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Redetermination of the crystal structure of 3,3,3-triphenylpropanoic acid, $C_{21}H_{18}O_2$ – Deposition of hydrogen atomic coordinates

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.42 × 0.21 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	28.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	22,458, 3952, 0.023
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3109
$N(\text{param})_{\text{refined}}$:	209
Programs:	Bruker [1, 2], SHELX [3], WinGX/ORTEP [4], Mercury [5], PLATON [6]

list of the atoms including atomic coordinates and displacement parameters.

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Abstract

$C_{21}H_{18}O_2$, triclinic, $P\bar{1}$ (no. 2), $a = 9.8213(6)$ Å, $b = 9.9212(6)$ Å, $c = 10.0400(6)$ Å, $\alpha = 65.510(2)^\circ$, $\beta = 63.539(2)^\circ$, $\gamma = 84.842(2)^\circ$, $V = 791.59(8)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0463$, $wR_{\text{ref}}(F^2) = 0.1101$, $T = 200$ K.

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The molecular structure is shown in the Figure. Table 1 contains crystallographic data, and Table 2 contains the

Source of material

The compound was obtained commercially (Aldrich). Crystals suitable for the diffraction study were taken directly from the product provided.

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å for aromatic carbon atoms, C–H 0.99 Å for the methylene group) and were included in the refinement in the riding model approximation, with $U(H)$ set to $1.2U_{\text{eq}}(C)$.

The H atom of the hydroxyl group was allowed to rotate with a fixed angle around the C–O bond to best fit the experimental electron density (HFIX 147 in the SHELX program suite [3]), with $U(H)$ set to $1.5U_{\text{eq}}(O)$.

Comment

While the first members of the homologous series of aliphatic carboxylic acids constitute a family of liquid

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.81381 (13)	0.95368 (14)	0.18355 (15)	0.0454 (3)
H1	0.900134	1.003734	0.132579	0.068*
O2	0.91625 (12)	0.87683 (14)	−0.01574 (15)	0.0456 (3)
C1	0.80645 (16)	0.88018 (16)	0.10321 (18)	0.0305 (3)
C2	0.64473 (15)	0.80895 (17)	0.17788 (18)	0.0310 (3)
H2A	0.579379	0.890711	0.164930	0.037*
H2B	0.610978	0.751898	0.297020	0.037*
C3	0.60741 (14)	0.70288 (15)	0.11586 (16)	0.0233 (3)
C11	0.43200 (14)	0.69591 (14)	0.17481 (16)	0.0235 (3)
C12	0.33224 (16)	0.66003 (16)	0.33964 (17)	0.0300 (3)
H12	0.372971	0.640437	0.414901	0.036*
C13	0.17508 (16)	0.65255 (18)	0.39514 (19)	0.0361 (3)
H13	0.109402	0.628144	0.507641	0.043*
C14	0.11298 (16)	0.68042 (18)	0.2877 (2)	0.0360 (3)
H14	0.005196	0.676079	0.325815	0.043*
C15	0.20923 (16)	0.71448 (17)	0.1251 (2)	0.0334 (3)
H15	0.167468	0.732618	0.050963	0.040*
C16	0.36747 (15)	0.72250 (15)	0.06865 (17)	0.0278 (3)
H16	0.432408	0.746449	−0.043904	0.033*
C21	0.65180 (15)	0.54540 (15)	0.19076 (16)	0.0261 (3)
C22	0.80182 (17)	0.52637 (19)	0.16792 (19)	0.0378 (4)
H22	0.874139	0.611566	0.115692	0.045*
C23	0.8473 (2)	0.3852 (2)	0.2201 (2)	0.0470 (4)
H23	0.950141	0.374804	0.203098	0.056*
C24	0.7445 (2)	0.2600 (2)	0.2965 (2)	0.0481 (5)
H24	0.776344	0.163443	0.330873	0.058*
C25	0.5952 (2)	0.27615 (19)	0.3225 (2)	0.0451 (4)
H25	0.523155	0.190391	0.376629	0.054*
C26	0.54960 (18)	0.41780 (17)	0.26968 (19)	0.0352 (3)
H26	0.446309	0.427323	0.288051	0.042*
C31	0.68874 (14)	0.76403 (14)	−0.07229 (16)	0.0234 (3)
C32	0.70684 (17)	0.91619 (16)	−0.17119 (19)	0.0337 (3)
H32	0.673764	0.984383	−0.121934	0.040*
C33	0.7721 (2)	0.96903 (18)	−0.3394 (2)	0.0450 (4)
H33	0.784257	1.073210	−0.404599	0.054*
C34	0.82012 (19)	0.87222 (19)	−0.41482 (19)	0.0418 (4)
H34	0.865532	0.909320	−0.530847	0.050*
C35	0.80099 (16)	0.72135 (17)	−0.31882 (18)	0.0315 (3)
H35	0.832393	0.653567	−0.368712	0.038*
C36	0.73593 (15)	0.66801 (15)	−0.14932 (17)	0.0259 (3)
H36	0.723429	0.563687	−0.084657	0.031*

substances at room temperature, the introduction of halogen atoms, additional hydroxyl groups or aromatic moieties in the backbone quickly results in a marked increase of melting points and subsequent tendency to form crystalline substances at ambient conditions which allows for the determination of metrical parameters on grounds of diffraction studies which enables comparative studies in case the acids are to be encountered as ligands in

coordination compounds. In continuation of our interest in the structures of functionalized carboxylic acids [7–14] the crystal and molecular structure of the title compound was determined to gain an understanding for the effects the introduction of three bulky phenyl groups on the terminal carbon atom will have. Structural information is at hand for a co-crystallizate of the title compound and 2-aminopyrimidine [15]. Furthermore, the structure of the title compound as such has been reported earlier [16], however, while the author included the hydrogen atoms during the refinement of the structure model no coordinates of the latter atoms were deposited. As this precludes an assessment of the influence hydrogen-supported interactions might have on the overall structure that go beyond the apparent patterns derivable from plain geometric positioning of these lighter atoms on their respective carrier atoms the present study – whose quality is higher than the data already at hand – was conducted to close this gap.

The title crystal structure shows the presence of a derivative of propanoic acid bearing three phenyl groups as substituents on the terminal carbon atom. C–O bond lengths of 1.2120(18) and 1.3179(17) Å are in good agreement with values reported for other carboxylic acids whose metrical parameters have been deposited with the Cambridge Structural Database [17]. Intracyclic C–C–C angles in all three phenyl rings show vast similarity with the smallest and the largest angle pairing up as 117.60(12)° and 121.17(13)°, 117.42(14)° and 121.39(15)° and 117.61(13)° and 121.40(13)°, respectively, with the smallest angle invariably found on the carbon atom directly bonded to the alkyl scaffold and the largest angle directly in *ortho*-position to the latter atom. The least-squares planes as defined by the respective carbon atoms of the three individual aromatic moieties enclose angles of 75.95(8)°, 79.14(8)° and 83.92(9)°.

In the crystal, classical hydrogen bonds of the O–H...O type are observed that give rise to the familiar pattern of centrosymmetric carboxylic acid dimer formation in between the carboxyl groups of two molecules. In terms of graph-set analysis [18, 19], the descriptor for these hydrogen bonds is $R_2^2(8)$ on the unary level. Furthermore, one can discuss the presence of four C–H... π contacts with three of the latter observed intramolecular between the propeller-like orientated phenyl groups. The last C–H... π contact extends the classical carboxylic acid dimers to infinite chains along [1 1 0]. π -Stacking is not a prominent feature in the crystal structure of the title compound as the shortest distance in between two centers of gravity was measured at 4.7613(10) Å.

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