

Fig. 1. Molecular structure and numbering scheme for tris-(2-pyridyl)phosphine. Hydrogen atoms are numbered according to the C atom to which they are bonded (Johnson, 1971).

reflections $\bar{5}14$, $\bar{6}14$ and $\bar{7}14$ were excluded from the final refinement owing to poor agreement. At convergence $R = 0.046$, $wR = 0.056$, $w = [\sigma^2(F) + 0.0028F^2]^{-1}$, $S = 1.47$, $(\Delta/\sigma)_{\max} \leq 0.001$, $(\Delta\rho)_{\max} = 0.94$, $(\Delta\rho)_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$; no extinction correction. Scattering factors for all atoms given in *SHELX76* (Sheldrick, 1976). All calculations on VAX11/785 computer system. Atomic parameters given in Table 1, bond distances and angles in Table 2;* the numbering scheme used is shown in Fig. 1.

Related literature. The title compound, $(2\text{-py})_3\text{P}$, has been shown by spectroscopic methods to function

* Lists of structure factors, thermal parameters, H-atom parameters and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44593 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

primarily as a tridentate ligand through the three N donor atoms, e.g. $[M\{(2\text{-py})_3\text{P}\}_2](\text{ClO}_4)_2$ for $M = \text{Mn}$, Co, Ni, Cu and Zn (Bogges & Zatkan, 1975), for $M = \text{Co}$ (Bogges & Zatkan, 1976; Hafeli & Keene, 1987) and for $M = \text{Fe}$ (Bogges, Hughes, Chew & Kemper, 1981). However, both monodentate P and bidentate P,N coordination have been reported for the $(2\text{-py})_3\text{P}$ ligand in a series of Ru and Rh compounds, e.g. $\text{RuHCl}\{(2\text{-py})_3\text{P}\}_3$ and $\text{RhCl}\{(2\text{-py})_3\text{P}\}_2$ (Kurtev, Ribola, Jones, Cole-Hamilton & Wilkinson, 1980). The only crystallographic characterization of a compound containing $(2\text{-py})_3\text{P}$ is that for the $[\text{Ru}\{(2\text{-py})_3\text{P}\}_2]^{2+}$ cation in which $(2\text{-py})_3\text{P}$ adopts the more common tridentate mode of coordination (Stephenson, Keene, Snow & Tiekink, 1988).

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Propane-1,2,3-tricarboxylic Acid (Tricarballic Acid)

BY JOHN C. BARNES* AND JOHN D. PATON

Chemistry Department, The University, Dundee DD1 4HN, Scotland

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Abstract. $\text{C}_6\text{H}_8\text{O}_6$, $M_r = 176.12$, orthorhombic, *Pbca*, $a = 11.89$ (2), $b = 23.96$ (3), $c = 5.38$ (2) \AA , $V = 1532.68 \text{ \AA}^3$, $Z = 8$, $D_x = 1.53 \text{ Mg m}^{-3}$, $\lambda(\text{Cu K}\alpha) = 1.5418 \text{ \AA}$, $\mu = 1.118 \text{ mm}^{-1}$, $F(000) = 736$, $T = 293 \text{ K}$, $R = 0.062$ for 603 reflexions. The molecule has no

unusual bond lengths or angles. Molecules are linked into double sheets by hydrogen bonding similar to that found in citric acid although the molecular conformations are different.

Experimental. Colourless crystals from water, very few of adequate diffraction quality. Preliminary

* To whom correspondence should be addressed.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) with e.s.d.'s in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
C1	7924 (7)	4178 (3)	-2324 (14)	29 (2)
C2	8269 (5)	3556 (3)	-2168 (12)	22 (2)
C3	7282 (5)	3145 (3)	-2056 (12)	24 (2)
C4	8836 (6)	4536 (3)	-3508 (14)	29 (2)
O5	9439 (5)	4375 (2)	-5181 (11)	51 (2)
O6	8879 (5)	5053 (2)	-2589 (12)	51 (2)
C7	9107 (5)	3454 (3)	-68 (14)	24 (2)
O8	9028 (4)	3070 (2)	1441 (9)	32 (1)
O9	9961 (4)	3819 (2)	-72 (10)	35 (1)
C10	6454 (6)	3233 (3)	74 (13)	24 (2)
O11	6558 (4)	3584 (2)	1704 (9)	32 (1)
O12	5606 (4)	2868 (2)	-14 (10)	34 (1)

Table 2. Interatomic distances (\AA) and angles ($^\circ$) and hydrogen-bond lengths (\AA)

C2-C1	1.546 (8)	C4-C1-C2	112.1 (6)
C4-C1	1.523 (10)	C3-C2-C1	114.7 (5)
C3-C2	1.533 (8)	C7-C2-C1	111.6 (6)
C7-C2	1.527 (9)	C7-C2-C3	111.5 (5)
C10-C3	1.525 (9)	C10-C3-C2	115.8 (5)
O5-C4	1.213 (8)	O5-C4-C1	123.4 (6)
O6-C4	1.334 (8)	O6-C4-C1	113.3 (6)
O8-C7	1.230 (8)	O6-C4-O5	123.2 (6)
O9-C7	1.340 (8)	O8-C7-C2	124.0 (6)
O11-C10	1.222 (8)	O9-C7-C2	112.9 (6)
O12-C10	1.334 (8)	O9-C7-O8	123.1 (7)
		O11-C10-C3	124.6 (6)
		O12-C10-C3	111.8 (6)
		O12-C10-O11	123.5 (6)
O5...O6 ⁱ	2.706 (7)		
O8...O12 ⁱⁱ	2.729 (8)		
O9...O11 ⁱⁱ	2.684 (8)		

Symmetry operations: (i) $2-x, 1-y, -(1+z)$; (ii) $1.5+x, y, 1.5-z$.

photographs indicated an orthorhombic system. Unit-cell dimensions were refined from $0kl$ and $hk0$ Weissenberg photographs. Intensity data from equi-inclination Weissenberg photographs of a crystal $0.5 \times 0.3 \times 0.1$ mm mounted initially on a (layers 0,1) and subsequently on c (layers 0-4). Intensities were measured by the SERC Microdensitometer Service, Daresbury Laboratory. 2045 measured reflexions gave 603 unique data, R_{int} 0.082, all of which were used in the refinement. Range of indices $0 \leq h \leq 13$, $0 \leq k \leq 27$, $0 \leq l \leq 6$, $2\theta_{max}$ 139° . Data corrected for Lorentz and polarization terms but not for absorption. All calculations were performed on the Dundee University DEC 10 computer using the program packages *SHELXS* (Sheldrick, 1986), *SHELX76* (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978). Atomic scattering factors were taken from *SHELX76*.

The direct-methods routine *TREF* showed all the non-hydrogen atoms. The structure was refined by conventional least-squares procedures and difference

syntheses with isotropic and then anisotropic thermal parameters for the non-hydrogen atoms. Skeletal (C-H) hydrogen atoms were introduced in calculated positions using one refined overall isotropic thermal parameter [$U = 0.039$ (9) \AA^2]. The carboxylic-acid hydrogen atoms were located on difference syntheses and refined with a second overall isotropic thermal parameter [$U = 0.078$ (18) \AA^2]. Final refinement minimizing $\sum w|F_o - |F_c||^2$, 120 refined parameters, $R = 0.062$, $wR = 0.078$, $w = 6.3331/[\sigma^2(F) + 0.000186F^2]$, mean shift/e.s.d. = 0.007, max. shift/e.s.d. = 0.036, max. $\Delta\rho = 0.34$, min. $\Delta\rho = -0.38$ $e \text{\AA}^{-3}$.

Final coordinates are given in Table 1, with bond lengths and angles in Table 2.* The molecule is shown in perspective view in Fig. 1, molecular packing is illustrated in Fig. 2.

Related literature. Comparison with anhydrous citric acid (Glusker, Minkin & Patterson, 1969), which differs only in the presence of a hydroxyl group on C2, shows a very similar pattern of hydrogen bonding (Fig. 2). The group C4, O5, O6 is linked to the same group of

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44605 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

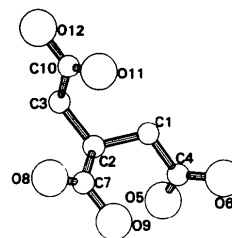


Fig. 1. Perspective view of the title compound.

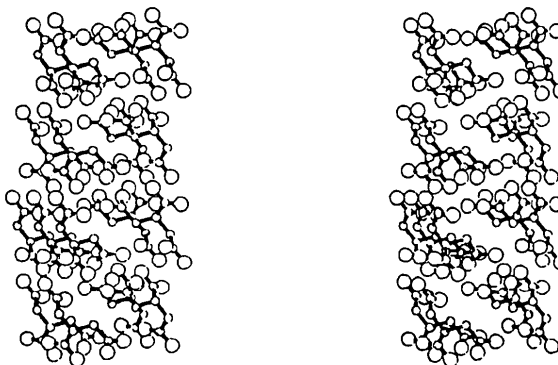


Fig. 2. Stereo pair showing hydrogen bonding in the title compound.

Table 3. Comparison of torsion angles ($^{\circ}$) for propane-1,2,3-tricarboxylic acid and citric acid

	Propane-1,2,3-tricarboxylic acid	Citric acid*
C4-C1-C2-C3	-155.6 (6)	-174.7
C4-C1-C2-C7	76.3 (5)	64.5
C2-C1-C4-O5	35.4 (6)	172.2
C2-C1-C4-O6	-147.1 (6)	-8.2
C1-C2-C3-C10	-57.0 (6)	171.1
C7-C2-C3-C10	71.1 (5)	68.7
C1-C2-C7-O8	133.3 (6)	111.4
C1-C2-C7-O9	-48.3 (5)	-68.0
C3-C2-C7-O8	3.6 (5)	51.0
C3-C2-C7-O9	-178.0 (6)	-129.6
C2-C3-C10-O11	-3.5 (5)	-50.4
C2-C3-C10-O12	178.1 (4)	129.6

* Data from Glusker, Minkin & Patterson (1969) with atoms renumbered.

another molecule across a centre of symmetry by two hydrogen bonds, and C7, O8, O9 is linked to C10, O11, O12 of a molecule related by a glide plane, again using two hydrogen bonds (Table 2). However, the conformations of tricarballic and citric acids are quite different. Torsion angles are compared in Table 3.

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Structure of 2,3,4-Tri-*O*-acetyl-*N*-(diacetyl-amino)- β -D-glucopyranurono-1,6-lactam

BY TOSHIO AKIMOTO, YASUKO TAKEDA* AND YOSHIHARU NAWATA†

Research Laboratories, Chugai Pharmaceutical Co. Ltd, Takada, Toshima, Tokyo 171, Japan

AND YOICHI IITAKA

Faculty of Pharmaceutical Sciences, University of Tokyo, Hongo, Bunkyo, Tokyo 113, Japan

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Abstract. $C_{16}H_{20}N_2O_{10}$, $M_r = 400.35$, orthorhombic, $P2_12_12_1$, $a = 11.581$ (1), $b = 15.153$ (1), $c = 10.787$ (1) Å, $V = 1892.9$ Å³, $Z = 4$, $D_x = 1.405$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 9.77$ cm⁻¹, $F(000) = 840$, $T = 298$ K, final $R = 0.035$ for 1933 unique reflections [$F_o^2 > 2\sigma(F_o^2)$]. Five-membered lactam ring of title compound is in the envelope form. The pyranose ring adopts a distorted 1C_4 (*D*) chair conformation, contrary to the boat form [$B_{0,3}$ (*D*)] observed in solution.

Experimental. Colorless prisms of the title compound were grown from ethanol solution. Crystal size 0.30 ×

0.28 × 0.13 mm, Enraf-Nonius CAD-4 κ -cradle diffractometer, Cu $K\alpha$ radiation, graphite monochromator, θ - 2θ scan with scan speed 2.06-4.12° min⁻¹ in θ , scan width (0.50 + 0.14tan θ)°. Range of indices $0 \leq h \leq 14$; $0 \leq k \leq 19$, $0 \leq l \leq 13$ ($2\theta < 150^\circ$). Lattice constants determined based on 25 2θ values ($21 < \theta < 47^\circ$). Variation of standard $< 1.5\%$; 2223 unique reflections measured; 1933 observed reflections with $F_o^2 > 2\sigma(F_o^2)$. Systematic absences $h00$, h odd; $0k0$, k odd; $00l$, l odd. No corrections for absorption. Structure solved by direct methods with *MULTAN* (Main, Woolfson & Germain, 1971). Refined by full-matrix least squares. The locations of all the H atoms were found on a difference-Fourier map. Non-H atoms refined with anisotropic thermal parameters, and H atoms with isotropic thermal parameters ($B = 5.5$ Å²: fixed). $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 1.0$

* Present address: Research Development Corporation of Japan, Koishikawa 4-14-24, Bunkyo, Tokyo 112, Japan.

† To whom all correspondence should be addressed.