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Crystal structure of 3-(3-(pyridin-3-yl)ureido) benzoic acid, $C_{13}H_{11}N_3O_3$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.25 × 0.23 × 0.21 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
$\theta_{\max}$, completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7453, 2045, 0.030
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1794
$N(\text{param})_{\text{refined}}$:	172
Programs:	Bruker programs [1], SHELX [2], SHELX [3], DIAMOND [4]

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Abstract

$C_{13}H_{11}N_3O_3$, Orthorhombic, $Pna2_1$ (no. 33), $a = 9.2062(6)$ Å, $b = 11.5258(7)$ Å, $c = 11.5263(7)$ Å, $V = 1223.04(13)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0377$, $wR_{\text{ref}}(F^2) = 0.1006$, $T = 293(2)$ K.

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The crystal 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 materials

A solution of pyridine-3-isocyanate (4 mmol) in ethanol (15 mL) was added to a stirred ethanol solution (15 mL) of *m*-aminobenzoic acid (4 mmol) in a three-necked flask. The reaction mixture was refluxed for 4 h, then the solution was

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.2743(3)	0.3323(2)	−0.0772(2)	0.0384(6)
H1	0.1810	0.3286	−0.0787	0.046*
N2	0.2597(3)	0.1570(2)	0.0141(2)	0.0370(6)
H2	0.1682	0.1713	0.0178	0.044*
N3	0.4868(3)	−0.0725(3)	0.1415(3)	0.0543(9)
O1	0.7452(3)	0.4245(2)	−0.2736(3)	0.0641(9)
H1A	0.8266	0.4289	−0.3023	0.096*
O2	0.7441(3)	0.6134(2)	−0.3080(3)	0.0695(9)
O3	0.4798(2)	0.23308(19)	−0.0301(3)	0.0527(7)
C1	0.6841(4)	0.5287(3)	−0.2727(3)	0.0476(9)
C2	0.5343(4)	0.5312(3)	−0.2218(3)	0.0399(8)
C3	0.4749(3)	0.4302(3)	−0.1753(3)	0.0374(7)
H3	0.5265	0.3609	−0.1794	0.045*
C4	0.3390(3)	0.4325(3)	−0.1230(3)	0.0361(7)
C5	0.2613(4)	0.5353(3)	−0.1212(3)	0.0510(10)
H5	0.1693	0.5375	−0.0881	0.061*
C6	0.3208(5)	0.6346(3)	−0.1688(4)	0.0613(11)
H6	0.2686	0.7037	−0.1671	0.074*
C7	0.4573(5)	0.6320(3)	−0.2187(3)	0.0532(10)
H7	0.4966	0.6993	−0.2502	0.064*
C8	0.3463(3)	0.2411(3)	−0.0308(3)	0.0338(6)
C9	0.3070(3)	0.0487(3)	0.0550(3)	0.0348(7)
C10	0.4423(4)	0.0318(3)	0.1048(3)	0.0449(9)
H10	0.5042	0.0950	0.1131	0.054*
C11	0.3976(4)	−0.1629(3)	0.1312(4)	0.0578(10)
H11	0.4304	−0.2358	0.1538	0.069*
C12	0.2598(4)	−0.1524(3)	0.0886(4)	0.0539(10)
H12	0.1979	−0.2161	0.0868	0.065*
C13	0.2141(4)	−0.0459(3)	0.0486(3)	0.0442(8)
H13	0.1215	−0.0374	0.0175	0.053*

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cooled to room temperature. The white precipitated residue was removed from the solution by filtration and then washed with ethanol and ether 3 times. Single crystals suitable for X-ray diffraction were obtained from an ethanol by slow evaporation at room temperature.

Experimental details

All hydrogen atoms were positioned geometrically, with the $d(\text{C}-\text{H}) = 0.97\text{--}0.99 \text{ \AA}$, $U_{\text{iso}}(\text{H}) = 1.2 \text{ times } U_{\text{eq}}(\text{C})$ and $U_{\text{iso}}(\text{H}) = 1.5 \text{ times } U_{\text{eq}}(\text{O})$.

Comment

Urea molecules are well studied in crystal engineering for the rational construction of supramolecular architectures and design of non-linear optical (NLO) materials for second harmonic generation (SHG) [5, 6].

In the crystal structure of the title compound, the pyridine ring and benzene ring are linked by an ureido moiety, forming a non-coplanar structure. The bond lengths and bond angles are in the normal ranges [7]. There are intramolecular hydrogen bonds (for example: C3—H3···O3) in the molecules. The carbonyl-oxygen atom in the carbamido group of one molecule forms hydrogen bonds (N1—H1···O3 and N2—H1···O3) with the two nitrogen atoms in the carbamido group of another molecule, which makes the molecule extended to form the one-dimensional polymeric (1D) structure. The three-dimensional (3D) structure is formed through

the extension of the hydrogen bond (O1—H1A···N3) of the one-dimensional (1D) structure.

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References

1. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2012).
2. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: *J. Appl. Cryst.* **42** (2009) 339–341.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
4. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Ver. 4.0. Crystal Impact, Bonn, Germany (2015).
5. George, S.; Nangia, A.; Lam, C. K.; Mak, T. C. W.; Nicoud, J. F.: Crystal engineering of urea α -network *via* I···O₂N synthon and design of SHG active crystal *N*-4-iodophenyl-*N'*-4'-nitrophenylurea. *Chem. Commun.* **10** (2004) 1202–1203.
6. Bai, X.; Jia, C.; Zhao, Y.; Yang, D.; Wang, S. C.; Li, A. Y.; Chan, Y. T.; Wang, Y. Y.; Yang, X. J.; Wu, B.: Peripheral templation-modulated interconversion between an A(4)L(6) tetrahedral anion cage and A(2)L(3) triple helicate with guest capture/release. *Angew. Chem. Int. Ed.* **57** (2018) 1851–1855.
7. Park, H.; Kim, J.; Kwon, E.; Kim, T. H.: Crystal structure of flucetosulfuron. *Acta Crystallogr. E* **73** (2017) 1439–1442.