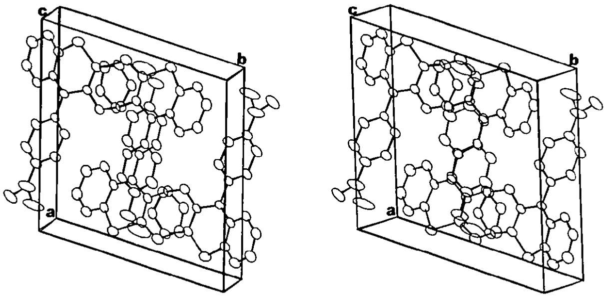

Fig. 3. Stereoscopic drawing of the molecular packing of *A* and *B* excluding hydrogen atoms, in the unit cell.

112·2 (1)° for *A* and *B* respectively and those between the planes of the phenyl ring and benzo ring are 92·4 (1) and 91·2 (1)° for *A* and *B* respectively. In (II), the dihedral angle between the planes of the phenyl ring and pyrido ring is 106·0 (1)° and that between the planes of the phenyl ring and benzo ring is 95·3 (1)°. The torsion angles about the N(10)—C(1′) link between the two ring systems and about the C(4′)—O(4′) of the methoxy group are also shown in Table 2. The torsion angles about the N(10)—C(1′) link between the two ring systems in (II) also have similar values. Therefore, there is no significant difference in the conformation of the two ring systems whether the methoxy substituent is at the C(2′) or C(4′) position.

The packing of the molecules in the unit cell is shown in the stereoscopic drawing in Fig. 3. Except for the close intermolecular contacts involving the methoxy carbon atoms, there are no other contacts less than van der Waals distances.

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Structure of a Polymorph of the 1/1 Addition Compound Between Benzoic Acid and 2,4-Diamino-5-(3,4,5-trimethoxybenzyl)pyrimidinium Benzoate (Trimethoprim Monobenzoate–Benzoic Acid 1/1 Complex, Form II), $C_{14}H_{19}N_4O_3^+.C_7H_5O_2^-.C_7H_6O_2$

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Abstract. $M_r = 534{\cdot}57$, triclinic, $P\bar{1}$, $a = 10{\cdot}192\,(1)$, $b = 14{\cdot}595\,(1)$, $c = 9{\cdot}457\,(1)$ Å, $\alpha = 97{\cdot}41\,(1)$, $\beta = 89{\cdot}69\,(1)$, $\gamma = 104{\cdot}57\,(1)°$, $V = 1349{\cdot}6\,(2)$ Å^3, $Z = 2$, $D_m = 1{\cdot}307\,(6)$, $D_x = 1{\cdot}315$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}89$ cm^{-1}, $F(000) = 564$, $T = 296$ K, $R = 0{\cdot}037$ for 2881 observed reflections. The trimethoprim cation is bonded to the benzoate anion through two N—H···O hydrogen bonds [N···O = 2·673 (2), 2·870 (2) Å], the other benzoic acid molecule being linked through a short O—H···O hydrogen bond [2·531 (2) Å] involving its carboxyl OH and an oxygen atom of the anion. The trimethoprim cation is in a twist conformation, as in the trimethoprim benzoate salt. Cyclic centrosymmetrical N—H···N and linear N—H···O hydrogen bonds are responsible for molecular packing.

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Introduction. Trimethoprim [5-(3,4,5-trimethoxybenzyl)-2,4-pyrimidinediamine, TMP] interacts with some aliphatic (*e.g.* formic, acetic, propanoic) and aromatic (*e.g.* salicylic, *m*-hydroxy- and *p*-hydroxybenzoic) acids *uniquely* in a 1:1 molar ratio. Using benzoic acid (BA), two products of different BA:TMP stoichiometry, 1:1 (BA–TMP) and 2:1 [$(BA)_2$–TMP, form II], can be prepared by recrystallization from aqueous solutions (Bettinetti, Caramella, Giordano, La Manna, Margheritis & Sinistri, 1983).

The structure of BA–TMP has already been published (Giuseppetti, Tadini, Bettinetti, Giordano & La Manna, 1984). A binding pattern similar to that found in BA–TMP was previously described for the acetate salt of TMP (Haltiwanger, 1971). More recently, analogous interactions have been brought to light in the crystal structures of other molecular complexes, *e.g.* *Escherichia coli* dihydrofolate reductase–TMP and chicken-liver dihydrofolate reductase–NADPH–TMP (Baker, Beddel, Champness, Goodford, Norrington, Smith & Stammers, 1981; Matthews & Volz, 1982; Baker, Beddel, Champness, Goodford, Norrington, Roth & Stammers, 1983; Li, Hansch, Matthews, Blaney, Landgridge, Delcamp, Susten & Freisheim, 1982). The ability of TMP to form 1:1 products is confirmed by other crystallographic investigations (Giuseppetti, Tadini, Bettinetti, Giordano & La Manna, 1980; Shimizu & Nishigaki, 1982) and is also shown by some antifolate TMP analogues (Cody & Zakrzewski, 1982; Cody, 1984).

BA, which interacts both in the 1:1 and 2:1 ratios with TMP (the 2:1 product existing in two polymorphic modifications), thus represents an exceptional partner for TMP.

Consequently, we thought it would be interesting to assess the molecular structure of $(BA)_2$–TMP, form II, in order to clarify whether we are dealing with a TMP dibenzoate salt or a TMP monobenzoate–benzoic acid complex. Valuable information for the correct interpretation of the binding mode of BA and TMP could also be achieved.

Experimental. Single crystal obtained by recrystallization from an aqueous solution of BA and TMP 2/1 mol/mol. Density measured with a helium pycnometer model 1302/1303 Micrometric Instrument Corp. Prismatic crystal ($0{\cdot}75 \times 0{\cdot}30 \times 0{\cdot}22$ mm). Philips PW 1100 four-circle diffractometer, graphite monochromator. 40 reflections ($2 < \theta < 25°$) used for measuring lattice parameters with Philips *LAT* routine. ω–2θ scan mode, scan speed $0{\cdot}09°\ s^{-1}$, scan width 1·8°, θ range 2–25° (intensities negligible at $\theta > 25°$). Three standard reflections every 180 min, mean intensity variation 1·5%, max. intensity variation 2·4%. 4759 independent data ($-12 \leq h \leq 12$, $-17 \leq k \leq 17$, $l \leq 11$, max. $\sin\theta/\lambda = 0{\cdot}59\ \text{Å}^{-1}$), 2881 observed with $I > 3\sigma(I)$. Corrections for Lorentz–polarization and experimental absorption, between 1·0002 and 1·0181, following North, Phillips & Mathews (1968). Structure solved by direct methods (*MULTAN*80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). F magnitudes used in least-squares refinement. Coordinates of hydrogen atoms calculated by geometrical considerations (*XANADU*: Roberts & Sheldrick, 1975), confirmed by ΔF synthesis showing random fluctuations between 0·05 and −0·04 e Å^{-3}; parameters refined: coordinates and anisotropic thermal parameters for non-hydrogen atoms, hydrogen atoms with isotropic temperature factors, scale factor and secondary extinction value [final $g = 2{\cdot}2\,(1) \times 10^{-4}$] calculated from the anisotropic coefficients following Coppens & Hamilton (1970). Final value of $R_{obs} = 0{\cdot}0365$, $R_{all} = 0{\cdot}074$, unit weights, $S = 0{\cdot}432$. Max. $\Delta/\sigma = 0{\cdot}009$ in last cycle of refinement. Atomic scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974). A locally modified version of *ORFLS* (Busing, Martin & Levy, 1962) and the program *PARST* (Nardelli, 1983) were used. Fig. 1 was drawn with *ORTEP*II (Johnson, 1976).

Discussion. Final atomic coordinates are given in Table 1.* Interatomic bond distances and angles, and the geometry of the hydrogen bonds and contacts are listed in Tables 2 and 3. As shown in Fig. 1, the title compound contains one BA anion linked to the N(1)-protonated TMP cation through two hydrogen-bond-type interactions, exactly like the BA molecule in BA–TMP (Giuseppetti *et al.*, 1984). Thus the $^{(+)}$N(1)—H(11)···$^{(-)}$O(11,1) interaction is stronger than the N(2)—H2(N2)···O(11,2) interaction (Table 3). Also, the twist-boat conformation of the ring system generated by the linkage between the TMP cation and BA anion (Cremer & Pople, 1975) together with the displacement of atoms with respect to its mean plane, and the ring angles and bonds lengths involving the N(1)-protonated atom, resemble common structural features.

The BA molecule is linked through a short hydrogen bond of 2·531 (2) Å from its carboxyl OH to the O(11,1) atom of the benzoate anion. The phenyl ring of this BA molecule is practically superimposed on the phenyl moiety of TMP (Figs. 1 and 2), the respective planes being almost parallel [the dihedral angle between them is 10·0 (1)°]. The distances between correspond-

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* Lists of structure factors, anisotropic temperature factors for non-hydrogen atoms, hydrogen coordinates and isotropic temperature coefficients, interatomic bond distances and angles involving hydrogen atoms, torsion angles and least-squares planes with deviations of the atoms from the planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42213 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

ing carbon atoms, *e.g.* C(19)···C(6*a*) = 3·814 (2) Å and C(22)···C(3*a*) = 3·770 (3) Å, fall in the range 3·960 (2)–3·572 (3) Å and the distance between the ring centers is 3·776 (3) Å.

This intramolecular arrangement might account for the peculiar behavior of BA with respect to other acids which interact with TMP only in the 1:1 ratio.

Table 1. *Fractional atomic coordinates* (×10⁴) *and equivalent isotropic temperature factors* (Å²), *with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
N(1)	2013 (2)	5149 (1)	3902 (2)	3·81 (5)
C(2)	1039 (2)	5394 (1)	3183 (2)	3·44 (6)
N(3)	689 (1)	5028 (1)	1831 (1)	3·45 (5)
C(4)	1302 (2)	4372 (1)	1200 (2)	3·41 (5)
C(5)	2342 (2)	4085 (1)	1899 (2)	3·35 (5)
C(6)	2660 (2)	4509 (1)	3250 (2)	3·79 (6)
C(7)	3068 (2)	3372 (1)	1215 (2)	3·99 (6)
N(2)	440 (2)	6023 (1)	3868 (2)	4·43 (5)
N(4)	899 (2)	4012 (1)	−141 (2)	4·27 (5)
C(1*a*)	2449 (2)	2337 (1)	1399 (2)	3·39 (5)
C(2*a*)	2971 (2)	1642 (1)	622 (2)	3·88 (6)
C(3*a*)	2534 (2)	704 (1)	858 (2)	3·98 (6)
C(4*a*)	1576 (2)	442 (1)	1890 (2)	3·67 (6)
C(5*a*)	1034 (2)	1134 (1)	2637 (2)	3·78 (6)
C(6*a*)	1455 (2)	2080 (1)	2387 (2)	3·71 (6)
O(3*a*)	2986 (2)	−35 (1)	152 (2)	5·79 (5)
O(4*a*)	1232 (2)	−483 (1)	2202 (2)	4·55 (4)
O(5*a*)	79 (2)	805 (1)	3606 (2)	5·14 (5)
C(8)	−459 (3)	1490 (2)	4473 (3)	5·46 (8)
C(9)	61 (3)	−1069 (2)	1416 (3)	5·73 (8)
C(10)	3683 (4)	121 (2)	−1123 (3)	7·60 (11)
O(11,1)	2666 (2)	5582 (1)	6684 (1)	4·81 (5)
O(11,2)	1454 (2)	6646 (1)	6749 (1)	4·88 (5)
C(11)	2212 (2)	6254 (1)	7328 (2)	3·71 (6)
C(12)	2671 (2)	6602 (1)	8863 (2)	3·30 (5)
C(13)	3392 (2)	6123 (1)	9607 (2)	3·56 (5)
C(14)	3818 (2)	6468 (2)	11010 (2)	4·25 (6)
C(15)	3526 (2)	7282 (2)	11663 (2)	4·74 (7)
C(16)	2815 (3)	7764 (2)	10939 (2)	5·23 (7)
C(17)	2377 (2)	7424 (2)	9540 (2)	4·51 (7)
O(18,1)	3899 (2)	4269 (1)	6462 (2)	5·07 (5)
O(18,2)	2044 (2)	3268 (1)	7155 (2)	5·85 (5)
C(18)	3122 (2)	3404 (2)	6544 (2)	4·12 (6)
C(19)	3649 (2)	2621 (1)	5797 (2)	3·71 (6)
C(20)	4742 (2)	2792 (2)	4914 (2)	4·78 (7)
C(21)	5154 (3)	2034 (2)	4191 (3)	6·19 (9)
C(22)	4504 (3)	1117 (2)	4344 (3)	6·12 (9)
C(23)	3418 (3)	939 (2)	5220 (3)	5·87 (8)
C(24)	2993 (2)	1689 (2)	5943 (2)	4·97 (7)

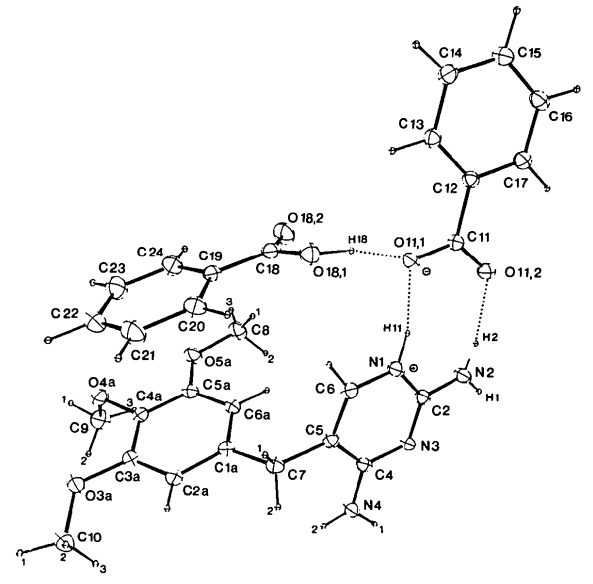

Fig. 1. Molecular conformation of the addition compound, form II: the H to O interactions are shown by dotted lines.

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

N(1)–C(2)	1·350 (3)	O(4*a*)–C(9)	1·429 (3)
N(1)–C(6)	1·358 (3)	O(5*a*)–C(8)	1·426 (3)
C(2)–N(3)	1·334 (2)	O(11,1)–C(11)	1·272 (2)
C(2)–N(2)	1·327 (3)	O(11,2)–C(11)	1·238 (3)
N(3)–C(4)	1·347 (2)	C(11)–C(12)	1·511 (2)
C(4)–C(5)	1·427 (3)	C(12)–C(13)	1·385 (3)
C(4)–N(4)	1·333 (2)	C(12)–C(17)	1·385 (3)
C(5)–C(6)	1·349 (2)	C(13)–C(14)	1·388 (3)
C(5)–C(7)	1·503 (3)	C(14)–C(15)	1·367 (3)
C(7)–C(1*a*)	1·515 (3)	C(15)–C(16)	1·369 (4)
C(1*a*)–C(2*a*)	1·388 (3)	C(16)–C(17)	1·386 (4)
C(1*a*)–C(6*a*)	1·389 (3)	O(18,1)–C(18)	1·322 (2)
C(2*a*)–C(3*a*)	1·376 (3)	O(18,2)–C(18)	1·222 (3)
C(3*a*)–C(4*a*)	1·396 (3)	C(18)–C(19)	1·480 (3)
C(3*a*)–O(3*a*)	1·372 (3)	C(19)–C(20)	1·380 (3)
C(4*a*)–C(5*a*)	1·385 (3)	C(19)–C(24)	1·380 (3)
C(4*a*)–O(4*a*)	1·377 (2)	C(20)–C(21)	1·380 (4)
C(5*a*)–C(6*a*)	1·389 (3)	C(21)–C(22)	1·361 (4)
C(5*a*)–O(5*a*)	1·374 (2)	C(22)–C(23)	1·371 (4)
O(3*a*)–C(10)	1·412 (4)	C(23)–C(24)	1·377 (4)
C(2)–N(1)–C(6)	119·9 (2)	C(1*a*)–C(6*a*)–C(5*a*)	119·6 (2)
N(1)–C(2)–N(2)	117·5 (2)	C(3*a*)–O(3*a*)–C(10)	117·7 (2)
N(1)–C(2)–N(3)	121·9 (2)	C(4*a*)–O(4*a*)–C(9)	113·2 (2)
N(3)–C(2)–N(2)	120·6 (2)	C(5*a*)–O(5*a*)–C(8)	117·6 (2)
C(2)–N(3)–C(4)	118·1 (2)	O(11,1)–C(11)–O(11,2)	123·3 (2)
N(3)–C(4)–N(4)	116·0 (2)	O(11,2)–C(11)–C(12)	119·2 (2)
N(3)–C(4)–C(5)	122·7 (2)	O(11,1)–C(11)–C(12)	117·4 (2)
C(5)–C(4)–N(4)	121·3 (2)	C(11)–C(12)–C(17)	119·4 (2)
C(4)–C(5)–C(7)	124·1 (2)	C(11)–C(12)–C(13)	121·4 (2)
C(4)–C(5)–C(6)	115·3 (2)	C(13)–C(12)–C(17)	119·2 (2)
C(6)–C(5)–C(7)	120·6 (2)	C(12)–C(13)–C(14)	120·1 (2)
N(1)–C(6)–C(5)	122·0 (2)	C(13)–C(14)–C(15)	120·2 (2)
C(5)–C(7)–C(1*a*)	116·3 (2)	C(14)–C(15)–C(16)	120·3 (2)
C(7)–C(1*a*)–C(6*a*)	121·8 (2)	C(15)–C(16)–C(17)	120·1 (2)
C(7)–C(1*a*)–C(2*a*)	118·2 (2)	C(12)–C(17)–C(16)	120·2 (2)
C(2*a*)–C(1*a*)–C(6*a*)	119·9 (2)	O(18,1)–C(18)–O(18,2)	122·6 (2)
C(1*a*)–C(2*a*)–C(3*a*)	120·2 (2)	O(18,1)–C(18)–C(19)	114·3 (2)
C(2*a*)–C(3*a*)–O(3*a*)	124·7 (2)	O(18,2)–C(18)–C(19)	123·1 (2)
C(2*a*)–C(3*a*)–C(4*a*)	120·5 (2)	C(18)–C(19)–C(24)	118·9 (2)
C(4*a*)–C(3*a*)–O(3*a*)	114·7 (2)	C(18)–C(19)–C(20)	122·0 (2)
C(3*a*)–C(4*a*)–O(4*a*)	120·0 (2)	C(20)–C(19)–C(24)	119·0 (2)
C(3*a*)–C(4*a*)–C(5*a*)	119·0 (2)	C(19)–C(20)–C(21)	119·7 (2)
C(5*a*)–C(4*a*)–O(4*a*)	120·9 (2)	C(20)–C(21)–C(22)	120·9 (2)
C(4*a*)–C(5*a*)–O(5*a*)	114·8 (2)	C(21)–C(22)–C(23)	119·8 (3)
C(4*a*)–C(5*a*)–C(6*a*)	120·7 (2)	C(22)–C(23)–C(24)	119·8 (2)
C(6*a*)–C(5*a*)–O(5*a*)	124·5 (2)	C(19)–C(24)–C(23)	120·7 (2)

Table 3. *Hydrogen-bonding and intermolecular-contact geometry*

Donor *R*H	Acceptor *R'*	*R*–H (Å)	*R*···*R'* (Å)	H···*R'* (Å)	*R*–H···*R'* (°)
N(1)···	O(11,1)	0·95 (2)	2·673 (2)	1·74 (2)	167 (2)
N(2)···	O(11,2)	0·92 (2)	2·870 (2)	1·97 (2)	168 (2)
O(18,1)···	O(11,1)	1·02 (3)	2·531 (2)	1·53 (3)	165 (2)
C(8)···	O(11,2)	1·05 (3)	3·460 (3)	2·51 (3)	151 (2)
N(2)···	O(18,2)	0·91 (2)	3·165 (3)	2·26 (2)	169 (2)
N(4)···	N(3)	0·97 (2)	2·980 (3)	2·02 (2)	168 (2)
N(4)···	O(18,2)	0·87 (2)	2·995 (2)	2·29 (2)	138 (2)
C(16)···	O(4*a*)	0·98 (2)	3·441 (3)	2·50 (2)	161 (2)

See Fig. 2 for the symmetry code.

It is hence evident that, as far as the TMP cation is concerned, its main binding ability remains localized along the N(1)—C(2)—N(2) aminopyrimidine moiety, as in BA–TMP and in other TMP compounds. In fact the protonation does not occur at the N(3) atom, as expected from its weak basic properties. Moreover, the N(3)—C(4)—N(4) portions are not involved in intramolecular hydrogen bonds; instead these portions are mutually engaged in intermolecular cyclic centrosymmetrically related hydrogen bonds, as shown in Fig. 2.

Each N(4) atom acts as a donor with its H(1) hydrogen and is also involved in hydrogen bonds with the O(18,2) oxygen atom of a second molecule *via* the H(2) hydrogen. The same O(18,2) atom acts as an acceptor in the hydrogen bond with the N(2)—H1(N2) group. Hence all the amino hydrogen atoms participate in the hydrogen bonding, as in BA–TMP. The short C(2)—N(2) and C(4)—N(4) amino bonds in both molecular compounds are consistent with their iminium character and proton-donor capability.

In Fig. 2, intermolecular C(sp^2)—H···O and C(sp^3)—H···O contacts, whose possible contribution to molecular packing in some crystal structures was recently emphasized (Berkovitch-Yellin & Leiserowitz, 1984), can also be seen. The intermolecular contacts and hydrogen bonds are listed in Table 3.

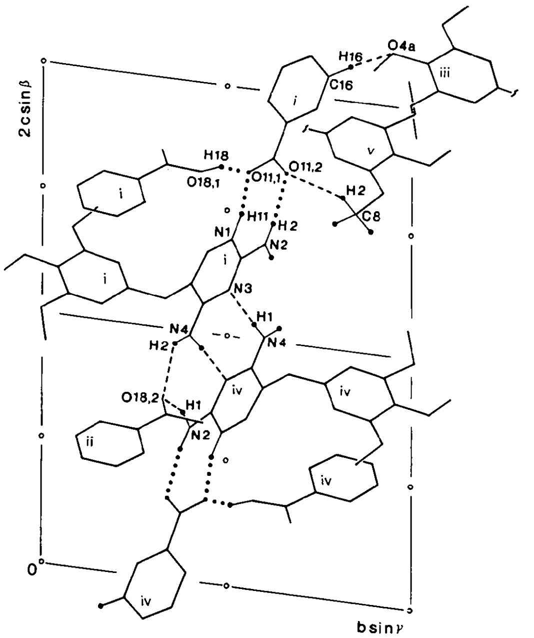

Fig. 2. View along **a** showing packing of molecules. Symmetry code: (i) x, y, z; (ii) $x, y, z-1$; (iii) $x, y+1, z+1$; (iv) $\bar{x}, \bar{y}+1, \bar{z}$; (v) $\bar{x}, \bar{y}+1, \bar{z}+1$.

The conformation adopted by TMP in this addition compound, in which the trimethoxyphenyl and pyrimidine rings make an angle of 101·2 (1)°, is described by τ_1 [C(4)—C(5)—C(7)—C(1*a*)] = −90·5 (2)° and τ_2 [C(5)—C(7)—C(1*a*)—C(2*a*)] = 171·0 (2)° torsion angles. These data resemble once more the situation found in BA–TMP where $\tau_1 = -68{\cdot}4\ (3)$, $\tau_2 = 149{\cdot}1\ (2)°$, and the angle between trimethoxyphenyl and pyrimidine rings = 96·0 (1)°. These results therefore demonstrate that the BA molecule bonded to TMP benzoate does not determine a substantial modification of the twist conformation of TMP, most frequently observed in other TMP structures (Oberhänsli, 1970; Haltiwanger, 1971; Koetzle & Williams, 1976; Shimizu & Nishigaki, 1982; Giuseppetti *et al.*, 1984), in TMP bound to avian dihydrofolate reductase (Matthews & Volz, 1982), and in an active analogue of TMP (Cody, 1984).

In conclusion, it may also be underlined that the diamino(trimethoxybenzyl)pyrimidine frame maintains the geometric and conformational features already found in TMP benzoate. In fact the addition compound decomposes by heating under vacuum producing BA (as sublimate) and BA–TMP (as residue).

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4-Phenyl-6,7,8,9-tetrahydro-1*H*-2,3-benzodiazepine 2-Oxide, $C_{15}H_{16}N_2O$

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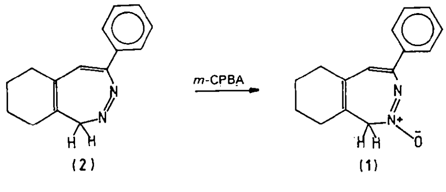
Abstract. $M_r = 240\cdot3$, monoclinic, $P2_1/c$, $a = 8\cdot062\,(2)$, $b = 13\cdot900\,(2)$, $c = 12\cdot007\,(2)$ Å, $\beta = 108\cdot180\,(17)°$, $V = 1278\cdot4$ Å^3, $Z = 4$, $D_x = 1\cdot248$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0\cdot71069$ Å, $\mu = 0\cdot74$ cm^{-1}, $F(000) = 512$, $T = 293$ K. Final $R = 0\cdot033$ from 1194 observed reflections. All N—C and C—C single and double bonds are close to expected values, though there is evidence of some delocalization round the seven-membered ring. The N=N and N—O bond lengths are 1·279 (2) and 1·268 (2) Å respectively. The boat conformation adopted by most 1,2-diazepine rings is unperturbed by the oxide ligand which serves to weaken the N=N double bond and lowers the activation energy for ring inversion.

Introduction. The title compound (1) was prepared by oxidation of 4-phenyl-6,7,8,9-tetrahydro-1*H*-2,3-benzodiazepine (2) using *m*-chloroperbenzoic acid (*m*-CPBA) (Argo, Robertson & Sharp, 1984). The barrier to ring inversion in the diazepine ring of the oxide (1) is substantially reduced by about 15 kJ mol^{-1} compared with the parent diazepine (2) as determined by variable-temperature ^{1}H NMR studies. A crystal-structure analysis was carried out to determine unambiguously the position of oxidation and also to see if there were any structural reasons for the lowering of activation energy for ring inversion.



Experimental. Crystal dimensions 0·3 × 0·25 × 0·25 mm; D_m not measured. Nonius CAD-4 diffractometer, graphite monochromator. 20 reflections used to measure lattice parameters. No correction for absorption. Intensities of 1777 unique reflections measured out to $\theta = 23°$, h −8 to 8, k 0 to 15, l 0 to 13, 1194 reflections with $I > 2\cdot5\sigma(I)$ used in refinement. Standard reflections $\bar{2}54$ and $\bar{2}2\bar{2}$ showed no measurable variation. Structure solved by direct methods (*SHELX*76, Sheldrick, 1976) and refined by full-matrix least squares on F. All hydrogen atoms located and positional and isotropic thermal parameters refined. Non-hydrogen atoms refined anisotropically. Weighting scheme $w = 1/[\sigma^2(F) + 0\cdot00035F^2]$ which gave final $R = 0\cdot033$, $wR = 0\cdot038$. Maximum ratio of least-squares shift to error in final refinement cycle 0·04. Maximum and minimum peak heights in final difference map 0·1 and −0·22 e Å^{-3}. No correction for secondary extinction. Atomic scattering factors from *SHELX*76.

Discussion. Table 1 contains positional and mean isotropic thermal parameters for all atoms.[*] Bond lengths, angles and torsion angles are given in Table 2. A drawing of the molecule is given in Fig. 1. A literature search using the Cambridge Structural Database (1984) revealed 20 crystal structures containing a diazepine seven-membered ring with adjacent nitrogen atoms. Unlike the widely studied 1,4-diazepines (Crippen, 1982), no pharmacological activity has been reported for the 1,2 ring system.

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[*] Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42233 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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