

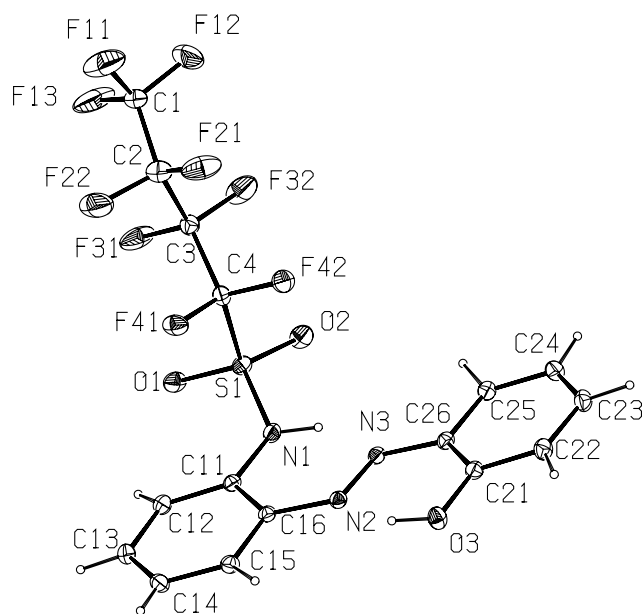
Crystal structure of nonafluoro- $\{N-[(E)-2-(2\text{-hydroxyphenyl})\text{diazenyl}]\text{-phenyl}\}$ -1-butanesulfonamide, $\text{C}_{16}\text{H}_{10}\text{F}_9\text{N}_3\text{O}_3\text{S}$

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Abstract

$\text{C}_{16}\text{H}_{10}\text{F}_9\text{N}_3\text{O}_3\text{S}$, monoclinic, $P121/c1$ (No. 14), $a = 5.6549(3)$ Å, $b = 12.2550(9)$ Å, $c = 27.170(6)$ Å, $\beta = 90.446(8)^\circ$, $V = 1882.8$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.088$, $wR_{\text{ref}}(F^2) = 0.261$, $T = 213$ 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 $\text{N}=\text{N}$ length 1.269(5) Å of the diazo compound lies within the normal accepted values. The dihedral angle $\text{C}16\text{--N}2=\text{N}3\text{--C}26$ is $177.7(3)^\circ$ indicating a *trans* $\text{N}=\text{N}$ double bond. The torsional angle between the two ring systems is found to be $3.6(3)^\circ$. Both the nitrogen atoms in the ortho-substituted product are in the proximity of hydrogens indicating hydrogen bonds. The spatial distance between $\text{O}3\text{--H}\cdots\text{N}(2)$ and $\text{N}1\text{--H}\cdots\text{N}3$ corresponds to 1.93(7) Å and 1.94(6) Å, respectively.

Table 1. Data collection and handling.

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

Table 2. Atomic coordinates and displacement parameters (in Å²).

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

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
O(3)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	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)	4e	−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)	4e	−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)	4e	−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)	4e	−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)	4e	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)	4e	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)	4e	−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)	4e	−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)	4e	−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)	4e	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)	4e	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)	4e	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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