The central five-membered ring is planar, the maximum distance from the best plane being 0·017 (9) Å. The two three-membered rings are *cis* to each other, while the angles formed by them with the central plane differ considerably [107·3 (6) and 111·9 (6)° for N1, C2, C7 and N4, C3, C5 respectively]. In addition the stereochemistry of two N centres is different. The N1—C8 and N4—C16 bonds are inclined at 48·8 (6) and 144·1 (6)° to the planes of the three-membered rings, giving evidence for the *exo,endo* modification of the molecule. Thus, in the crystal, the molecule is not symmetrical. The molecules are linked by van der Waals forces only.

This work was supported by project RP.II.10. from the Polish Ministry of Science and Higher Education.

References

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MAJCHRZAK, M. W., KOTEŁKO, A. & LAMBERT, J. B. (1983). *Org. Magn. Reson.* **21**, 706–710.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1988). C**44**, 334–337

Structure of Tris(2-hydroxyethyl)ammonium 3,5-Dinitrobenzoate

BY GUIDO P. M. VAN MIER AND JAN A. KANTERS

Laboratorium voor Kristal- en Structuurchemie, Rijksuniversiteit Utrecht, Transitorium 3, *Padualaan* 8, 3584 *CH Utrecht, The Netherlands*

AND NARINDER S. POONIA

Research and Industrial Training Institute, Vishnupuri, Indore 452001, *India*

(*Received* 14 *July* 1987; *accepted* 29 *October* 1987)

Abstract. $C_6H_{16}NO_3^+.C_7H_3N_2O_6^-$, $M_r = 361{\cdot}31$, monoclinic, $P2_1/n$, $a = 12{\cdot}929$ (3), $b = 7{\cdot}160$ (2), $c = 18{\cdot}131$ (4) Å, $\beta = 105{\cdot}55$ (2)°, $V = 1617{\cdot}0$ (7) Å^3, $Z = 4$, $D_x = 1{\cdot}484$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54184$ Å, $\mu = 9{\cdot}87$ cm^{-1}, $F(000) = 760$, $T = 298$ K. $R = 0{\cdot}0480$ for 2621 unique observed reflections. In the acid–base complex the H atom of 3,5-dinitrobenzoic acid (DNB) is transferred to the N atom of tris(2-hydroxyethyl)amine (TEA). In the TEA residue, the conformation about the three C—C bonds is *gauche*, causing encapsulation of the transferred H atom. This H atom forms a weak trifurcated *intra*molecular H bond with the terminal O atoms. *Inter*molecular O—H···O hydrogen bonds link screw-axis-related TEA residues into an infinite chain along **b**. The two remaining TEA O—H donors are hydrogen bonded to the O atoms of the carboxyl groups of two DNB residues that are situated on opposite sides of the TEA chain.

Introduction. Several acid–base complexes between phenols and tertiary amines have been investigated in solution and in the solid state by infrared, nuclear quadrupole resonance methods and dipole measurements with the aim of studying the proton transfer of the system $\mathrm{O{-}H\cdots N \longleftrightarrow O^-\cdots H{-}N^+}$ (Ratajczak & Sobczyk, 1969; Grech, Kalenik, Malarski & Sobczyk, 1983; Zundel & Fritsch, 1984; Huyskens & Zeegers-Huyskens, 1964). According to the Huyskens & Zeegers-Huyskens (1964) model the proton transfer equilibrium constant K_{PT} can be related to the ΔpK_a value *via*

$$\log K_{\mathrm{PT}} = c'\Delta pK_a + c''$$

where ΔpK_a is the difference of the acid dissociation constants of the base and the acid, and c' and c'' are constants depending on the chosen series of complexes and the environment respectively. Only a few structure determinations have been performed on these types of complexes consisting of a proton donor and a proton acceptor. Clearly, there are two types of such complexes, depending on whether the H bond is *inter-* or *intra*molecular. In the complex pentachlorophenol–aniline (van Bellingen, Germain, Piret & Van Meerssche, 1971) the acidic proton is transferred to the base with the formation of an *inter*molecular $\mathrm{N^+{-}N\cdots O^-H}$ bond. In the complexes of triethanolamine (TEA) with 2-(methylphenoxy)acetic acid (Starova, Frank-Kamenetskaya, Fundamenskii, Semenova & Voronkov, 1981) and 4-chlorophenylthioacetic acid (Shklover, Gridunova, Struchkov, Voronkov, Kryukova & Mirskova, 1983) the $\mathrm{N{-}H^+}$ group is involved in weak *intra*molecular H-bond interactions with the O atoms of

0108-2701/88/020334-04$03.00

the hydroxyl groups of TEA. The same holds for the recently reported structure of the complex 1,8-bis(dimethylamine)naphthalene-2,4-dinitroimidazolate (DMAN.DNI) (Głowiak, Malarski, Sobczyk & Grech, 1987), where the N—H proton is transferred from DNI to DMAN and the H bond is *intra*molecular between the N atoms of DMAN.

We now report the X-ray analysis of the complex of 3,5-dinitrobenzoic acid and tris(2-hydroxyethyl)amine (DNB.TEA), in which, based on the difference of the pK_a values [DNB: $pK_a = 3{\cdot}1$ (Gupta & Soni, 1965); TEA: $pK_a = 7{\cdot}8$ (Perrin, 1965)], a transfer of the acidic proton may be expected. Our primary interest is to examine whether the hydrogen bond is *intra*molecular between the N and the acceptor O atoms of the TEA residue, as in the TEA complexes mentioned above.

Experimental. A colourless crystal (dimensions $1{\cdot}17 \times 0{\cdot}39 \times 0{\cdot}22$ mm) was used for data collection on an Enraf–Nonius CAD-4F diffractometer with Ni-filtered Cu $K\alpha$ radiation. Cell constants were derived from the angular settings of 24 reflections with $26{\cdot}8 < \theta < 29{\cdot}6°$. The intensity data of 3545 unique reflections were collected within one quarter of the reflection sphere: $-15 \leq h \leq 15$, $-8 \leq k \leq 0$ and $0 \leq l \leq 22$; $2\theta_{\max} = 140°$.

ω–2θ scan mode with $\Delta\omega = (0{\cdot}60 + 0{\cdot}15\tan\theta)°$. Three reference reflections (200, 020, 004) were measured every hour and showed a variation of less than 1·4%. Lorentz and polarization corrections were applied, resulting in 2621 observed reflections with $I > 2{\cdot}5\sigma(I)$.

The structure was solved by direct methods (*SHELX*84; Sheldrick, 1984) and subsequent Fourier analysis. All H atoms were located on difference maps. In the final cycles of blocked full-matrix least-squares refinement on F, 302 parameters were varied, including overall scale factor, positional and anisotropic thermal parameters for the non-H atoms, and positional and isotropic thermal parameters for H atoms. The final R value was 0·0480, $wR = 0{\cdot}0711$, $w = 3{\cdot}737/[\sigma^2(F_o) + 0{\cdot}000257(F_o)^2]$, $S = 2{\cdot}95$, $\langle\Delta/\sigma\rangle = 0{\cdot}04$, $(\Delta/\sigma)_{\max} = 0{\cdot}08$. Max. and min. residual density in the final difference Fourier map 0·43 and $-0{\cdot}12$ e Å^{-3} respectively. Three strong reflections (021, 301 and 004) which apparently suffered from extinction were given zero weights.

The scattering factors for non-H atoms were taken from Cromer & Mann (1968), those for H atoms from Stewart, Davidson & Simpson (1965), anomalous-dispersion factors from Cromer & Liberman (1970). Calculations were performed with *SHELX*76 (refinement) (Sheldrick, 1976) and the *EUCLID* package (geometric calculations and illustrations) (Spek, 1982).

Discussion. The final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2), *with e.s.d.'s in parentheses*

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

	x	y	z	$U_{\rm eq}$(Å^2)
3,5-Dinitrobenzoate				
O(1)	0·6121 (1)	−0·2039 (3)	0·4380 (1)	0·061 (1)
O(2)	0·5104 (1)	−0·1163 (3)	0·3242 (1)	0·064 (1)
O(3)	0·6808 (1)	−0·1222 (4)	0·1115 (1)	0·089 (1)
O(4)	0·8492 (2)	−0·1539 (4)	0·1334 (1)	0·095 (1)
O(5)	1·0059 (1)	−0·2027 (3)	0·4800 (1)	0·077 (1)
O(6)	1·0687 (1)	−0·0546 (3)	0·3979 (1)	0·068 (1)
N(1)	0·7690 (1)	−0·1349 (3)	0·1547 (1)	0·052 (1)
N(2)	0·9957 (1)	−0·1280 (3)	0·4181 (1)	0·053 (1)
C(1)	0·6977 (1)	−0·1423 (3)	0·3409 (1)	0·040 (1)
C(2)	0·6881 (2)	−0·1394 (3)	0·2627 (1)	0·041 (1)
C(3)	0·7802 (2)	−0·1325 (3)	0·2375 (1)	0·041 (1)
C(4)	0·8824 (2)	−0·1263 (3)	0·2864 (1)	0·043 (1)
C(5)	0·8888 (1)	−0·1294 (3)	0·3637 (1)	0·042 (1)
C(6)	0·7991 (2)	−0·1393 (3)	0·3918 (1)	0·043 (1)
C(7)	0·5984 (1)	−0·1547 (3)	0·3710 (1)	0·045 (1)
Tris(2-hydroxyethyl)ammonium				
O(7)	0·4316 (1)	0·0501 (2)	0·1865 (1)	0·053 (1)
O(8)	0·1695 (1)	0·3772 (2)	0·1316 (1)	0·054 (1)
O(9)	0·1787 (2)	0·0101 (3)	0·2454 (1)	0·074 (1)
N(3)	0·2165 (1)	0·0046 (2)	0·1031 (1)	0·037 (1)
C(8)	0·3080 (2)	−0·0353 (4)	0·0691 (1)	0·057 (1)
C(9)	0·4073 (2)	−0·0923 (3)	0·1299 (1)	0·057 (1)
C(10)	0·1301 (1)	0·1160 (3)	0·0471 (1)	0·046 (1)
C(11)	0·1550 (2)	0·3195 (3)	0·0549 (1)	0·052 (1)
C(12)	0·1718 (2)	−0·1634 (3)	0·1309 (1)	0·062 (1)
C(13)	0·1155 (2)	−0·1099 (4)	0·1906 (2)	0·074 (1)

Bond distances, angles and selected torsion angles are listed in Table 2.* A perspective view of the complex with atomic numbering is shown in Fig. 1.

As was expected from the difference of the pK_a values of the constituents, the complex consists of a DNB anion and a TEA cation. The conformation of the cation is analogous to that of the neutral amine (Voegele, Fischer & Weiss, 1974*a*,*b*; Voegel, Thierry & Weiss, 1974) with a tris-*gauche* conformation about the C—C bonds (Table 2). This *endo* conformation causes encapsulation of the H atom on the central N atom within the 'lantern' with approximate C_3 symmetry (Fig. 1). The torsion angles about the C—C bonds are −55·5 (3), −55·0 (2) and −47·5 (8)°, which are within the range reported for several free tris[2-hydroxyethyl]amines (Voegele, Fischer & Weiss, 1974*a*,*b*; Voegel, Thierry & Weiss, 1974) and the protonated forms (Starova *et al.*, 1981; Shklover *et al.*, 1983).

As a consequence of the *endo* conformation the N···O contacts are short (Table 3) and well within the limits that are normal for N···O hydrogen bonds. However, in view of the unfavourable N—H···O angles, these contacts may not be considered as normal

---

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and the complete internal geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44497 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

hydrogen bonds, but at the most as a weak trifurcated H-bond system. Like in the TEA complexes mentioned in *Introduction*, there is no *inter*molecular H bond between N—H$^+$ and O$^-$, which are strong donors and acceptors respectively. Apparently, the encapsulation of the N—H$^+$ group by the polar hydroxyl groups, resulting from the *endo* conformation, prevents the formation of such a bond. However, disruption of the tris-*gauche* conformation, which would enable the formation of an *inter*molecular H bond, implies overruling of the so-called *gauche* effect (Wolfe, 1972; Whangbo & Wolfe, 1976; Razenberg, Kanters, Schoone & Duisenberg, 1983), which stabilizes conformations having the maximum number of *gauche* interactions between adjacent polar groups.

Table 2. *Interatomic distances* (Å), *bond angles* (°) *and selected torsion angles* (°)

3,5-Dinitrobenzoate

C(1)—C(2)	1·390 (3)	C(7)—O(2)	1·254 (2)
C(2)—C(3)	1·386 (4)	C(3)—N(1)	1·469 (3)
C(3)—C(4)	1·381 (3)	N(1)—O(3)	1·201 (2)
C(4)—C(5)	1·382 (3)	N(1)—O(4)	1·207 (3)
C(5)—C(6)	1·388 (3)	C(5)—N(2)	1·467 (2)
C(6)—C(1)	1·386 (3)	N(2)—O(5)	1·218 (3)
C(1)—C(7)	1·526 (2)	N(2)—O(6)	1·219 (2)
C(7)—O(1)	1·231 (3)		

C(1)—C(2)—C(3)	119·2 (2)	C(2)—C(3)—N(1)	118·6 (2)
C(2)—C(3)—C(4)	123·3 (2)	C(4)—C(3)—N(1)	118·2 (2)
C(3)—C(4)—C(5)	116·0 (2)	C(3)—N(1)—O(3)	118·8 (2)
C(4)—C(5)—C(6)	123·0 (2)	C(3)—N(1)—O(4)	118·1 (2)
C(5)—C(6)—C(1)	119·4 (2)	O(3)—N(1)—O(4)	123·1 (2)
C(6)—C(1)—C(2)	119·3 (2)	C(4)—C(5)—N(2)	118·1 (2)
C(2)—C(1)—C(7)	120·8 (2)	C(6)—C(5)—N(2)	118·9 (2)
C(6)—C(1)—C(7)	119·9 (2)	C(5)—N(2)—O(5)	117·8 (2)
C(1)—C(7)—O(1)	117·2 (1)	C(5)—N(2)—O(6)	117·8 (2)
C(1)—C(7)—O(2)	116·3 (2)	O(5)—N(2)—O(6)	124·4 (2)
O(1)—C(7)—O(2)	126·5 (2)		

O(1)—C(7)—C(1)—C(6)	−16·1 (3)	O(4)—N(1)—C(3)—C(2)	−170·3 (2)
O(2)—C(7)—C(1)—C(6)	165·0 (2)	O(5)—N(2)—C(5)—C(6)	26·9 (3)
O(3)—N(1)—C(3)—C(2)	8·0 (3)	O(6)—N(2)—C(5)—C(6)	−154·2 (2)

Tris(2-hydroxyethyl)ammonium

N(3)—C(8)	1·501 (3)	C(11)—O(8)	1·414 (3)
C(8)—C(9)	1·507 (3)	N(3)—C(12)	1·480 (3)
C(9)—O(7)	1·421 (3)	C(12)—C(13)	1·507 (4)
N(3)—C(10)	1·519 (2)	C(13)—O(9)	1·399 (4)
C(10)—C(11)	1·491 (3)		

C(8)—N(3)—C(10)	110·2 (1)	N(3)—C(10)—C(11)	110·4 (1)
C(8)—N(3)—C(12)	114·1 (2)	C(10)—C(11)—O(8)	110·3 (2)
C(10)—N(3)—C(12)	111·7 (1)	N(3)—C(12)—C(13)	110·2 (2)
N(3)—C(8)—C(9)	111·2 (2)	C(12)—C(13)—O(9)	111·3 (2)
C(8)—C(9)—O(7)	108·3 (2)		

C(10)—N(3)—C(8)—C(9)	162·8 (2)	C(10)—N(3)—C(12)—C(13)	−77·3 (2)
C(12)—N(3)—C(8)—C(9)	−70·6 (2)	N(3)—C(8)—C(9)—O(7)	−55·5 (3)
C(8)—N(3)—C(10)—C(11)	−85·5 (2)	N(3)—C(10)—C(11)—O(8)	−55·0 (2)
C(12)—N(3)—C(10)—C(11)	146·6 (2)	N(3)—C(12)—C(13)—O(9)	−47·5 (3)
C(8)—N(3)—C(12)—C(13)	156·9 (2)		

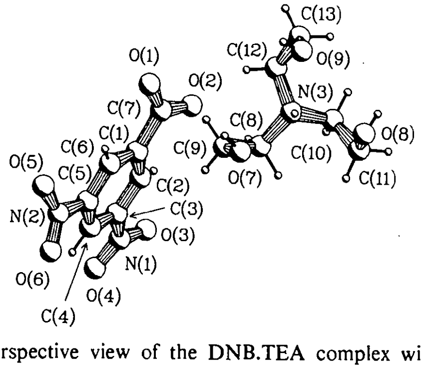

Fig. 1. Perspective view of the DNB.TEA complex with atomic numbering.

Table 3. *Hydrogen-bonding geometry* (*D: donor; A: acceptor*)

D—H···*A*	*D*—H (Å)	H···*A* (Å)	*D*···*A* (Å)	*D*—H···*A* (°)
N(3)—H(3)···O(7)	0·88 (2)	2·37 (3)	2·806 (2)	111 (2)
N(3)—H(3)···O(8)	0·88 (2)	2·36 (2)	2·815 (2)	113 (2)
N(3)—H(3)···O(9)	0·88 (2)	2·25 (2)	2·752 (3)	116 (2)
O(7)—H(7)···O(2)	0·96 (2)	1·75 (2)	2·707 (3)	178 (2)
O(8)—H(8)···O(2^{ii})	0·78 (4)	1·88 (4)	2·656 (2)	171 (3)
O(9)—H(9)···O(8^{i})	0·84 (4)	1·89 (4)	2·717 (3)	166 (4)

Symmetry code: (i) 0·5−*x*, −0·5+*y*, 0·5−*z*; (ii) 0·5−*x*, 0·5+*y*, 0·5−*z*.

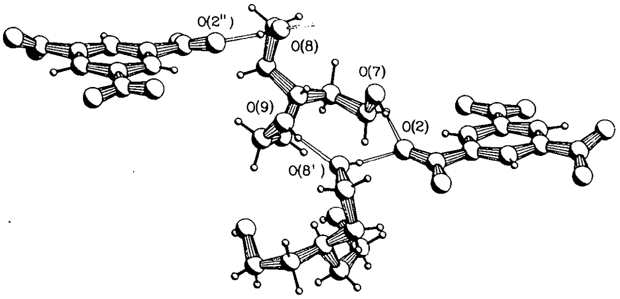

Fig. 2. A view of the intermolecular hydrogen-bonding scheme of the DNB.TEA complex as seen approximately along the *c* axis: (i) denotes symmetry code 0·5−*x*, −0·5+*y*, 0·5−*z*, and (ii) 0·5−*x*, 0·5+*y*, 0·5−*z*.

The carboxyl-group O(2) atoms of two DNB residues, related by the screw axis, accept an H bond from O(7) and O(8) of TEA, whereas O(9) of TEA donates an H bond to O(8) of TEA at (0·5−*x*, −0·5+*y*, 0·5−*z*). This last H bond generates an infinite chain of TEA residues along the **b** direction. This chain is flanked on both sides by DNB residues each of which is doubly H bonded to two different TEA residues (Fig. 2).

With respect to the phenyl ring, which is planar within 0·002 Å, the carboxyl group and the nitro groups N(1)O(3)O(4) and N(2)O(5)O(6) are rotated 8·6 (1), 27·6 (1) and 16·8 (1)° respectively.

References

Bellingen, I. van, Germain, G., Piret, P. & Van Meerssche, M. (1971). *Acta Cryst.* B**27**, 560–564.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Głowiak, T., Malarski, Z., Sobczyk, L. & Grech, E. (1987). *J. Mol. Struct.* **157**, 329–337.

Grech, E., Kalenik, J., Malarski, Z. & Sobczyk, L. (1983). *J. Chem. Soc. Faraday Trans.* 1, **79**, 2005–2012.

GUPTA, S. L. & SONI, R. N. (1965). *J. Ind. Chem. Soc.* **42**, 377–380.

HUYSKENS, P. & ZEEGERS-HUYSKENS, T. (1964). *J. Chim. Phys. Phys.-Chim. Biol.* **61**, 81–86.

PERRIN, D. D. (1965). *Dissociation Constants of Organic Bases in Aqueous Solution*. London: Butterworth.

RATAJCZAK, H. & SOBCZYK, L. (1969). *J. Chem. Phys.* **50**, 556–557.

RAZENBERG, H., KANTERS, J. A., SCHOONE, J. C. & DUISENBERG, A. J. M. (1983). *Acta Cryst.* C**39**, 114–117.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1984). *SHELX84*. Program for crystal structure determination. Univ. of Göttingen, Federal Republic of Germany.

SHKLOVER, V. E., GRIDUNOVA, G. V., STRUCHKOV, YU. T., VORONKOV, M. G., KRYUKOVA, YU. T. & MIRSKOVA, A. N. (1983). *Dokl. Akad. Nauk SSSR*, **269**, 387–390.

SPEK, A. L. (1982). *The EUCLID Package*. In *Computational Crystallography*, edited by D. SAYRE, p. 528. Oxford: Clarendon Press.

STAROVA, G. L., FRANK-KAMENETSKAYA, O. V., FUNDAMENSKII, V. S., SEMENOVA, N. V. & VORONKOV, M. G. (1981). *Sov. Phys. Dokl.* **26**, 911–913.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

VOEGELE, J. C., FISCHER, J. & WEISS, R. (1974*a*). *Acta Cryst.* B**30**, 62–65.

VOEGELE, J. C., FISCHER, J. & WEISS, R. (1974*b*). *Acta Cryst.* B**30**, 66–69.

VOEGEL, J. C., THIERRY, J. C. & WEISS, R. (1974). *Acta Cryst.* B**30**, 70–75.

WHANGBO, M.-H. & WOLFE, S. (1976). *Can. J. Chem.* **54**, 963–968.

WOLFE, S. (1972). *Acc. Chem. Res.* **5**, 102–111.

ZUNDEL, G. & FRITSCH, J. (1984). *J. Phys. Chem.* **88**, 6295–6302.

Acta Cryst. (1988). C**44**, 337–340

Studies in Nonlinear Optical Materials: Structure of Di-2-menthyl 2-(*N*,*N*′-Dimethyl-2-imidazolidinylidene)malonate Monohydrate

BY D. KANAGAPUSHPAM AND K. VENKATESAN

Department of Organic Chemistry, Indian Institute of Science, Bangalore 560012, *India*

AND T. S. CAMERON

Department of Chemistry, Dalhousie University, Halifax, NS, Canada B3H 3J5

(*Received* 3 *June* 1987; *accepted* 1 *October* 1987)

Abstract. $C_{28}H_{48}N_2O_4.H_2O$, $M_r = 494{\cdot}7$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}634$ (2), $b = 11{\cdot}370$ (2), $c = 34{\cdot}167$ (4) Å, $V = 2966$ (2) Å^3, $Z = 4$, $D_m = 1{\cdot}095$, $D_x = 1{\cdot}108$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}43$ cm^{-1}, $F(000) = 1088{\cdot}0$, $T = 293$ K, $R = 0{\cdot}061$ for 1578 significant reflections. The second-harmonic-generation (SHG) efficiency of this compound is negligible (1/100th of the urea standard). The observed low second-order nonlinear response has been attributed to the unfavourable packing of the molecules in the crystal lattice.

Introduction. As part of a long-range research programme on developing organic compounds exhibiting good nonlinear optical properties (Kanagapushpam & Venkatesan, 1987; Kanagapushpam, Padmanabhan & Venkatesan, 1987), we have investigated the molecular geometry and correlation between structure and second-harmonic generation of the title compound. This compound was prepared by following the procedure of Ericsson, Sandström & Wennerbeck (1970) using di-2-menthyl malonate as the active methylene compound.

Experimental. Single crystals of the title compound obtained from acetone by slow evaporation, approximate dimensions 0·30 × 0·30 × 0·35 mm. D_m by flotation. Preliminary ocillation and Weissenberg photographs indicated that the crystals were orthorhombic. Lattice parameters refined by a least-squares fit to settings of 25 accurately centred reflections. Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan mode, scan speed 1° min^{-1}, $\theta \leq 23°$. During data collection three standard reflections (1,4,11, 156, 404) showed statistical variations within ±7% and the data were corrected for this. 2146 reflections collected, *hkl* range, *h*: 0 to 8, *k*: 0 to 12, *l*: 0 to 37; 1578 significant, $|F_o| \geq 3\sigma|F_o|$. Data not corrected for absorption. Structure solved by direct methods (*MULTAN*80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The *E* map computed using the second-best set with ABSFOM = 1·0715, PSIZERO = 1·729, RESID = 26·23, CFOM = 2·6768 yielded the structure. Full-matrix least-squares refinement (*SHELX*76; Sheldrick, 1976) of a scale factor, positional and anisotropic thermal parameters for non-H atoms and positional and

0108-2701/88/020337-04$03.00