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# Phenylacetic Acid

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**Abstract.** $C_8H_8O_2$, $M_r = 136 \cdot 1$, monoclinic, $P2_1/a$, $a = 10 \cdot 201$ (5), $b = 4 \cdot 9568$ (14), $c = 14 \cdot 437$ (10) Å, $\beta = 99 \cdot 17$ (5)°, $V = 720 \cdot 6$ (6) Å$^3$, $Z = 4$, $D_m = 1 \cdot 25$ (2), $D_x = 1 \cdot 255$ g cm$^{-3}$, Mo $K\alpha$ ($\lambda = 0 \cdot 71073$ Å), $\mu = 0 \cdot 084$ mm$^{-1}$, $F(000) = 288$, $T = 295$ K, $R = 0 \cdot 0412$, $wR = 0 \cdot 0388$ 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 $1 \cdot 13 \times 0 \cdot 70 \times 0 \cdot 25$ mm. Nicolet *R3m/V* diffractometer, Mo $K\alpha$ radiation, graphite monochromator; lattice parameters obtained by least-squares analysis of 28 reflections in the range $20 \cdot 0 \leq 2\theta(\text{Mo}) \leq 30 \cdot 0°$; data collected in $\theta/2\theta$ scan mode up to $50 \cdot 0°$ in $2\theta$; $0 \leq h \leq 12$, $0 \leq k \leq 5$, 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 $1 \cdot 7\%$. Structure solved by direct methods. Non-H atoms refined anisotropically, H atoms refined isotropically. Function minimized was $\sum w(F_o - F_c)^2$, $w = [\sigma^2(F) + 0 \cdot 00001F^2]^{-1}$. Final $R = 0 \cdot 0412$, $wR = 0 \cdot 0388$, $S = 3 \cdot 467$ for 903 reflections, 123 parameters, $(\Delta/\sigma)_{max} = 0 \cdot 199$, $\Delta\rho_{max} = 0 \cdot 14$, $\Delta\rho_{min} = 0 \cdot 00$ 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* (Å$^2 \times 10^3$) *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.
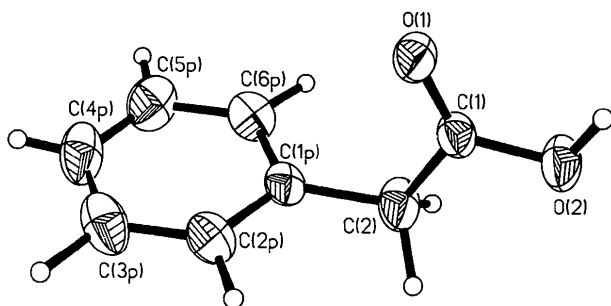
Fig. 1. View of a single molecule of phenylacetic acid. Thermal ellipsoids at the 50% probility level, with H atoms of arbitrary size.

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
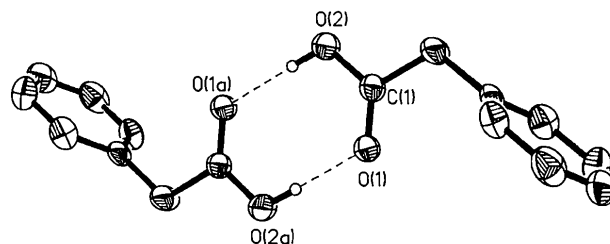


Fig. 2. Hydrogen bonding between pairs of phenylacetic acid molecules. Atoms labelled 'a' are related to the 'parent' atoms by inversion through the center at the origin.

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$_2$)$_6$N$_4$CH$_3$]I, $M_r = 282\cdot13$, monoclinic, $P2_1$, $a = 6\cdot630$ (2), $b = 7\cdot254$ (2), $c = 10\cdot878$ (2) Å, $\beta = 105\cdot04$ (7)°, $V = 505\cdot3$ (2) Å$^3$, $Z = 2$, $D_x = 1\cdot854$ Mg m$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·7107 Å, $\mu = 3\cdot093$ mm$^{-1}$, $F(000) = 276$, $T = 293$ (2) K, final $R = 0\cdot048$ 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$^-$.