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Crystal structure of 4-(3-(pyridin-3-yl)ureido) benzoic acid – adipic acid (2/1), C₁₆H₁₆N₃O₅

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.25 × 0.24 × 0.21 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.11 mm ⁻¹
Diffractometer, scan mode:	D8/APEX2, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4933, 2663, 0.016
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2131
$N(\text{param})_{\text{refined}}$:	217
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

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Abstract

C₁₃H₁₁N₃O₃ · C₃H₅O₂, triclinic, $P\bar{1}$ (no. 2), $a = 9.6481(6)$ Å, $b = 10.0123(6)$ Å, $c = 10.1667(6)$ Å, $\alpha = 116.680(2)^\circ$, $\beta = 113.268(2)^\circ$, $\gamma = 95.809(2)^\circ$, $V = 758.31(8)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0426$, $wR_{\text{ref}}(F^2) = 0.1212$, $T = 293(2)$ 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.

Source of material

A solution of pyridine-3-isocyanate (8 mmol) in ethanol (25 mL) was added to a stirred ethanol solution (30 mL) of *p*-aminobenzoic acid (8 mmol) in a three-necked flask.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.9537(2)	0.3885(2)	0.0968(2)	0.0416(5)
C2	0.9199(2)	0.3146(2)	0.1820(2)	0.0397(4)
C3	0.7715(2)	0.2072(2)	0.1142(2)	0.0423(5)
H3	0.6892	0.1775	0.0085	0.051*
C4	0.7439(2)	0.1438(2)	0.2007(2)	0.0440(5)
H4	0.6436	0.0726	0.1542	0.053*
C5	0.8681(2)	0.1875(2)	0.3590(2)	0.0399(4)
C6	1.0166(2)	0.2942(2)	0.4263(2)	0.0472(5)
H6	1.0998	0.3229	0.5311	0.057*
C7	1.0421(2)	0.3579(2)	0.3402(3)	0.0476(5)
H7	1.1418	0.4306	0.3878	0.057*
C8	0.7317(2)	0.0177(2)	0.4217(2)	0.0411(5)
C9	0.6736(2)	−0.1015(2)	0.5708(2)	0.0389(4)
C10	0.5186(2)	−0.2033(2)	0.4473(2)	0.0457(5)
H10	0.4685	−0.2074	0.3457	0.055*
C11	0.7411(2)	−0.1003(2)	0.7199(2)	0.0446(5)
H11	0.8451	−0.0314	0.8038	0.053*
C12	0.4403(3)	−0.2983(3)	0.4787(3)	0.0525(5)
H12	0.3361	−0.3681	0.3975	0.063*
C13	0.5152(3)	−0.2905(2)	0.6288(3)	0.0499(5)
H13	0.4602	−0.3557	0.6475	0.060*
C14	0.8047(2)	−0.2519(2)	0.0846(2)	0.0431(5)
C15	0.6331(2)	−0.3584(2)	−0.0187(2)	0.0414(5)
H15A	0.6118	−0.4346	−0.1318	0.050*
H15B	0.5649	−0.2946	−0.0288	0.050*
C16	0.5873(2)	−0.4482(2)	0.0530(2)	0.0435(5)
H16A	0.6533	−0.5142	0.0608	0.052*
H16B	0.6097	−0.3725	0.1668	0.052*
N1	0.7691(2)	0.0014(2)	0.5581(2)	0.0487(5)
H1	0.8621	0.0619	0.6457	0.058*
N2	0.8551(2)	0.1330(2)	0.4592(2)	0.0500(5)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H2	0.9367	0.1787	0.5592	0.060*
N3	0.6643(2)	−0.19289(19)	0.7491(2)	0.0460(4)
O1	0.60740(17)	−0.06004(16)	0.28759(16)	0.0515(4)
O2	0.83928(17)	0.33400(17)	−0.05790(17)	0.0523(4)
H2A	0.8676	0.3811	−0.0962	0.078*
O3	1.08046(18)	0.49416(18)	0.16600(18)	0.0582(4)
O4	0.85402(16)	−0.17033(17)	0.03139(18)	0.0519(4)
H4A	0.7919	−0.1238	0.0090	0.078*
O5	0.89897(18)	−0.2404(2)	0.21382(19)	0.0677(5)

The reaction mixture was refluxed for 7 h, after that the solution was cooled to room temperature. The white precipitated residue was washed with ether and ethanol 3 times. The white solids (0.2 mmol) were dissolved in 15 mL methanol. The adipic acid (0.1 mmol) was added. Single crystals were obtained from methanol by slow evaporation at room temperature.

Experimental details

The structure was solved using Charge Flipping and refined with the ShelXL [3] refinement. All hydrogen atoms were positioned geometrically, with the $d(\text{C}–\text{H}) = 0.97–0.99 \text{ \AA}$, $U_{\text{iso}}(\text{H}) = 1.2 \text{ times } U_{\text{eq}}(\text{C})$ and $U_{\text{iso}}(\text{H}) = 1.5 \text{ times } U_{\text{eq}}(\text{O})$.

Comment

As a double hydrogen bond donor, ureido derivatives may form double hydrogen bonds to a Y-type hydrogen bond binding site for example acetate or tetrahedral anions (such as SO_4^{2-}) [5]. It is more likely that a hydrogen bond may be formed between ureido and ureido, or between ureido and other molecules, etc., which may assemble into supramolecular compounds. This phenomenon has caused some attention [6].

In the crystal structure of the title compound, the asymmetric unit of the title compound consists of one formula unit of 4-(3-(pyridin-3-yl)ureido) benzoic acid and one half adipic acid (see the figure). The pyridine ring and benzoic

acid fragment are linked by an ureido moiety, forming a non-coplanar structure. The dihedral angle between the rings is 176.04° . The bond lengths and bond angles are in the normal ranges [7, 8]. There are some weak non-classical intramolecular hydrogen bonds (for example: $\text{C4}–\text{H4} \cdots \text{O1}$, $\text{C10}–\text{H10} \cdots \text{O1}$) in the molecules. The carboxyl group and carboxyl group of the title compound form a dimer through the double hydrogen bond ($\text{O2}–\text{H2A} \cdots \text{O3}$), and the nitrogen atom on the urea group and the nitrogen atom on the pyridine ring form a hydrogen bond ($\text{N1}–\text{H1} \cdots \text{O5}$, $\text{N2}–\text{H1} \cdots \text{O5}$ and $\text{O4}–\text{H4A} \cdots \text{N3}$) with the carboxyl group of adipic acid respectively to expand into a two-dimensional network structure.

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