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5,5′-Diethoxy-α,α′-dimethyl-α,α′-azinodi-*o*-cresol

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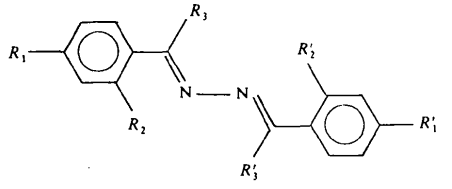
Abstract. $C_{20}H_{24}N_2O_4$, M_r = 356·40, monoclinic, $P2_1/n$, a = 10·9430 (3), b = 13·9325 (5), c = 6·0102 (2) Å, β = 90·653 (2)°, U = 916·3 (1) Å^3, Z = 2, D_c = 1·291 Mg m^{-3}; final R = 0·061 for 907 observed reflexions. The molecule, containing a crystallographic centre of symmetry, is nearly planar with two intramolecular OH···N hydrogen bonds forming chelate rings. The molecules are in a face-to-face close-packing arrangement; accordingly, this compound can be classified as a potential thermochromic luminescent material.

Introduction. This analysis was undertaken to determine the influence of the R substituents in the general skeleton (I) on some structural features, like molecular planarity and crystal packing, conditioning solid-state thermochromism and photochromism.



(I)

Crystals of the title compound were provided by Professor Melendez (University of Zaragoza, Spain). A yellow needle was used to collect 2674 independent reflexions ($\theta \leq 30°$) on a four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation (λ = 0·71069 Å). No intensity decay was observed. After Lorentz and polarization corrections, 907 reflexions with $I > 4\sigma(I)$ were considered as observed.

The structure was solved by *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977). Scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1974). After anisotropic refinement of non-H atoms, a difference map revealed the positions of all H atoms. Final full-matrix least-squares anisotropic refinement (fixed isotropic for H) converged to R = 0·061. A weighting scheme was chosen to prevent bias on $\langle w\Delta^2F\rangle$ *vs* $\langle F_o\rangle$ or *vs* $\langle \sin\theta/\lambda\rangle$ (Martinez-Ripoll & Cano, 1975), giving a final R_w = 0·072. Table 1 shows the final atomic parameters.*

Table 1. *Atomic parameters for* $C_{20}H_{24}N_2O_4$

Listed coordinates have been multiplied by 10^4 (10^3 for H). $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$. The overall isotropic thermal parameter for H atoms is U = 0·063 Å^2.

	x	y	z	U_{eq} (Å$^2\times10^3$)
C(1)	4595 (3)	3762 (3)	1030 (7)	37 (2)
C(2)	5826 (3)	3662 (3)	315 (8)	40 (2)
C(3)	6106 (3)	3099 (3)	−1499 (8)	45 (2)
C(4)	5197 (3)	2607 (3)	−2637 (8)	42 (3)
C(5)	3984 (3)	2682 (4)	−2000 (8)	45 (3)
C(6)	3711 (4)	3262 (3)	−211 (8)	44 (3)
C(7)	4276 (3)	4349 (3)	2951 (7)	40 (3)
C(8)	2948 (4)	4491 (5)	3518 (10)	58 (3)
C(9)	4705 (4)	1533 (4)	−5637 (9)	50 (3)
C(10)	5380 (6)	991 (5)	−7418 (11)	67 (4)
N	5165 (3)	4727 (3)	4083 (5)	42 (2)
O(1)	6760 (2)	4108 (2)	1388 (6)	54 (2)
O(2)	5592 (2)	2059 (2)	−4371 (5)	54 (2)
H(1)	646 (6)	439 (5)	251 (11)	
H(3)	696 (5)	303 (4)	−200 (10)	
H(5)	336 (5)	236 (5)	−276 (10)	
H(6)	287 (5)	332 (4)	24 (10)	
H(81)	242 (6)	443 (5)	238 (11)	
H(82)	273 (5)	405 (5)	473 (11)	
H(83)	273 (6)	505 (5)	411 (11)	
H(91)	424 (6)	109 (5)	−468 (11)	
H(92)	412 (5)	201 (4)	−630 (10)	
H(101)	595 (6)	52 (5)	−678 (10)	
H(102)	571 (5)	146 (5)	−859 (11)	
H(103)	475 (6)	73 (5)	−823 (11)	

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35212 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Discussion. Fig. 1 shows the molecular structure. Table 2 lists bond lengths and angles showing significant departures from the aromatic bond lengths. Table 3 shows the torsion angles. The molecule contains a crystallographic centre of symmetry, departures from 2/*m* symmetry being considerable. As is shown in Table 3, the molecule is nearly planar though the torsion angle C(2)–C(1)–C(7)–N of −4·5 (6)° implies a separation between the two benzene rings of 0·296 (5) Å. An internal hydrogen bond of 2·545 (5) Å, binding the phenolic OH group to the nearest N atom of the azine chain, is also described in Table 2. Fig. 2 shows the face-to-face molecular packing in this crystal structure.

Taking into account all these results, and according to the scheme given by Arcovito, Bonamico, Domenicano & Vaciago (1969), we conclude that this structure can be classified as a potential thermochromic luminescent solid.

Table 2. *Bond lengths* (Å) *and angles* (°)

Average e.s.d.'s are 0·007 Å and 0·2° (0·08 Å and 2° when H atoms are involved).

1–2	1·426	2–1–6	116·1
1–6	1·400	2–1–7	121·9
1–7	1·459	6–1–7	122·1
2–3	1·381	1–2–3	120·9
2–O(1)	1·354	1–2–O(1)	121·5
3–4	1·382	3–2–O(1)	117·6
4–5	1·390	2–3–4	120·6
4–O(2)	1·365	3–4–5	120·6
5–6	1·381	3–4–O(2)	115·1
7–8	1·510	5–4–O(2)	124·4
7–N	1·293	4–5–6	118·4
9–10	1·510	1–6–5	123·5
9–O(2)	1·429	1–7–8	119·4
N–N′	1·390	1–7–N	117·4
O(1)–H(1)	0·85	8–7–N	123·2
O(1)···N	2·545	10–9–O(2)	107·5
N···H(1)	1·78	2–O(1)–H(1)	108
		4–O(2)–9	118·4
		7–N–N′	116·0
		O(1)–H(1)···N	149

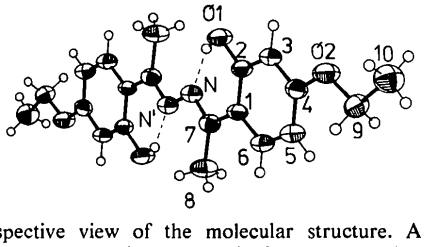

Fig. 1. Perspective view of the molecular structure. A crystallographic centre of symmetry lies between N and N′.

Table 3. *Torsion angles* (°)

Average e.s.d.'s are 0·6° (5° when H atoms are involved).

6–1–2–3	−0·2	3–4–O(2)–9	−179·7
1–2–3–4	−1·0	4–O(2)–9–10	−179·7
2–3–4–5	1·0	2–1–7–N	−4·5
3–4–5–6	0·2	6–1–7–8	−5·2
4–5–6–1	−1·5	1–7–N–N′	−179·3
5–6–1–2	1·5	1–2–O(1)–H(1)	3

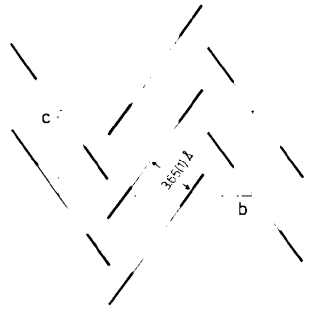

Fig. 2. Schematic representation of the crystal packing viewed along [100]. Only the two benzene rings and the azine chain are represented, by thick and thin lines respectively.

Most of the calculations were carried out with XRAY 70 (Stewart, Kundell & Baldwin, 1970). We thank Professor S. García-Blanco for facilities and the staff of the Centro de Proceso de Datos del Ministerio de Educación, Madrid, for the use of the Univac 1108 computer.

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