Crystal structure of 2,11-bis(isopropylimino)-5,5,6,6,7,7,8,8-octafluoro-dodeca-2,10-dien-4,9-dione, $C_{18}H_{24}F_8N_2O_2$

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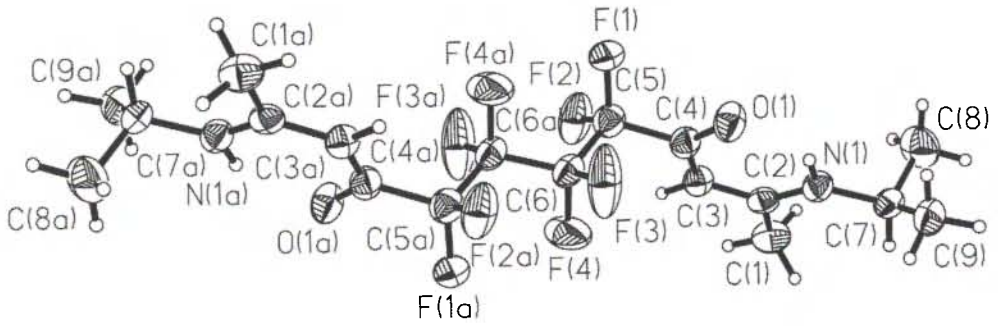

Abstract

$C_{18}H_{24}F_8N_2O_2$, triclinic, *P*1 (No. 2), a = 6.382(2) Å, b = 8.857(1) Å, c = 10.061(2) Å, α = 75.84(1)°, β = 81.49(2)°, γ = 75.35(2)°, V = 531.2 Å^3, Z = 1, $R(F)$ = 0.063, $wR_{ref}(F^2)$ = 0.171, T = 173 K.

Source of material

To a solution of 2.0 g (20 mmol) 2-isopropyliminopropane in 20 ml diethyl ether 2.9 g (10 mmol) perfluoroadipinic acid fluoride in 20 ml diethyl ether was added. After stirring the mixture for 12 hours the reaction was quenched by addition of a saturated solution af sodium hydrogencarbonate (3 × 20 ml). The organic layer was separated, dried over $MgSO_4$ and all volatiles were removed under reduced pressure. The crude product was recrystallized from n-hexane/THF (2:1).

Discussion

The unit cell shows parallel stacks of molecules, which contain strong intramolecular hydrogen bonding between N(1) and O(1) with 261.7 pm giving a nearly planar six-membered ring. The respective C—N and C—C distances in the enamino ketone framework are in between a single and a double bond (N1—C2 = 138.8 pm, C2—C3 = 139.1 pm, C3—C4 = 138.2 pm) indicating a delocalization with sp^2-hybridized carbon atoms [1]. The C=O bond length of the carbonyl group however is in common with values found in literature [2]. The angles at the carbonyl function (O1-C4-C3 = 126.7° and O1-C4-C5 = 115.5°) deviate from the expected value of 120° [3].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.40 × 0.50 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.39 cm^{-1}
Diffractometer, scan mode:	Siemens P4, 2θ/ω
$2\theta_{max}$:	45°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1788, 1314
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 968
$N(param)_{refined}$:	147
Programs:	SHELX-97 [3], SHELXTL [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1A)	2*i*	-0.3086	0.281	1.0369	0.081(6)
H(1B)	2*i*	-0.3088	0.4401	0.9198	0.081(6)
H(1C)	2*i*	-0.2516	0.4325	1.0712	0.081(6)
H(1)	2*i*	0.266(9)	0.183(6)	1.020(5)	0.07(2)
H(7)	2*i*	-0.0571	0.2573	1.2312	0.06(1)
H(8A)	2*i*	-0.1169	0.0224	1.1848	0.081(6)
H(8B)	2*i*	-0.0416	-0.0117	1.3362	0.081(6)
H(8C)	2*i*	0.1282	-0.0676	1.2141	0.081(6)
H(9A)	2*i*	0.3904	0.0924	1.2433	0.081(6)
H(9B)	2*i*	0.2318	0.1307	1.3757	0.081(6)
H(9C)	2*i*	0.2943	0.2737	1.2573	0.081(6)
H(3)	2*i*	-0.0178	0.4097	0.7562	0.04(1)

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	2*i*	–0.2383(7)	0.3710(5)	1.0003(5)	0.037(3)	0.053(3)	0.063(3)	–0.008(2)	0.000(2)	–0.022(2)
C(2)	2*i*	–0.0015(7)	0.3086(5)	0.9591(4)	0.037(3)	0.033(2)	0.041(3)	–0.013(2)	–0.003(2)	–0.014(2)
N(1)	2*i*	0.1266(6)	0.2218(4)	1.0546(3)	0.038(2)	0.041(2)	0.036(2)	–0.011(2)	0.001(2)	–0.011(2)
C(7)	2*i*	0.0683(8)	0.1743(5)	1.2029(4)	0.055(3)	0.041(2)	0.033(2)	–0.008(2)	0.009(2)	–0.012(2)
C(8)	2*i*	0.0039(9)	0.0154(6)	1.2375(6)	0.072(4)	0.058(3)	0.071(4)	–0.030(3)	0.009(3)	–0.004(3)
C(9)	2*i*	0.2636(9)	0.1671(6)	1.2763(5)	0.091(4)	0.053(3)	0.040(3)	–0.020(3)	–0.017(3)	–0.009(2)
C(3)	2*i*	0.0785(7)	0.3434(5)	0.8215(4)	0.034(3)	0.040(2)	0.037(2)	–0.007(2)	–0.009(2)	–0.010(2)
C(4)	2*i*	0.2922(7)	0.2857(5)	0.7757(4)	0.044(3)	0.040(2)	0.035(2)	–0.015(2)	–0.006(2)	–0.009(2)
O(1)	2*i*	0.4408(5)	0.2007(4)	0.8482(3)	0.039(2)	0.062(2)	0.037(2)	–0.004(2)	–0.002(2)	–0.007(1)
C(5)	2*i*	0.3588(7)	0.3275(5)	0.6193(4)	0.041(3)	0.046(3)	0.040(3)	–0.005(2)	–0.003(2)	–0.011(2)
F(1)	2*i*	0.5040(5)	0.2038(3)	0.5810(3)	0.110(3)	0.042(2)	0.048(2)	–0.003(2)	0.021(2)	–0.015(1)
F(2)	2*i*	0.1855(5)	0.3562(4)	0.5465(2)	0.071(2)	0.106(2)	0.040(2)	–0.049(2)	–0.016(1)	–0.007(1)
C(6)	2*i*	0.4577(7)	0.4747(5)	0.5755(4)	0.037(3)	0.048(3)	0.032(2)	–0.005(2)	–0.006(2)	–0.013(2)
F(3)	2*i*	0.6138(6)	0.4571(5)	0.6531(3)	0.107(3)	0.161(3)	0.048(2)	–0.094(3)	–0.039(2)	0.026(2)
F(4)	2*i*	0.3010(6)	0.5983(3)	0.6052(3)	0.130(3)	0.045(2)	0.096(2)	–0.014(2)	0.072(2)	–0.025(2)

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