Tai Xi-Shi, Zhou Xiao-Jing, Liu Li-Li, Zhang Ai-Ling* and Wang Li-Hua

The crystal structure of 4-amino-*N'*-(4-aminobenzoyl)benzohydrazide monohydrate, $C_{14}H_{16}N_4O_3$

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Abstract

$C_{14}H_{16}N_4O_3$, orthorhombic, $P2_12_12$ (no. 18), $a = 8.0968(7)$ Å, $b = 16.6829(17)$ Å, $c = 5.3097(5)$ Å, $V = 717.22(12)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0420$, $wR_{ref}(F^2) = 0.0816$, $T = 293(2)$ 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.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.13 \times 0.12 \times 0.11$ mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffractometer, scan mode:	SuperNova,
θ_{max} , completeness:	27.5°, 97%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	2291, 1490, 0.025
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1224
$N(param)_{refined}$:	110
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

*Corresponding author: Zhang Ai-Ling, College of Chemistry and Chemical Engineering, Weifang University, Weifang, Shandong 261061, P. R. China, e-mail: zhangal@wfu.edu.cn

Tai Xi-Shi, Zhou Xiao-Jing, Liu Li-Li and Wang Li-Hua, College of Chemistry and Chemical Engineering, Weifang University, Weifang, Shandong 261061, P. R. China. <https://orcid.org/0000-0002-0050-1900> (T. Xi-Shi)

Source of material

The synthetic method of 4-amino-*N'*-(4-aminobenzoyl)benzohydrazide monohydrate is as following: the mixture of 0.0755 g 4-aminobenzoic hydrazide (0.5 mmol), 0.0531 g thiocarbonylhydrazide (0.5 mmol) and neodymium(III) nitrate hexahydrate were dissolved in 20 mL (water-ethanol = 1:1) and continually stirred at 70 °C for 5.5 h. After cooled to room temperature, the solution was filtered to a small beaker. The colorless crystals of 4-amino-*N'*-(4-aminobenzoyl)benzohydrazide monohydrate were obtained by slow evaporation in 30 days.

Experimental details

The hydrogen atoms were positioned geometrically ($C-H = 0.93$ Å, $N-H = 0.84-0.91$ Å and $O-H = 0.85$ Å). Their U_{iso} values were set to $1.2U_{eq}$ or $1.5U_{eq}$ of the parent atoms.

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

Atom	x	y	z	U_{iso}^*/U_{eq}
O1	0.2071 (2)	0.46321 (10)	0.9185 (3)	0.0388 (5)
N1	0.0086 (3)	0.45863 (12)	0.6211 (4)	0.0344 (6)
H1	-0.065 (4)	0.4362 (16)	0.535 (5)	0.043 (9)*
N2	0.2120 (3)	0.08664 (13)	0.7009 (4)	0.0340 (6)
H21	0.244 (3)	0.0603 (16)	0.843 (5)	0.041*
H22	0.143 (3)	0.0603 (16)	0.613 (5)	0.041*
C1	0.0663 (3)	0.29030 (15)	0.5576 (5)	0.0258 (6)
H1A	0.0053	0.3163	0.4337	0.031*
C2	0.0859 (3)	0.20834 (15)	0.5456 (5)	0.0271 (6)
H2	0.0370	0.1796	0.4153	0.033*
C3	0.1785 (3)	0.16808 (15)	0.7274 (4)	0.0266 (6)
C4	0.2468 (3)	0.21238 (16)	0.9245 (5)	0.0308 (6)
H4	0.3063	0.1863	1.0499	0.037*
C5	0.2271 (3)	0.29412 (16)	0.9353 (5)	0.0303 (6)
H5	0.2751	0.3226	1.0669	0.036*
C6	0.1366 (3)	0.33522 (15)	0.7532 (4)	0.0249 (6)
C7	0.1201 (3)	0.42302 (15)	0.7733 (4)	0.0284 (6)
O2	0.5000	0.5000	0.6775 (5)	0.0486 (8)
H2O	0.4233	0.4862	0.7777	0.073*

Comment

Benzamide compounds may exhibit good biological activity in antitumor activity [5, 6], FGFR1 inhibitors [7], antibacterial agent [8], antialzheimer agent [9], and uPA inhibitor [10]. In our previous work, some benzamide compounds have been synthesized and structurally characterized [11, 12]. In this work, we synthesized a new benzamide compound, 4-amino-*N'*-(4-aminobenzoyl)benzohydrazide monohydrate, and its structure has also been determined.

The molecular structure of 4-amino-*N'*-(4-aminobenzoyl)benzohydrazide is shown in the Figure. The compound consists of one 4-amino-*N'*-(4-aminobenzoyl) benzohydrazide monohydrate molecule and one water molecule. The bond distance of C–O is 1.241(3) Å (C7–O1), which is similar to the C–O bond length [13, 14], indicating the C–O bond is double bond. The dihedral angle between two benzene rings ((C1–C2–C3–C4–C5–C6) and (C1A–C2A–C3A–C4A–C5A–C6A)) is 65.7°. In the crystal packing, the molecules form a 3D structure through N–H⋯O and O–H⋯O intermolecular hydrogen bonds.

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

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