

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)
$$B_{\text{eq}} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	B_{eq}
Br	0.4214 (2)	0.2614 (1)	0.0837 (1)	7.88 (5)
F	0.9791 (9)	0.0089 (3)	-0.8383 (3)	7.2 (2)
O(1)	0.2653 (8)	0.5349 (3)	-0.3994 (3)	4.3 (2)
O(2)	-0.1891 (8)	0.2246 (4)	-0.3658 (3)	5.6 (2)
N(1)	-0.0045 (9)	0.3928 (4)	-0.3623 (3)	3.6 (2)
N(2)	0.3114 (8)	0.3686 (3)	-0.4864 (3)	3.3 (2)
C(1)	0.243 (1)	0.3185 (7)	-0.0273 (4)	5.0 (3)
C(2)	0.095 (1)	0.2450 (6)	-0.0579 (5)	5.7 (4)
C(3)	-0.037 (1)	0.2874 (6)	-0.1370 (5)	5.2 (3)
C(4)	-0.023 (1)	0.4003 (5)	-0.1880 (4)	3.6 (2)
C(5)	0.126 (1)	0.4702 (5)	-0.1558 (5)	4.8 (3)
C(6)	0.260 (1)	0.4296 (7)	-0.0761 (5)	5.8 (4)
C(7)	-0.161 (1)	0.4434 (5)	-0.2763 (5)	4.2 (3)
C(8)	0.200 (1)	0.4414 (5)	-0.4155 (4)	3.5 (2)
C(9)	0.189 (1)	0.2677 (5)	-0.4795 (4)	3.6 (3)
C(10)	-0.026 (1)	0.2867 (5)	-0.3981 (4)	3.9 (3)
C(11)	0.239 (1)	0.1705 (5)	-0.5315 (4)	4.0 (3)
C(12)	0.437 (1)	0.1328 (5)	-0.6104 (4)	3.9 (3)
C(13)	0.634 (1)	0.1969 (5)	-0.6530 (5)	4.6 (3)
C(14)	0.814 (1)	0.1560 (5)	-0.7285 (5)	4.8 (3)
C(15)	0.801 (1)	0.0499 (6)	-0.7633 (5)	5.0 (3)
C(16)	0.612 (1)	-0.0175 (5)	-0.7250 (5)	5.3 (3)
C(17)	0.432 (1)	0.0252 (5)	-0.6497 (5)	4.7 (3)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Br—C(1)	1.893 (6)	F—C(15)	1.352 (8)
O(1)—C(8)	1.224 (6)	O(2)—C(10)	1.212 (7)
N(1)—C(7)	1.459 (8)	N(1)—C(8)	1.377 (7)
N(1)—C(10)	1.379 (7)	N(2)—C(8)	1.350 (7)
N(2)—C(9)	1.398 (7)	C(1)—C(2)	1.38 (1)
C(1)—C(6)	1.35 (1)	C(2)—C(3)	1.36 (1)
C(3)—C(4)	1.378 (9)	C(4)—C(5)	1.369 (8)
C(4)—C(7)	1.492 (8)	C(5)—C(6)	1.38 (1)
C(9)—C(10)	1.477 (8)	C(9)—C(11)	1.345 (8)
C(11)—C(12)	1.435 (8)	C(12)—C(13)	1.396 (9)
C(12)—C(17)	1.394 (8)	C(13)—C(14)	1.361 (9)
C(14)—C(15)	1.359 (9)	C(15)—C(16)	1.37 (1)
C(16)—C(17)	1.37 (1)		
C(7)—N(1)—C(8)	123.7 (4)	N(2)—C(9)—C(10)	105.2 (4)
C(7)—N(1)—C(10)	125.7 (5)	N(2)—C(9)—C(11)	131.4 (5)
C(8)—N(1)—C(10)	110.5 (4)	C(10)—C(9)—C(11)	123.4 (5)
C(8)—N(2)—C(9)	110.5 (4)	O(2)—C(10)—N(1)	125.0 (5)
Br—C(1)—C(2)	119.2 (5)	O(2)—C(10)—C(9)	129.6 (5)
Br—C(1)—C(6)	120.4 (5)	N(1)—C(10)—C(9)	105.4 (5)
C(2)—C(1)—C(6)	120.4 (6)	C(9)—C(11)—C(12)	132.7 (5)
C(1)—C(2)—C(3)	119.0 (6)	C(11)—C(12)—C(13)	125.1 (5)
C(2)—C(3)—C(4)	121.8 (6)	C(11)—C(12)—C(17)	118.2 (5)
C(3)—C(4)—C(5)	117.6 (5)	C(13)—C(12)—C(17)	116.6 (5)
C(3)—C(4)—C(7)	121.1 (5)	C(12)—C(13)—C(14)	121.8 (6)
C(5)—C(4)—C(7)	121.3 (5)	C(13)—C(14)—C(15)	119.0 (6)
C(4)—C(5)—C(6)	121.7 (6)	F—C(15)—C(14)	119.2 (6)
C(1)—C(6)—C(5)	119.6 (7)	F—C(15)—C(16)	118.5 (6)
N(1)—C(7)—C(4)	112.2 (5)	C(14)—C(15)—C(16)	122.2 (6)
O(1)—C(8)—N(1)	124.6 (5)	C(15)—C(16)—C(17)	118.0 (6)
O(1)—C(8)—N(2)	127.1 (5)	C(12)—C(17)—C(16)	122.3 (6)
N(1)—C(8)—N(2)	108.3 (4)		

Data were corrected for Lp effects. The structure was solved by direct methods. H atoms were found in difference syntheses and included as fixed contributors with an overall isotropic displacement parameter that refined to $U_{\text{iso}} = 0.067 (5) \text{\AA}^2$. The refinement was by blocked-matrix least-squares methods. Programs used were: *SHELXS86* (Sheldrick, 1985) and *SHELX76* (Sheldrick, 1976). Most of the calculations were performed on a VAX 6420 computer at the Instituto de Física e Química de São Carlos.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1003). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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8,16-Methano-16H-dinaphtho[2,1-d:1',2'-g]-[1,3]dioxocine-2,14-diol

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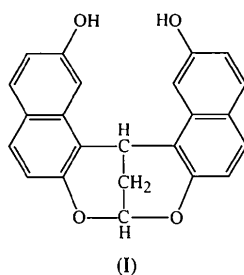
Abstract

The symmetry of the chemical structure of the title compound, $\text{C}_{23}\text{H}_{16}\text{O}_4$, is not preserved in the crystal structure; two conformational isomers are present as required by the centrosymmetry of the space group. The naphthalene components are splayed away from each other and twisted in order to separate the aromatic H atoms H14 and H26. The chevron-shaped molecules are stacked on top of one another. Four such stacks (two antiparallel pairs) are apparent when the unit cell is viewed along the z axis. Both the conformation of

the molecule and the orientation of the oxocine (O1 and O7) and phenolic (O18 and O27) O atoms, as defined by the vector from C4 to C6 (the methano bridgehead atoms), are reversed between adjacent molecules within each stack. This orientation reversal stacks the oxocine O atoms and the phenolic O atoms from neighboring molecules roughly on top of each other and places all O atoms, along with all hydrogen bonds, near the planes $y = \pm \frac{a}{2}$. The two intermolecular hydrogen bonds per molecule involve only the phenolic O atoms. Energy minimization of the crystal structure with *MOPAC* [Stewart (1983). QCPE No. 455. Indiana Univ., USA] yields much the same structure. The proximity of H atoms H14 and H26 to each other and the phenolic rings of the opposite naphthalene components, even with the distortion, is sufficient to explain the downfield shift of 1.1 p.p.m. observed for these protons in NMR spectrum.

Comment

The title compound, (I), was the major product isolated from the reaction of malondialdehyde bis(diethyl acetal) and 2,7-dihydroxynaphthalene in an attempt to synthesize 6-hydroxyphenalenone with improved yields. The usual three-carbon source in this reaction is glycerol, which gives the desired product with 6% yields. The reaction with malondialdehyde bis(diethyl acetal) also produces the desired product with similarly poor yields. X-ray analysis of the major product was undertaken to verify the chemical structure and to explain the downfield shift of 1.1 p.p.m. from the expected value of 7.0 p.p.m. of the aromatic H atoms H14 and H26 in the NMR spectrum (Callegari & Edwards, unpublished results).



The X-ray structure of the title compound is consistent with the chemical structure deduced from NMR analysis. The basis for the NMR shift is apparent from the crystal structure. The distance between H atoms H14 and H26 (2.09 Å) and the distances from these H atoms to the centers of the phenolic rings of the opposite naphthalene components (3.96 and 4.26 Å, respectively) are short enough to produce the downfield shift of 1.1 p.p.m. from to 8.1 p.p.m. observed in the NMR spectrum. These H atoms, located by difference Fourier synthe-

sis, are essentially coplanar ($\pm 1^\circ$) with the phenol rings to which they are attached, as shown in Table 2.

The most pronounced feature of the crystal structure is the distortion and asymmetry. A symmetric reference structure was generated by the molecular-modeling package *INSIGHTII/DISCOVER* (version 2.90; Biosym Technologies, San Diego, CA, USA) with all nonbonded interactions ignored in order to examine the distortion. The aromatic H atoms H14 and H26 were 0.91 Å apart in the nonphysical (no steric effects) reference structure, while they were found to be 2.09 Å apart in the crystal structure. This difference between the reference and experimental distances is the result of three coordinated deformations: a distortion of the bicyclic [3.3.1] methanodioxocin connector between the two naphthalene components, a twist of the naphthalene components, and splay between the naphthalene components.

The distortion of the connector, while small, is not random and does accentuate other alterations. The twist of the naphthalene components is the most noticeable deformation. The dihedral angles C2—C3—C22—C26 and C8—C9—C10—C14 are $-169.9(5)$ and $175.7(4)^\circ$, respectively. The twists of the two naphthalene components are of opposite handedness, which displaces H14 and H26 in the same direction (into the plane of the paper in Fig. 1) and away from each other. The splay between the two naphthalene components is also increased, as shown by the fact that the C4—C3—C22 and C4—C9—C10 bond angles are greater than 120° [$124.5(3)$ and $125.2(4)^\circ$, respectively], while the O1—C2—C19 and O7—C8—C13 bond angles are less than 120° [$113.4(4)$ and $112.7(4)^\circ$, respectively]. The connector and ring dihedral angles and the splay bond angles are listed in Table 2. None of the deformations is spectacular, but this is not necessary given the relatively small displacement needed and the distance of several bonds over which most distortions act.

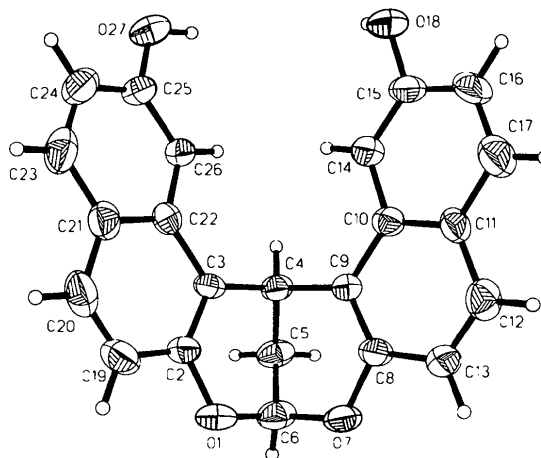


Fig. 1. Displacement ellipsoid plot (50% probability) with atom-numbering scheme. H atoms bear the same number as the atom to which they are bonded.

The unit cell contains portions of 12 molecules. Fig. 2 shows complete representations of these molecules placed about their common unit cell to illustrate the intricacies of the packing. The centrosymmetric space group $P2_1/c$ requires equal amounts of the two conformational isomers. The conformation of the molecule at (x, y, z) , as defined by the atomic coordinates listed in Table 1, is the same as that of the molecule at $(-x, y + \frac{1}{2}, -z + \frac{1}{2})$, while the conformation of that at $(-x, -y, -z)$ is the same as that at $(x, -y + \frac{1}{2}, z + \frac{1}{2})$. The chevron-shaped molecules are stacked on top of one another along the z axis. Four stacks of two antiparallel pairs are present. The CH_2 (C4—C5—C6) bridge points down in the two stacks based on (x, y, z) and $(x, -y + \frac{1}{2}, z + \frac{1}{2})$, but up in the two stacks based on $(-x, y + \frac{1}{2}, -z + \frac{1}{2})$ and $(-x, -y, -z)$ when the structure is viewed down the $+z$ axis. Both the conformation of the molecule and the orientation of the oxocine (O1 and O7) and phenolic (O18 and O27) O atoms, as defined by the direction of the vector from C4 to C6, alternate between adjacent molecules within each stack. This orientation reversal stacks the oxocine and phenolic O atoms of neighboring molecules roughly on top of each other and places all O atoms, along with all hydrogen bonds, near the planes $y = \pm \frac{z}{2}$. Two intermolecular hydrogen bonds per molecule occur between O18 and symmetry-related OH27 and OH18 hydroxy groups, generated by the transformation $(-x, 1 - y, -z)$. The hydrogen bond between O18 and OH27 (2.84 Å, 172.9°) is stronger than that between O18 and OH18 (3.09 Å, 152.4°).

The crystallographic coordinates listed in Table 1 were input into the semi-empirical package *MOPAC* 6.0 (Stewart, 1983) to see how the geometry would optimize in the absence of packing forces. It was thought that an isolated molecule might adjust the hinge angles between the two naphthalene components (C3—C4—C9 and O1—C6—O7), but this was not the case. The distance between the H atoms H14 and H26 decreased slightly from 2.09 Å to 2.01 Å. The crystal and *MOPAC* structures are basically quite similar.

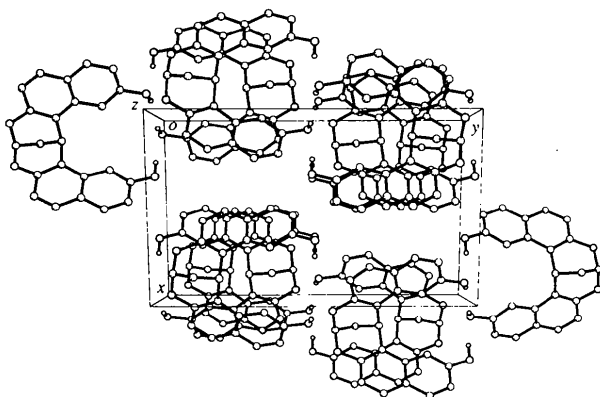


Fig. 2. Packing diagram viewed down the $+z$ axis.

Experimental

The title compound was prepared by the reaction of malondi-aldehyde bis(diethyl acetal) and 2,7-dihydroxynaphthalene. Details of the synthesis will be provided upon request (Callegari & Edwards, unpublished results).

Crystal data

$\text{C}_{23}\text{H}_{16}\text{O}_4$	Cu $K\alpha$ radiation
$M_r = 356.40$	$\lambda = 1.5418 \text{ \AA}$
Monoclinic	Cell parameters from 16 reflections
$P2_1/c$	$\theta = 4.2\text{--}27.7^\circ$
$a = 10.458 (2) \text{ \AA}$	$\mu = 0.789 \text{ mm}^{-1}$
$b = 16.969 (3) \text{ \AA}$	$T = 298 \text{ K}$
$c = 9.739 (2) \text{ \AA}$	Prism
$\beta = 104.93 (3)^\circ$	$0.20 \times 0.12 \times 0.10 \text{ mm}$
$V = 1670.0 (6) \text{ \AA}^3$	Amber
$Z = 4$	
$D_x = 1.417 \text{ Mg m}^{-3}$	

Data collection

Siemens $R3m/V$ diffractometer	$\theta_{\text{max}} = 50^\circ$
$\theta/2\theta$ scans	$h = -10 \rightarrow 10$
Absorption correction: none	$k = 0 \rightarrow 16$
1708 measured reflections	$l = 0 \rightarrow 9$
1708 independent reflections	3 standard reflections monitored every 100 reflections
1283 observed reflections	intensity decay: 1%
$[F > 3\sigma(F)]$	

Refinement

Refinement on F	$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
$R = 0.0561$	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
$wR = 0.0730$	Extinction correction:
$S = 1.25$	$F^* = F[1 + (0.002\chi \times F^2/\sin 2\theta)]^{-1/4}$
1283 reflections	Extinction coefficient:
251 parameters	$\chi = 0.017 (2)$
Only positions of H18 and H27 refined	Atomic scattering factors from <i>SHELXTL-Plus</i> (Sheldrick, 1990)
$w = 1/[\sigma^2(F) + 0.0021F^2]$	
$(\Delta/\sigma)_{\text{max}} = 0.001$	

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

U_{iso} for H atoms, $U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$ for all others.

	x	y	z	$U_{\text{iso}}/U_{\text{eq}}$
O1	0.2782 (3)	0.9247 (2)	0.1933 (4)	0.058 (1)
C2	0.3249 (4)	0.8495 (3)	0.2383 (5)	0.039 (2)
C3	0.2684 (4)	0.7816 (2)	0.1741 (4)	0.031 (2)
C4	0.1421 (4)	0.7891 (2)	0.0543 (4)	0.034 (2)
C5	0.1519 (5)	0.8648 (2)	-0.0273 (5)	0.043 (2)
C6	0.1662 (5)	0.9316 (3)	0.0745 (5)	0.047 (2)
O7	0.0521 (3)	0.9398 (2)	0.1291 (3)	0.049 (1)
C8	-0.0145 (5)	0.8713 (3)	0.1500 (5)	0.038 (2)
C9	0.0209 (4)	0.7972 (2)	0.1151 (4)	0.031 (2)
C10	-0.0600 (4)	0.7334 (2)	0.1387 (4)	0.035 (2)
C11	-0.1632 (4)	0.7472 (3)	0.2062 (5)	0.040 (2)
C12	-0.1923 (5)	0.8252 (3)	0.2393 (5)	0.048 (2)
C13	-0.1209 (5)	0.8864 (3)	0.2088 (5)	0.046 (2)
C14	-0.0405 (5)	0.6542 (3)	0.0988 (5)	0.045 (2)
C15	-0.1176 (5)	0.5947 (3)	0.1280 (6)	0.060 (2)
C16	-0.2167 (5)	0.6077 (3)	0.1982 (6)	0.069 (3)

C17	-0.2385 (5)	0.6830 (3)	0.2361 (6)	0.063 (2)
O18	-0.1003 (4)	0.5172 (2)	0.0880 (5)	0.083 (2)
C19	0.4340 (5)	0.8502 (3)	0.3582 (6)	0.054 (2)
C20	0.4882 (5)	0.7811 (3)	0.4135 (5)	0.053 (2)
C21	0.4412 (4)	0.7088 (3)	0.3490 (5)	0.043 (2)
C22	0.3336 (4)	0.7081 (3)	0.2229 (5)	0.034 (2)
C23	0.5019 (5)	0.6356 (3)	0.4017 (5)	0.054 (2)
C24	0.4630 (5)	0.5661 (3)	0.3315 (6)	0.057 (2)
C25	0.3623 (5)	0.5667 (3)	0.2054 (5)	0.045 (2)
C26	0.2987 (4)	0.6349 (2)	0.1528 (5)	0.035 (2)
O27	0.3329 (4)	0.4956 (2)	0.1386 (4)	0.065 (2)
H14	0.0271	0.6426	0.0512	0.080
H26	0.2290	0.6332	0.0667	0.080
H18	-0.072 (6)	0.508 (4)	0.021 (7)	0.080
H27	0.256 (6)	0.496 (3)	0.063 (6)	0.080

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles, and torsion angles have been deposited with the IUCr (Reference: SZ1012). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 2. Selected geometric parameters (°)

Bond angles			
Splay			
C4—C3—C22	124.5 (3)	C4—C9—C10	125.2 (4)
O1—C2—C19	113.4 (4)	O7—C8—C13	112.7 (4)
Hinge			
C3—C4—C9	110.5 (3)	O1—C6—O7	107.3 (4)
Dihedral angles			
Connector			
C22—C3—C4—C9	98.8 (5)	C3—C4—C9—C10	-93.3 (4)
O1—C2—C3—C22	174.4 (5)	O7—C8—C9—C10	-177.9 (3)
C6—O1—C2—C19	-178.7 (5)	C6—O7—C8—C13	177.6 (3)
C2—O1—C6—C5	-28.1 (6)	C5—C6—O7—C8	34.0 (5)
O1—C2—C3—C4	-4.7 (7)	O7—C8—C9—C4	1.8 (6)
C6—O1—C2—C3	0.1 (7)	C6—O7—C8—C9	-2.8 (5)
Naphthalene components			
C2—C3—C22—C26	-169.9 (5)	C8—C9—C10—C14	175.7 (4)
C4—C3—C22—C26	9.1 (7)	C4—C9—C10—C14	-4.1 (6)
C20—C21—C22—C26	173.4 (5)	C14—C10—C11—C12	-175.9 (4)
C23—C21—C22—C3	177.0 (5)	C9—C10—C11—C17	-176.6 (4)
C19—C2—C3—C22	-7.0 (8)	C13—C8—C9—C10	1.6 (6)
C3—C2—C19—C20	0.7 (9)	C9—C8—C13—C12	2.5 (6)
C2—C19—C20—C21	3.1 (9)	C11—C12—C13—C8	-2.8 (6)
C19—C20—C21—C23	176.7 (5)	C17—C11—C12—C13	-179.2 (4)
C20—C21—C23—C24	-174.4 (5)	C12—C11—C17—C16	176.5 (4)
C21—C23—C24—C25	0.3 (9)	C15—C16—C17—C11	-0.1 (7)
C23—C24—C25—C26	-2.2 (9)	C14—C15—C16—C17	1.3 (8)
C24—C25—C26—C22	1.0 (8)	C10—C14—C15—C16	-0.6 (7)
C3—C22—C26—C25	-178.7 (5)	C9—C10—C14—C15	177.7 (4)
H atoms H14 and H26			
C21—C22—C26—H26	-178.0 (3)	C11—C10—C14—H14	178.7 (2)
C24—C25—C26—H26	-179.0 (4)	H14—C14—C15—C16	179.4 (4)

All H atoms were located by difference Fourier synthesis. Only the positions of H18 and H27 were refined, as free rotation is possible around the C—OH bonds.

Data collection: *SHELXTL-Plus* (Sheldrick, 1990). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus* and *INSIGHTII/DISCOVER* (Biosym Technologies, San Diego, CA, USA).

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1-(*p*-Carbamoylphenyl)-3,3-dimethyl-triazene, an Antitumour Agent

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Abstract

In the crystal structure of the title compound, 4-(3,3-dimethyl-1-triazeno)benzamide, C₉H₁₂N₄O, (2), the N=N double bond [1.282 (8) Å] is 0.030 Å shorter than the N—N single bond [1.312 (8) Å], but both bonds are shorter than an isolated N—N single bond suggesting that there is double-bond character in each N—N bond, although it is unequally distributed. The molecule adopts a *trans* geometry around the N=N bond, but there is a significant deviation from planarity between the benzene ring and the plane of the triazene moiety. Compound (2) forms chains in the solid state in which the molecules are linked by C=O...H—N hydrogen bonds between carbamoyl groups. These chains are cross-linked into sheets by hydrogen bonding between the second N—H moiety and triazene units in adjacent chains.

Comment

1-Aryl-3,3-dialkyltriazenes, Ar—N=N—NR₂, represent a well known class of antitumour agents (Vaughan, 1990). The most familiar member of this drug family is DTIC [5-(3,3-dimethyltriazene-1-yl)imidazole-4-