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

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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

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($\times 10^3$) *for non*-H *atoms*

E.s.d.'s are given in parentheses.

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

	x	y	z	U_{eq}(Å^2)
O(1)	9880 (7)	4277 (4)	1849 (1)	62 (2)
O(2)	8724 (7)	3248 (3)	1355 (1)	63 (2)
O(3)	9867 (7)	150 (4)	1909 (1)	60 (2)
O(4)	8921 (7)	1052 (3)	1371 (1)	63 (2)
N(1)	12247 (10)	2759 (5)	2353 (2)	64 (3)
N(2)	9895 (10)	1912 (5)	2580 (2)	68 (3)
C(1)	10683 (11)	2290 (5)	2262 (2)	54 (3)
C(2)	9961 (8)	2209 (6)	1875 (2)	46 (2)
C(3)	13553 (12)	3133 (7)	2086 (3)	87 (4)
C(4)	12569 (13)	2690 (8)	2763 (3)	94 (4)
C(5)	10895 (16)	2156 (7)	2934 (2)	93 (4)
C(6)	8071 (15)	1514 (8)	2617 (3)	102 (4)
C(7)	9543 (9)	3308 (6)	1700 (2)	42 (2)
C(8)	8121 (10)	4309 (6)	1171 (2)	53 (3)
C(9)	6445 (11)	4740 (6)	1360 (2)	61 (3)
C(10)	5701 (11)	5806 (7)	1160 (3)	73 (3)
C(11)	5525 (12)	5561 (7)	729 (3)	86 (4)
C(12)	7149 (10)	5100 (7)	537 (2)	65 (3)
C(13)	7852 (10)	4030 (6)	748 (2)	52 (3)
C(14)	9482 (12)	3461 (7)	544 (2)	73 (3)
C(15)	9004 (13)	2915 (11)	146 (3)	117 (5)
C(16)	11011 (15)	4216 (9)	534 (4)	142 (6)
C(17)	4006 (13)	6255 (8)	1353 (3)	108 (5)
C(18)	9596 (9)	1061 (6)	1726 (2)	48 (3)
C(19)	8372 (10)	−34 (5)	1191 (2)	51 (3)
C(20)	9798 (10)	−515 (6)	942 (2)	60 (3)
C(21)	9269 (13)	−1578 (6)	707 (2)	71 (3)
C(22)	7672 (13)	−1300 (7)	474 (2)	76 (4)
C(23)	6155 (11)	−825 (8)	739 (3)	77 (3)
C(24)	6720 (10)	256 (6)	965 (2)	54 (3)
C(25)	5303 (13)	829 (8)	1206 (3)	87 (4)
C(26)	4557 (12)	−10 (9)	1516 (2)	99 (4)
C(27)	3894 (13)	1380 (9)	969 (3)	110 (5)
C(28)	10828 (15)	−2061 (8)	453 (2)	104 (4)
W(1)	7490 (8)	4536 (5)	2505 (1)	99 (2)

Table 2. *Bond distances* (Å) *and angles* (°) *involving non*-H *atoms with their e.s.d.'s in parentheses*

O(1)–C(7)	1·241 (8)	C(9)–C(10)	1·503 (11)
O(2)–C(7)	1·336 (8)	C(10)–C(11)	1·505 (14)
O(2)–C(8)	1·436 (8)	C(10)–C(17)	1·539 (13)
O(3)–C(18)	1·227 (8)	C(11)–C(12)	1·497 (12)
O(4)–C(18)	1·318 (8)	C(12)–C(13)	1·513 (10)
O(4)–C(19)	1·442 (7)	C(13)–C(14)	1·566 (11)
N(1)–C(1)	1·344 (11)	C(14)–C(15)	1·539 (13)
N(1)–C(3)	1·417 (12)	C(14)–C(16)	1·449 (14)
N(1)–C(4)	1·424 (12)	C(19)–C(20)	1·486 (10)
N(2)–C(1)	1·314 (10)	C(19)–C(24)	1·515 (10)
N(2)–C(5)	1·457 (11)	C(20)–C(21)	1·506 (10)
N(2)–C(6)	1·470 (13)	C(21)–C(22)	1·490 (13)
C(1)–C(2)	1·435 (10)	C(21)–C(28)	1·572 (13)
C(2)–C(7)	1·421 (10)	C(22)–C(23)	1·566 (13)
C(2)–C(18)	1·428 (10)	C(23)–C(24)	1·514 (12)
C(4)–C(5)	1·531 (15)	C(24)–C(25)	1·508 (12)
C(8)–C(9)	1·515 (11)	C(25)–C(26)	1·535 (13)
C(8)–C(13)	1·494 (10)	C(25)–C(27)	1·485 (14)

C(7)–O(2)–C(8)	119·6 (5)	C(10)–C(11)–C(12)	114·8 (7)
C(18)–O(4)–C(19)	120·9 (5)	C(11)–C(12)–C(13)	111·5 (6)
C(1)–N(1)–C(3)	126·5 (7)	C(8)–C(13)–C(12)	109·8 (6)
C(1)–N(1)–C(4)	111·0 (7)	C(8)–C(13)–C(14)	114·1 (6)
C(3)–N(1)–C(4)	121·9 (7)	C(12)–C(13)–C(14)	113·7 (6)
C(1)–N(2)–C(5)	112·6 (7)	C(13)–C(14)–C(15)	111·8 (7)
C(1)–N(2)–C(6)	127·3 (7)	C(13)–C(14)–C(16)	113·9 (7)
C(5)–N(2)–C(6)	118·9 (7)	C(15)–C(14)–C(16)	114·1 (8)
N(1)–C(1)–N(2)	110·2 (6)	O(3)–C(18)–O(4)	121·9 (6)
N(1)–C(1)–C(2)	125·4 (6)	O(3)–C(18)–C(2)	123·8 (6)
N(2)–C(1)–C(2)	124·4 (7)	O(4)–C(18)–C(2)	114·3 (6)
C(1)–C(2)–C(7)	114·7 (6)	O(4)–C(19)–C(20)	110·3 (5)
C(1)–C(2)–C(18)	117·5 (6)	O(4)–C(19)–C(24)	105·8 (5)
C(7)–C(2)–C(18)	127·6 (6)	C(20)–C(19)–C(24)	113·5 (6)
N(1)–C(4)–C(5)	104·7 (7)	C(19)–C(20)–C(21)	113·8 (6)
N(2)–C(5)–C(4)	101·3 (7)	C(20)–C(21)–C(22)	109·5 (7)
O(1)–C(7)–O(2)	120·3 (6)	C(20)–C(21)–C(28)	111·8 (7)
O(1)–C(7)–C(2)	124·2 (6)	C(22)–C(21)–C(28)	113·5 (7)
O(2)–C(7)–C(2)	115·5 (6)	C(21)–C(22)–C(23)	111·7 (7)
O(2)–C(8)–C(9)	110·8 (6)	C(22)–C(23)–C(24)	111·4 (7)
O(2)–C(8)–C(13)	106·8 (5)	C(19)–C(24)–C(23)	108·7 (6)
C(9)–C(8)–C(13)	111·4 (6)	C(19)–C(24)–C(25)	114·4 (6)
C(8)–C(9)–C(10)	112·7 (6)	C(23)–C(24)–C(25)	115·1 (7)
C(9)–C(10)–C(11)	109·2 (7)	C(24)–C(25)–C(26)	112·0 (7)
C(9)–C(10)–C(17)	113·0 (7)	C(24)–C(25)–C(27)	113·8 (8)
C(11)–C(10)–C(17)	113·9 (8)	C(26)–C(25)–C(27)	111·7 (8)

isotropic thermal parameters for H atoms (all the H atoms were fixed stereochemically) converged to $R = 0{\cdot}061$, $wR = 0{\cdot}077$, $S = 1{\cdot}2$. The function minimized was $\sum w(|F_o| - |F_c|)^2$ where $w = 0{\cdot}9309/[\sigma^2(|F|)+0{\cdot}003542|F|^2]$. In the final cycle of refinement, Δ/σ for non-H atoms is about 0·06 and for H atoms about 0·12, $\Delta\rho_{\max} = +0{\cdot}29$, $\Delta\rho_{\min} = -0{\cdot}21$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. The atomic positional and thermal parameters for non-H atoms are listed in Table 1.* Bond lengths and angles are presented in Table 2. Fig. 1 is a view of the molecule.

There is extensive delocalization involving the donor imidazolidine ring and the acceptor menthoxycarbonyl groups as reflected in the molecular dimensions. The C(1)=C(2) distance is 1·435 (10) Å which is very much greater than that of the C=C bond in ethylene [1·336 (2) Å; Bartell, Roth, Hollowell, Kuchitsu & Young, 1965]. There is a corresponding reduction in the lengths of donor–ethylenic and acceptor–ethylenic bonds: N(1)–C(1) and N(2)–C(1) are 1·344 (11) and 1·314 (10) Å, respectively, which are shorter than the 1·452 (2) Å reported for an N–C_{sp^2} bond (Ammon, Mazzocchi, Regan & Colicelli, 1979). On the acceptor side, the C(2)–C(7) and C(2)–C(18) distances of 1·421 (10) and 1·428 (10) Å, respectively, are significantly shorter than the 1·487 (5) Å reported for a C_{sp^2}–C_{sp^2} single bond (Shmueli, Shanan-Atidi, Horwitz & Shvo, 1973). Because of the delocalization, the acceptor part of the molecule assumes a carbanion-like structure. The menthoxycarbonyl groups assume the *Z,Z* conformation (Sandström, 1983).

The steric interaction involving the methyl groups at the N atoms and the menthoxycarbonyl groups results in a large rotation about the C(1)=C(2) bond. The twist angle measured as the angle between the planes defined by N(6), C(1), N(2) and by C(7), C(2), C(18) is 64·8 (8)°. It is of interest to note that in 3-(1,3-dimethyl-2-imidazolidinylidene)-2,4-pentanedione (Adhikesavalu & Venkatesan, 1983*a*), which has two acetyl groups as the acceptors, the C=C bond length is 1·468 (3) Å and the twist angle is 72·9 (3)°. In methyl 2-(1,3-dimethyl-2-imidazolinylidene)-3-oxobutyrate

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44422 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Adhikesavalu & Venkatesan, 1983*b*), the C=C bond length is 1·464 (2) Å and the twist angle is 62·6 (3)°. It is noteworthy that the C=C bond lengths do not differ very significantly in the three push–pull ethylenes discussed above, although the twist angle about the C=C bond differs considerably.

The average C–C distance in the cyclohexane rings is 1·515 (12) Å and the mean C–C–C valence angle is 110·3 (6)°. These values are in good agreement with those in menthyl 2-[bis(dimethylamino)methylene]-3-oxobutyrate (Kanagapushpam, Padmanabhan & Venkatesan, 1987). The torsion angles within the six-membered rings indicate that both are in the chair conformation and the non-H atoms take up the equatorial positions.

The packing of the molecules viewed down the *a* axis is displayed in Fig. 2. The crystal structure is stabilized by hydrogen bonding. The water O atom is hydrogen bonded to O(3) of the menthoxycarbonyl group. The hydrogen-bonding parameters are $O(3)\cdots W(1^{i}) =$ 2·865 (7), $O(3)\cdots H2(W^{i}) =$ 1·99 (5) Å and $O(3)\cdots H2(W^{i})-W(1^{i}) =$ 136 (4)° [(i) = $2-x, y-\frac{1}{2}, \frac{1}{2}-z$].

Crystal packing–SHG efficiency correlation

Because of the considerable delocalization of π electrons in this molecule, one would expect it to possess a strong second-order hyperpolarizability and hence good SHG efficiency. The SHG measurements carried out on a powder sample of the title compound (Eaton & Wang, 1986) show, however, the SHG efficiency to be negligible (0·01 times urea). With the CNDO/2 method (Pople & Segal, 1966), the calculated dipole moment was found to be ~17·9 × 10^{-30} C m. From the nature of the charge distribution, it is reasonable to assume that the charge-transfer axis is essentially along the C(1)–C(2) bond. In this molecule, the charge-transfer axis makes 86·9, 68·2 and 22·0° with axes *a*, *b* and *c*, respectively. For the point group 222 there are three nonvanishing nonlinear coefficients. Away from resonance, which corresponds to the experimental conditions, the Kleinman (1962) relations apply and lead to a single component,

$$d_{14} = d_{15} = d_{36} = d.$$

In this molecule, the charge-transfer axis is almost perpendicular (86·9°) to the *a* axis. According to the oriented-gas model (Chemla, Oudar & Jerphagnon, 1975),

$$d = 4\,Nf^{3} \cos(X, a) \cos(X, b) \cos(X, c)\, \beta_{XXX},$$

where *X* is the charge-transfer axis. Since the charge-transfer axis is almost perpendicular to the *a* axis, the cosine projection factor cos (*X*, *a*) cancels out, resulting in very low SHG activity of the crystal. Another factor which is of importance for SHG activity is the requirement of high $\Delta\mu$, the difference between excited charge transfer and ground-state dipole moment. Knowledge of $\Delta\mu$ is not available for this molecule. Even if $\Delta\mu$ were large, the molecular packing in the crystals is clearly not favourable for the optimization of second-order nonlinear response.

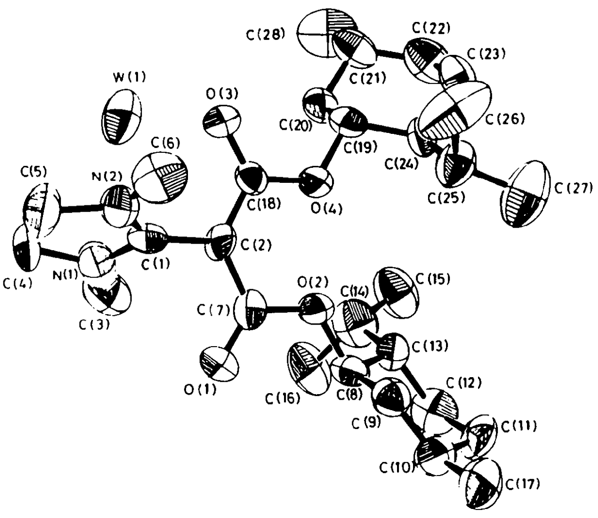

Fig. 1. A perspective view of the molecule showing atom numbering.

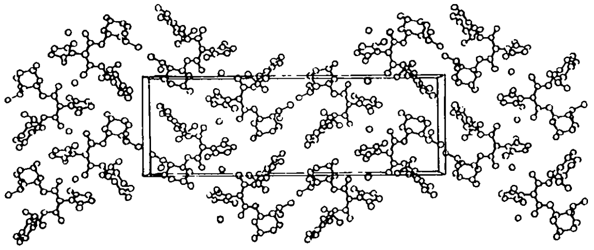

Fig. 2. Packing of the molecules in the unit cell viewed down **a**.

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Structure de la Nitro-7 Benzoxasélénole-2,1 One-3

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Abstract. 7-Nitro-2,1-benzoxaselenol-3-one, $C_7H_3NO_4Se$, $M_r = 244\cdot1$, tetragonal, $P4_1$ or $P4_3$, $a = b = 7\cdot591$ (2), $c = 26\cdot574$ (2) Å, $V = 1531\cdot5$ (9) Å^3, $Z = 8$, $D_x = 2\cdot116$ g cm^{-3}, λ(Mo $K\alpha$) = $0\cdot7107$ Å, $\mu = 51\cdot95$ cm^{-1}, $F(000) = 944$, $T = 290$ K, m.p. = 443 K, final $R = 0\cdot056$ for 1322 observed reflections. The compound is an anhydride of carboxylic and selenic acid moieties with ring closure through an Se—O bond. This bond with the oxygen of the carboxy group is 1·977 (9) Å in molecule *A* and 1·918 (9) Å in *B*. An Se···O bonding interaction with an O atom of the nitro group is also observed: 2·347 (9) (*A*) and 2·500 (9) Å (*B*). All atoms are coplanar. The cohesion of the crystal is the result of van der Waals interactions.

Introduction. Lors de synthèses d'hétérocycles séléniés à cinq chaînons, la nitro-7 benzoxasélénole-2,1 one-3 a été isolée avec un rendement de 2%. Différentes modifications des conditions opératoires ont permis d'améliorer celui-ci jusqu'à 75%. La structure chimique du composé a été établie par spectrométrie de masse, résonance magnétique nucléaire ^{1}H, ^{13}C et ^{77}Se (δ = 1401,8), infra-rouge et par analyse élémentaire. Il s'agit d'une nouvelle fonction: un anhydride cyclique mixte entre un acide carboxylique et un acide sélénique. L'étude radiocristallographique a été entreprise pour confirmer cette structure inconnue auparavant.

Partie expérimentale. Cristallisé dans le toluène. Cristal incolore: 0,4 × 0,5 × 0,2 mm. Paramètres de la maille déterminés à partir de 27 réflexions ($14 \leq \theta \leq 20°$). Diffractomètre Siemens. 1582 réflexions mesurées, $2\theta \leq 50°$, monochromateur à lame graphite, balayage ω, 1372 réflexions indépendantes, $-5 \leq h \leq 6$, $0 \leq k \leq 9$, $0 \leq l \leq 31$, $R_{\text{int}} = 0{,}028$. Réflexions de référence: $1040 \leq F_o(2\bar{1}7) \leq 1090$, $1730 \leq F_o(\bar{2}17) \leq 1780$. Corrections d'absorption par la méthode empirique de North, Phillips & Mathews (1968): coefficients de transmission compris entre 0,11 et 0,28. Structure déterminée avec *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Affinement basé sur F (matrice entière des équations normales), avec *SHELX76* (Sheldrick, 1976). Facteurs de diffusion sont ceux de *SHELX*. Facteurs de température anisotropes affinés pour tous les atomes excepté les hydrogènes. Facteur B global affiné pour les H = 7 (2) Å^2. Facteur R final = 0,056 pour 1322 réflexions observées positives. $wR = 0{,}059$ avec $w = 1/[\sigma^2(F_o) + 0{,}003916F_o^2]$. $\Delta/\sigma \leq 0{,}1$. Valeurs minimale et maximale de la densité électronique résiduelle: $-0{,}7$ et $0{,}6$ e Å^{-3}. Les coordonnées fractionnaires et les $B_{\text{éq}}$ des atomes non-hydrogéne sont données dans le Tableau 1.* La Fig. 1 est une vue de la molecule *A* avec la numérotation des atomes.

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* Les listes des facteurs de structure, les facteurs d'agitation thermique anisotrope, les coordonnées des atomes H, les distances aux plans *P*1 et *P*2 et les distances intermoléculaires ont été déposées au dépôt d'archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 44493: 11 pp.). On peut en obtenir des copies en s'adressant à: The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

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