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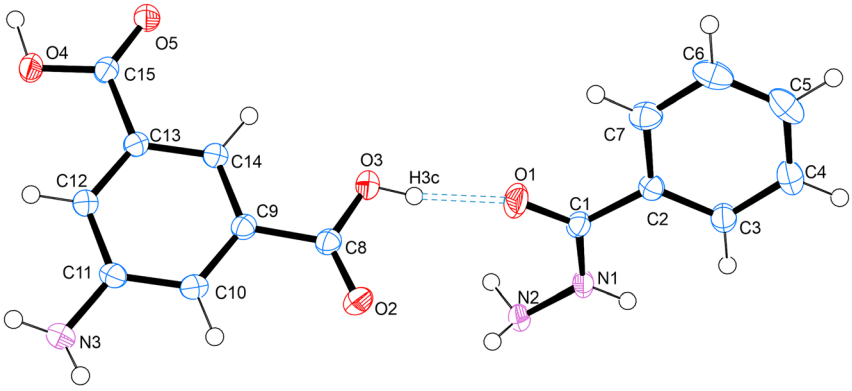
The crystal structure of the co-crystal composed of benzhydrazide and 5-aminoisophthalic acid, $C_8H_7NO_4{\cdot}C_7H_8N_2O$



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Abstract

$C_8H_7NO_4{\cdot}C_7H_8N_2O$, monoclinic, $P2_1/n$ (no. 14), a = 18.3690(6) Å, b = 3.8010(1) Å, c = 21.5232(7) Å, β = 108.867(1)°, V = 1422.02(8) Å^3, Z = 4, $R_{gt}(F)$ = 0.0340, $wR_{ref}(F^2)$ = 0.0959, T = 173 K.

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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:	Yellow block
Size:	0.38 × 0.24 × 0.13 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.11 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	28.0°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	64276, 3409, 0.052
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3,194
$N(param)_{refined}$:	234
Programs:	Bruker,[1] SHELX,[2,3] ORTEP,[4] WinGX,[5] PLATON[6]

1 Source of materials

Benzhydrazide and 5-amino-isophthalic acid were both purchased commercially and not purified any further. An amount of 0.0214 g of benzhydrazide (0.157 mmol) and 0.0132 g 5-aminoisophthalic acid (0.0728 mmol) were added to a vial and dissolved in 3 ml of AP-grade methanol. The mixture was stirred and heated to 100 °C for 1 min, and then placed in the fridge (8 °C) overnight. The solution was then allowed to slowly evaporate at room temperature with cap being left slightly open. Yellow block crystals formed after three days.

2 Experimental details

C-bound hydrogen atoms were located in the difference map then positioned geometrically and were allowed to ride on their respective parent atoms with thermal displacement parameters 1.2 times of the parent C atom. The coordinates

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.67077 (5)	0.4839 (3)	0.52327 (5)	0.0220 (2)
C2	0.64854 (5)	0.3871 (3)	0.58163 (5)	0.0215 (2)
C3	0.69557 (6)	0.4382 (3)	0.64611 (5)	0.0259 (2)
H3C	0.745701	0.533785	0.65502	0.031*
C4	0.66878 (7)	0.3486 (3)	0.69729 (5)	0.0317 (2)
H4A	0.700354	0.387623	0.741369	0.038*
C5	0.59646 (7)	0.2029 (3)	0.68470 (6)	0.0351 (3)
H5	0.578847	0.139029	0.720076	0.042*
C6	0.54983 (7)	0.1501 (3)	0.62075 (7)	0.0354 (3)
H6	0.500146	0.050023	0.612111	0.043*
N1	0.73497 (5)	0.6721 (2)	0.53332 (4)	0.02459 (19)
H1	0.7645 (8)	0.737 (4)	0.5720 (7)	0.03*
N2	0.75807 (5)	0.7979 (3)	0.48085 (4)	0.02526 (19)
O1	0.63121 (5)	0.3933 (2)	0.46747 (4)	0.0358 (2)
C7	0.57550 (6)	0.2429 (3)	0.56932 (6)	0.0292 (2)
H7	0.543162	0.20826	0.525346	0.035*
C8	0.62663 (5)	0.2308 (3)	0.31247 (5)	0.0231 (2)
C9	0.59276 (5)	0.1998 (3)	0.23993 (5)	0.02112 (19)
C10	0.63734 (5)	0.0515 (3)	0.20534 (5)	0.0227 (2)
H10	0.688242	−0.024467	0.22824	0.027*
C11	0.60787 (5)	0.0135 (3)	0.13737 (5)	0.0226 (2)
C12	0.53323 (6)	0.1313 (3)	0.10492 (5)	0.0238 (2)
H12	0.512502	0.109934	0.058529	0.029*
C13	0.48881 (5)	0.2795 (3)	0.13957 (5)	0.0219 (2)
C14	0.51800 (5)	0.3132 (3)	0.20753 (5)	0.0215 (2)
H14	0.487512	0.411739	0.23136	0.026*
C15	0.40987 (6)	0.4131 (3)	0.10450 (5)	0.0244 (2)
N3	0.65209 (5)	−0.1260 (3)	0.10164 (5)	0.0287 (2)
O2	0.68833 (4)	0.1043 (2)	0.34397 (4)	0.03175 (19)
O3	0.58273 (4)	0.4088 (2)	0.33915 (4)	0.03042 (19)
H3	0.6050 (9)	0.414 (4)	0.3824 (9)	0.046*
O4	0.39040 (5)	0.3872 (3)	0.03990 (4)	0.0378 (2)
O5	0.36787 (4)	0.5381 (2)	0.13213 (4)	0.03225 (19)
H3A	0.6925 (10)	−0.244 (4)	0.1253 (8)	0.044 (4)*
H3B	0.6236 (10)	−0.231 (5)	0.0637 (9)	0.049 (4)*
H2A	0.7175 (9)	0.918 (4)	0.4520 (7)	0.035 (4)*
H2B	0.7696 (9)	0.614 (4)	0.4586 (8)	0.045 (4)*
H4	0.3381 (11)	0.491 (5)	0.0177 (9)	0.064 (5)*

and isotropic displacement parameters of the N-bound and O-bound H atoms involved in hydrogen bonding interactions were allowed to refine freely. Diagrams and publication material were generated using ORTEP-3,[4] WinGX[5] and PLATON.[6]

3 Comment

Benzhydrazide also known as benzohydrazide and its derivatives have become popular due to their biological properties, such as anti-bacterial, anti-fungal, anti-cancer and anti-tubercular activity.[7] Benzhydrazide in its pure form crystallizes in the $P2_1/n$ space group.[8]

5-aminoisophthalic acid, a dicarboxylic acid with an amino group is used a lot in the metal organic frameworks of co-ordination chemistry,[9] but is also used in co-crystal engineering.[10] The 5-aminoisophthalic acid can react with a number of aldehydes to form Schiff bases, due to the amino group, by removing water molecules.[11] 5–Aminoisophthalic acid in its pure form crystallizes in the *Pbcn* space group.[12]

The benzhydrazide·5-aminoisophthalic acid asymmetric unit contains one molecule of benzhydrazide and one molecule of 5-aminoisophthalic acid (see the figure). There is a single hydrogen bond between the two molecules making a discrete hydrogen bond via O1···H3c–O3.

With regard to packing, each benzhydrazide molecule is hydrogen bonded to four 5-aminoisophthalic acid molecules via O1···H3c–O3, O2···H2a–N2, N3···H2b–N2, N2···H4a–O4 and O5···H1–N1. Each aminoterephthalic acid is hydrogen bonded to four benzhydrazide molecules and two aminoterephthalic acid molecules.

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