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Crystal structure of nonafluoro-{*N*-[(*E*)-2-(2-hydroxyphenyl)diazenyl]-phenyl}-1-butanesulfonamide, $C_{16}H_{10}F_9N_3O_3S$

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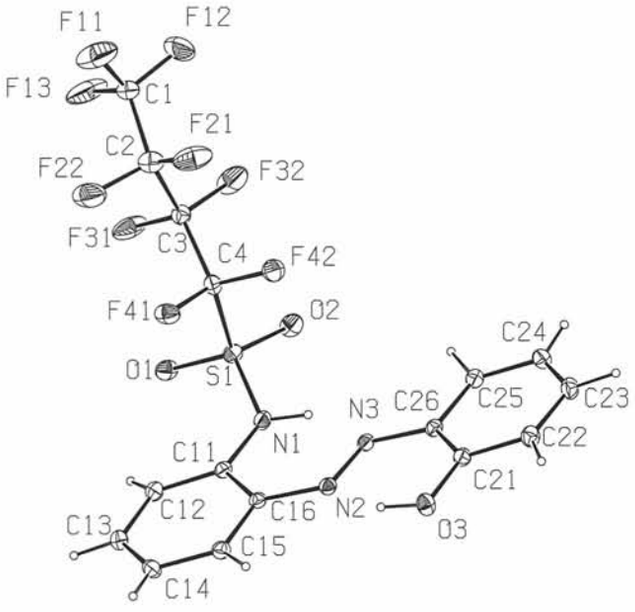
Received September 17, 2003, accepted and available on-line November 17, 2003; CCDC-No. 219149



Abstract

$C_{16}H_{10}F_9N_3O_3S$, monoclinic, $P121/c1$ (No. 14), $a = 5.6549(3)$ Å, $b = 12.2550(9)$ Å, $c = 27.170(6)$ Å, $\beta = 90.446(8)°$, $V = 1882.8$ Å^3, $Z = 4$, $R_{gt}(F) = 0.088$, $wR_{ref}(F^2) = 0.261$, $T = 213$ K.

Source of material

The title compound was prepared from 1-[(nonafluorobutyl)-sulfonyl]-1*H*-1,2,3-benzotriazole and phenol in toluene in the presence of sodium hydride as described in [1].

Discussion

The N=N length 1.269(5) Å of the diazo compound lies within the normal accepted values. The dihedral angle C16–N2=N3–C26 is 177.7(3)° indicating a *trans* N=N double bond. The torsional angle between the two ring systems is found to be 3.6(3)°. Both the nitrogen atoms in the ortho-substituted product are in the proximity of hydrogens indicating hydrogen bonds. The spatial distance between O3-H···N(2) and N1-H···N3 corresponds to 1.93(7) Å and 1.94(6) Å, respectively.

Table 1. Data collection and handling.

Crystal:	orange needle, size 0.15 × 0.15 × 0.75 mm
Wavelength:	Cu K_α radiation (1.54184 Å)
μ:	26.19 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω
$2\theta_{max}$:	129.9°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4527, 3075
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2735
$N(param)_{refined}$:	329
Programs:	SHELXS-97 [2], SHELXL-97 [3], PLATON [4]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	–0.40(1)	0.452(5)	–0.060(2)	0.07(2)
H(2)	4*e*	–0.83(1)	0.194(4)	–0.026(2)	0.06(1)
H(3)	4*e*	–0.69(1)	–0.266(5)	0.170(2)	0.06(2)
H(4)	4*e*	–0.13(1)	0.402(5)	–0.004(2)	0.06(2)
H(5)	4*e*	–0.23(1)	–0.014(6)	0.144(2)	0.08(2)
H(6)	4*e*	–0.87(1)	0.021(6)	0.040(2)	0.07(2)
H(7)	4*e*	–0.364(9)	–0.169(4)	0.186(2)	0.05(1)
H(8)	4*e*	–0.92(1)	–0.195(5)	0.102(2)	0.07(2)
H(9)	4*e*	–0.18(1)	0.169(5)	0.074(2)	0.06(2)
H(10)	4*e*	–0.72(1)	0.347(5)	–0.069(2)	0.07(2)

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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}
O(3)	4*e*	–0.8946(6)	–0.0445(3)	0.0505(1)	0.052(2)	0.058(2)	0.070(2)	–0.013(2)	–0.008(2)	0.013(2)
C(21)	4*e*	–0.7440(9)	–0.0772(4)	0.0861(2)	0.052(3)	0.045(2)	0.049(2)	–0.002(2)	0.005(2)	–0.002(2)
C(22)	4*e*	–0.800(1)	–0.1683(4)	0.1138(2)	0.056(3)	0.052(3)	0.064(3)	–0.010(2)	0.007(3)	0.005(2)
C(23)	4*e*	–0.652(1)	–0.2051(4)	0.1505(2)	0.067(4)	0.048(3)	0.063(3)	0.000(2)	0.013(3)	0.009(2)
C(24)	4*e*	–0.440(1)	–0.1506(5)	0.1609(2)	0.072(4)	0.058(3)	0.052(3)	0.008(3)	–0.004(3)	0.007(2)
C(25)	4*e*	–0.3810(9)	–0.0605(4)	0.1339(2)	0.056(3)	0.046(3)	0.054(3)	0.001(2)	–0.001(2)	–0.002(2)
C(26)	4*e*	–0.5293(8)	–0.0226(4)	0.0961(2)	0.048(3)	0.040(2)	0.047(2)	–0.000(2)	0.006(2)	–0.004(2)
N(2)	4*e*	–0.5844(7)	0.1081(3)	0.0381(1)	0.046(2)	0.042(2)	0.045(2)	–0.004(2)	0.004(2)	–0.002(2)
N(3)	4*e*	–0.4498(7)	0.0716(3)	0.0719(1)	0.047(2)	0.038(2)	0.045(2)	0.000(2)	0.003(2)	–0.004(2)
C(11)	4*e*	–0.3124(8)	0.2696(4)	0.0241(2)	0.047(3)	0.042(2)	0.043(2)	–0.002(2)	0.004(2)	–0.005(2)
C(12)	4*e*	–0.273(1)	0.3617(4)	–0.0040(2)	0.059(3)	0.047(3)	0.059(3)	–0.010(2)	–0.002(2)	–0.001(2)
C(13)	4*e*	–0.432(1)	0.3923(4)	–0.0401(2)	0.066(3)	0.047(3)	0.056(3)	–0.005(2)	0.003(2)	0.005(2)
C(14)	4*e*	–0.632(1)	0.3313(4)	–0.0493(2)	0.061(3)	0.058(3)	0.045(3)	–0.002(2)	–0.003(2)	0.007(2)
C(15)	4*e*	–0.6711(9)	0.2372(4)	–0.0230(2)	0.049(3)	0.051(3)	0.048(2)	–0.006(2)	0.001(2)	–0.002(2)
C(16)	4*e*	–0.5146(8)	0.2053(3)	0.0141(2)	0.048(3)	0.040(2)	0.039(2)	–0.001(2)	0.006(2)	–0.005(2)
N(1)	4*e*	–0.1531(8)	0.2336(3)	0.0615(2)	0.058(3)	0.041(2)	0.058(2)	–0.004(2)	–0.006(2)	–0.003(2)
C(1)	4*e*	–0.149(2)	0.5696(6)	0.2545(2)	0.122(6)	0.074(4)	0.054(3)	–0.005(4)	0.003(3)	–0.009(3)
F(11)	4*e*	–0.320(1)	0.6289(7)	0.2748(3)	0.191(7)	0.209(7)	0.155(5)	0.042(6)	–0.010(5)	–0.099(6)
F(12)	4*e*	–0.089(1)	0.5115(5)	0.2906(2)	0.210(7)	0.154(5)	0.080(3)	–0.036(5)	–0.054(4)	0.013(3)
F(13)	4*e*	0.014(2)	0.6305(6)	0.2427(2)	0.33(1)	0.192(7)	0.129(4)	–0.175(8)	0.110(6)	–0.098(5)
C(2)	4*e*	–0.254(2)	0.5049(7)	0.2132(3)	0.135(8)	0.096(5)	0.069(4)	–0.002(5)	0.008(4)	–0.011(4)
F(21)	4*e*	–0.4279(8)	0.4449(6)	0.2267(2)	0.084(3)	0.198(6)	0.113(3)	–0.032(3)	0.029(3)	–0.070(4)
F(22)	4*e*	–0.350(2)	0.5810(6)	0.1810(2)	0.32(1)	0.163(6)	0.106(4)	0.145(7)	–0.084(5)	–0.040(4)
C(3)	4*e*	–0.082(1)	0.4394(4)	0.1808(2)	0.060(3)	0.055(3)	0.058(3)	–0.003(2)	–0.001(2)	0.002(2)
F(31)	4*e*	0.055(1)	0.5106(5)	0.1598(2)	0.197(6)	0.138(4)	0.104(3)	–0.115(4)	0.071(3)	–0.059(3)
F(32)	4*e*	0.046(1)	0.3795(4)	0.2112(2)	0.176(5)	0.115(4)	0.165(5)	0.065(4)	–0.120(4)	–0.054(3)
C(4)	4*e*	–0.1952(9)	0.3632(4)	0.1426(2)	0.055(3)	0.049(3)	0.055(3)	–0.002(2)	–0.001(2)	0.007(2)
F(41)	4*e*	–0.3634(6)	0.4169(3)	0.1177(1)	0.069(2)	0.095(2)	0.064(2)	0.026(2)	–0.017(2)	–0.010(2)
F(42)	4*e*	–0.2985(7)	0.2798(3)	0.1655(1)	0.093(3)	0.074(2)	0.071(2)	–0.028(2)	0.017(2)	0.002(2)
S(1)	4*e*	0.0117(2)	0.3058(1)	0.09715(4)	0.0461(8)	0.0517(7)	0.0567(7)	–0.0082(5)	–0.0039(5)	–0.0050(5)
O(1)	4*e*	0.1050(7)	0.3965(3)	0.0717(1)	0.070(2)	0.071(2)	0.063(2)	–0.033(2)	0.005(2)	–0.008(2)
O(2)	4*e*	0.1576(7)	0.2326(3)	0.1244(2)	0.057(2)	0.073(2)	0.088(3)	0.007(2)	–0.022(2)	–0.011(2)

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