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Redetermination of 3-hydroxy-2-naphthoic acid

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.070
Data-to-parameter ratio = 18.0

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

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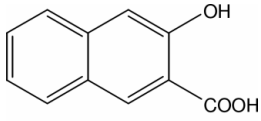
The title compound, $C_{11}H_8O_3$, previously reported by Gupta & Dutta [*Cryst. Struct. Commun.* (1975), **4**, 37–40] has been rerefined against new intensity data. Geometric parameters of the C and O atoms agree quite well. However, the positions of the hydroxyl H atoms differ slightly. Furthermore, the results of the present structure determination are of significantly higher precision.

Comment

3-Hydroxy-2-naphthoic acid, (I), also known as β-oxy-naphthoic acid (BONA, BONS), is produced industrially in a 1000 ton scale. It is used for syntheses of red azo pigments (Herbst & Hunger, 1997). Our intention was to synthesize its Cu salt from $CuSO_4$ and 3-hydroxy-2-naphthoic acid. However, it turned out that the resulting crystals were composed of the starting material 3-hydroxy-2-naphthoic acid.



(I)

A perspective view of (I) is shown in Fig. 1. The original structure was reported by Gupta & Dutta (1975). The geometric parameters of the C and O atoms of both determinations agree quite well, but the positions of the hydroxyl H atoms differ slightly. A least-squares fit between all non-H atoms of the two structures gives an r.m.s. deviation of

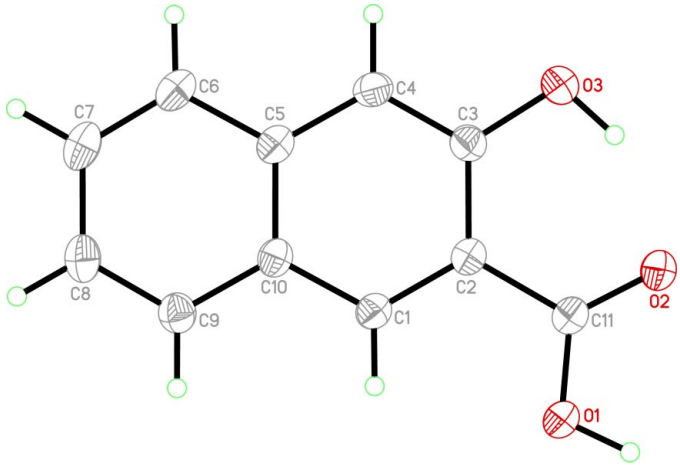


Figure 1
Perspective view of (I), with the atom-numbering scheme. Displacement ellipsoids are at the 50% probability level.

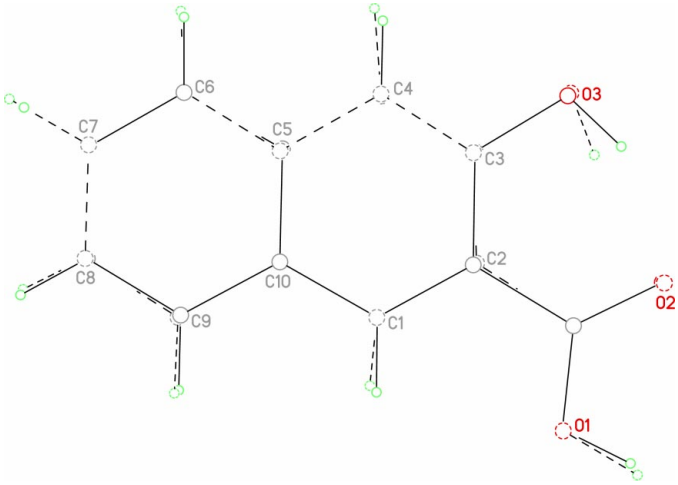


Figure 2
Least-squares fit of the present structure determination (full lines) with that performed by Gupta & Dutta (1975) (dashed lines).

0.0337 Å (Fig. 2). In addition, the present work is of significantly improved precision. The molecules are essentially planar (r.m.s. deviation for all non-H atoms: 0.0265 Å) and crystallize as hydrogen-bonded dimers. Furthermore, an intramolecular hydrogen bond is formed (Table 1).

Experimental

In a test tube, a spatula tip of 3-hydroxy-2-naphthoic acid was dissolved in diethyl ether. In a second test tube, a saturated solution of copper(II) sulfate was prepared. Then, a layer of the 3-hydroxy-2-naphthoic acid solution was placed over the $CuSO_4$ solution in the test tube. After 4 d, the diethyl ether had evaporated and small yellow crystals of (I) were obtained.

Crystal data

$C_{11}H_8O_3$
M_r = 188.17
Monoclinic, $P2_1/c$
a = 9.9942 (10) Å
b = 11.6591 (11) Å
c = 7.6298 (9) Å
β = 104.392 (9)°
V = 861.15 (16) Å^3
Z = 4
D_x = 1.451 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9721 reflections
θ = 3.5–29.8°
μ = 0.11 mm^{-1}
T = 173 (2) K
Plate, light yellow
0.36 × 0.34 × 0.15 mm

Data collection

Stoe IPDS II two-circle diffractometer
ω scans
Absorption correction: none
12620 measured reflections
2450 independent reflections
1540 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 29.8°
$h = -13 \rightarrow 13$
$k = -16 \rightarrow 16$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.070
S = 0.96
2450 reflections
136 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.011 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H11···O2^{i}	0.936 (19)	1.715 (19)	2.6484 (11)	174.3 (18)
O3—H10···O2	0.929 (18)	1.787 (18)	2.6326 (11)	149.9 (16)

Symmetry code: (i) $2-x, 1-y, 2-z$.

All H atoms were located in difference Fourier syntheses. H atoms bonded to C atoms were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C)], using a riding model with C—H_{methyl} = 0.95 Å. H atoms bonded to O atoms were refined freely. The O—H lengths are in the range 0.929 (18)–0.936 (19) Å.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991).

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