Crystal structure of 2-amino-*N*,3,5-trimethylbenzamide, $C_{10}H_{14}N_2O$

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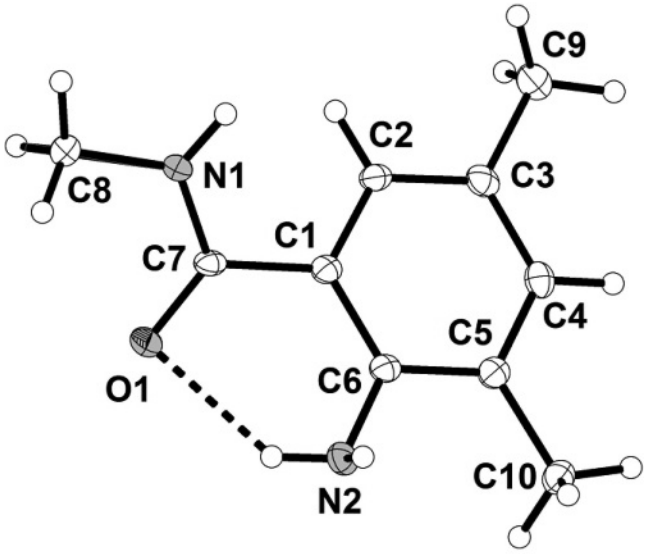

Abstract

$C_{10}H_{14}N_2O$, monoclinic, $P2_1/n$ (no. 14), a = 9.9191(8) Å, b = 4.9955(4) Å, c = 19.1981(13) Å, β = 92.509(7)°, V = 950.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.0644, $wR_{ref}(F^2)$ = 0.1828, T = 100 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.1×0.1×0.2 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	0.82 cm^{-1}
Diffractometer, scan mode:	SuperNova, Dual, Cu at zero, Atlas, ω
$2\theta_{max}$:	52.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3884, 1866
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1446
$N(param)_{refined}$:	122
Programs:	OLEX2 [1], SHELX [2]

Source of material

Several synthetic methods for anthranilamide have been reported in the literature [3, 4]. The title compound was obtained by stirring 6,8-dimethyl-1*H*-benzo[*d*][1,3]oxazine-2,4-dione with aqueous methylamine in THF at room temperature. 6,8-dimethyl-1*H*-benzo[*d*][1,3]oxazine-2,4-dione may also be prepared by the reaction of 2-amino-3,5-dimethylbenzoic acid with triphosgene in THF at room temperature in good yield [5]. The title compound (0.2 g) was dissolved in ethanol (30 ml) at room temperature. Colourless block-shaped crystals were obtained through slow evaporation within two weeks.

Experimental details

All H atoms were placed at calculated positions.

Discussion

Anthranilamide-based derivatives exhibit interesting biological activities such as antibacterial, antifungal, antiviral, antimalarial and insecticidal activities. We report here the crystal structure of 2-amino-*N*,3,5-trimethylbenzamide, an very important organic intermediate in the synthesis of medicines, agricultural chemicals and animal drugs [6, 7]. In the title structure, the benzene ring and the methylamide are linked to each other with N1–C7–O1 moiety enclose a diheral angle of ~36°. There is one intramolecular hydrogen bond (see the Fig.) Intermolecular hydrogen bonds connect adjacent molecules in the *b* direction to chains.

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	0.5558	0.7836	0.5500	0.023
H(4)	4*e*	0.9457	0.5563	0.5616	0.025
H(8A)	4*e*	0.1863	0.6030	0.6288	0.036
H(8B)	4*e*	0.2101	0.8732	0.6724	0.036
H(8C)	4*e*	0.2331	0.5890	0.7095	0.036
H(9A)	4*e*	0.7514	0.8236	0.4441	0.038
H(9B)	4*e*	0.8827	0.9250	0.4878	0.038
H(9C)	4*e*	0.7402	1.0672	0.4976	0.038
H(10A)	4*e*	1.0276	0.2280	0.6388	0.035
H(10B)	4*e*	0.9205	0.0027	0.6578	0.035
H(10C)	4*e*	0.9563	0.2385	0.7120	0.035
H(1)	4*e*	0.4191	0.8453	0.6365	0.024
H(2A)	4*e*	0.6972	0.2524	0.7483	0.025
H(2B)	4*e*	0.5940	0.1217	0.7090	0.025

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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)	4*e*	0.5969(2)	0.4950(5)	0.6230(1)	0.019(1)	0.012(1)	0.021(1)	–0.001(1)	–0.0023(9)	–0.0034(9)
C(2)	4*e*	0.6262(2)	0.6759(5)	0.5701(1)	0.021(1)	0.014(1)	0.021(1)	0.001(1)	–0.0056(9)	0.0004(9)
C(3)	4*e*	0.7558(3)	0.7022(5)	0.5462(1)	0.024(1)	0.017(1)	0.021(1)	–0.005(1)	–0.003(1)	–0.001(1)
C(4)	4*e*	0.8565(2)	0.5410(5)	0.5773(1)	0.020(1)	0.021(1)	0.022(1)	–0.005(1)	0.0008(9)	–0.005(1)
C(5)	4*e*	0.8321(2)	0.3597(5)	0.6299(1)	0.020(1)	0.015(1)	0.022(1)	–0.001(1)	–0.0027(9)	–0.0046(9)
C(6)	4*e*	0.7003(2)	0.3345(5)	0.6537(1)	0.020(1)	0.012(1)	0.019(1)	–0.0006(9)	–0.0033(9)	–0.0028(9)
C(7)	4*e*	0.4556(2)	0.4682(5)	0.6460(1)	0.021(1)	0.012(1)	0.020(1)	–0.000(1)	–0.0053(9)	–0.0005(9)
C(8)	4*e*	0.2414(2)	0.6890(5)	0.6660(1)	0.018(1)	0.021(1)	0.035(2)	0.002(1)	0.002(1)	0.004(1)
C(9)	4*e*	0.7851(3)	0.8964(5)	0.4889(1)	0.028(1)	0.025(1)	0.024(1)	–0.005(1)	0.000(1)	0.001(1)
C(10)	4*e*	0.9438(2)	0.1927(5)	0.6625(1)	0.020(1)	0.022(1)	0.028(1)	0.002(1)	–0.001(1)	–0.002(1)
N(1)	4*e*	0.3817(2)	0.6920(4)	0.6473(1)	0.018(1)	0.013(1)	0.029(1)	–0.0012(8)	–0.0006(8)	0.0038(8)
N(2)	4*e*	0.6777(2)	0.1681(4)	0.7100(1)	0.021(1)	0.020(1)	0.020(1)	–0.0043(9)	–0.0019(8)	0.0004(8)
O(1)	4*e*	0.4083(2)	0.2489(3)	0.66312(9)	0.0220(9)	0.0137(9)	0.036(1)	–0.0019(7)	0.0014(7)	0.0018(7)

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