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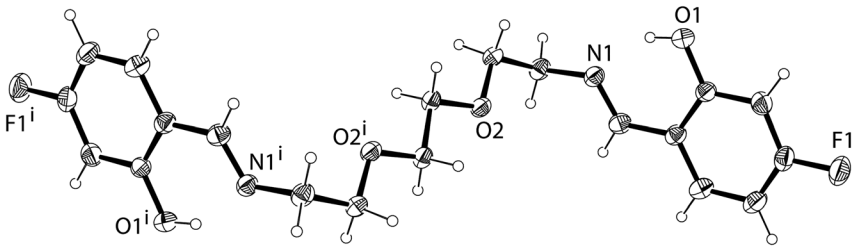
Crystal structure of *N*,*N*′-bis(4-fluoro-salicylaldehyde)-3,6-dioxa-1,8-diaminooctane, $C_{20}H_{22}F_2N_2O_4$



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Abstract

$C_{20}H_{22}F_2N_2O_4$, triclinic, $P\bar{1}$ (no. 2), a = 4.7005(2) Å, b = 10.2279(3) Å, c = 10.9917(3) Å, α = 63.1510(10)°, β = 83.369(2)°, γ = 83.014(2)°, V = 466.88(3) Å^3, Z = 1, $R_{gt}(F)$ = 0.0361, $wR_{ref}(F^2)$ = 0.1018, T = 173.0 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Orange block
Size	0.14 × 0.13 × 0.11 mm
Wavelength:	Cu *K*α radiation (1.54178 Å)
μ:	0.94 mm^{-1}
Diffractometer, scan mode:	Bruker D8 Venture, φ and ω
θ_{max}, completeness:	72.1°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5378, 1812, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1625
$N(param)_{refined}$:	128
Programs:	Bruker [1], SHELX [2], Olex2 [3]

Source of material

All chemicals were purchased from commercial sources and used without further purification. The title compound was prepared following the literature procedures [4, 5]. A flask was charged with 4-fluoro-2-hydroxy-benzaldehyde (280.22 mg, 2 mmol) and 1,8-diamino-3,6-dioxaoctane (148.21 mg, 1 mmol) and 20 mL methanol. The mixture was stirred at 238 K for 6 h. After cooling to room temperature, the mixture was filtered, washed successively with methanol and methanol/hexane (1:4), respectively. The isolated product was dried under reduced pressure and purified by recrystallization with methanol solution. Several clear orange crystals were obtained.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

In the past decades, various Schiff base compounds and their derivatives have been designed and synthesized through different strategies. For example, Salen-type compound [6], Salamo-type compound [7] and so on. These compounds and their derivatives have excellent applications in materials science [8], biochemistry [9] and coordination chemistry [10]. The Schiff base compounds form stable metal complexes with one, two or more transition metal centers [11, 12], and rare earth metals [13].

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
F1	0.9087 (2)	0.95953 (9)	0.37137 (9)	0.0681 (3)
O1	0.4406 (2)	0.68236 (10)	0.79894 (9)	0.0500 (3)
H1	0.327936	0.615097	0.836080	0.075*
O2	0.26550 (18)	0.16591 (9)	0.94055 (8)	0.0415 (2)
N1	0.1157 (2)	0.48530 (11)	0.81784 (10)	0.0401 (3)
C1	0.4911 (3)	0.71494 (13)	0.66568 (12)	0.0394 (3)
C2	0.6761 (3)	0.82331 (13)	0.58499 (14)	0.0476 (3)
H2	0.763873	0.874412	0.622674	0.057*
C3	0.7281 (3)	0.85425 (14)	0.44989 (14)	0.0480 (3)
C4	0.6085 (3)	0.78502 (15)	0.38838 (13)	0.0509 (3)
H4	0.652106	0.808851	0.294496	0.061*
C5	0.4224 (3)	0.67951 (16)	0.46897 (13)	0.0490 (3)
H5	0.333138	0.631312	0.428880	0.059*
C6	0.3613 (3)	0.64133 (13)	0.60785 (12)	0.0397 (3)
C7	0.1663 (3)	0.52811 (14)	0.68993 (12)	0.0421 (3)
H7	0.073585	0.484701	0.646429	0.051*
C8	−0.0805 (3)	0.37050 (14)	0.89368 (13)	0.0447 (3)
H8A	−0.156143	0.341219	0.829930	0.054*
H8B	−0.245025	0.409331	0.935891	0.054*
C9	0.0682 (3)	0.23775 (13)	1.00365 (12)	0.0414 (3)
H9A	0.171781	0.268451	1.058767	0.050*
H9B	−0.075274	0.169673	1.065146	0.050*
C10	0.3923 (3)	0.03154 (13)	1.03798 (11)	0.0403 (3)
H10A	0.243123	−0.037841	1.088844	0.048*
H10B	0.488978	0.048919	1.104164	0.048*

The crystal structure of the title compound was analyzed by X-ray diffraction (*cf*. the figure), in which all bond lengths and bond angles are within the normal ranges [7]. There is one strong intramolecular O1—H1···N1 hydrogen bond interaction (d(N1···H1) = 1.85 Å, d(O1—H1) = 0.84 Å and d(O1···N1) = 2.5953(13) Å) in the title structure at both ends of the centrosymmetric molecule. In addition, there are two intermolecular C2—H2···F1#2 (#2 $-x+2, -y+2, -z+1$) and C4—H4···O2#3 (#3 $-x+1, -y+1, -z+1$) hydrogen bonds, which are (d(F1#2···H1) = 2.45 Å, d(C2—H2) = 0.95 Å, d(C2···F1#2) = 3.3474(17) Å) and (d(O2#3···H4) = 2.47 Å, d(C4—H4) = 0.95 Å, d(C4···O2#3) = 3.4078(15) Å), respectively.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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