end of the scan ($2\theta$ scan range: $2\theta$Mo $K\alpha_1$ − 1·0° to $2\theta$Mo $K\alpha_2$ + 1·0°) for a total time equal to one-half the scan time. During data collection, the intensities of three standard reflections were measured every 97 reflections; no significant variation in intensity was observed. The data were reduced to $F^2$ and $\sigma(F^2)$ (Schmonsees, 1974); no absorption correction was made [$\mu$(Mo $K\alpha$) = 6·0 mm$^{-1}$]. Standard deviations were assigned as follows: $\sigma(I) = [\sigma_{\text{counter}}(I)^2 + (0{\cdot}04I)^2]^{1/2}$ where $\sigma_{\text{counter}} = (I + K^2B)^{1/2}$, $I$ = net intensity, $B$ = total background counts and $K$ = ratio of scan time to background time. The 936 data with $F_o^2 > 2{\cdot}5\sigma(F_o^2)$ were used in the structure solution and refinement.

The lattice constants and their standard deviations were determined by a least-squares refinement of 15 reflections with $2\theta > 15°$.

The structure was determined by heavy-atom techniques and was refined by least-squares methods (Busing, Martin & Levy, 1962). The scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Real and imaginary anomalous-dispersion corrections were made for the Br atoms. No correction was applied for extinction. Unobserved reflections were not included in the refinements.

There is one molecule in the asymmetric unit. The positional coordinates of the Br atoms were determined from a Patterson map and the remaining non-H atoms were located from an electron density map calculated with phases determined by the Br atoms. Refinement was completed with anisotropic temperature factors and inclusion of H atoms at fixed positions (Zalkin, 1974). The H atoms were relocated after each cycle of refinement with C—H distances of 1·0 Å. The largest positional parameter and temperature factor shifts in the final cycle of refinement were 0·10$\sigma$ and 0·24$\sigma$, respectively. The final value of $R$ was 0·044 and the final difference map showed no feature larger than 0·57 e Å$^{-3}$.

Table 1. *Final atomic positional parameters* (×10$^4$) *and the equivalent value of the anisotropic temperature factor coefficients for the non-*H *atoms*

| | $x$ | $y$ | $z$ | $B_{eq}$ (Å$^2$) |
|---|---|---|---|---|
| Br | 1662 (1) | 4956 (1) | −1183 (1) | 5·98 |
| Br′ | 5362 (1) | 8124 (1) | 5477 (1) | 5·64 |
| C(1) | 2317 (11) | 5061 (11) | 1486 (10) | 3·82 |
| C(2) | 1446 (10) | 3955 (11) | 703 (9) | 3·04 |
| C(3) | 1037 (11) | 3754 (11) | −511 (10) | 3·63 |
| C(4) | 199 (12) | 2733 (13) | −1269 (11) | 4·26 |
| C(5) | −250 (12) | 1867 (13) | −787 (11) | 4·83 |
| C(6) | 140 (13) | 2055 (13) | 420 (12) | 5·08 |
| C(7) | 978 (11) | 3051 (12) | 1159 (10) | 3·90 |
| C(1′) | 3362 (11) | 6050 (12) | 3446 (9) | 3·75 |
| C(2′) | 3542 (11) | 5958 (11) | 4672 (10) | 3·39 |
| C(3′) | 4386 (11) | 6820 (11) | 5662 (10) | 3·51 |
| C(4′) | 4561 (12) | 6750 (12) | 6786 (10) | 4·45 |
| C(5′) | 3911 (13) | 5793 (15) | 6972 (11) | 4·95 |
| C(6′) | 3062 (11) | 4910 (13) | 6032 (12) | 4·93 |
| C(7′) | 2896 (11) | 5009 (12) | 4875 (10) | 4·20 |
| O | 2530 (7) | 5054 (7) | 2657 (6) | 4·01 |

$B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\,\mathbf{a}_i\cdot\mathbf{a}_j$ (Hamilton, 1959).

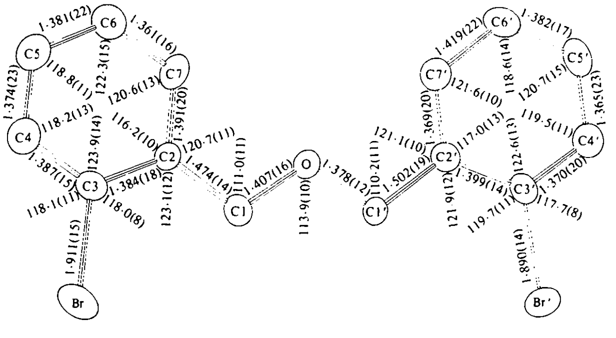

Fig. 1. A perspective drawing of *o,o′*-dibromodibenzyl ether, showing bond distances (Å) and angles (°) in the molecule.

**Discussion.** The final atomic parameters for all non-H atoms are given in Table 1. All atoms are in the general position. The numbers in parentheses are the standard deviations in the least significant figure as estimated from the least-squares analysis.* A perspective drawing of the molecule is given in Fig. 1 with associated bond distances and angles and their estimated standard deviations (Johnson, 1965; Busing, Martin & Levy, 1964).

The two halves of the molecule are not related by symmetry. The molecule is approximately planar. The largest displacement of an atom [C(5)] from the best plane is 0·16 Å. The largest displacements of an atom from the best plane of the benzyl moieties are 0·017 Å [for C(1′)–C(7′) and Br′] and 0·025 Å [for C(1)–C(7) and Br]. The normals to the planes of the two benzyl moieties make an angle of 4·2° with each other. The packing of the molecules is illustrated in Fig. 2. The shortest intermolecular distance that involves a non-H atom and an H atom is 2·87 Å between C(4′) and an H atom on the methylene carbon [C(1′)] related by the center of symmetry at $\frac{1}{2},\frac{1}{2},\frac{1}{2}$. The shortest intermolecular distance involving two H atoms is 2·54 Å.

In the two-atom separation case (dibenzyl and related compounds) the phenyl rings lie in parallel

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* Tables of structure factors, final hydrogen atom positions and anisotropic temperature factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35873 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

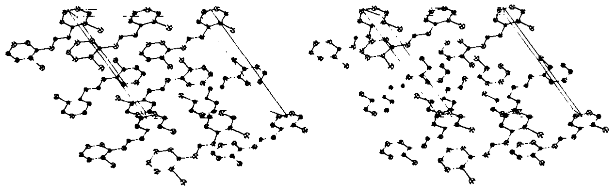

Fig. 2. Packing of the *o,o'*-dibromodibenzyl ether molecules viewed down *b*. Atoms are drawn with 25% probability thermal ellipsoids. The *c* axis is across the page.

planes approximately perpendicular to the best plane of the central bonds of the molecule (observed angles 72–89°, Corey, 1979). In the title compound the observed configuration is probably favored because it minimizes interactions with the lone pairs of oxygen electrons.

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# Anilino-2 Méthyl-4 *N*-Méthyl-anilino-5 Thiazole-1,3 et Anilino-2 *N*-Méthyl-anilino-5 Phényl-4 Thiazole-1,3 (Deux Dérivés du Thiazole-1,3)

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(*Reçu le* 8 *septembre* 1980, *accepté le* 17 *décembre* 1980)

**Abstract.** $C_{17}H_{17}N_3S$ (2-anilino-4-methyl-5-*N*-methylanilino-1,3-thiazole), monoclinic, $C2/c$, $a = 22\cdot844$ (8), $b = 5\cdot634$ (2), $c = 25\cdot291$ (11) Å, $\beta = 108\cdot15$ (3)°, $V = 3093$ (4) Å$^3$, $M_r = 295$, $Z = 8$, $D_x = 1\cdot26$ Mg m$^{-3}$. $R = 0\cdot039$ for 1652 reflections. $C_{22}H_{19}N_3S$ (2-anilino-5-*N*-methylanilino-4-phenyl-1,3-thiazole), monoclinic, $P2_1/n$, $a = 5\cdot921$ (2), $b = 14\cdot642$ (13), $c = 21\cdot516$ (13) Å, $\beta = 94\cdot21$ (4)°, $V = 1860$ (2) Å$^3$, $M_r = 357$, $Z = 4$, $D_x = 1\cdot27$ Mg m$^{-3}$. $R = 0\cdot044$ for 1836 reflections. The thiazole ring is planar in both compounds. The data indicate a resonance interaction between the exocyclic nitrogen and the ring.

**Introduction.** Les molécules dont la structure est décrite dans le présent article sont les dérivés méthyl-4 (I) et phényl-4 (II) de l'anilino-2 *N*-méthyl-anilino-5 thiazole-1,3. Ces composés ont été obtenus par addition d'isothiocyanate de phényle aux dérivés monosubstitués en 3 (méthyl ou phényl) de la *N*-méthylanilino-2 azirine. Pour ce type d'azirines, la structure des produits d'addition est intéressante à établir car elle indique que l'ouverture du cycle se produit au niveau de la liaison N(1)—C(2) alors que dans les dérivés disubstitués en 3, l'ouverture a lieu par rupture de la liaison N(1)—C(3) (Germain, Declercq, Touillaux, Van Meerssche & Henriet, 1979).

Les spectres de diffraction ont été relevés sur un diffractomètre Syntex $P2_1$ équipé d'un monochromateur au graphite et produisant une radiation incidente Mo $K\alpha$ de longueur d'onde $\lambda = 0{,}71069$ Å.

Pour le composé (I), l'exploration du réseau réciproque s'est faite jusqu'à une valeur de $2\theta_{\max} = 47°$ et 1652 parmi les 2282 réflexions mesurées ont été retenues. Pour le composé (II), les intensités de 2770 réflexions ont été mesurées jusqu'à une valeur de

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