

Structures of *E*- and *Z*-Isomers of 1-Methyl-5-[(2,4,6-trimethylphenyl)methylene]imidazolidine-2,4-dione

BY MICHAEL G. B. DREW

Department of Chemistry, The University, Whiteknights, Reading RG6 2AD, England

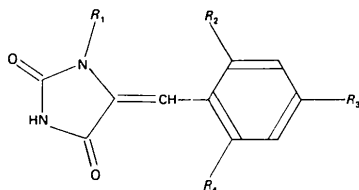
AND K. F. MOK, K. P. ANG AND S. F. TAN

Department of Chemistry, National University of Singapore, Kent Ridge, Singapore 0511

(Received 6 November 1986; accepted 19 December 1986)

Abstract. $C_{14}H_{16}N_2O_2$, $M_r = 244.30$, monoclinic, $P2_1/c$, for both isomers. *E*-isomer: $a = 8.203(7)$, $b = 8.278(7)$, $c = 18.939(9) \text{ \AA}$, $\beta = 90.2(2)^\circ$, $U = 1286.0 \text{ \AA}^3$, $Z = 4$, $D_m = 1.25(3)$, $D_x = 1.26 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.7107 \text{ \AA}$, $\mu = 0.50 \text{ cm}^{-1}$, $F(000) = 520$, room temperature, final $R = 0.062$ for 1168 [$I > 3\sigma(I)$] independent observed reflections. *Z*-isomer: $a = 13.288(9)$, $b = 8.026(7)$, $c = 12.727(9) \text{ \AA}$, $\beta = 95.6(2)^\circ$, $U = 1350.9 \text{ \AA}^3$, $Z = 4$, $D_m = 1.16(3)$, $D_x = 1.20 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.7107 \text{ \AA}$, $\mu = 0.48 \text{ cm}^{-1}$, $F(000) = 520$, room temperature, final $R = 0.082$ for 1019 [$I > 3\sigma(I)$] independent observed reflections. Owing to the presence of methyl groups in the *ortho* positions of the benzene ring, the plane of this ring is rotated away from that of the imidazolidine-2,4-dione ring but the angle of rotation is smaller in the case of the *E*-isomer at $57.1(3)^\circ$ than in the case of the *Z*-isomer in which the two planes are almost perpendicular at $84.2(3)^\circ$.

Introduction. Although compounds of imidazolidine-2,4-dione (also known as hydantoin) substituted at the 5-position are well documented (*Kirk-Othmer Encyclopedia of Chemical Technology*, 1980), those with a methylene linkage at this position are relatively less common. We have previously reported the structures of the *Z*-isomers of two 5-arylmethylenehydantoin in a class of compounds shown in the diagram: $R_1 = R_2 = R_4 = \text{H}$, $R_3 = \text{OCH}_3$ (Drew, Mok, Ang & Tan, 1987a) and $R_1 = \text{CH}_3$, $R_2 = R_3 = R_4 = \text{H}$ (Drew, Mok, Ang & Tan, 1987b). Both the *E*- and *Z*-isomers of the title compound are members of this class with $R_1 = R_2 = R_3 = R_4 = \text{CH}_3$.



Experimental. Compounds prepared as described elsewhere (Tan, Ang & Fong, 1986). Density measured by flotation in $\text{CCl}_4/\text{ligroin}$. Preliminary cell constants established from precession photographs. Crystals of approximate size $0.2 \times 0.2 \times 0.4$ for *E*- and $0.2 \times 0.2 \times 0.1$ mm for *Z*-isomer mounted on Stoe Stadi-2 diffractometer each to rotate about the b axis. Cell dimensions by measurement of about 20 high-angle axial reflections (2θ range 30 to 40°). Intensity data collected *via* variable-width ω scan, background count every 20 s, step-scan rate $0.033^\circ \text{ s}^{-1}$, width $(2.0 + \sin\mu/\tan\theta)$ for *E*-isomer and $(3.0 + \sin\mu/\tan\theta)$ for *Z*-isomer. Absorption and extinction corrections not applied. Standard reflections $2k2$ measured every 20 measurements for each layer: no significant variation in intensity in *E*-isomer; decay of ca 20% in *Z*-isomer, catered for by scaling reflections within each layer according to the standard reflections and giving each layer a separate scale factor in subsequent refinement. For *E*-isomer 2482 unique data measured with $2\theta_{\text{max}} 50^\circ$ ($-9 \leq h \leq 9$, $0 \leq k \leq 9$, $0 \leq l \leq 22$); 1168 data with $I > 3\sigma(I)$ used in subsequent calculations. For *Z*-isomer 2482 data measured with $2\theta_{\text{max}} 50^\circ$ ($0 \leq h \leq 15$, $0 \leq k \leq 9$, $-15 \leq l \leq 15$); 1019 data with $I > 3\sigma(I)$ used in subsequent calculations. Structure solved by direct methods using *SHELX76* (Sheldrick, 1976) on Amdahl V7 computer. Subsequent refinements and calculations also used *SHELX76*. Non-hydrogen atoms refined anisotropically before placing H atoms in trigonal or tetrahedral positions. Thermal parameters of H atoms on N(3) and C(6) allowed to refine individually while those on methyl groups constrained to refine as rigid groups with common parameters. Weighting scheme $w = 1/[\sigma^2(F) + 0.002F^2]$, $\sigma(F)$ taken from counting statistics. For *E*-isomer final $R(F) 0.062$ ($wR 0.071$, $S 0.66$) and final shift/e.s.d. < 0.03 . For *Z*-isomer final $R(F) 0.082$ ($wR 0.096$, $S 2.41$) and final shift/e.s.d. < 0.06 ; the relatively high R value due to poor quality of crystal used but considered satisfactory. In final difference map, max. and min. peaks 0.15 and -0.19 e \AA^{-3} for

E-isomer and 0.22 and -0.17 e \AA^{-3} for *Z*-isomer. Scattering factors from *International Tables for X-ray Crystallography* (1974). Atomic coordinates are given in Tables 1 and 2, interatomic distances and angles in Tables 3 and 4.*

* 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 43657 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters with *e.s.d.*'s in parentheses for the *E*-isomer

$$U_{eq} = \frac{1}{3} \sum_i \sum_j \mathbf{a}_i \cdot \mathbf{a}_j U_{ij}$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}(\text{\AA}^2 \times 1000)$
N(1)	8869 (4)	2101 (4)	3567 (2)	69 (4)
C(2)	9211 (5)	1336 (5)	4184 (2)	65 (5)
N(3)	10732 (4)	668 (5)	4113 (2)	68 (4)
C(4)	11350 (5)	883 (5)	3452 (2)	60 (4)
C(5)	10121 (5)	1897 (5)	3075 (2)	60 (4)
C(6)	10092 (6)	2481 (6)	2420 (2)	68 (5)
C(7)	11271 (5)	2403 (6)	1846 (2)	72 (5)
C(8)	10742 (6)	1831 (6)	1187 (2)	77 (5)
C(9)	11770 (7)	1843 (6)	619 (3)	83 (6)
C(10)	13329 (6)	2397 (6)	672 (3)	92 (6)
C(11)	13829 (6)	2990 (6)	1319 (3)	86 (6)
C(12)	12849 (5)	3003 (6)	1904 (3)	76 (5)
C(13)	9043 (6)	1220 (7)	1080 (3)	76 (6)
C(14)	14419 (9)	2421 (10)	29 (3)	129 (9)
C(15)	13489 (6)	3760 (7)	2572 (3)	85 (6)
C(16)	7327 (6)	2873 (7)	3429 (3)	87 (6)
O(17)	8355 (4)	1260 (4)	4704 (2)	88 (4)
O(18)	12605 (3)	361 (4)	3230 (2)	81 (4)

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters with *e.s.d.*'s in parentheses for the *Z*-isomer

$$U_{eq} = \frac{1}{3} \sum_i \sum_j \mathbf{a}_i \cdot \mathbf{a}_j U_{ij}$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}(\text{\AA}^2 \times 1000)$
N(1)	-1659 (4)	4172 (7)	2941 (4)	62 (6)
C(2)	-1080 (6)	4526 (9)	3838 (6)	73 (8)
N(3)	-151 (5)	3814 (7)	3769 (5)	71 (7)
C(4)	-95 (6)	3096 (9)	2822 (6)	74 (9)
C(5)	-1097 (6)	3333 (8)	2253 (5)	64 (8)
C(6)	-1326 (7)	2853 (9)	1285 (6)	73 (9)
C(7)	-2286 (6)	3044 (9)	619 (5)	71 (9)
C(8)	-3072 (8)	1935 (11)	664 (7)	97 (11)
C(9)	-3952 (8)	2170 (12)	26 (8)	93 (13)
C(10)	-4075 (7)	3437 (13)	-679 (7)	90 (12)
C(11)	-3286 (7)	4494 (10)	-752 (7)	89 (11)
C(12)	-2389 (6)	4339 (9)	-132 (6)	75 (9)
C(13)	-2947 (9)	446 (15)	1406 (9)	152 (18)
C(14)	-5060 (8)	3669 (20)	-1391 (10)	126 (17)
C(15)	-1491 (8)	5498 (12)	-251 (10)	107 (14)
C(16)	-2650 (6)	4926 (11)	2713 (7)	81 (10)
O(17)	-1322 (4)	5360 (6)	4574 (4)	78 (6)
O(18)	633 (5)	2398 (8)	2534 (4)	102 (8)

Discussion. The structures of the *E*- and *Z*-isomers are shown in Figs. 1 and 2 together with the atomic numbering scheme. In hydantoins with one or two groups substituted at the 5-position the imidazolidine ring is planar with the N(1)–C(5) and C(4)–C(5) distances in the ranges 1.45 to 1.48 Å and 1.51 to 1.55 Å respectively and the N(1)–C(5)–C(4) angle in the 99 to 101° range (Camerman & Camerman, 1971; Florencio, Smith-Verdier & Garcia-Blanco, 1978*a,b*; Fujiwara & Van der Veen, 1979; Koch, Germain, Declercq & Dusaosoy, 1975; Smith-Verdier, Florencio

Table 3. Bond lengths (Å) and angles (°) with *e.s.d.*'s in parentheses for the *E*-isomer

N(1)–C(2)	1.357 (5)	C(7)–C(8)	1.402 (7)
N(1)–C(5)	1.399 (5)	C(7)–C(12)	1.391 (6)
N(1)–C(16)	1.440 (5)	C(8)–C(9)	1.369 (6)
C(2)–N(3)	1.371 (6)	C(8)–C(13)	1.496 (6)
C(2)–O(17)	1.214 (5)	C(9)–C(10)	1.362 (7)
N(3)–C(4)	1.364 (5)	C(10)–C(11)	1.382 (7)
C(4)–C(5)	1.492 (6)	C(10)–C(14)	1.514 (7)
C(4)–O(18)	1.194 (5)	C(11)–C(12)	1.371 (6)
C(5)–C(6)	1.332 (6)	C(12)–C(15)	1.506 (7)
C(6)–C(7)	1.458 (6)		
C(2)–N(1)–C(5)	111.5 (3)	C(6)–C(7)–C(8)	118.3 (4)
C(2)–N(1)–C(16)	122.8 (4)	C(6)–C(7)–C(12)	123.0 (4)
C(5)–N(1)–C(16)	125.4 (4)	C(8)–C(7)–C(12)	118.4 (4)
N(1)–C(2)–N(3)	106.8 (4)	C(7)–C(8)–C(9)	120.5 (5)
N(1)–C(2)–O(17)	127.2 (4)	C(7)–C(8)–C(13)	121.3 (4)
N(3)–C(2)–O(17)	126.0 (4)	C(9)–C(8)–C(13)	118.2 (5)
C(2)–N(3)–C(4)	112.2 (4)	C(8)–C(9)–C(10)	121.7 (5)
N(3)–C(4)–C(5)	105.1 (4)	C(9)–C(10)–C(11)	117.5 (4)
N(3)–C(4)–O(18)	126.9 (4)	C(9)–C(10)–C(14)	120.2 (5)
C(5)–C(4)–O(18)	128.1 (4)	C(11)–C(10)–C(14)	122.3 (5)
N(1)–C(5)–C(4)	104.2 (3)	C(10)–C(11)–C(12)	123.1 (5)
N(1)–C(5)–C(6)	124.6 (4)	C(7)–C(12)–C(11)	118.8 (5)
C(4)–C(5)–C(6)	131.2 (4)	C(7)–C(12)–C(15)	122.5 (4)
C(5)–C(6)–C(7)	132.0 (4)	C(11)–C(12)–C(15)	118.6 (4)

Table 4. Bond lengths (Å) and angles (°) with *e.s.d.*'s in parentheses for the *Z*-isomer

N(1)–C(2)	1.343 (9)	C(7)–C(8)	1.379 (11)
N(1)–C(5)	1.380 (8)	C(7)–C(12)	1.409 (10)
N(1)–C(16)	1.453 (9)	C(8)–C(9)	1.369 (12)
C(2)–N(3)	1.372 (9)	C(8)–C(13)	1.522 (13)
C(2)–O(17)	1.219 (8)	C(9)–C(10)	1.355 (13)
N(3)–C(4)	1.345 (9)	C(10)–C(11)	1.358 (12)
C(4)–C(5)	1.464 (10)	C(10)–C(14)	1.530 (13)
C(4)–O(18)	1.206 (8)	C(11)–C(12)	1.369 (11)
C(5)–C(6)	1.299 (9)	C(12)–C(15)	1.533 (12)
C(6)–C(7)	1.468 (11)		
C(2)–N(1)–C(5)	110.0 (5)	C(6)–C(7)–C(8)	121.9 (7)
C(2)–N(1)–C(16)	121.0 (6)	C(6)–C(7)–C(12)	119.3 (7)
C(5)–N(1)–C(16)	127.8 (6)	C(8)–C(7)–C(12)	118.7 (7)
N(1)–C(2)–N(3)	107.6 (6)	C(7)–C(8)–C(9)	119.5 (8)
N(1)–C(2)–O(17)	126.7 (6)	C(7)–C(8)–C(13)	119.7 (9)
N(3)–C(2)–O(17)	125.7 (7)	C(9)–C(8)–C(13)	120.8 (9)
C(2)–N(3)–C(4)	111.4 (6)	C(8)–C(9)–C(10)	122.5 (8)
N(3)–C(4)–C(5)	105.1 (6)	C(9)–C(10)–C(11)	118.0 (8)
N(3)–C(4)–O(18)	126.0 (7)	C(9)–C(10)–C(14)	121.6 (10)
C(5)–C(4)–O(18)	128.9 (6)	C(11)–C(10)–C(14)	120.3 (10)
N(1)–C(5)–C(4)	105.8 (5)	C(10)–C(11)–C(12)	122.5 (8)
N(1)–C(5)–C(6)	130.9 (7)	C(7)–C(12)–C(11)	118.7 (7)
C(4)–C(5)–C(6)	123.2 (7)	C(7)–C(12)–C(15)	119.3 (7)
C(5)–C(6)–C(7)	128.8 (7)	C(11)–C(12)–C(15)	122.0 (7)

& García-Blanco, 1977, 1979). However, when the substitution at the 5-position is a methylene group the bond distances decrease to 1.37–1.40 and 1.47–1.50 Å but the corresponding angle increases to 104–106° (Drew, Mok, Ang & Tan, 1987*a,b*; Thieme & Haedicke, 1978). Both isomers of the title compound conform to these observations: the imidazolidine ring is planar; the N(1)–C(5) and C(4)–C(5) distances are 1.399 (5) and 1.492 (6) Å in the *E*-isomer and 1.380 (8) and 1.464 (10) Å respectively in the *Z*-isomer; the N(1)–C(5)–C(4) angle is 104.2 (3) and 105.8 (5)° in the *E*- and *Z*-isomers respectively. The slightly longer bond distances and smaller bond angle in the *E*-isomer (relative to the *Z*-isomer) have also been found for 5-benzylidene-1-methylhydantoin (Drew, Mok, Ang & Tan, 1987*b,c*). Differences between the two isomers also occur in the two remaining angles at the C(5) position (see Table 5). The N(1)–C(5)–C(6) and C(4)–C(5)–C(6) angles are 124.6 (4) and 131.2 (4)° for the *E*-isomer and 130.9 (7) and 123.2 (7)° for the *Z*-isomer respectively. Thus the angle *cis* to the phenyl group is the larger in both isomers due to the steric effect of the phenyl ring despite this being lessened by its rotation out of the plane of the hydantoin ring. This phenomenon has also been observed in the case of 5-benzylidene-1-methylhydantoin (Drew, Mok, Ang & Tan, 1987*b,c*). Note that in both cases C(5) is coplanar with atoms N(1), C(4), C(6) to within experimental error.

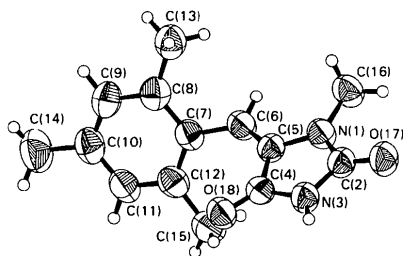


Fig. 1. The structure of the *E*-isomer of the title molecule. Thermal ellipsoids are shown at 50% probability.

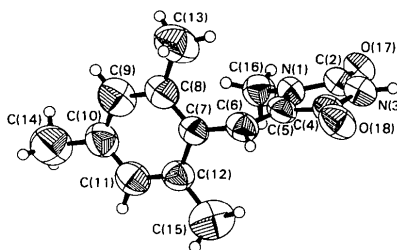


Fig. 2. The structure of the *Z*-isomer of the title molecule. Thermal ellipsoids are shown at 50% probability.

Bond distances and angles in the phenyl ring are normal. The C(6)–C(7) bond lengths are 1.458 (6) and 1.468 (11) Å in the *E*- and *Z*-isomers respectively. These distances are shorter than that expected for a single bond and partial double-bond character is indicated.

For both isomers the molecule is essentially made up of two planes of atoms (the imidazolidine-2,4-dione ring and the phenyl ring) as shown in Table 6. However, owing to the presence of a methyl group at the N(1) position of the five-membered ring and at both *ortho* positions of the phenyl ring which would otherwise cause severe strain, the plane of one ring is rotated away from that of the other. This angle of rotation is 57.1 (3) and 84.2 (3)° for the *E*- and the *Z*-isomer respectively. A larger angle of rotation for the *Z*-isomer has also been found in 5-benzylidene-1-methylimidazolidine-2,4-dione, for which the angles are 8.7 and 45.9° for the two isomers.

With the presence of two carbonyl oxygen atoms and one imine hydrogen atom in the hydantoin moiety, hydrogen bonds are expected and are indeed observed in nearly all hydantoin with no substitution at the N(3) position. For both *E*- and *Z*-isomers hydrogen bonding

Table 5. Angles (°) around the C(5) atom in some 5-arylmethenehydantoin

	(1)	(2)	(3)	(4)	(5)
N(1)–C(5)–C(4)	104.2 (3)	105.8 (6)	104.4 (13)	105.5 (2)	105.1 (2)
N(1)–C(5)–C(6)	124.5 (4)	130.9 (7)	122.0 (14)	132.7 (2)	131.8 (2)
C(4)–C(5)–C(6)	131.3 (4)	123.2 (7)	133.3 (15)	121.8 (2)	123.0 (2)

Note: (1) *E*-isomer of title molecule; (2) *Z*-isomer of title molecule; (3) *E*-isomer of 5-benzylidene-1-methylhydantoin (Drew *et al.*, 1987*c*); (4) *Z*-isomer of 5-benzylidene-1-methylhydantoin (Drew *et al.*, 1987*b*); (5) *Z*-isomer of 5-[(4-methoxyphenyl)methylene]hydantoin (Drew *et al.*, 1987*a*).

Table 6. Least-squares planes for *E*- and *Z*-isomers

Distances of atoms from the two planes are given in Å. Atoms not contributing to the plane are marked with an asterisk.

E-isomer:

Plane 1. N(1) 0.01 (1), C(2) –0.02 (1), N(3) 0.02 (1), C(4) –0.02 (1), C(5) 0.01 (1), C(6)* –0.01 (1), C(7)* 0.00 (1), C(10)* 0.01 (1), C(14)* 0.02 (1), C(16)* –0.10 (1), O(17)* –0.05 (1), O(18)* –0.07 (1).

Plane 2. C(6)* 0.10 (1), C(7) –0.01 (1), C(8) 0.00 (1), C(9) 0.01 (1), C(10) –0.01 (1), C(11) 0.01 (1), C(12) 0.00 (1), C(13)* 0.04 (1), C(14)* 0.02 (1), C(15)* 0.10 (1).

Angle between plane 1 and plane 2: 57.1 (3)°.

Z-isomer:

Plane 1. N(1) –0.02 (1), C(2) 0.02 (1), N(3) –0.02 (1), C(4) 0.00 (1), C(5) 0.01 (1), C(6)* 0.08 (1), C(7)* 0.12 (1), C(10)* 0.26 (1), C(14)* 0.34 (1), C(16)* 0.17 (1), O(17)* 0.11 (1), O(18)* –0.00 (1).

Plane 2. C(6)* 0.01 (1), C(7) 0.02 (1), C(8) –0.02 (1), C(9) 0.00 (1), C(10) 0.01 (1), C(11) –0.01 (1), C(12) –0.01 (1), C(13)* –0.10 (1), C(14)* 0.02 (1), C(15)* –0.08 (1).

Angle between plane 1 and plane 2: 84.2 (3)°.

Note: Plane 1 is that of the imidazolidine ring and 2 the benzene ring.

occurs through the H(31) and O(17) atoms. N(3)—H(31)···O(17ⁱ) and H(31)···O(17ⁱ) distances and N(3)—H(31)···O(17ⁱ) angle [symmetry element (i): 2-x, -y, 1-z for *E*- and -x, 1-y, 1-z for *Z*-isomer] are 2.85 (1), 2.01 (6) Å and 165 (6)° for the *E*-isomer and 2.81 (1), 1.92 (8) Å and 163 (8)° for the *Z*-isomer respectively.

References

- CAMERMAN, A. & CAMERMAN, N. (1971). *Acta Cryst.* B27, 2205–2211.
- DREW, M. G. B., MOK, K. F., ANG, K. P. & TAN, S. F. (1987a). *Acta Cryst.* C43, 743–745.
- DREW, M. G. B., MOK, K. F., ANG, K. P. & TAN, S. F. (1987b). *Acta Cryst.* C43, 745–748.
- DREW, M. G. B., MOK, K. F., ANG, K. P. & TAN, S. F. (1987c). *Acta Cryst.* C43. In the press.
- FLORENCIO, F., SMITH-VERDIER, P. & GARCÍA-BLANCO, S. (1978a). *Acta Cryst.* B34, 1317–1321.
- FLORENCIO, F., SMITH-VERDIER, P. & GARCÍA-BLANCO, S. (1978b). *Acta Cryst.* B34, 2220–2223.
- FUJIWARA, H. & VAN DER VEEN, J. M. (1979). *J. Chem. Soc. Perkin Trans.* 2, pp. 659–663.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- Kirk-Othmer Encyclopedia of Chemical Technology* (1980). 3rd ed., Vol. 12. New York: John Wiley.
- KOCH, M. H., GERMAIN, G., DECLERCQ, J.-P. & DUSAUSOY, Y. (1975). *Acta Cryst.* B31, 2547–2549.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- SMITH-VERDIER, P., FLORENCIO, F. & GARCÍA-BLANCO, S. (1977). *Acta Cryst.* B33, 3381–3385.
- SMITH-VERDIER, P., FLORENCIO, F. & GARCÍA-BLANCO, S. (1979). *Acta Cryst.* B35, 216–217.
- TAN, S., ANG, K. P., & FONG, Y. F. (1986). *J. Chem. Soc. Perkin Trans.* 2. In the press.
- THIEME, P. C. & HAEDICKE, E. (1978). *Justus Liebig's Ann. Chem.* 2, pp. 227–237.

Acta Cryst. (1987). C43, 972–974

Structure of 9-Nitro-7*H*-dibenzo[*a,k*]anthracen-7-one

BY SHOJI FUJISAWA, ISAO OONISHI AND JUNJI AOKI

Department of Chemistry, Faculty of Science, Toho University, Funabashi, Chiba 274, Japan

AND YUJI OHASHI

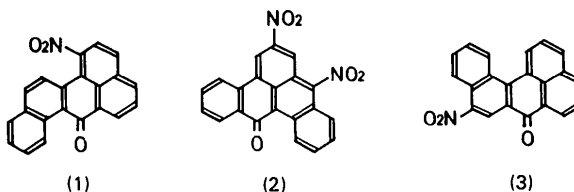
Department of Chemistry, Faculty of Science, Ochanomizu University, Otsuka 2-1-1, Bunkyo-ku, Tokyo 112, Japan

(Received 20 November 1986; accepted 22 December 1986)

Abstract. C₂₁H₁₁NO₃, *M_r* = 325.3, monoclinic, *P*2₁/*a*, *a* = 23.349 (9), *b* = 8.577 (4), *c* = 7.180 (3) Å, β = 97.62 (4)°, *V* = 1424.7 (10) Å³, *Z* = 4, *D_m* = 1.49, *D_x* = 1.517 Mg m⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 0.109 mm⁻¹, *F*(000) = 672, *T* = 298 K. Final *R* = 0.069 for 2226 independent reflections. The molecule is greatly distorted from a planar structure owing to the overcrowding of H atoms. The mean planes of the two naphthalene moieties make an angle of 24.95 (5)°. The NO₂ group rotates around the C—N bond from a coplanar conformation. The torsion angle O—N—C—C is 30.1 (3)°.

Introduction. Aromatic nitro compounds such as 1-nitro-7*H*-benzo[*h,i*]chrysen-7-one (1) (Fujisawa, Oonishi, Aoki & Ohashi, 1987) and 6,8-dinitro-13*H*-dibenzo[*a,de*]anthracen-13-one (2) (Fujisawa, Oonishi, Aoki & Ohashi, 1986) have been extensively used as starting materials in the synthesis of polycyclic aromatic compounds. It is often difficult, however, to assign the positions of the nitro groups by chemical

procedures. The present study reports the structure of the title compound (3).



Experimental. Orange plate-like crystals from chlorobenzene solution; *D_m* by flotation in ZnCl₂ solution; systematic absences: *h*0*l*, *h* = 2*n* + 1, 0*k*0, *k* = 2*n* + 1; crystal dimensions 0.45 × 0.45 × 0.20 mm; Rigaku AFC-6 diffractometer; graphite monochromator; cell parameters refined by least-squares method on the basis of 20 independent 2θ values, 20 < 2θ < 26°; intensity measurement performed up to 2θ = 50°; *h* -31 to 32, *k* 0 to 12 and *l* 0 to 10; ω-2θ scan, scan speed 2° min⁻¹ (2θ), scan width (1.2 + 0.35 tan θ)°; background 5 s