

Moore & Rege, 1986). The structure of ingenol triacetate, a modified derivative of the natural product ingenol-3-hexadecanoate, has been examined (Zechmeister, Brandl & Hoppe, 1970; Paquette, Nitz, Ross & Springer, 1984). There are many examples in the literature of cycloheptane-cyclopentene fused rings (e.g. McPhail & Sim, 1973), especially as sesquiterpenoids with an additional lactone ring. Ring conformations have been discussed by Toromanoff (1980) and asymmetry parameters are described by Ladd & Palmer (1985).

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### Structure of *N,N*-Dimethyl-9-anthrylacamide\*

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**Abstract.**  $C_{18}H_{17}NO$ ,  $M_r = 263.33$ , monoclinic,  $P2_1/n$ ,  $a = 13.124$  (6),  $b = 9.636$  (3),  $c = 12.203$  (5) Å,  $\beta = 113.84$  (2)°,  $V = 1412$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.24$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 0.083$  mm<sup>-1</sup>,  $F(000) = 263$ ,  $T = 295$  K,  $R = 0.055$  for 1200 reflections [ $|F_o| > 3\sigma(F_o)$ ]. The structure consists of two planes of an anthracene  $\pi$  system and an *N,N*-dimethylacetamido moiety. The planes are nearly perpendicular because of steric repulsion between the H atoms at C(1) and C(8) and the *N,N*-dimethylcarbamoylmethyl group, the dihedral angle being 82.1 (1)°. More steric repulsion between the 9-anthryl and dimethylamino groups might give the conformer, with these groups on the opposite sides of the CH<sub>2</sub>—CO bond axis.

**Experimental.** The dehydration coupling of 9-anthrylacetic acid with dimethylamine gave the title compound, which will be reported in detail elsewhere. Single crystals of the compound were prepared by slow evaporation of a dichloromethane–hexane solution. A pale-yellow plate crystal with dimensions 0.44 × 0.52 × 0.26 mm was used for the X-ray diffractometry. Enraf–Nonius CAD-4 diffractometer operating at 50 kV/26 mA, graphite-monochromated

Mo  $K\alpha$  radiation. Accurate cell parameters by least-squares fit for 25 reflections with  $15 < 2\theta < 25^\circ$ . Intensity data collected by  $\theta$ – $2\theta$  scan, scan rates varied 1.2–4.1° min<sup>-1</sup>, scan range (°) 1.0 + 0.5 tan  $\theta$ ,  $2\theta_{\text{max}} = 60^\circ$ ,  $h = -18$  to 18,  $k = 0$  to 13,  $l = 0$  to 17. Three standard reflections monitored every 7200 s showed no crystal movement or decay. Intensities corrected for absorption effects by numerical Gaussian integration method, transmission factor range = 0.980–0.963; 4625 reflections measured, 1200 reflections with  $|F_o| > 3\sigma(F_o)$  observed.

The structure was solved by direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), and refined on  $F$  by the full-matrix least-squares technique (*RADIEL*; Coppens, Guru Row, Leung, Stevens, Becker & Yang, 1979). All non-H atoms refined anisotropically. H atoms were located by difference synthesis and refined isotropically. Weighting scheme  $w = [\sigma(F_o)^2 + (0.040|F_o|)^2]^{-1}$ ; extinction correction applied yielding a secondary-extinction coefficient  $g$  of  $3.1$  (3) × 10<sup>-4</sup> (Zachariasen, 1967),  $R(F) = 0.055$ ,  $wR(F) = 0.073$ , GOF = 1.35 for 1200 independent reflections, 250 parameters refined. Maximum and minimum peak heights in difference Fourier map were 0.19 and  $-0.13$  e Å<sup>-3</sup>, max. shift/e.s.d. = 0.51. Atomic scattering factors and anomalous-scattering corrections from *International Tables for X-ray Crys-*

\* 2-(9-Anthryl)-*N,N*-dimethylacetamide.

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Table 1. Fractional coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2$ ) for the non-H atoms

	$B_{eq} = (4/3)\sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$			
	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}$
O	8625 (2)	-905 (2)	8169 (2)	6.3 (1)
N	7758 (2)	101 (3)	9204 (2)	5.7 (1)
C(1)	9164 (3)	2314 (3)	7242 (3)	5.3 (1)
C(2)	10040 (3)	3077 (4)	7329 (4)	6.2 (1)
C(3)	10425 (4)	3057 (4)	6416 (4)	7.4 (2)
C(4)	9908 (3)	2258 (4)	5427 (5)	7.2 (2)
C(5)	7007 (4)	-1122 (4)	3201 (3)	6.3 (1)
C(6)	6130 (4)	-1910 (4)	3108 (4)	6.7 (1)
C(7)	5716 (3)	-1887 (4)	3997 (4)	6.3 (1)
C(8)	6176 (3)	-1052 (3)	4965 (3)	5.3 (1)
C(9)	7631 (2)	661 (3)	6124 (3)	4.3 (1)
C(10)	8429 (3)	581 (4)	4313 (3)	6.0 (1)
C(11)	8567 (2)	1455 (3)	6229 (3)	4.4 (1)
C(12)	8965 (3)	1434 (3)	5308 (3)	5.3 (1)
C(13)	7114 (2)	-192 (3)	5122 (3)	4.5 (1)
C(14)	7519 (3)	-229 (3)	4210 (3)	5.2 (1)
C(15)	7228 (3)	734 (4)	7112 (3)	4.7 (1)
C(16)	7931 (3)	-90 (3)	8205 (3)	4.6 (1)
C(m1)	8419 (6)	-719 (6)	10625 (4)	7.8 (2)
C(m2)	6864 (4)	940 (6)	9265 (5)	7.5 (2)

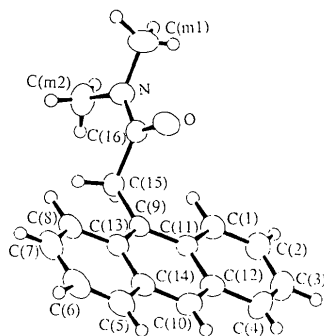
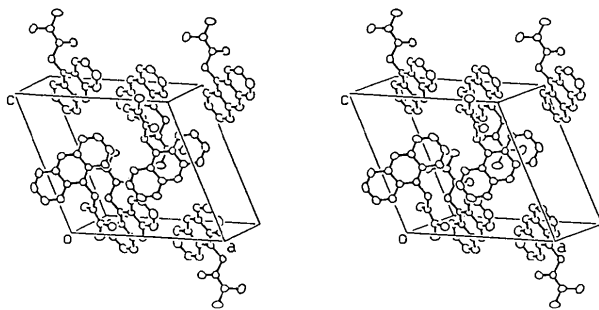
Fig. 1. ORTEP drawing of *N,N*-dimethyl-9-anthrylacetamide with the atomic numbering scheme; thermal ellipsoids are at the 30% probability level.

Fig. 2. A stereoscopic view of the crystal structure.

*tallography* (1974, Vol. IV). All the calculations were carried out on a HITAC M680 computer of the Institute for Molecular Science. Deviations from the least-squares planes and their e.s.d.'s were calculated by the procedure proposed by Ito (1981).

Table 2. Relevant interatomic distances ( $\text{\AA}$ ) and angles ( $^\circ$ )

O—C(16)	1.217 (4)	C(6)—C(7)	1.396 (8)
N—C(16)	1.340 (5)	C(7)—C(8)	1.353 (5)
N—C(m1)	1.464 (5)	C(8)—C(13)	1.431 (5)
N—C(m2)	1.451 (7)	C(9)—C(11)	1.408 (4)
C(1)—C(2)	1.332 (5)	C(9)—C(13)	1.400 (4)
C(1)—C(11)	1.430 (4)	C(9)—C(15)	1.502 (5)
C(2)—C(3)	1.398 (8)	C(10)—C(12)	1.398 (4)
C(3)—C(4)	1.360 (6)	C(10)—C(14)	1.390 (5)
C(4)—C(12)	1.427 (6)	C(11)—C(12)	1.418 (5)
C(5)—C(6)	1.345 (6)	C(13)—C(14)	1.414 (5)
C(5)—C(14)	1.428 (5)	C(15)—C(16)	1.506 (4)
C(16)—N—C(m1)	118.2 (3)	C(1)—C(11)—C(12)	116.4 (3)
C(16)—N—C(m2)	124.1 (3)	C(9)—C(11)—C(12)	120.5 (3)
C(m1)—N—C(m2)	117.3 (4)	C(4)—C(12)—C(10)	121.8 (4)
C(2)—C(1)—C(11)	122.8 (4)	C(4)—C(12)—C(11)	119.6 (3)
C(1)—C(2)—C(3)	120.6 (4)	C(10)—C(12)—C(11)	118.6 (3)
C(2)—C(3)—C(4)	120.2 (4)	C(8)—C(13)—C(9)	123.0 (3)
C(3)—C(4)—C(12)	120.5 (5)	C(8)—C(13)—C(14)	117.2 (3)
C(6)—C(5)—C(14)	120.1 (4)	C(9)—C(13)—C(14)	119.8 (3)
C(5)—C(6)—C(7)	120.9 (4)	C(5)—C(14)—C(10)	120.3 (4)
C(6)—C(7)—C(8)	120.8 (4)	C(5)—C(14)—C(13)	119.8 (3)
C(7)—C(8)—C(13)	121.2 (4)	C(10)—C(14)—C(13)	119.9 (3)
C(11)—C(9)—C(13)	119.8 (3)	C(9)—C(15)—C(16)	113.5 (3)
C(11)—C(9)—C(15)	118.6 (3)	O—C(16)—N	121.8 (3)
C(13)—C(9)—C(15)	121.6 (3)	O—C(16)—C(15)	120.5 (3)
C(12)—C(10)—C(14)	121.4 (4)	N—C(16)—C(15)	117.7 (3)
C(1)—C(11)—C(9)	123.1 (3)		

Atomic parameters are given in Table 1.\* Relevant interatomic distances and angles are presented in Table 2. Fig. 1 shows the ORTEP (Johnson, 1965) drawing with the atom numbering. A stereoscopic view of the crystal structure is shown in Fig. 2.

**Related literature.** The crystal structure of the related compound, 9-anthryl- $\text{CH}_2\text{CH}=\text{C}(\text{CHO})$ -9-anthryl, was reported by Axelsson, Becker, Skelton, Sörenson & White (1988) and the angle between the planes of the former 9-anthryl  $\pi$  system and the olefinic double bond was 86 or 74°.

\* Lists of deviations of atoms from the least-squares planes, structure factors, anisotropic thermal parameters for the non-H atoms, and positional and isotropic thermal parameters of the H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53345 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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