N(1)	0.5635 (3)	−0.2274 (3)	0.8785 (2)	0.0282 (9)
N(2)	0.4598 (3)	−0.2989 (3)	1.4089 (2)	0.0250 (8)
C(1)	0.3085 (3)	−0.0436 (3)	0.9502 (2)	0.0224 (9)
C(2)	0.3941 (3)	−0.0836 (3)	0.8955 (2)	0.0233 (9)
C(3)	0.4702 (3)	−0.1829 (3)	0.9320 (2)	0.0230 (9)
C(4)	0.4624 (3)	−0.2449 (3)	1.0203 (2)	0.0254 (10)
C(5)	0.3789 (3)	−0.2006 (3)	1.0757 (2)	0.0276 (10)
C(6)	0.3025 (3)	−0.1011 (3)	1.0405 (3)	0.0268 (10)
C(7)	0.6564 (3)	−0.0679 (3)	1.5706 (2)	0.0222 (9)
C(8)	0.5680 (3)	−0.1586 (3)	1.5399 (2)	0.0223 (9)
C(9)	0.5533 (3)	−0.2045 (3)	1.4453 (2)	0.0219 (9)
C(10)	0.6260 (3)	−0.1644 (3)	1.3797 (2)	0.0249 (9)
C(11)	0.7123 (3)	−0.0703 (4)	1.4104 (3)	0.031 (1)
C(12)	0.7270 (3)	−0.0232 (3)	1.5051 (3)	0.029 (1)

Table 2. *Selected geometric parameters (Å, °)*

S(1)—O(1)	1.449 (2)	S(2)—O(7)	1.467 (2)
S(1)—O(2)	1.458 (2)	S(2)—C(7)	1.777 (3)
S(1)—O(3)	1.457 (2)	O(4)—C(4)	1.350 (4)
S(1)—C(1)	1.767 (3)	O(8)—C(10)	1.357 (4)
S(2)—O(5)	1.457 (2)	N(1)—C(3)	1.465 (4)
S(2)—O(6)	1.457 (2)	N(2)—C(9)	1.463 (4)
O(1)—S(1)—O(2)	111.7 (2)	S(1)—C(1)—C(2)	120.7 (2)
O(1)—S(1)—O(3)	113.8 (2)	S(1)—C(1)—C(6)	118.9 (2)
O(1)—S(1)—C(1)	107.1 (1)	N(1)—C(3)—C(2)	120.6 (3)
O(2)—S(1)—O(3)	109.7 (1)	N(1)—C(3)—C(4)	117.2 (3)
O(2)—S(1)—C(1)	107.3 (1)	O(4)—C(4)—C(3)	116.4 (3)
O(3)—S(1)—C(1)	107.0 (1)	O(4)—C(4)—C(5)	124.9 (3)
O(5)—S(2)—O(6)	114.3 (1)	S(2)—C(7)—C(8)	120.6 (2)
O(5)—S(2)—O(7)	112.0 (1)	S(2)—C(7)—C(12)	119.7 (3)
O(5)—S(2)—C(7)	105.8 (1)	N(2)—C(9)—C(8)	121.1 (3)
O(6)—S(2)—O(7)	111.3 (1)	N(2)—C(9)—C(10)	116.8 (3)
O(6)—S(2)—C(7)	106.2 (1)	O(8)—C(10)—C(9)	116.7 (3)
O(7)—S(2)—C(7)	106.6 (1)	O(8)—C(10)—C(11)	124.9 (3)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N(1)—H(4)···O(5^{i})	1.01 (3)	1.75 (3)	2.758 (4)	171 (3)
N(1)—H(5)···O(9^{ii})	0.94 (3)	1.93 (3)	2.869 (4)	173 (3)
N(1)—H(6)···O(3^{iii})	0.91 (3)	1.92 (3)	2.814 (4)	166 (3)
O(4)—H(7)···O(9^{iv})	0.80 (3)	1.95 (4)	2.723 (4)	165 (4)
N(2)—H(11)···O(3^{v})	0.92 (3)	1.94 (3)	2.852 (4)	167 (3)
N(2)—H(12)···O(6^{vi})	0.97 (3)	2.01 (3)	2.774 (4)	134 (3)
N(2)—H(13)···O(2^{vii})	0.93 (3)	1.93 (3)	2.800 (4)	156 (3)
O(8)—H(14)···O(1^{iii})	0.77 (3)	1.97 (3)	2.741 (3)	176 (4)
O(9)—H(16)···O(7^{viii})	0.81 (3)	1.93 (4)	2.734 (4)	169 (4)

Symmetry codes: (i) $x, y, z-1$; (ii) $x-\frac{1}{2}, -\frac{1}{2}-y, z-\frac{1}{2}$; (iii) $1-x, -y, 2-z$; (iv) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{5}{2}-z$; (v) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{5}{2}-z$; (vi) $1-x, -y, 3-z$; (vii) $\frac{1}{2}+x, -\frac{1}{2}-y, \frac{1}{2}+z$; (viii) $2-x, -y, 3-z$.

All H atoms were located on difference electron density maps and their positions refined with fixed isotropic B values equal to 1.2 times those of the attached atoms at the time of their inclusion, with bond distances O—H 0.77 (3)–0.85 (3), N—H 0.91 (3)–1.01 (3) and C—H 0.90 (3)–0.98 (3) Å.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1991). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1983). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, least-squares-planes data, torsion angles and intermolecular distances involving both H and non-H atoms have been deposited with the IUCr (Reference: BK1189). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Gilmore, C. J. (1983). *MITHRIL. Computer Program for the Automatic Solution of Crystal Structures from X-ray Data*. Department of Chemistry, University of Glasgow, Scotland.

Gunderman, B. J. & Squattrito, P. J. (1994). *Inorg. Chem.* **33**, 2924–2931.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*, revised edition. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1991). *TEXSAN. TEXRAY Structure Analysis Package*, revised edition. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Shubnell, A. J. & Squattrito, P. J. (1994). *Acta Cryst.* C**50**, 1296–1299.

Zachariasen, W. H. (1968). *Acta Cryst.* A**24**, 212–216.

Acta Cryst. (1996). C**52**, 942–944

A Photoproduct Derived from 9-Benzyl-Substituted Dibenzobarrelene†

THOMAS MATHEW,[a] S. AJAYA KUMAR,[a] SURESH DAS,[a] NIGAM P. RATH[b]* AND M. V. GEORGE[c]‡

[a]*Photochemistry Research Unit, Regional Research Laboratory (CSIR), Trivandrum 695 019, India,* [b]*Department of Chemistry, University of Missouri-St. Louis, St. Louis, MO 63121, USA, and* [c]*Radiation Laboratory, University of Notre Dame, Notre Dame, IN 46556, USA*

(*Received 28 July 1994; accepted 4 October 1995*)

Abstract

Photolysis of 11,12-dibenzoyl-9-benzyl-9,10-ethenoanthracene in the absence of oxygen, gives 9a,10-dibenzoyl-9-benzylidene-4b,9,9a,10-tetrahydroindeno[1,2-*a*]indene, $C_{37}H_{26}O_2$, (3). The structure of (3) has been unambiguously established through X-ray crystallographic analysis.

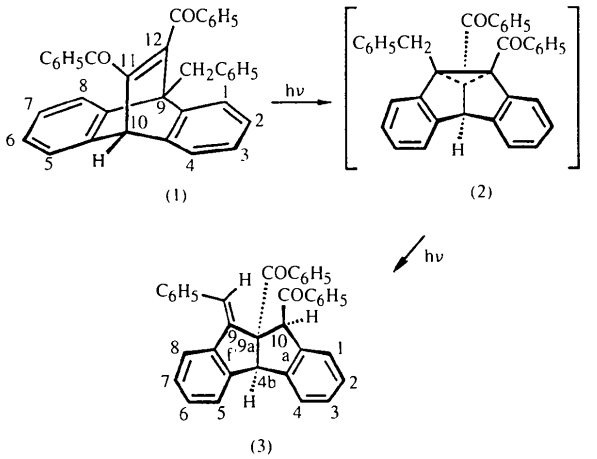
Comment

We reported earlier that the partial photolysis of 11,12-dibenzoyl-9-benzyl-9,10-ethenoanthracene, (1), in the absence of air, gives exclusively the 9-benzylidene-dibenzopentalene (3), derived from the dibenzosemibullvalene precursor (2) (Pratapan, Ashok, Cyr, Das & George, 1987).

† Contribution No. NDRL-3741 from the Notre Dame Radiation Laboratory and No. TTLT-PRU-53 from the Regional Research Laboratory (CSIR), Trivandrum 695 019, India.

‡ Also at the Regional Research Laboratory (CSIR), Trivandrum 695 019, India.

©1996 International Union of Crystallography
Printed in Great Britain – all rights reserved



In view of the implications on the regioselectivity exerted by the 9-benzyl substituent in the photorearrangement of (1), it was necessary to establish the structure of the photoproduct (3) unambiguously. The structure of (3) has now been determined by X-ray crystallographic analysis and confirms our earlier suggestion that the observed photoproduct (3) is a secondary product, derived from the initially formed dibenzosemibullvalene (2).

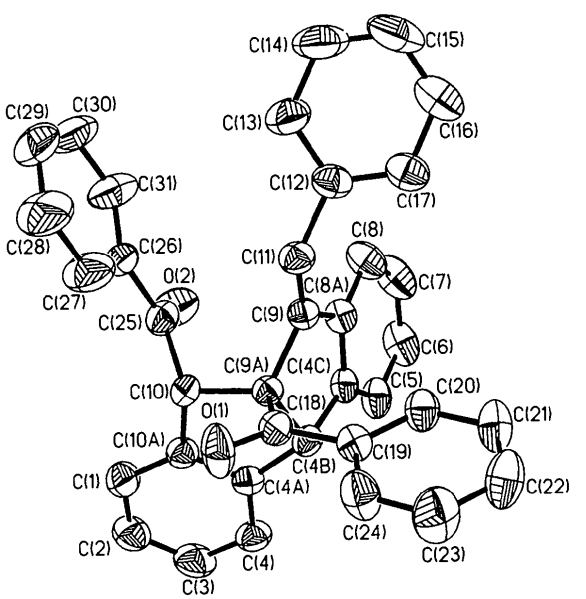


Fig. 1. Projection view of compound (3) showing 50% probability ellipsoids. Only one of the two molecules in the asymmetric unit is shown for clarity.

Experimental

Colorless crystals of the title compound (3) were obtained by recrystallization from a 1:1 mixture of chloroform and methanol.

Crystal data

$C_{37}H_{26}O_2$
M_r = 502.6
Cu $K\alpha$ radiation
λ = 1.54178 Å
Triclinic
$P\bar{1}$
a = 9.498 (2) Å
b = 14.443 (3) Å
c = 20.318 (4) Å
α = 78.56 (3)°
β = 85.43 (3)°
γ = 88.77 (3)°
V = 2723.0 (10) Å^3
Z = 4
D_x = 1.226 Mg m^{-3}
D_m not measured
Cell parameters from 25 reflections
θ = 7.5–12.5°
μ = 0.580 mm^{-1}
T = 298 K
Rhombic
0.4 × 0.3 × 0.2 mm
Colorless

Data collection

Siemens $P4$ diffractometer
$\omega/2\theta$ scans
Absorption correction: none
8843 measured reflections
7270 independent reflections
5594 observed reflections [$F > 3.0\sigma(F)$]
R_{int} = 0.0545
θ_{max} = 56.5°
$h = -1 \rightarrow 10$
$k = -15 \rightarrow 15$
$l = -22 \rightarrow 22$
3 standard reflections monitored every 50 reflections
intensity decay: <6%

Refinement

Refinement on F
R = 0.0571
wR = 0.0796
S = 1.01
5594 reflections
703 parameters
$w = 1/[\sigma^2(F) + 0.0040F^2]$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O(1)	0.1151 (2)	0.7178 (2)	0.3598 (1)	0.060 (1)
O(2)	−0.2444 (2)	0.9303 (2)	0.2363 (1)	0.063 (1)
C(1)	−0.2013 (3)	0.9373 (2)	0.3992 (2)	0.049 (1)
C(2)	−0.2968 (4)	0.9384 (2)	0.4542 (2)	0.057 (1)
C(3)	−0.3784 (4)	0.8604 (3)	0.4813 (2)	0.059 (1)
C(4)	−0.3675 (3)	0.7794 (2)	0.4542 (2)	0.046 (1)
C(4A)	−0.2730 (3)	0.7788 (2)	0.3984 (1)	0.036 (1)
C(4B)	−0.2455 (3)	0.7009 (2)	0.3588 (1)	0.034 (1)
C(4C)	−0.3527 (3)	0.6936 (2)	0.3090 (1)	0.036 (1)
C(5)	−0.4973 (3)	0.6794 (2)	0.3223 (2)	0.046 (1)
C(6)	−0.5803 (3)	0.6804 (2)	0.2692 (2)	0.057 (1)
C(7)	−0.5208 (3)	0.6958 (3)	0.2041 (2)	0.061 (1)
C(8)	−0.3764 (3)	0.7073 (2)	0.1902 (2)	0.052 (1)
C(8A)	−0.2912 (3)	0.7045 (2)	0.2437 (1)	0.036 (1)
C(9)	−0.1365 (3)	0.7154 (2)	0.2424 (1)	0.035 (1)
C(9A)	−0.1079 (3)	0.7329 (2)	0.3121 (1)	0.032 (1)
C(10)	−0.0911 (3)	0.8410 (2)	0.3131 (1)	0.035 (1)
C(10A)	−0.1908 (3)	0.8570 (2)	0.3717 (1)	0.038 (1)
C(11)	−0.0334 (3)	0.7120 (2)	0.1944 (1)	0.039 (1)
C(12)	−0.0417 (3)	0.6903 (2)	0.1272 (2)	0.044 (1)
C(13)	0.0338 (4)	0.7456 (3)	0.0725 (2)	0.066 (1)
C(14)	0.0316 (6)	0.7246 (3)	0.0089 (2)	0.087 (2)
C(15)	−0.0423 (5)	0.6492 (3)	−0.0009 (2)	0.082 (2)
C(16)	−0.1156 (4)	0.5930 (3)	0.0528 (2)	0.070 (2)
C(17)	−0.1146 (3)	0.6133 (2)	0.1171 (2)	0.054 (1)
C(18)	0.0195 (3)	0.6769 (2)	0.3418 (1)	0.036 (1)
C(19)	0.0254 (3)	0.5713 (2)	0.3525 (1)	0.037 (1)
C(20)	−0.0645 (3)	0.5167 (2)	0.3253 (2)	0.043 (1)

C(21)	−0.0502 (3)	0.4196 (2)	0.3371 (2)	0.054 (1)
C(22)	0.0503 (3)	0.3760 (2)	0.3776 (2)	0.061 (1)
C(23)	0.1398 (4)	0.4290 (3)	0.4048 (2)	0.068 (2)
C(24)	0.1286 (4)	0.5255 (2)	0.3917 (2)	0.057 (1)
C(25)	−0.1234 (3)	0.9061 (2)	0.2470 (2)	0.041 (1)
C(26)	−0.0053 (3)	0.9420 (2)	0.1955 (2)	0.044 (1)
C(27)	0.1345 (4)	0.9345 (3)	0.2091 (2)	0.079 (2)
C(28)	0.2390 (5)	0.9726 (4)	0.1606 (2)	0.104 (2)
C(29)	0.2050 (6)	1.0182 (3)	0.0993 (2)	0.092 (2)
C(30)	0.0674 (6)	1.0248 (3)	0.0839 (2)	0.084 (2)
C(31)	−0.0385 (5)	0.9878 (3)	0.1321 (2)	0.067 (1)
O(1′)	−0.7344 (2)	0.2284 (2)	0.3441 (1)	0.066 (1)
O(2′)	−0.3330 (2)	0.4389 (2)	0.2401 (1)	0.064 (1)
C(1′)	−0.4211 (3)	0.4232 (2)	0.4063 (2)	0.049 (1)
C(2′)	−0.3435 (4)	0.4130 (2)	0.4624 (2)	0.057 (1)
C(3′)	−0.2706 (4)	0.3301 (2)	0.4843 (2)	0.053 (1)
C(4′)	−0.2730 (3)	0.2563 (2)	0.4499 (1)	0.043 (1)
C(4A′)	−0.3499 (3)	0.2666 (2)	0.3938 (1)	0.034 (1)
C(4B′)	−0.3646 (3)	0.1970 (2)	0.3482 (1)	0.032 (1)
C(4C′)	−0.2426 (3)	0.1956 (2)	0.2963 (1)	0.032 (1)
C(5′)	−0.1006 (3)	0.1829 (2)	0.3070 (2)	0.042 (1)
C(6′)	−0.0029 (3)	0.1877 (3)	0.2518 (2)	0.055 (1)
C(7′)	−0.0470 (3)	0.2083 (3)	0.1876 (2)	0.060 (1)
C(8′)	−0.1874 (3)	0.2206 (2)	0.1761 (2)	0.052 (1)
C(8A′)	−0.2870 (3)	0.2119 (2)	0.2314 (1)	0.035 (1)
C(9′)	−0.4425 (3)	0.2235 (2)	0.2334 (1)	0.033 (1)
C(9A′)	−0.4902 (3)	0.2369 (2)	0.3049 (1)	0.031 (1)
C(10′)	−0.5057 (3)	0.3433 (2)	0.3124 (1)	0.035 (1)
C(10B)	−0.4240 (3)	0.3491 (2)	0.3725 (1)	0.036 (1)
C(11′)	−0.5329 (3)	0.2211 (2)	0.1871 (1)	0.039 (1)
C(12′)	−0.5059 (3)	0.1996 (2)	0.1191 (1)	0.042 (1)
C(13′)	−0.5710 (5)	0.2521 (3)	0.0656 (2)	0.069 (2)
C(14′)	−0.5511 (6)	0.2309 (3)	0.0020 (2)	0.094 (2)
C(15′)	−0.4671 (6)	0.1558 (3)	−0.0082 (2)	0.088 (2)
C(16′)	−0.4043 (4)	0.1015 (3)	0.0446 (2)	0.077 (2)
C(17′)	−0.4236 (3)	0.1227 (3)	0.1081 (2)	0.055 (1)
C(18′)	−0.6291 (3)	0.1842 (2)	0.3309 (1)	0.037 (1)
C(19′)	−0.6382 (3)	0.0795 (2)	0.3406 (1)	0.035 (1)
C(20′)	−0.5371 (3)	0.0223 (2)	0.3149 (1)	0.039 (1)
C(21′)	−0.5555 (3)	−0.0741 (2)	0.3256 (2)	0.046 (1)
C(22′)	−0.6730 (3)	−0.1153 (2)	0.3639 (2)	0.050 (1)
C(23′)	−0.7738 (3)	−0.0601 (2)	0.3900 (2)	0.048 (1)
C(24′)	−0.7569 (3)	0.0372 (2)	0.3784 (1)	0.042 (1)
C(25′)	−0.4548 (3)	0.4129 (2)	0.2488 (2)	0.041 (1)
C(26′)	−0.5568 (3)	0.4479 (2)	0.1971 (2)	0.045 (1)
C(27′)	−0.5045 (4)	0.4909 (2)	0.1333 (2)	0.064 (1)
C(28′)	−0.5974 (6)	0.5269 (3)	0.0840 (2)	0.079 (2)
C(29′)	−0.7403 (6)	0.5199 (3)	0.0996 (2)	0.089 (2)
C(30′)	−0.7909 (5)	0.4779 (4)	0.1613 (2)	0.097 (2)
C(31′)	−0.7020 (4)	0.4415 (3)	0.2107 (2)	0.070 (2)

Table 2. *Selected geometric parameters (Å, °)*

O(2)—C(25)	1.217 (4)	O(1)—C(18)	1.213 (4)
C(10)—C(10A)	1.514 (4)	C(10)—C(9A)	1.577 (4)
C(10)—C(25)	1.527 (4)	C(10A)—C(1)	1.381 (5)
C(10A)—C(4A)	1.381 (4)	C(4A)—C(4B)	1.515 (4)
C(4B)—C(4C)	1.510 (4)	C(4B)—C(9A)	1.574 (4)
C(8A)—C(9)	1.479 (4)	C(4C)—C(8A)	1.387 (4)
C(9)—C(11)	1.332 (4)	C(9)—C(9A)	1.532 (4)
C(25)—C(26)	1.499 (4)	C(9A)—C(18)	1.537 (4)
C(11)—C(12)	1.470 (4)	C(18)—C(19)	1.498 (4)
C(10A)—C(10)—C(9A)	104.2 (2)	C(10A)—C(10)—C(25)	112.3 (2)
C(9A)—C(10)—C(25)	113.4 (2)	C(10)—C(10A)—C(1)	126.7 (2)
C(10)—C(10A)—C(4A)	112.1 (3)	C(10A)—C(4A)—C(4B)	111.4 (2)
C(4)—C(4A)—C(4B)	128.4 (3)	C(4A)—C(4B)—C(4C)	115.3 (2)
C(4A)—C(4B)—C(9A)	104.4 (2)	C(4C)—C(4B)—C(9A)	103.0 (2)
C(4C)—C(8A)—C(9)	110.7 (3)	C(4B)—C(4C)—C(8A)	111.9 (2)
C(8A)—C(9)—C(9A)	105.9 (2)	C(8)—C(8A)—C(9)	129.4 (3)
C(9A)—C(9)—C(11)	122.4 (3)	C(8A)—C(9)—C(11)	131.7 (3)
C(10)—C(9A)—C(9)	112.7 (2)	C(10)—C(9A)—C(4B)	105.3 (2)
C(10)—C(9A)—C(18)	109.9 (2)	C(4B)—C(9A)—C(9)	105.7 (2)
C(9)—C(9A)—C(18)	113.4 (2)	C(4B)—C(9A)—C(18)	109.4 (2)
O(2)—C(25)—C(26)	119.6 (3)	O(2)—C(25)—C(10)	120.5 (3)
C(25)—C(26)—C(27)	122.9 (3)	C(10)—C(25)—C(26)	119.9 (3)
O(1)—C(18)—C(9A)	120.0 (3)	O(1)—C(18)—C(19)	118.5 (2)
C(9A)—C(18)—C(19)	121.4 (2)	C(9)—C(11)—C(12)	129.2 (3)

©1996 International Union of Crystallography
Printed in Great Britain – all rights reserved

The data reduction, structure solution and refinement were carried out using *SHELXTL-Plus* (VMS) (Sheldrick, 1991). The structure was solved by direct methods and refined successfully in the triclinic space group $P\bar{1}$, with two unique molecules per asymmetric unit. All non-H atoms were refined anisotropically to convergence, whereas H atoms were included in their calculated positions with fixed isotropic displacement parameters.

We thank the Council of Scientific and Industrial Research, Government of India (TM, SAK, SD and MVG), NSF (CHE-9309690), the Missouri Research Board and the Center for Molecular Electronics of the University of Missouri-St. Louis (NPR), the Jawaharlal Nehru Centre for Advanced Scientific Research (MVG) and the Office of Basic Energy (MVG, in part) for financial support of this work.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, together with an ellipsoid plot of the second molecule in the asymmetric unit, have been deposited with the IUCr (Reference: AS1145). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Pratapan, S., Ashok, K., Cyr, D. R., Das, P. K. & George, M. V. (1987). *J. Org. Chem.* **52**, 5512–5517.

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.3 for Siemens Crystallographic Research Systems. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1996). C**52**, 944–947

1-Iodo-2-methoxy-7-naphthyl Acetate

PHILIPPE PRINCE,[a] FRANK R. FRONCZEK[a] AND RICHARD D. GANDOUR[b]*

[a]*Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, USA, and* [b]*Department of Chemistry, Virginia Polytechnic Institute and State University, Blacksburg, VA 24061-0212, USA*

(*Received 2 February 1994; accepted 25 July 1995*)

Abstract

The asymmetric unit of the title compound, $C_{13}H_{11}IO_3$, contains two independent molecules. The naphthalene ring systems of the molecules have average deviations from planarity of 0.029 (13) and 0.017 (9) Å with maximum deviations of 0.042 (11) and 0.034 (9) Å, respectively. The methyl parts of the methoxy groups are *anti*