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Redetermination of 3-hydroxybenzamide

Lars Kr. Hansen,[a]* German L. Perlovich[b,c] and Annette Bauer-Brandl[b]

[a]Department of Chemistry, University of Tromsø, 9037 Tromsø, Norway, [b]Department of Pharmaceutics and Biopharmaceutics, University of Tromsø, 9037 Tromsø, Norway, and [c]Institute of Solution Chemistry, Russian Academy of Sciences, 153045 Ivanovo, Russian Federation

Correspondence e-mail: larsk@chem.uit.no

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.033
Data-to-parameter ratio = 9.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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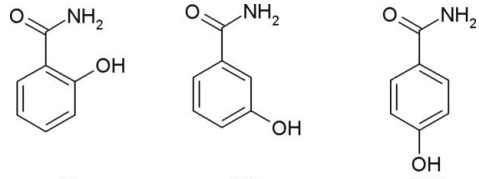
The crystal structure of the title compound, $C_7H_7NO_2$, has been redetermined [Katsube, Y. (1966). *Bull. Chem. Soc. Jpn*, **39**, 2576–2588] to higher precision and with the hydrogen-bonding scheme established.

Comment

Hydroxybenzamides (I), (II) and (III) are often used as pro-drug compounds to model various physico-chemical processes of the drug molecules. Their different hydrogen-bonding patterns help to establish their structures and determine their solubilities.



(I) (II) (III)

The crystal structure of 2-hydroxybenzamide (salicylamide), (I), has been described in detail in the literature (Sasada *et al.*, 1964; Pertlik, 1990), whereas the structure of 4-hydroxybenzamide, (III), has not been reported at all. The structure of 3-hydroxybenzamide, (II), was studied some time ago by Katsube (1966) to moderate precision. Here we present a high-precision redetermination of (II) (Fig. 1) and describe its hydrogen-bonding scheme (Table 1).

The bond lengths and angles for (II) are within their normal ranges (Allen *et al.*, 1987). The data obtained by us for the non-H atoms are consistent with Katsube's, but improved by about a factor of twenty in precision. For example, C7—O1 = 1.245 (2) Å, compared with 1.24 (7) Å in Katsube's study. The dihedral angle between the mean plane of the aromatic ring and the plane of C7/N1/O1 is 22.9 (2)°.

The packing of (II) is shown in Fig. 2. The molecules form (101) layers held together by N—H···O and O—H···O hydrogen bonds. The layers interact with each other by van der Waals forces. The hydrogen-bond network can be described by the graph set assignments introduced by Etter (1990) as $C(4)$, $C(8)$, and $R_2^2(14)$.

Experimental

A commercial sample of 3-hydroxybenzamide (Sigma–Aldrich Co. Ltd, St Louis, USA) was used. Crystals of (II) were grown by slow evaporation of a methanol solution.

Crystal data

$C_7H_7NO_2$
M_r = 137.14
Monoclinic, $P2_1/n$
a = 10.873 (5) Å
b = 5.064 (2) Å
c = 11.641 (5) Å
β = 92.414 (11)°
V = 640.4 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293.1 K
0.50 × 0.20 × 0.10 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.938, T_{max} = 0.989
5804 measured reflections
1707 independent reflections
1103 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.033
S = 1.80
1103 reflections
119 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H8···O2^{i}	0.93 (2)	2.07 (2)	2.990 (2)	168 (2)
N1—H7···O1^{ii}	0.90 (2)	2.15 (2)	2.988 (2)	153 (2)
O2—H6···O1^{iii}	0.97 (2)	1.86 (2)	2.798 (2)	164 (2)

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $x, y+1, z$; (iii) $-x+2, -y+1, -z+1$.

The crystals were of poor quality and weakly diffracting, which accounts for the low fraction of measured reflections. The H atoms were located in difference maps and their positions and U_{iso} values were freely refined [C—H = 0.963 (18)–1.007 (18) Å].

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2005); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEX* (McArdle, 1993) and *ORTEPIII* (Burnett & Johnson, (1996); software used to prepare material for publication: *CrystalStructure*.

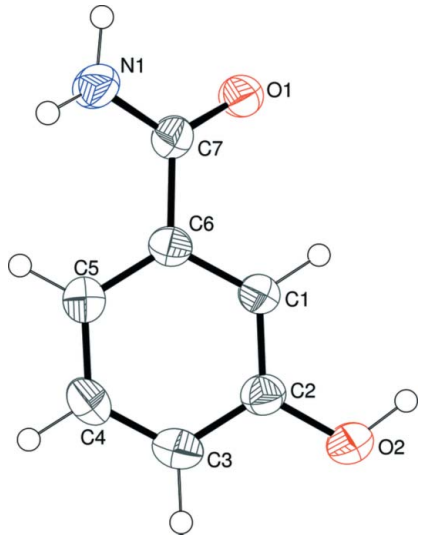


Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 40% probability level (arbitrary spheres for the H atoms).

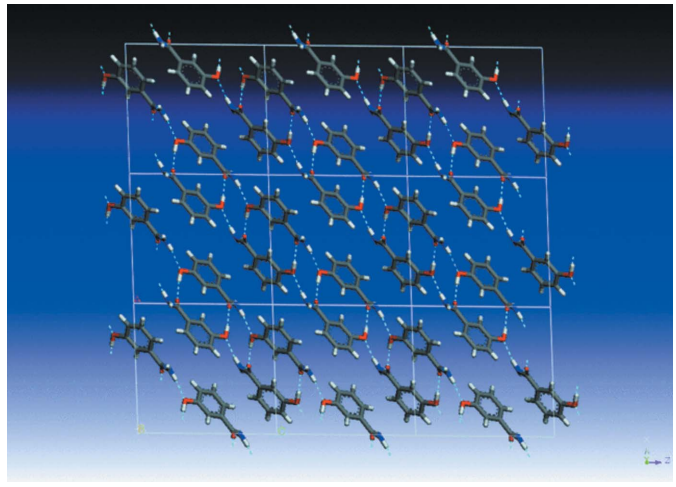


Figure 2
The packing of (I) with hydrogen bonds indicated by dashed lines.

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