

1986

CARBA-BICYCLOMYCIN I

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Phenylacetic Acid

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Abstract. $C_8H_8O_2$, $M_r = 136.1$, monoclinic, $P2_1/a$, $a = 10.201$ (5), $b = 4.9568$ (14), $c = 14.437$ (10) Å, $\beta = 99.17$ (5)°, $V = 720.6$ (6) Å³, $Z = 4$, $D_m = 1.25$ (2), $D_x = 1.255$ g cm⁻³, Mo $K\alpha$ ($\lambda = 0.71073$ Å), $\mu = 0.084$ mm⁻¹, $F(000) = 288$, $T = 295$ K, $R = 0.0412$, $wR = 0.0388$ for 903 unique observed reflections. The compound is formed by decarboxylation of phenylmalonic acid by CaO in aqueous medium under mild conditions. The hydrogen-bonding pattern in the crystals is the centrosymmetric head-to-tail form normally seen in carboxylic acids.

Experimental. Colorless hexagonal plates obtained from the apparent decarboxylation of phenylmalonic acid in the presence of CaO in aqueous medium. D_m by flotation. Crystal of dimensions $1.13 \times 0.70 \times 0.25$ mm. Nicolet R3m/V diffractometer, Mo $K\alpha$ radiation, graphite monochromator; lattice parameters obtained by least-squares analysis of 28 reflections in the range $20.0 \leq 2\theta(\text{Mo}) \leq 30.0^\circ$; data collected in $\theta/2\theta$ scan mode up to 50.0° in 2θ ; $0 \leq h \leq 12$, $0 \leq k \leq 5$, and $-17 \leq l \leq 17$; 1513 total reflections, 1270 independent reflections [$F > 6\sigma(F)$]. Absorption correction not applied. The intensities of four standard reflections, measured after every 50 reflections, exhibited a maximum fluctuation of 1.7%. Structure solved by direct methods. Non-H atoms refined anisotropically, H atoms refined isotropically. Function minimized was $\sum w(F_o - F_c)^2$, $w = [\sigma^2(F) + 0.00001F^2]^{-1}$. Final $R = 0.0412$, $wR = 0.0388$, $S = 3.467$ for 903 reflections, 123 parameters, $(\Delta/\sigma)_{\max} = 0.199$, $\Delta\rho_{\max} = 0.14$, $\Delta\rho_{\min} = 0.00$ e Å⁻³. All calculations and scattering factors using the Nicolet SHELXTL-Plus system (Sheldrick, 1987).

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Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for phenylacetic acid

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O(1)	897 (1)	524 (3)	1053 (1)	89 (1)
O(2)	1099 (2)	-2850 (3)	102 (1)	96 (1)
C(1)	1439 (2)	-1534 (4)	881 (1)	70 (1)
C(2)	2533 (3)	-2869 (6)	1530 (2)	100 (1)
C(1P)	3055 (2)	-1297 (4)	2392 (1)	71 (1)
C(2P)	4031 (2)	542 (5)	2384 (2)	91 (1)
C(3P)	4541 (3)	2009 (6)	3155 (3)	115 (1)
C(4P)	4065 (4)	1611 (7)	3969 (2)	119 (1)
C(5P)	3111 (4)	-194 (8)	4012 (2)	119 (1)
C(6P)	2579 (3)	-1688 (6)	3227 (2)	99 (1)

Table 2. Bond lengths (Å) and angles (°) for phenylacetic acid

O(1)—C(1)	1.205 (3)	O(2)—C(1)	1.298 (3)
C(1)—C(2)	1.493 (4)	C(2)—C(1P)	1.493 (4)
C(1P)—C(2P)	1.351 (3)	C(1P)—C(6P)	1.383 (4)
C(2P)—C(3P)	1.361 (4)	C(3P)—C(4P)	1.355 (5)
C(4P)—C(5P)	1.331 (6)	C(5P)—C(6P)	1.389 (4)
O(1)—C(1)—O(2)	122.4 (2)	O(1)—C(1)—C(2)	124.6 (2)
O(2)—C(1)—C(2)	113.0 (2)	C(1)—C(2)—C(1P)	115.1 (2)
C(2)—C(1P)—C(2P)	120.7 (2)	C(2)—C(1P)—C(6P)	121.7 (2)
C(2P)—C(1P)—C(6P)	117.6 (2)	C(1P)—C(2P)—C(3P)	122.7 (3)
C(2P)—C(3P)—C(4P)	119.2 (3)	C(3P)—C(4P)—C(5P)	120.2 (3)
C(4P)—C(5P)—C(6P)	121.0 (3)	C(1P)—C(6P)—C(5P)	119.3 (3)

Atomic parameters are listed in Table 1,† and interatomic distances and angles are presented in Table 2. A view of a single molecule, giving the atomic

† Lists of structure amplitudes, anisotropic thermal parameters, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54013 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

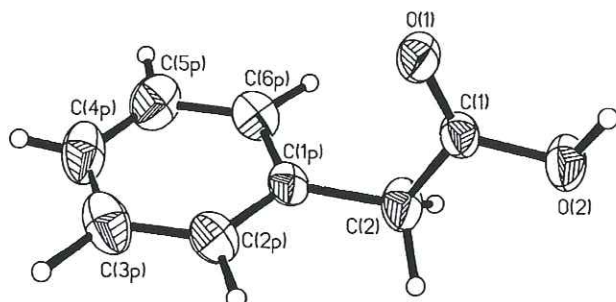


Fig. 1. View of a single molecule of phenylacetic acid. Thermal ellipsoids at the 50% probability level, with H atoms of arbitrary size.

numbering scheme, is shown in Fig. 1, and the intermolecular hydrogen-bonding pattern is depicted in Fig. 2.

Related literature. Decarboxylations of carboxylic acids are not uncommon (Fitzpatrick & Hopgood, 1974; de Meester & Hodgson, 1976, 1978). The structure of phenylacetic acid was initially solved by Patterson (1927) as one of his pioneering crystallographic studies, and the structure of a potassium salt has been reported (Manojlović & Speakman, 1968). Carboxylic acids generally form centrosymmetric hydrogen-bonded pairs (Haas & Brenner, 1966) and, as is shown in Fig. 2, phenylacetic acid is

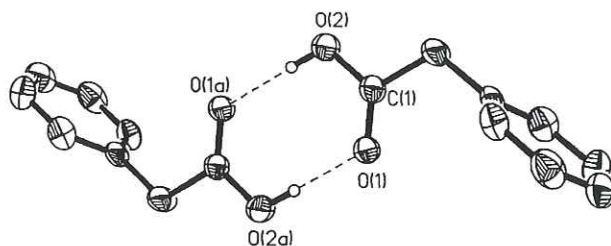


Fig. 2. Hydrogen bonding between pairs of phenylacetic acid molecules. Atoms labelled 'a' are related to the 'parent' atoms by inversion through the center at the origin.

normal in this regard. In the present case, the $O(2) \cdots O(1a)$ and $H \cdots O(1a)$ distances are 2.679 (3) and 1.61 (3) Å, while the associated $O(2)-H \cdots O(1a)$ angle is 174 (2)°.

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Structure of *N*-Methylurotropinium Iodide

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Abstract. 1-Methyl-1,3,5,7-tetraazatricyclo[3.3.1.1^{3,7}]-decan-1-ium iodide, $[(CH_2)_6N_4CH_3]I$, $M_r = 282.13$, monoclinic, $P2_1$, $a = 6.630$ (2), $b = 7.254$ (2), $c = 10.878$ (2) Å, $\beta = 105.04$ (7)°, $V = 505.3$ (2) Å³, $Z = 2$, $D_x = 1.854$ Mg m⁻³, $\lambda(Mo K\alpha) = 0.7107$ Å, $\mu = 3.093$ mm⁻¹, $F(000) = 276$, $T = 293$ (2) K, final $R = 0.048$ for 2734 observed reflections. The structure

consists of *N*₁-methylurotropinium cations and I⁻ anions. The C—N distances and bond angles (C—N—C and N—C—N) vary from 1.415 (8) to 1.572 (8) Å and from 103.0 (5) to 115.9 (5)°, respectively. I⁻ is surrounded by seven H atoms at distances in the range 2.90 (5)–3.29 (5) Å. N atoms are 4.391 (7) Å away from I⁻.

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