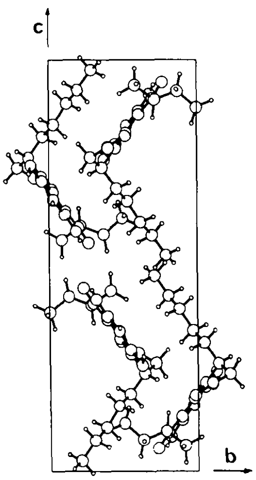

Fig. 2. *PLUTO* (Motherwell, 1972) drawing of the packing of molecules in the unit cell.

molecule related by $2 - x, \frac{1}{2} + y, \frac{1}{2} - z$ is almost parallel to the benzene ring with transformed C(12) 3·693 (7) Å above a point approximately midway between C(5) and C(7). The operation $2 - x, -\frac{1}{2} + y, \frac{1}{2} - z$ produces contacts on the opposite side of the ring between C(6) and transformed C(4) of 3·470 (5) Å, and between C(8*A*) and transformed O(5) of 3·628 (5) Å. Thus the 'L-shaped' molecules can fill space efficiently.

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Structure of 2-Amino-3,5-dibromo-*N*-cyclohexyl-*N*-methylbenzenemethanamine–1,2-Benzisothiazol-3(2*H*)-one 1,1-Dioxide (1:1), $C_{14}H_{20}Br_2N_2.C_7H_5NO_3S$

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Abstract. $M_r = 559{\cdot}3$, orthorhombic, *Pbca*, $a = 15{\cdot}374\,(2)$, $b = 25{\cdot}330\,(4)$, $c = 11{\cdot}630\,(13)$ Å, $U = 4529{\cdot}0$ Å^3, $Z = 8$, $D_x = 1{\cdot}641$, $D_m = 1{\cdot}632$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 4{\cdot}03$ mm^{-1}, $F(000) = 2256$. Final $R = 0{\cdot}095$ for 2577 observed reflections. 2-Amino-3,5-dibromo-*N*-cyclohexyl-*N*-methylbenzenemethanamine (ABCMBMA) and 1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide (*o*-sulfobenzoimide, OSBI) are alternately linked by hydrogen bonds [N(1)–H(1)···O(2), N(2)–H···N(3)] and a permanent dipole–induced-dipole interaction to form an infinite chain in the **a** direction.

Introduction. ABCMBMA is an expectorant drug and OSBI is an artificial sweetener. This investigation was undertaken as part of a series of our structural studies of molecular complexes between different drugs.

Experimental. Suitable crystals (m.p. 459 K) for diffraction analysis grown *via* slow evaporation of an ethanol solution of equimolar quantities of ABCMBMA and OSBI, D_m measured by flotation in $CCl_4/CHBr_3$, crystal 0·2 × 0·3 × 0·4 mm; preliminary Weissenberg photographs revealed systematic absences $0kl$ for $k = 2n + 1$, $h0l$ for $l = 2n + 1$, $hk0$ for $h = 2n + 1$, and indicated the centrosymmetric orthorhombic space group *Pbca* (No. 61); cell dimensions derived from least-squares treatment of the setting angles for 18 reflections, Rigaku four-circle diffractometer, Mo $K\alpha$ radiation, graphite monochromator, no correction made for absorption; one independent octant of data out to $2\theta = 55°$ collected using ω–2θ scan technique; three reference reflections (measured periodically) showed no significant variation in their intensities; 2577 reflections considered observed [$F_o \geq 3\sigma(F_o)$]; structure

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solved by heavy-atom method, Br positions deduced from a three-dimensional Patterson synthesis, remaining atoms located in a subsequent Fourier synthesis; structure refined by full-matrix least-squares calculations (against F_o, unit weight) with parameters grouped in two blocks including anisotropic temperature factors for the non-H atoms to yield R = 0·095; positions of H atoms derived from a D synthesis and from geometrical calculations, and included as fixed contributions with $B_{\rm iso}$'s of their parent atoms; maximum ratio of the last shift in any atomic parameter to its e.s.d. was 0·30; final difference synthesis showed no anomalies; atomic scattering factors and anomalous-dispersion correction terms for Br and S atoms taken from *International Tables for X-ray Crystallography* (1974); calculations and drawing of figures carried out on a HITAC M-200H computer at the Computer Center of the University of Tokyo, using programs *UNICS* (1967) and *ORTEP* (Johnson, 1965).

Discussion. Final atomic coordinates are listed in Table 1.*

Table 1. *Positional parameters and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

	x	y	z	$B_{\rm eq}$(Å^2)*
ABCMBMA				
Br(1)	0·5667 (1)	0·04883 (8)	0·1097 (3)	5·18 (6)
Br(2)	0·2099 (1)	0·09766 (9)	0·1050 (2)	5·39 (7)
C(1)	0·4331 (9)	0·1921 (6)	0·0912 (15)	2·55 (38)
C(2)	0·5010 (10)	0·1543 (6)	0·1022 (18)	3·22 (44)
C(3)	0·4796 (9)	0·1026 (6)	0·1063 (21)	3·97 (52)
C(4)	0·3930 (10)	0·0835 (7)	0·1048 (25)	5·57 (66)
C(5)	0·3277 (9)	0·1240 (7)	0·1008 (20)	4·26 (52)
C(6)	0·3455 (9)	0·1744 (6)	0·0959 (17)	3·14 (44)
N(1)	0·5867 (7)	0·1699 (6)	0·1058 (21)	5·78 (64)
C(7)	0·4469 (9)	0·2499 (7)	0·0795 (13)	2·75 (43)
N(2)	0·4429 (7)	0·2669 (5)	−0·0438 (11)	2·07 (27)
C(8)	0·5236 (8)	0·2512 (8)	−0·1105 (15)	3·25 (48)
C(9)	0·4237 (10)	0·3247 (6)	−0·0605 (14)	2·59 (41)
C(10)	0·4888 (11)	0·3600 (7)	0·0069 (20)	4·28 (54)
C(11)	0·4715 (13)	0·4173 (9)	−0·0207 (24)	5·96 (73)
C(12)	0·3799 (14)	0·4350 (8)	0·0057 (23)	5·40 (68)
C(13)	0·3142 (12)	0·3973 (8)	−0·0465 (21)	5·01 (63)
C(14)	0·3319 (10)	0·3394 (7)	−0·0229 (17)	3·48 (49)
OSBI				
S	0·7232 (2)	0·2924 (2)	0·2070 (4)	2·35 (8)
N(3)	0·8258 (7)	0·2964 (5)	0·1785 (12)	2·56 (37)
O(1)	0·7065 (7)	0·2636 (5)	0·3091 (13)	4·56 (36)
O(2)	0·6769 (7)	0·2734 (5)	0·1071 (12)	3·82 (33)
C(15)	0·7871 (12)	0·4405 (7)	0·2079 (17)	3·99 (50)
C(16)	0·7119 (15)	0·4708 (7)	0·2305 (20)	4·75 (60)
C(17)	0·6346 (13)	0·4428 (8)	0·2518 (22)	4·86 (60)
C(18)	0·6283 (10)	0·3879 (7)	0·2478 (19)	3·84 (56)
C(19)	0·7048 (8)	0·3600 (6)	0·2212 (12)	1·99 (33)
C(20)	0·7831 (9)	0·3860 (6)	0·2032 (14)	2·41 (39)
C(21)	0·8546 (8)	0·3465 (6)	0·1820 (14)	2·49 (40)
O(3)	0·9301 (6)	0·3608 (5)	0·1707 (11)	4·18 (37)

*$B_{\rm eq}$ defined according to Hamilton (1959).

Fig. 1 shows the molecular structure with the numbering of the atoms and the bond distances, Table 2 gives the bond angles. Table 3† contains the best planes and deviations of the atoms from them.

So far as we know, this analysis is the first X-ray crystallographic investigation of the ABCMBMA part. In the ABCMBMA moiety, the bond lengths C(2)–C(3) 1·351 (21) and C(5)–C(6) 1·307 (23) Å seem rather short for a normal C$\overset{...}{-}$C bond 1·397 Å, and observed angles Br(1)–C(3)–C(4) 114·6 (11)° and Br(2)–C(5)–C(4) 114·1 (12)° are significantly different from 120°.

N(2) receives the H atom which has been released from N(3) of OSBI.

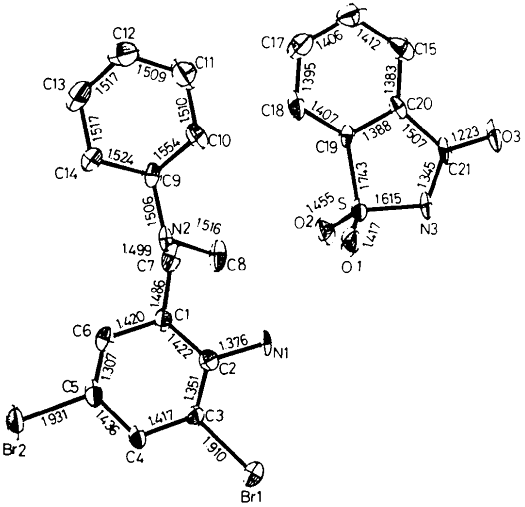

Fig. 1. Molecular structure and bond lengths (Å) for the title compound. E.s.d.'s are 0·011–0·030 Å.

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

ABCMBMA			
C(2)–C(1)–C(6)	118·7 (14)	C(1)–C(6)–C(5)	120·6 (14)
C(2)–C(1)–C(7)	124·5 (13)	C(1)–C(7)–N(2)	111·4 (13)
C(6)–C(1)–C(7)	116·7 (13)	C(7)–N(2)–C(8)	112·4 (11)
C(1)–C(2)–C(3)	118·5 (14)	C(7)–N(2)–C(9)	114·3 (12)
C(1)–C(2)–N(1)	120·8 (14)	C(8)–N(2)–C(9)	110·4 (12)
C(3)–C(2)–N(1)	120·7 (14)	N(2)–C(9)–C(10)	111·6 (13)
Br(1)–C(3)–C(2)	121·4 (11)	N(2)–C(9)–C(14)	112·5 (13)
Br(1)–C(3)–C(4)	114·6 (11)	C(10)–C(9)–C(14)	108·1 (14)
C(2)–C(3)–C(4)	124·0 (14)	C(9)–C(10)–C(11)	109·4 (16)
C(3)–C(4)–C(5)	114·4 (15)	C(10)–C(11)–C(12)	114·0 (18)
Br(2)–C(5)–C(4)	114·1 (12)	C(11)–C(12)–C(13)	110·7 (18)
Br(2)–C(5)–C(6)	122·4 (12)	C(12)–C(13)–C(14)	114·6 (17)
C(4)–C(5)–C(6)	123·6 (14)	C(9)–C(14)–C(13)	110·5 (14)
OSBI			
N(3)–S–O(1)	112·4 (7)	C(17)–C(18)–C(19)	116·8 (15)
N(3)–S–O(2)	109·7 (7)	S–C(19)–C(18)	130·5 (11)
N(3)–S–C(19)	96·7 (7)	S–C(19)–C(20)	108·1 (10)
O(1)–S–O(2)	114·4 (8)	C(18)–C(19)–C(20)	121·3 (14)
O(1)–S–C(19)	113·4 (8)	C(15)–C(20)–C(19)	120·4 (14)
O(2)–S–C(19)	108·9 (7)	C(15)–C(20)–C(21)	129·6 (14)
S–N(3)–C(21)	112·0 (10)	C(19)–C(20)–C(21)	110·0 (13)
C(16)–C(15)–C(20)	120·9 (16)	N(3)–C(21)–C(20)	113·0 (11)
C(15)–C(16)–C(17)	116·8 (17)	N(3)–C(21)–O(3)	126·1 (13)
C(16)–C(17)–C(18)	123·8 (18)	C(20)–C(21)–O(3)	120·9 (14)

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* Lists of structure factors, anisotropic thermal parameters of the non-H atoms, positional parameters of the H atoms, Tables 3 and 4 and Fig. 3 have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38298 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† See previous footnote.

Some selected torsion angles are reported in Table 4.* The cyclohexyl ring of ABCMBMA has the chair conformation. Torsion angles C(2)–C(1)–C(7)–N(2) (ψ_1), C(1)–C(7)–N(2)–C(9) (ψ_2) and C(7)–N(2)–C(9)–C(10) (ψ_3) are 97·7 (17), 158·9 (8) and 54·3 (14)°, respectively. In order to get the information about the freedom of rotation with regard to the torsion angles ψ_1 and ψ_2, conformational analysis was performed.

Since it seems that the two Br atoms have little effect on the torsion angles ψ_1 and ψ_2, the structure in which Br atoms were replaced by H atoms was used for the potential-energy calculations. ψ_3 was kept fixed at the experimental value of 54·3°. Two energy-minimum regions exist in Fig. 2.

The observed conformation (⊕) lies in the left-hand valley. In the OSBI moiety, the bond lengths S–N(3) and N(3)–C(21), and the angle S–N(3)–C(21) are 1·615 (11), 1·345 (19) Å and 112·0 (10)°, respectively. These values are similar to those found in our previous report: 2,4-diamino-5-(3,4,5-trimethoxybenzyl)pyrimidine(DTMBP)–OSBI (1:1) monohydrate [1·618 (5), 1·346 (7) Å and 111·2 (4)°, Shimizu & Nishigaki, 1982], and are appreciably different from 1·663 (2), 1·375 (2) Å and 115·0 (2)° found by Bart (1968) and 1·663 (4), 1·369 (5) Å and 115·1° found by Okaya (1969) for OSBI not forming a molecular complex.

These results demonstrate that the H has been released from N(3).

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* See deposition footnote.

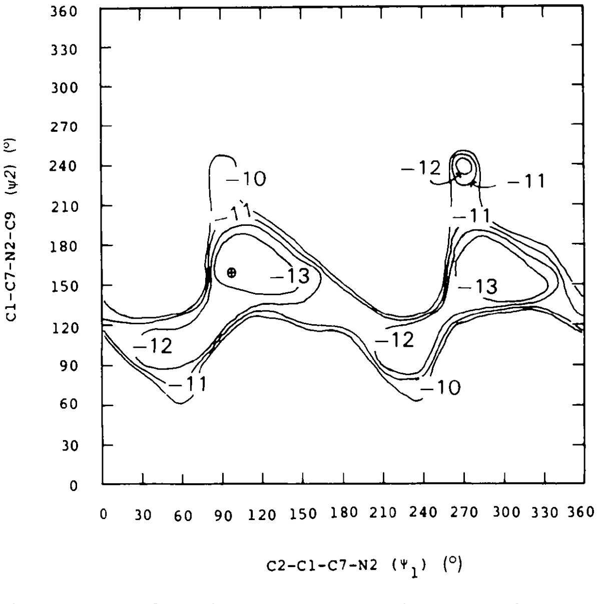


Fig. 2. The conformational-energy map. The contour levels are quantified in nominal units of kcal mol^{-1} (1 kcal mol^{-1} ≡ 4·2 kJ mol^{-1}). The observed conformation is marked by a cross.

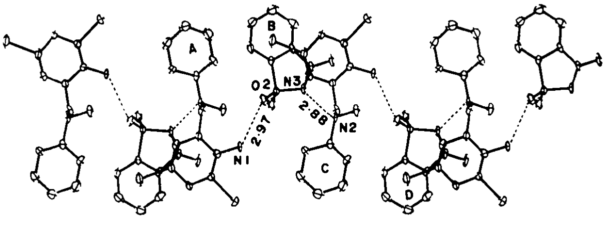
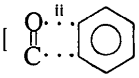


Fig. 4. Hydrogen-bonding diagram. Distances in Å. *A*: ABCMBMA; *B*: OSBI.

Fig. 3* depicts the unit cell of the title compound. Fig. 4 illustrates the hydrogen-bonding scheme.

In the present crystal, ABCMBMA and OSBI are alternately linked to form an endless chain (*–A–B–C–D–*) by two hydrogen bonds [N(1)···O(2) = 2·97 (2), N(2^{i})···N(3) = 2·88 (2) Å; (i) $\frac{1}{2}+x$, $\frac{1}{2}-y$, $-z$]. The dihedral angle between the phenyl ring [C(1)–C(6), *A*] and the OSBI (*B*) plane is 14·2 (1)°.

O(3) of OSBI in our previous structure (Shimizu & Nishigaki, 1982) accepts two hydrogen bonds from DTMBP. In the present crystal, however, it is interesting to note that the O(3) of OSBI does not participate in any hydrogen bonding. It is situated over the benzene ring of the partner molecule ABCMBMA, and a permanent dipole–induced-dipole interaction exists between this carbonyl C(21)=O(3) of OSBI and the benzene ring of ABCMBMA [, (ii) $-\frac{1}{2}+x$, $\frac{1}{2}-y$, $-z$: O(3)···benzene plane, 3·15 (1) Å; C(21)···benzene plane, 3·24 (2) Å]. There are also two weak electrostatic attractions between slightly negatively charged Br atoms and the positively charged H atoms [Br(1)···H(15^{iii})–C(15) = 2·83 Å, (iii) $\frac{3}{2}-x$, $-\frac{1}{2}+y$, z; Br(2)···H(101^{ii})–C(10) = 2·96 Å, (ii) $-\frac{1}{2}+x$, $\frac{1}{2}-y$, $-z$].

We thank Professor Kenji Osaki and Dr Tooru Taga of Kyoto University for their useful suggestions during this project.

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* See deposition footnote.

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