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Crystal structure of N^1,N^2 -bis(2-fluorobenzyl) benzene-1,2-diamine, $C_{20}H_{18}F_2N_2$

Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	$0.42 \times 0.38 \times 0.35$ mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm $^{-1}$
Diffractometer, scan mode:	SMART, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8005, 2957, 0.104
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1455
$N(\text{param})_{\text{refined}}$:	236
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

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Abstract

$C_{20}H_{18}F_2N_2$, monoclinic, $P2_1/c$ (no. 14), $a = 12.4233(11)$ Å, $b = 7.3805(7)$ Å, $c = 18.9531(16)$ Å, $\beta = 104.109(3)^\circ$, $V = 104.109(3)$ Å 3 , $Z = 4$, $R_{\text{gt}}(F) = 0.0464$, $wR_{\text{ref}}(F^2) = 0.1201$, $T = 296(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of o-phenylene diamine (0.541 g, 5 mmol), 1-bromopropane (0.615 g, 5 mmol) and potassium carbonate (0.828 g, 6 mmol) was stirred in ethanol at room temperature. After the reaction was completed (monitored

by TLC, eluent, dichloromethane/petroleum ether, 1/1, v/v), the solvent was removed and the residue was extracted with dichloromethane (3 $\times$ 40 mL), dried over anhydrous sodium sulfate and purified by silica gel column chromatography (eluent, dichloromethane/petroleum ether, 1/1, v/v) to afford the white solid. Yield, 645 mg, 40%. The title compound was dissolved in dichloromethane, and the solvent was evaporated slowly at room temperature. After three days, colorless crystals were obtained.

Experimental details

Data reduction was carried out using SAINT+ and SADABS [1]. The structure was determined by intrinsic phasing routines in the SHELXT program [2] and refined by full-matrix least-squares methods in SHELXL [3] by using Olex 2 [4]. All of the hydrogen atoms were placed in the calculated positions.

Comment

Phenylamine derivatives are an important class of organic compounds, which have a wide range of applications in medicine and chemical industry [5, 6]. For instance, phenylamines are widely used as antimicrobial drugs, anti-inflammatory analgesics and antileptic drugs in the field of medicine, and as insecticides and fungicides in the field of pesticides [7]. Additionally, phenylamines can

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.1777 (3)	0.4570 (5)	0.55252 (15)	0.0748 (8)
H1 ^a	0.1486	0.5423	0.5788	0.090*
C2	0.1201 (3)	0.2998 (5)	0.53252 (16)	0.0874 (10)
H2	0.0527	0.2806	0.5445	0.105*
C3	0.1624 (3)	0.1705 (5)	0.49465 (15)	0.0862 (10)
H3	0.1244	0.0624	0.4813	0.103*
C4	0.2623 (3)	0.2024 (4)	0.47644 (14)	0.0792 (9)
H4	0.2912	0.1173	0.4499	0.095*
C5	0.3174 (3)	0.3599 (5)	0.49794 (14)	0.0701 (8)
H5 ^b	0.3855	0.3780	0.4869	0.084*
C6	0.2772 (2)	0.4947 (4)	0.53542 (12)	0.0591 (7)
C7	0.3394 (2)	0.6679 (4)	0.55588 (12)	0.0676 (8)
H7A	0.3716	0.7063	0.5166	0.081*
H7B	0.2886	0.7616	0.5634	0.081*
C8	0.5056 (2)	0.7805 (4)	0.64599 (12)	0.0508 (6)
C9	0.4969 (2)	0.9535 (4)	0.61579 (13)	0.0659 (8)
H9	0.4365	0.9826	0.5778	0.079*
C10	0.5774 (3)	1.0823 (4)	0.64200 (17)	0.0771 (9)
H10	0.5709	1.1975	0.6215	0.093*
C11	0.6665 (3)	1.0414 (4)	0.69791 (18)	0.0805 (9)
H11	0.7205	1.1287	0.7152	0.097*
C12	0.6766 (2)	0.8709 (4)	0.72870 (14)	0.0660 (7)
H12	0.7375	0.8445	0.7667	0.079*
C13	0.5976 (2)	0.7386 (3)	0.70390 (12)	0.0510 (6)
C14	0.7040 (2)	0.5046 (3)	0.78625 (12)	0.0603 (7)
H14A	0.6874	0.3922	0.8079	0.072*
H14B	0.7201	0.5955	0.8244	0.072*
C15	0.8058 (2)	0.4773 (3)	0.75771 (11)	0.0489 (6)
C16	0.9073 (2)	0.4380 (4)	0.80387 (12)	0.0613 (7)
C17	1.0027 (2)	0.4067 (4)	0.78206 (15)	0.0778 (9)
H17	1.0687	0.3803	0.8159	0.093*
C18	0.9990 (3)	0.4149 (4)	0.70898 (16)	0.0838 (9)
H18	1.0627	0.3954	0.6925	0.101*
C19	0.9000 (3)	0.4524 (4)	0.66086 (14)	0.0755 (9)
H19	0.8967	0.4566	0.6113	0.091*
C20	0.8048 (2)	0.4841 (3)	0.68435 (13)	0.0609 (7)
H20	0.7389	0.5104	0.6504	0.073*
F1 ^a	0.4115 (3)	0.3927 (5)	0.48346 (14)	0.0980 (14)
F1A ^b	0.1450 (4)	0.5726 (6)	0.5906 (3)	0.113 (2)
F2	0.91046 (14)	0.4293 (2)	0.87692 (7)	0.0986 (6)
N1	0.42681 (18)	0.6445 (4)	0.62191 (10)	0.0590 (6)
H1A	0.454 (2)	0.539 (4)	0.6348 (14)	0.079 (10)*
N2	0.6066 (2)	0.5608 (3)	0.73116 (11)	0.0560 (6)
H2A	0.545 (3)	0.527 (4)	0.7344 (15)	0.092 (11)*

^a Occupancy: 0.579(5), ^b Occupancy: 0.421(5).

also be used to synthesize some important medicinal intermediates and other chemicals with important application value in organic synthesis [8]. Although

*N*¹,*N*²-bis(2-fluorobenzyl)benzene-1,2-diamine is a known compound, its crystal structure has not been reported yet. Therefore, in consideration of the importance of this compound it was synthesized from commercial material, and also its crystal structure will be reported.

There is one molecule in the asymmetric unit (see the Figure). The bond lengths and angles in the title molecule are in normal ranges. In a word, the title compound was synthesized under mild conditions, which can be readily used as a key intermediate to develop new drugs, novel reagents and advanced materials.

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