

Diethyl 6,7,8,9,10,11-Hexahydro-9,14-dioxo-7,10-ethano-7,10-cycloocteno[*b*]-naphthalenedicarboxylate

BY J. BERNSTEIN AND S. YAKIR-ZAMIR

Department of Chemistry, Ben-Gurion University of the Negev, Beer Sheva 84105, Israel

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Abstract. $C_{24}H_{24}O_6$, $M_r = 408.19$, monoclinic, $P2_1/n$, $a = 13.31$ (1), $b = 11.694$ (9), $c = 13.54$ (1) Å, $\beta = 86.6^\circ$, $V = 2104$ (2) Å³, $Z = 4$, $D_x = 1.289$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.55$ cm⁻¹, $F(000) = 864$, $T = 293$ K, final $R = 0.053$ for 2381 observed unique reflections, $wR = 0.051$.

Experimental. The title compound prepared recently by S. Bittner and H. Strul of this Department, has two diastereomers (I) and (II), which could not be distinguished by usual chemical analytical techniques. X-ray diffraction analysis was carried out to determine which of the two diastereomers was obtained. Presumably, this would be the energetically more stable isomer.

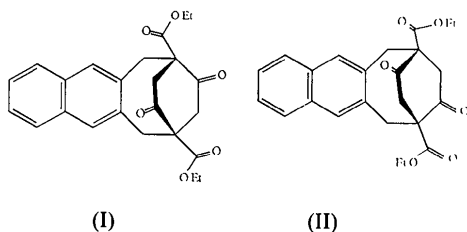


Table 1. Fractional atomic coordinates ($\times 10^4$) and U_{eq} ($\times 10^4$) with *e.s.d.*'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq} (Å ²)
O(1)	2436 (2)	-1598 (2)	6151 (1)	787 (12)
O(2)	2575 (1)	-3505 (2)	2533 (1)	605 (9)
O(3)	2936 (1)	182 (1)	4575 (1)	586 (9)
O(4)	1305 (2)	533 (2)	4693 (2)	915 (15)
O(5)	4287 (1)	-4355 (1)	3669 (2)	630 (10)
O(6)	3163 (2)	-5716 (2)	4074 (2)	793 (13)
C(1)	756 (2)	-3876 (2)	4153 (2)	434 (12)
C(2)	364 (2)	-2785 (2)	4157 (2)	468 (12)
C(3)	-481 (2)	-2550 (3)	3606 (2)	538 (14)
C(4)	-911 (2)	-3372 (3)	3067 (2)	585 (15)
C(5)	-957 (3)	-5357 (3)	2457 (2)	654 (17)
C(6)	-586 (3)	-6437 (3)	2427 (2)	702 (18)
C(7)	243 (2)	-6717 (3)	2961 (2)	646 (17)
C(8)	691 (2)	-5912 (2)	3517 (2)	531 (15)
C(9)	326 (2)	-4761 (2)	3572 (2)	449 (12)
C(10)	-522 (2)	-4494 (2)	3025 (2)	509 (13)
C(11)	765 (2)	-1795 (3)	4737 (2)	543 (15)
C(12)	1894 (2)	-1438 (2)	4509 (2)	453 (12)
C(13)	2526 (2)	-1948 (2)	5304 (2)	537 (14)
C(14)	3209 (2)	-2909 (3)	4995 (3)	594 (16)
C(15)	2693 (2)	-3717 (2)	4279 (2)	456 (12)
C(16)	2517 (2)	-3043 (2)	3336 (2)	424 (12)
C(17)	2301 (2)	-1781 (2)	3462 (2)	450 (12)
C(18)	1650 (2)	-4135 (3)	4759 (2)	513 (15)
C(19)	1984 (2)	-126 (2)	4606 (2)	560 (13)
C(20)	3147 (2)	1401 (2)	4708 (2)	628 (16)
C(21)	4240 (2)	1557 (3)	4515 (3)	869 (21)
C(22)	3379 (2)	-4727 (3)	4003 (2)	562 (14)
C(23)	5000 (2)	-5249 (3)	3337 (4)	839 (21)
C(24)	5973 (2)	-4706 (3)	3057 (3)	937 (23)

Single crystals were obtained by slow evaporation from ethanol. A cube-shaped transparent crystal of side 0.4 mm, m.p. 472 K, was used for collecting the data on a four-circle Syntex $P\bar{1}$ diffractometer at $T = 291$ K. Lattice parameters were obtained from least-squares analysis of 15 reflections, $4 < 2\theta < 23^\circ$; graphite-monochromated Mo $K\alpha$ radiation, space group by absences, ω - 2θ scan, $\theta_{\max} = 25^\circ$, $h = 0$ to 13, $k = 0$ to 15, $l = -15$ to 15; 3590 unique reflections measured, 2384 regarded as observed [$I \geq 3\sigma(I)$] with $R_{\text{int}} = 0.021$. No absorption corrections applied. Three reflections ($\bar{2}00$, $1\bar{2}\bar{2}$ and $\bar{2}35$) monitored every 60 reflections during data collection indicated no crystal deterioration.

The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1986) and refined *via* least squares on F by *SHELXL76* (Sheldrick, 1976). Scattering factors were taken from Cromer & Waber (1974). Minimization with anisotropic thermal

parameters of $\sum w(|F_o| - |F_c|)$ with $w = 1$ converged at $R = 0.056$; at this stage all the H atoms (except the ethoxy hydrogens) were located on successive difference maps and included in the refinement. The positions of the ethoxy H atoms were calculated and refined until convergence, final $R = 0.0526$, $wR = 0.0505$, weighting scheme $w = 1.7591/[\sigma^2(F) + 0.001 F^2]$; 123 parameters, max. $\Delta/\sigma = 0.004$ for all atoms; residual $\Delta\rho$ densities in difference maps range from 0.26 to -0.28 e Å⁻³.

Atomic parameters are listed in Table 1,* bond distances and bond angles are presented in Table 2. The *ORTEP* diagram (Johnson, 1976) showing the atomic numbering is given in Fig. 1.

* 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 53445 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses

O(1)—C(13)	1.217 (3)	C(5)—C(10)	1.414 (4)
O(2)—C(16)	1.212 (3)	C(6)—C(7)	1.394 (5)
O(3)—C(19)	1.316 (3)	C(7)—C(8)	1.365 (4)
O(3)—C(20)	1.466 (3)	C(8)—C(9)	1.431 (3)
O(4)—C(19)	1.188 (4)	C(9)—C(10)	1.421 (4)
O(5)—C(22)	1.338 (3)	C(11)—C(12)	1.573 (4)
O(5)—C(23)	1.465 (4)	C(12)—C(13)	1.526 (4)
O(6)—C(22)	1.194 (4)	C(12)—C(17)	1.540 (3)
C(1)—C(2)	1.378 (3)	C(12)—C(19)	1.545 (3)
C(1)—C(9)	1.439 (4)	C(13)—C(14)	1.490 (4)
C(1)—C(18)	1.516 (4)	C(14)—C(15)	1.544 (4)
C(2)—C(3)	1.413 (4)	C(15)—C(16)	1.530 (4)
C(2)—C(11)	1.514 (4)	C(15)—C(18)	1.576 (4)
C(3)—C(4)	1.354 (4)	C(15)—C(22)	1.526 (4)
C(4)—C(10)	1.410 (4)	C(16)—C(17)	1.511 (3)
C(5)—C(6)	1.356 (5)	C(20)—C(21)	1.474 (4)
		C(23)—C(24)	1.472 (4)

C(12)—C(13)—O(1)	120.4 (2)	C(14)—C(13)—O(1)	122.9 (3)
C(15)—C(16)—O(2)	120.9 (2)	C(17)—C(16)—O(2)	122.5 (2)
C(20)—O(3)—C(19)	117.0 (2)	O(4)—C(19)—O(3)	123.4 (2)
C(12)—C(19)—O(3)	110.4 (2)	C(21)—C(20)—O(3)	107.0 (2)
C(12)—C(19)—O(4)	126.2 (3)	C(23)—O(5)—C(22)	115.3 (2)
O(6)—C(22)—O(5)	123.4 (3)	C(15)—C(22)—O(5)	110.3 (3)
C(24)—C(23)—O(5)	108.4 (3)	C(15)—C(22)—O(6)	126.3 (3)
C(9)—C(1)—C(2)	120.3 (2)	C(18)—C(1)—C(2)	119.5 (2)
C(3)—C(2)—C(1)	119.4 (3)	C(11)—C(2)—C(1)	124.4 (2)
C(18)—C(1)—C(9)	120.2 (2)	C(8)—C(9)—C(1)	124.0 (2)
C(10)—C(9)—C(1)	118.7 (2)	C(15)—C(18)—C(1)	114.6 (2)
C(11)—C(2)—C(3)	116.2 (2)	C(4)—C(3)—C(2)	121.4 (3)
C(12)—C(11)—C(2)	117.6 (2)	C(10)—C(4)—C(3)	121.1 (3)
C(5)—C(10)—C(4)	121.7 (3)	C(9)—C(10)—C(4)	119.0 (2)
C(10)—C(5)—C(6)	121.4 (3)	C(7)—C(6)—C(5)	120.1 (3)
C(9)—C(10)—C(5)	119.4 (2)	C(8)—C(7)—C(6)	120.6 (3)
C(9)—C(8)—C(7)	121.3 (3)	C(10)—C(9)—C(8)	117.2 (2)
C(13)—C(12)—C(11)	108.6 (2)	C(17)—C(12)—C(11)	113.0 (2)
C(19)—C(12)—C(11)	109.0 (2)	C(17)—C(12)—C(13)	111.6 (2)
C(19)—C(12)—C(13)	106.2 (2)	C(14)—C(13)—C(12)	116.7 (2)
C(19)—C(12)—C(17)	108.2 (2)	C(16)—C(17)—C(12)	114.4 (2)
C(15)—C(14)—C(13)	110.6 (2)	C(16)—C(15)—C(14)	107.7 (2)
C(18)—C(15)—C(14)	110.2 (2)	C(22)—C(15)—C(14)	110.2 (2)
C(18)—C(15)—C(16)	109.2 (2)	C(22)—C(15)—C(16)	108.3 (2)
C(17)—C(16)—C(15)	116.5 (2)	C(22)—C(15)—C(18)	111.2 (2)

Related literature. Molecular mechanics calculations (*MODEL*, Steliou, 1986) indicate that the diastereomer found here (I), has lower steric energy than isomer (II) by 0.8 kcal mol⁻¹ (3.36 kJ mol⁻¹), in accordance with the assumption that it would crystallize preferentially. Large anisotropic thermal

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Structure of 2,2-Dinitro-1,3-propanediyl Bis[(*N*-pentafluorosulfanyl)carbamate] (I) and 2-Fluoro-2,2-dinitroethyl (*N*-Pentafluorosulfanyl)carbamate (II)

BY RICHARD GILARDI, JUDITH L. FLIPPEN-ANDERSON AND CLIFFORD GEORGE

Laboratory for the Structure of Matter, Naval Research Laboratory, Washington, DC 20375, USA

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Abstract. (I), C₃H₆F₁₀N₄O₈S₂, *M_r* = 504.3, orthorhombic, *Pna*2₁, *a* = 10.665 (2), *b* = 16.097 (3), *c* = 9.803 (1) Å, *V* = 1682.8 (4) Å³, *Z* = 4, *D_x* = 1.990 Mg m⁻³, λ(Mo *K*α) = 0.71073 Å, μ = 0.45 mm⁻¹, *F*(000) = 1000, *T* = 293 K, final *R* =

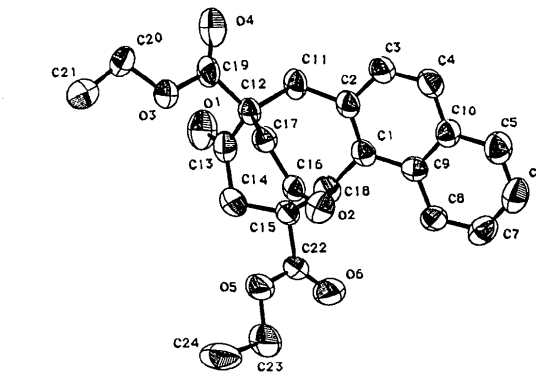


Fig. 1. ORTEP drawing of the molecule showing atomic numbering.

parameters of the ethoxy C atoms (0.063–0.094 Å²) indicate a free movement of these moieties in the solid state.

We appreciate the help of Professor Robert Glazer who performed the *MODEL* calculations together with Hava Strul, and of Michael Dorfman for technical assistance.

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0.033, *wR* = 0.035 for 1545 independent observed reflections. (II), C₃H₃F₆N₃O₆S, *M_r* = 323.1, orthorhombic, *P2₁2₁2₁*, *a* = 9.709 (2), *b* = 23.682 (6), *c* = 9.156 (3) Å, *V* = 2105.4 (9) Å³, *Z* = 8 (two molecules in asymmetric unit), *D_x* = 2.039 Mg m⁻³, λ(Mo *K*α)