

and H) 1.02; U_{ij} (C and N) 1.12; bond lengths (not involving H) 1.18; angles (not involving H) 0.80. **G** finds the methyl group to be disordered with occupation factors of 0.6 and 0.4. The methyl hydrogens found by **H** are those for which **G** finds the highest occupation factor. A difference map with structure factors from **H**'s final refinement showed peaks close to the positions of H(77) and H(88) but none corresponding to H(99).

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The Structure of 2-Methyl-2H-naphtho[1,8-*de*]triazine

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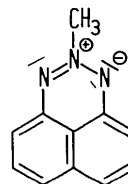
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Abstract. $C_{11}H_9N_3$, $M_r = 183.2$, monoclinic, space group $P2_1/a$, $a = 15.385$ (7), $b = 17.344$ (13), $c = 3.955$ (2) Å, $\gamma = 122.12$ (3)°, $Z = 4$, $D_o = 1.35$, $D_x = 1.36$ Mg m⁻³, μ (Cu $K\alpha$) = 0.689 mm⁻¹. Refinement with 1690 independent reflexions yielded an R of 0.065 for 1070 observed reflexions ($I > 2\sigma_I$). The molecule shows $mm2$ symmetry. The CH₃ group is disordered by rotation about the C–N bond resulting in two statistically occupied positions of the H atoms. In this way the disordered CH₃ group also fulfils approximately the $mm2$ symmetry. Bond lengths and angles are in agreement with those given by Hazell & Mariezcurrena [*Acta Cryst.* (1980). **B36**, 3140–3142].

Introduction. To compare the structures of sulfur-diimides with molecules containing the zwitterionic N_3R moiety, which is isoelectronic with the SN_2 unit, we have, among other structures (Gieren, Lamm, Haddon, Kaplan, Perkins & Flowerday, 1980), carried out an X-ray analysis of the title compound (I) (Perkins, 1964). When our analysis was finished we

heard from A. Hazell and R. Mariezcurrena, that they had also solved the structure of (I) (Hazell & Mariezcurrena, 1980).



(I)

(I) crystallizes from light petroleum in the form of blue-black elongated plates in the monoclinic space group $P2_1/a$. The crystal was 0.10 × 0.03 × 0.85 mm. To determine the lattice constants and for data collection a punch-tape-controlled diffractometer (Siemens AED) was used. Transformation into the space group $P2_1/c$ gives the lattice constants $a = 15.929$ (14), $b = 3.955$ (2), $c = 15.385$ (7) Å, $\beta = 112.76$ (5)°. These values are within the limits of error

$[2\sigma_A, \sigma_A = (\sigma_1^2 + \sigma_2^2)^{0.5}]$ identical with those of Hazell & Mariezcurrena (1980). With Cu $K\alpha$ radiation the intensities of 1690 independent reflexions ($\theta_{\max} = 70^\circ$, $\theta/2\theta$ scan, five-point measurement) were determined. 620 of these were unobserved ($I < 2\sigma_I$).

The structure was solved by direct methods with PHASDT (Zechmeister, 1969), part of our modified version (Hoppe, Gassmann & Zechmeister, 1970) of XRAY 67 (Stewart, 1967), which was mainly used for the other calculations. An E Fourier synthesis calculated with the 130 highest E values, taking the signs of the phase set with the second best figure of merit, showed the positions of all non-hydrogen atoms. During the least-squares refinement, first with isotropic and then anisotropic thermal parameters, difference syntheses revealed all the H atoms including those of a second orientation of the CH_3 group. The occupancy factors for the H atoms in the two orientations were estimated as 0.6 [H(7),H(8),H(9)] and 0.4 [H(77),H(88),H(99)] taking into account the peak heights in the difference maps and also the assumption that H atoms should have almost equal temperature parameters. Refinement (full-matrix, unit weights) of the positional and thermal parameters (H isotropic, all other atoms anisotropic) yielded $R = \sum |F_o| - |F_c| / \sum |F_o| = 0.065$. In the last stages 15 reflexions with $\Delta F = ||F_o| - |F_c|| > 3$ were not used for the calculation of parameter shifts. The unobserved reflexions ($|F_o| = 4\sigma_F$) were only included if $|F_o| - |F_c| < 0$. Scattering factors for N and C were taken from *International Tables for X-ray Crystallography* (1962) and for H from Stewart, Davidson & Simpson (1965). The positional parameters of all atoms and the isotropic temperature parameters are given in Table 1.† The atomic coordinates given there (\mathbf{r}) can be transformed into those of Hazell & Mariezcurrena (1980) (\mathbf{r}^*) with the following equation:

$$\mathbf{r}^* = \begin{pmatrix} 0 & -1 & 0 \\ 0 & 0 & -1 \\ 1 & -1 & 0 \end{pmatrix} \cdot \mathbf{r} + \begin{pmatrix} 0.5 \\ 1 \\ 0 \end{pmatrix}.$$

Discussion. The molecule is planar with the exception of H atoms of the disordered CH_3 group. The molecule shows $mm2$ symmetry, which is almost fulfilled by two statistical orientations of the CH_3

Table 1. Positional parameters and isotropic temperature parameters (equivalent values for non-hydrogen atoms)

The e.s.d.'s given in parentheses refer to the least significant digit.

	x	y	z	B_{eq}/B (\AA^2)
N(1)	0.3914 (3)	0.3866 (2)	0.7548 (10)	4.3 (2)
N(2)	0.2492 (3)	0.3788 (2)	0.4837 (10)	4.4 (2)
N(3)	0.3359 (3)	0.4170 (2)	0.6412 (10)	4.3 (2)
C(1)	0.2044 (3)	0.2857 (3)	0.4183 (11)	3.7 (2)
C(2)	0.1113 (4)	0.2374 (4)	0.2584 (13)	4.8 (2)
C(3)	0.0672 (4)	0.1436 (4)	0.2076 (14)	4.7 (2)
C(4)	0.1148 (4)	0.0999 (4)	0.3109 (14)	4.7 (3)
C(5)	0.2667 (5)	0.1092 (4)	0.5959 (14)	5.5 (3)
C(6)	0.3583 (5)	0.1602 (4)	0.7486 (14)	5.3 (3)
C(7)	0.4020 (4)	0.2537 (4)	0.8049 (12)	4.8 (2)
C(8)	0.3496 (3)	0.2932 (3)	0.6961 (11)	3.6 (2)
C(9)	0.2543 (3)	0.2413 (3)	0.5319 (10)	3.4 (2)
C(10)	0.2096 (4)	0.1465 (3)	0.4749 (11)	4.0 (2)
C(11)	0.3817 (5)	0.5156 (4)	0.7087 (20)	5.3 (3)
H(1)	0.080 (3)	0.269 (3)	0.191 (12)	4 (1)
H(2)	-0.003 (3)	0.109 (3)	0.106 (12)	5 (1)
H(3)	0.398 (4)	0.136 (3)	0.814 (14)	6 (1)
H(4)	0.469 (3)	0.282 (3)	0.905 (12)	4 (1)
H(5)	0.085 (4)	0.045 (4)	0.278 (16)	8 (2)
H(6)	0.239 (4)	0.055 (3)	0.576 (14)	6 (1)
H(7)	0.448 (6)	0.545 (5)	0.859 (21)	3 (2)
H(8)	0.324 (7)	0.514 (6)	0.764 (28)	4 (2)
H(9)	0.415 (7)	0.553 (6)	0.487 (25)	6 (2)
H(77)	0.455 (9)	0.545 (7)	0.677 (33)	2 (2)
H(88)	0.366 (10)	0.544 (8)	0.898 (32)	4 (3)
H(99)	0.355 (11)	0.560 (9)	0.584 (39)	6 (3)

group. The two orientations are roughly such that one H atom is nearly in the molecular plane. The only exceptions from the molecular symmetry concerning bond distances and angles are the angles defined by H(1) and H(4), but it is not clear if the differences of 3.1 and 2.8 σ_A respectively are due to systematic errors. Fig. 1 shows the bond distances (not corrected for thermal vibration) and angles averaged by molecular symmetry compared with the values obtained by Hazell & Mariezcurrena (1980). Within the limits of error ($\Delta \leq 1.6 \sigma_A$) both results are identical.

The mean N—N distance in the zwitterionic N_3R unit is 1.295 \AA and the C—N distance in the triazine skeleton 1.405 \AA . The N—N distances are elongated by only 0.06 \AA compared with a N=N double bond. The relatively long C—N distances of the N_3R system to the naphthalene skeleton, which are only 0.04 \AA shorter than a C—N single bond of type sp^2-sp^2 , show only a small π interaction between these two fragments.

In the crystal structure (Fig. 2) the molecules form columnar stacks in the \mathbf{c} direction in which the distance between the molecular planes is 3.482 \AA . Within the stacks the molecules are inclined by 28° to the ab plane. As can be seen from Fig. 2 the CH_3 groups protrude into gaps between the columns. These gaps allow the two statistical orientations of the CH_3 groups,

† Listings of observed and calculated structure factor amplitudes, of the anisotropic thermal parameters of the non-hydrogen atoms and a list of bond distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35570 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

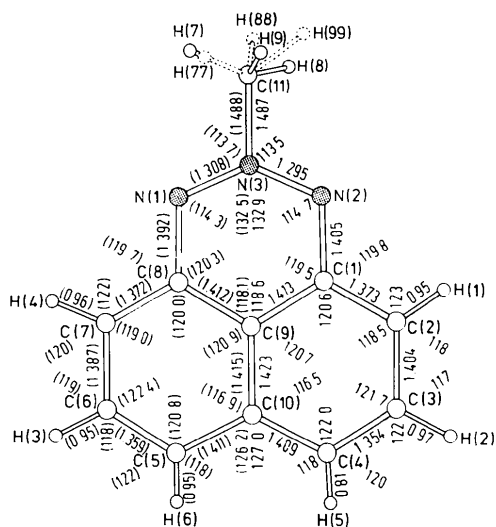


Fig. 1. Molecular structure of 2-methyl-2H-naphtho[1,8-*de*]triazine. The bond lengths and angles are mean values averaged over the molecular symmetry and those in parentheses are the corresponding values of Hazell & Mariezcurrena (1980). The e.s.d.'s are between 0.006 (0.007) and 0.010 (0.011) Å for bond distances and between 0.4 (0.6) and 0.6 (0.9)° for bond angles with the exception of those defined by H atoms, where they are 0.06 (0.05–0.10) Å and 3–5 (4–7)° respectively.

for both CH₃ group orientations show almost identical intermolecular environments in the crystal packing.

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Structure of 3,5-Diformylbicyclo[5.4.1]dodeca-2,5,7,9,11-pentaene

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Abstract. C₁₄H₁₂O₂, orthorhombic, *Pccn*, $a = 13.738$ (2), $b = 13.472$ (2), $c = 11.658$ (2) Å, $Z = 8$, $D_c = 1.307$, $D_m = 1.306$ Mg m⁻³ (floatation), $\mu(\text{Mo } K\alpha) = 0.094$ mm⁻¹, $F(000) = 896$. The structure has been solved by an iterative least-squares rigid-body process, and refined to $R = 0.062$ for 1243 independent reflexions. Bond lengths of the structure indicate an open

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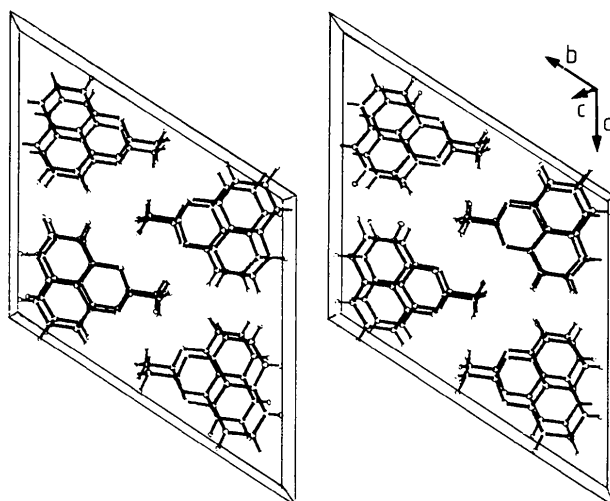


Fig. 2. Stereoscopic view of the crystal packing. At the CH₃ group only one statistical position for the H atoms is shown.

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14 π -electron system. Of the six strongest intermolecular interactions, four are of the type O...H, one is C...C and the last is C...H.

Introduction. The present analysis forms part of a structural study of bridged annulenes. We report here the structure of the title compound (hereafter ALD).

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