ORGANIC COMPOUNDS

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An Octafluoronaphthalene:Indene (1:2) Adduct

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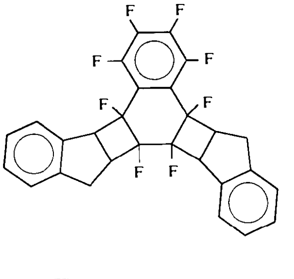
Abstract

The structure contains discrete molecules of the 1:2 adduct of octafluoronaphthalene with indene: (4bα,4cα,8bβ,8cβ,13bβ,13cβ,13dα,13eα)-4*c*,5,6,7,8,-8b,13c,13d-octafluoro-4b,4c,8b,8c,9,13b,13c,13d,13e,-14-decahydrobis(indeno[1′,2′:3,4]cyclobuta)[1,2-*a*:1′,2′-*c*]naphthalene [*Chem. Abstr.* (1992). **116**, 3468*f*]. The bond lengths and angles are normal for this type of compound and there are no unusual close intermolecular contacts in the structure.

Comment

The present structure determination is one in a series of investigations of fluoroorganic compounds prepared in the Department of Chemistry and Chemical Technology, University of Ljubljana, Slovenia (Golič & Leban, 1978*a*,*b*, 1980*a*,*b*, 1981). The compound (Fig. 1)† is the result of the photochemical reaction between octafluoronaphthalene and indene (Zupančič & Šket, 1992). It was obtained from the initially formed [2+2] *endo* adduct, which has a *cis* orientation of the H atoms in the cyclobutane ring, by an *exo* attack from the second indene molecule to the double bond in the dihydronapthalene derivative, resulting in a *trans* orientation of the H atoms. All distances are generally as expected.



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† It should be noted that the numbering scheme used differs from the numbering scheme in *Chemical Abstracts*.

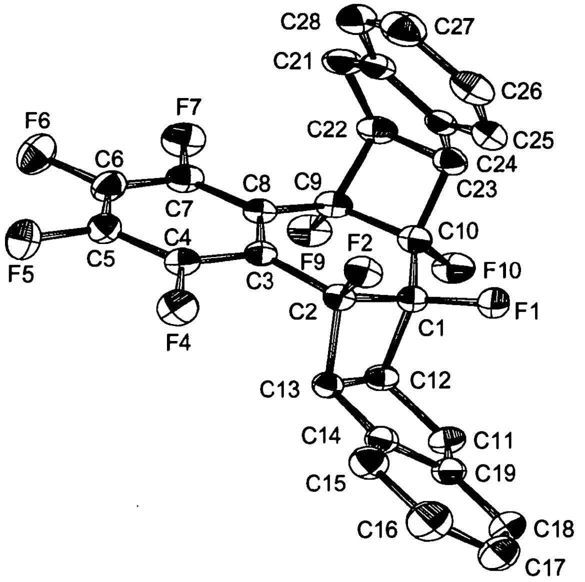

Fig. 1. *ORTEP* (Johnson, 1965) view of the molecule with that atomic numbering scheme. Displacement ellipsoids are plotted at the 30% probability level.

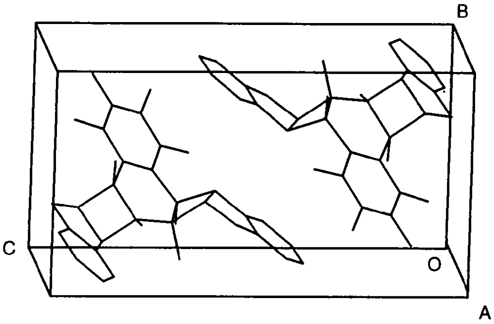

Fig. 2. The unit cell.

Experimental

Crystal data

$C_{28}H_{16}F_8$
$M_r = 504.41$
Triclinic
$P\bar{1}$
$a = 7.546$ (1) Å
$b = 9.629$ (1) Å
$c = 15.876$ (2) Å
$\alpha = 83.16$ (1)°
$\beta = 81.17$ (1)°
$\gamma = 71.23$ (1)°
$V = 1076.3$ (2) Å^3
$Z = 2$
$D_x = 1.556$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 10$–13°
$\mu = 0.138$ mm^{-1}
$T = 293$ (2) K
Transparent prisms
0.62 × 0.54 × 0.08 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
Variable rate $\omega/2\theta$ scans
11 972 measured reflections
5166 independent reflections
3165 observed reflections [$I>2\sigma(I)$]
$R_{\rm int} = 0.0224$
$\theta_{\rm max} = 27.97°$

$h = -9 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -20 \rightarrow 20$
3 standard reflections monitored every 500 reflections
frequency: 180 min
intensity variation: 1.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0401$
$wR(F^2) = 0.1055$
$S = 1.110$
5166 reflections
342 parameters
Only H-atom U's refined
Calculated weights $w=1/[\sigma^2(F_o^2)+(0.0711P)^2+0.1142P]$ where $P=(F_o^2+2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.060$

$\Delta\rho_{\rm max} = 0.283$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.231$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0143 (25)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	$U_{\rm eq}$
F1	0.75049 (15)	0.10821 (11)	0.67969 (7)	0.0525 (3)
F2	0.38010 (14)	0.18913 (10)	0.67246 (6)	0.0456 (3)
F4	0.09840 (15)	0.44915 (13)	0.63055 (7)	0.0557 (3)
F5	−0.1566 (2)	0.66943 (15)	0.71277 (9)	0.0698 (4)
F6	−0.0771 (2)	0.75598 (15)	0.85601 (9)	0.0815 (4)
F7	0.2544 (2)	0.62527 (14)	0.91328 (7)	0.0687 (4)
F9	0.6177 (2)	0.52155 (14)	0.83326 (7)	0.0640 (3)
F10	0.8708 (2)	0.2871 (2)	0.77937 (7)	0.0657 (3)
C1	0.6591 (2)	0.2544 (2)	0.69662 (11)	0.0406 (4)
C2	0.4540 (2)	0.3023 (2)	0.67439 (10)	0.0353 (3)
C3	0.3165 (2)	0.4179 (2)	0.72845 (9)	0.0353 (3)
C4	0.1408 (2)	0.4882 (2)	0.70138 (10)	0.0412 (4)
C5	0.0081 (3)	0.6012 (2)	0.74264 (12)	0.0489 (4)
C6	0.0496 (3)	0.6456 (2)	0.81436 (12)	0.0527 (5)
C7	0.2204 (3)	0.5778 (2)	0.84300 (11)	0.0487 (4)
C8	0.3583 (2)	0.4647 (2)	0.80093 (10)	0.0393 (4)
C9	0.5484 (3)	0.4038 (2)	0.83298 (11)	0.0454 (4)
C10	0.6899 (2)	0.2749 (2)	0.78478 (11)	0.0449 (4)
C11	0.8698 (3)	0.2925 (2)	0.55299 (11)	0.0501 (4)
C12	0.7054 (2)	0.3601 (2)	0.62095 (10)	0.0425 (4)
C13	0.5191 (2)	0.3695 (2)	0.58656 (10)	0.0381 (4)
C14	0.5854 (3)	0.2696 (2)	0.51427 (10)	0.0414 (4)
C15	0.4768 (3)	0.2228 (2)	0.46743 (12)	0.0527 (5)
C16	0.5663 (4)	0.1349 (3)	0.40117 (14)	0.0672 (6)
C17	0.7595 (4)	0.0942 (3)	0.38158 (13)	0.0689 (6)
C18	0.8678 (3)	0.1401 (2)	0.42773 (12)	0.0605 (5)
C19	0.7807 (3)	0.2288 (2)	0.49475 (11)	0.0458 (4)
C21	0.3882 (3)	0.2798 (3)	0.96921 (11)	0.0607 (5)
C22	0.5648 (3)	0.3034 (2)	0.91714 (11)	0.0555 (5)
C23	0.6615 (3)	0.1687 (2)	0.86284 (11)	0.0502 (5)
C24	0.5101 (3)	0.0968 (2)	0.86700 (11)	0.0454 (4)
C25	0.5116 (3)	−0.0206 (2)	0.82386 (12)	0.0542 (5)
C26	0.3581 (3)	−0.0729 (2)	0.83912 (14)	0.0630 (6)
C27	0.2047 (3)	−0.0074 (3)	0.8965 (2)	0.0683 (6)
C28	0.2023 (3)	0.1094 (3)	0.93984 (14)	0.0640 (6)
C29	0.3564 (3)	0.1608 (2)	0.92563 (11)	0.0503 (5)

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

F1—C1	1.392 (2)	C10—C23	1.544 (2)
F2—C2	1.379 (2)	C11—C19	1.508 (3)
F4—C4	1.341 (2)	C11—C12	1.537 (2)
F5—C5	1.335 (2)	C12—C13	1.557 (2)
F6—C6	1.342 (2)	C13—C14	1.507 (2)
F7—C7	1.344 (2)	C14—C15	1.390 (3)
F9—C9	1.394 (2)	C14—C19	1.392 (3)
F10—C10	1.396 (2)	C15—C16	1.381 (3)
C1—C10	1.499 (2)	C16—C17	1.377 (3)
C1—C2	1.550 (2)	C17—C18	1.376 (3)
C1—C12	1.552 (2)	C18—C19	1.391 (3)
C2—C3	1.505 (2)	C21—C29	1.506 (3)
C2—C13	1.544 (2)	C21—C22	1.523 (3)
C3—C4	1.389 (2)	C22—C23	1.556 (3)
C3—C8	1.397 (2)	C23—C24	1.505 (3)
C4—C5	1.372 (3)	C24—C25	1.386 (3)
C5—C6	1.374 (3)	C24—C29	1.391 (3)
C6—C7	1.365 (3)	C25—C26	1.383 (3)
C7—C8	1.395 (3)	C26—C27	1.383 (3)
C8—C9	1.508 (2)	C27—C28	1.380 (3)
C9—C10	1.545 (3)	C28—C29	1.383 (3)
C9—C22	1.549 (2)		
F1—C1—C10	108.37 (14)	C1—C10—C23	122.7 (2)
F1—C1—C2	110.25 (13)	F10—C10—C9	108.94 (14)
C10—C1—C2	118.35 (14)	C1—C10—C9	118.80 (14)
F1—C1—C12	110.98 (13)	C23—C10—C9	89.15 (13)
C10—C1—C12	118.97 (14)	C19—C11—C12	103.59 (14)
C2—C1—C12	88.71 (12)	C11—C12—C1	116.79 (15)
F2—C2—C3	109.14 (13)	C11—C12—C13	107.59 (14)
F2—C2—C13	115.87 (12)	C1—C12—C13	88.97 (12)
C3—C2—C13	111.65 (13)	C14—C13—C2	116.91 (13)
F2—C2—C1	115.31 (13)	C14—C13—C12	103.34 (14)
C3—C2—C1	114.32 (13)	C2—C13—C12	88.73 (12)
C13—C2—C1	89.57 (12)	C15—C14—C19	120.7 (2)
C4—C3—C8	118.2 (2)	C15—C14—C13	128.1 (2)
C4—C3—C2	117.53 (14)	C19—C14—C13	111.17 (15)
C8—C3—C2	124.19 (15)	C16—C15—C14	118.7 (2)
F4—C4—C5	117.5 (2)	C17—C16—C15	120.8 (2)
F4—C4—C3	119.69 (15)	C18—C17—C16	120.8 (2)
C5—C4—C3	122.7 (2)	C17—C18—C19	119.5 (2)
F5—C5—C4	120.9 (2)	C18—C19—C14	119.5 (2)
F5—C5—C6	120.4 (2)	C18—C19—C11	128.6 (2)
C4—C5—C6	118.7 (2)	C14—C19—C11	111.83 (15)
F6—C6—C7	119.9 (2)	C29—C21—C22	103.4 (2)
F6—C6—C5	120.1 (2)	C21—C22—C9	119.9 (2)
C7—C6—C5	119.9 (2)	C21—C22—C23	107.8 (2)
F7—C7—C6	117.7 (2)	C9—C22—C23	88.56 (13)
F7—C7—C8	120.2 (2)	C24—C23—C10	119.04 (14)
C6—C7—C8	122.1 (2)	C24—C23—C22	103.3 (2)
C7—C8—C3	118.3 (2)	C10—C23—C22	89.24 (14)
C7—C8—C9	118.61 (15)	C25—C24—C29	120.4 (2)
C3—C8—C9	123.0 (2)	C25—C24—C23	128.7 (2)
F9—C9—C8	107.25 (15)	C29—C24—C23	110.8 (2)
F9—C9—C10	112.84 (14)	C26—C25—C24	119.4 (2)
C8—C9—C10	115.11 (13)	C27—C26—C25	119.9 (2)
F9—C9—C22	113.34 (13)	C28—C27—C26	121.0 (2)
C8—C9—C22	118.25 (15)	C27—C28—C29	119.2 (2)
C10—C9—C22	89.47 (14)	C28—C29—C24	120.0 (2)
F10—C10—C1	107.32 (14)	C28—C29—C21	128.0 (2)
F10—C10—C23	108.54 (14)	C24—C29—C21	111.9 (2)

All H atoms were located in the difference map and included in the refinement at calculated positions with individual isotropic displacement parameters. A single crystal was sealed in a glass capillary and the space group determined from intensity statistics. Intensity data were collected with a maximum scan time of 60 s and a scan width of $(0.7 + 0.3\tan\theta)°$; background was measured for 1/4 of the scan time at each scan limit. Data were corrected for intensity variation and Lp effects, but absorption was ignored as a result of the small linear absorption coefficient. The initial structure model was refined by full-matrix least squares. All calculations performed on the VAX 8550 cluster at University Computer Centre, Ljubljana, under

VMS 5.2. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1075). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
Golič, L. & Leban, I. (1978*a*). *Cryst. Struct. Commun.* **7**, 47–52.
Golič, L. & Leban, I. (1978*b*). *Cryst. Struct. Commun.* **7**, 53–57.
Golič, L. & Leban, I. (1980*a*). *Acta Cryst.* **B36**, 1520–1522.
Golič, L. & Leban, I. (1980*b*). *Cryst. Struct. Commun.* **9**, 739–744.
Golič, L. & Leban, I. (1981). *Cryst. Struct. Commun.* **10**, 221–226.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Mallinson, P. R. & Muir, K. W. (1985). *J. Appl. Cryst.* **18**, 51–53.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.
Sheldrick, G. M. (1993). *SHELXL93. Program for Crystal Structure Refinement.* Univ. of Göttingen, Germany.
Zupančič, N. & Šket, B. (1992). *J. Chem. Soc. Perkin Trans.* 1, pp. 179–180.

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A Novel Dioxabicyclo[3.3.1]nonane, a Key Intermediate in the Synthesis of Erythronolide B *seco*-Acid

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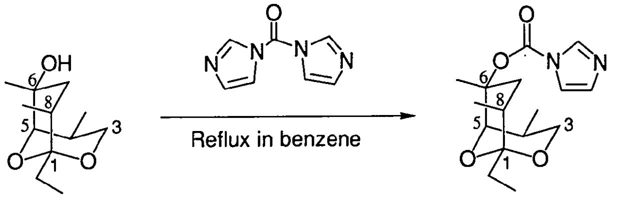
Abstract

In the title compound, (1*R*,4*S*,5*R*,6*R*,8*R*)-1-ethyl-4,6,8-trimethyl-2,9-dioxabicyclo[3.3.1]nonan-6-yl 1-imidazolecarboxylate, $C_{16}H_{24}N_2O_4$, the 2,9-dioxabicyclo[3.3.1]nonane ring system assumes a double-chair conformation. Bond angles around the ring are enlarged compared to normal tetrahedral values to alleviate some of the overcrowding which results from close intramolecular H···H and C···C contacts. The *N*-imidazolylcarbonyloxy group is nearly planar with dihedral angles of 5.2 (2) and 6.0 (2)° between the imidazole and carbonyl groups for molecules 1 and 2, respectively.

Comment

The title compound, (II), was synthesized by refluxing 1-ethyl-4,6,8-trimethyl-2,9-dioxabicyclo[3.3.1]nonan-6-ol, (I), with 1,1′-carbonyldiimidazole in benzene (Martin, Pacofsky, Gist & Lee, 1989). The crystal structure determination of (II) was undertaken as part of a project aimed at the total syntheses of erythromycins A and B utilizing novel synthetic strategies (Martin *et al.*, 1989).



(I) (II)

There are two independent molecules in the asymmetric unit. They will be referred to as molecule 1 and molecule 2. Atoms of molecule 2 have labels appended with a prime. There are no significant differences in geometry or conformation between the two molecules. The dioxabicyclo[3.3.1]nonane ring system is found to have a double-chair conformation. This conformation results in the close proximity of the H atoms in the 3*a* and 7*a* positions. Constraining the bond lengths and angles to idealized values for bicyclo[3.3.1]nonane would result in the distance between these H atoms being 0.76 Å (Peters, Baas, van de Graaf, van der Toorn & van Bekkum, 1978). In the present case, the distances are 1.96 (5) and 1.98 (4) Å for molecules 1 and 2, respectively. The molecule adjusts to minimize these close contacts by expanding the appropriate angles at atoms around the ring system. In the present example, angles C4—C5—C6 and C1—O2—C3 are enlarged significantly. The angle at C4—C5—C6 is 121.9 (3) and 122.3 (3)° for molecules 1 and 2, respectively, while C1—O2—C3 is 116.8 (2) and 117.6 (3)°, respectively. Although the double-chair is the usual conformation for such a molecule lacking bulky groups at the 3*a* and/or the 7*a* positions (Peters *et al.*, 1978), the presence of axial Me groups at C6 and C8 was expected to force the ring into a chair–boat conformation. The C13···C14 contacts are the same for both molecules [3.356 (6) Å for molecule 1, 3.355 (5) Å for molecule 2]. Even though