Table 3. *Comparison of torsion angles* (°) *for propane-1,2,3-tricarboxylic acid and citric acid*

	Propane-1,2,3-tri-carboxylic 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 tricarballylic and citric acids are quite different. Torsion angles are compared in Table 3.

Although the calcium salt occurs widely in vegetables such as sugar beet, few metal complexes of tricarballylic acid have been reported. These often have smaller solution formation constants than the corresponding citrates and precipitate as fine powders at relatively low pH (Barnes & Bristow, 1969). Attempts in this laboratory to prepare single-crystal specimens of these complexes have not yet been successful.

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

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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, $V = 1892.9$ Å^3, $Z = 4$, $D_x = 1.405$ g cm^{-3}, λ(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 ×

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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 + 0·14tanθ)°. Range of indices $0 \le h \le 14$, $0 \le k \le 19$, $0 \le l \le 13$ ($2\theta < 150°$). Lattice constants determined based on 25 2θ values (21 < θ < 47°). 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 ($B = 5.5$ Å^2: fixed). $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 1.0$

for $|F_o| < 907\cdot9$, $w = (907\cdot9/F_o)^2$ for $|F_o| \geq 907\cdot9$. Final $R = 0\cdot035$, $wR = 0\cdot032$, $S = 4\cdot6$ for 334 variables, secondary-extinction factor (g) $1\cdot10$ (3) $\times$ 10^{-6} $[|F_o| = |F_c|/(1 + gIc)]$; $\Delta/\sigma < 0\cdot35$, largest peak in final ΔF map $+0\cdot16$ e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs: Enraf–Nonius *SDP* (Frenz, 1984), *ORTEP*II (Johnson, 1976). The structure of the title compound is shown in Fig. 1, crystal structure in Fig. 2. Positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1, bond distances and angles are listed in Table 2.*

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* Lists of anisotropic thermal parameters, H-atom coordinates, torsion angles, least-squares planes and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44594 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
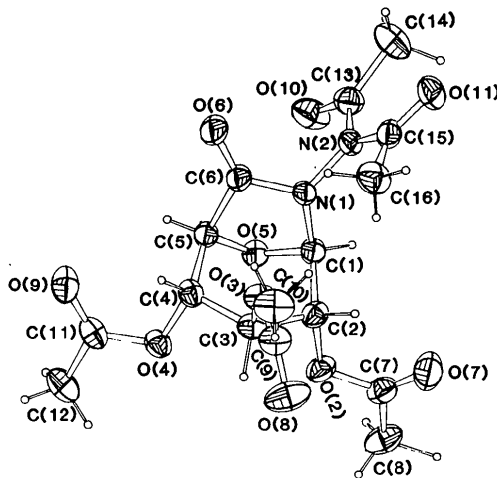

Fig. 1. A perspective view of the molecule with the numbering scheme.
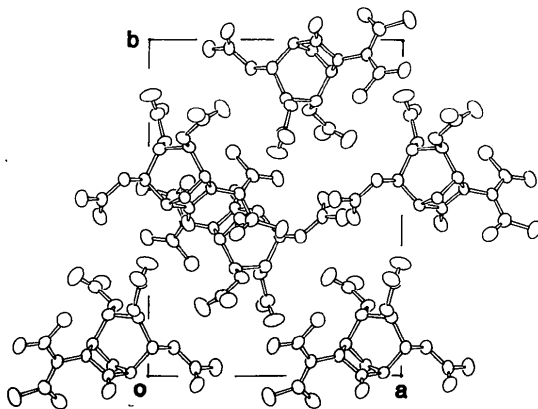

Fig. 2. The crystal structure projected along the c axis.

Table 1. *Final fractional coordinates and equivalent isotropic temperature factors for non-H atoms with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\Sigma_i \Sigma_j B_{ij}\mathbf{a}_i\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
C(1)	0·2803 (2)	0·4068 (2)	0·5684 (2)	3·21 (5)
C(2)	0·3428 (3)	0·3185 (2)	0·5593 (2)	3·41 (5)
C(3)	0·4598 (3)	0·3252 (2)	0·4922 (3)	3·42 (5)
C(4)	0·5168 (2)	0·4164 (2)	0·4925 (2)	3·30 (5)
C(5)	0·4309 (2)	0·4905 (2)	0·5146 (2)	3·21 (5)
C(6)	0·3428 (2)	0·4988 (2)	0·4101 (3)	3·47 (5)
C(7)	0·2902 (3)	0·2371 (2)	0·7381 (3)	4·13 (6)
C(8)	0·3161 (4)	0·2236 (2)	0·8721 (3)	5·60 (8)
C(9)	0·4685 (3)	0·2218 (2)	0·3263 (3)	4·28 (7)
C(10)	0·4594 (3)	0·2143 (2)	0·1883 (3)	5·47 (8)
C(11)	0·6860 (2)	0·4762 (2)	0·5822 (3)	4·04 (6)
C(12)	0·7639 (3)	0·4737 (3)	0·6911 (3)	5·46 (8)
C(13)	0·0938 (3)	0·5346 (2)	0·4579 (3)	4·33 (6)
C(14)	−0·0241 (3)	0·5630 (3)	0·4189 (4)	6·25 (9)
C(15)	0·0922 (3)	0·4019 (2)	0·3154 (3)	4·06 (6)
C(16)	0·1694 (3)	0·3335 (2)	0·2602 (3)	5·24 (8)
N(1)	0·2559 (2)	0·4401 (2)	0·4425 (2)	3·17 (4)
N(2)	0·1423 (2)	0·4575 (2)	0·4055 (2)	3·42 (4)
O(2)	0·3675 (2)	0·2921 (1)	0·6855 (2)	4·00 (4)
O(3)	0·4411 (2)	0·3051 (1)	0·3624 (2)	3·78 (4)
O(4)	0·5994 (2)	0·4157 (1)	0·5924 (2)	3·72 (4)
O(5)	0·3599 (2)	0·4689 (1)	0·6188 (2)	3·21 (3)
O(6)	0·3457 (2)	0·5456 (2)	0·3206 (2)	5·16 (5)
O(7)	0·2123 (2)	0·2050 (2)	0·6837 (2)	6·71 (6)
O(8)	0·4952 (3)	0·1658 (1)	0·3974 (2)	6·64 (6)
O(9)	0·6935 (2)	0·5260 (2)	0·4966 (2)	5·10 (5)
O(10)	0·1506 (2)	0·5759 (2)	0·5307 (2)	5·67 (5)
O(11)	−0·0066 (2)	0·4112 (2)	0·2835 (2)	5·32 (5)

Table 2. *Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses*

C(1)–C(2)	1·524 (4)	C(7)–O(7)	1·181 (4)
C(1)–N(1)	1·476 (3)	C(9)–C(10)	1·496 (5)
C(1)–O(5)	1·424 (3)	C(9)–O(3)	1·358 (4)
C(2)–C(3)	1·540 (4)	C(9)–O(8)	1·186 (4)
C(2)–O(2)	1·447 (3)	C(11)–C(12)	1·481 (5)
C(3)–C(4)	1·532 (4)	C(11)–O(4)	1·363 (4)
C(3)–O(3)	1·449 (3)	C(11)–O(9)	1·196 (4)
C(4)–C(5)	1·519 (4)	C(13)–C(14)	1·493 (5)
C(4)–O(4)	1·441 (3)	C(13)–N(2)	1·415 (4)
C(5)–C(6)	1·525 (4)	C(13)–O(10)	1·200 (4)
C(5)–O(5)	1·431 (3)	C(15)–C(16)	1·492 (5)
C(6)–N(1)	1·387 (4)	C(15)–N(2)	1·411 (4)
C(6)–O(6)	1·199 (3)	C(15)–O(11)	1·203 (4)
C(7)–C(8)	1·490 (4)	N(1)–N(2)	1·400 (3)
C(7)–O(2)	1·348 (4)		
C(2)–C(1)–N(1)	109·4 (2)	O(3)–C(9)–O(8)	122·7 (3)
C(2)–C(1)–O(5)	107·3 (2)	C(12)–C(11)–O(4)	111·6 (3)
N(1)–C(1)–O(5)	104·4 (2)	C(12)–C(11)–O(9)	125·7 (3)
C(1)–C(2)–C(3)	113·0 (2)	O(4)–C(11)–O(9)	122·6 (3)
C(1)–C(2)–O(2)	106·0 (2)	C(14)–C(13)–N(2)	119·3 (3)
C(3)–C(2)–O(2)	106·7 (2)	C(14)–C(13)–O(10)	122·4 (3)
C(2)–C(3)–C(4)	115·9 (2)	N(2)–C(13)–O(10)	118·3 (3)
C(2)–C(3)–O(3)	108·0 (2)	C(16)–C(15)–N(2)	116·3 (3)
C(4)–C(3)–O(3)	104·8 (2)	C(16)–C(15)–O(11)	122·5 (3)
C(3)–C(4)–C(5)	112·7 (2)	N(2)–C(15)–O(11)	121·2 (3)
C(3)–C(4)–O(4)	106·3 (2)	C(1)–N(1)–C(6)	108·2 (2)
C(5)–C(4)–O(4)	108·8 (2)	C(1)–N(1)–N(2)	120·4 (2)
C(4)–C(5)–C(6)	112·5 (2)	C(6)–N(1)–N(2)	119·3 (2)
C(4)–C(5)–O(5)	109·3 (2)	C(13)–N(2)–C(15)	127·2 (2)
C(6)–C(5)–O(5)	102·4 (2)	C(13)–N(2)–N(1)	114·5 (2)
C(5)–C(6)–N(1)	104·3 (2)	C(15)–N(2)–N(1)	118·1 (2)
C(5)–C(6)–O(6)	128·6 (3)	C(2)–O(2)–C(7)	115·9 (2)
N(1)–C(6)–O(6)	127·1 (3)	C(3)–O(3)–C(9)	115·9 (2)
C(8)–C(7)–O(2)	111·1 (3)	C(4)–O(4)–C(11)	115·0 (2)
C(8)–C(7)–O(7)	125·4 (3)	C(1)–O(5)–C(5)	102·9 (2)
O(2)–C(7)–O(7)	123·5 (3)		
C(10)–C(9)–O(3)	109·9 (3)		
C(10)–C(9)–O(8)	127·4 (3)		

$C_{16}H_{20}N_2O_{10}$

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Structure of 1,6-Anhydro-4-carboxymethyl-2,4-dideoxy-2-fluoro-β-D-allo-hexopyranose-γ-lactone

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Abstract. $C_8H_9FO_4$, $M_r = 188 \cdot 16$, orthorhombic, $P2_12_12_1$, $a = 7 \cdot 5071$ (10), $b = 8 \cdot 9402$ (8), $c = 11 \cdot 7375$ (20) Å, $V = 787 \cdot 8$ Å^3, $Z = 4$, $D_x = 1 \cdot 57$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1 \cdot 5418$ Å, $\mu = 1 \cdot 119$ mm^{-1}, $F(000) = 392$, room temperature, $R = 0 \cdot 039$ for 1159 observed reflexions. The anhydro and lactone rings are on opposite sides of the pyranose ring such that the molecule has an overall chair conformation. The dioxolane and lactone rings adopt envelope conformations with O(2) and C(4) respectively at the flap. By comparison with cyclohexane, O(2) shows enhanced puckering and C(3) is flattened. The torsion angles about the bonds to O(2) are *ca* |76°| and those about bonds to C(3) are *ca* |36°|. Therefore, the F–C(2)–C(3)–O(3) torsion angle is positive, 37·4 (2)°, but considerably less than 60°. Intramolecular non-bonded distances of interest with respect to NMR long-range coupling are F···C(4) 3·129 (3), F···C(7) 3·096 (3), F···H(7A) 2·755 (3) Å and F··· H(7B) 4·084 (3) Å.

Experimental. Colourless crystal, dimensions 0·10 × 0·20 × 0·10 mm. Enraf–Nonius CAD-4 diffractometer, graphite monochromator, Cu $K\alpha$ radiation. Cell dimensions from setting angles of 25 independent reflexions with $\theta \simeq 22°$. 885 unique intensities measured, with $2\theta < 70°$, as θ–2θ scans. Range of hkl 0–9, 0–10, 0–14, 811 independent reflexions with $I > 2 \cdot 5\sigma(I)$. Two reference reflexions monitored period-

ically showed no significant variation in intensity. No absorption correction. Structure was determined with *MITHRIL* (Gilmore, 1984) and H atoms located in difference Fourier maps with *SHELX* (Sheldrick, 1976). Full-matrix least-squares calculations on F with anisotropic thermal parameters for C, F and O atoms and isotropic thermal parameters for H atoms converged at R 0·039, wR 0·042, $\Delta/\sigma < 0·03$, $w = 49 \cdot 3/\sigma^2(|F_o|)$. Final $\Delta\rho$ max. 0·07, min. 0·14 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). Molecular diagrams and geometries were generated by the *GX* package (Mallinson & Muir, 1985) and all computer programs were run on a Honeywell CP6 mainframe. Atomic coordinates

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses and equivalent values of the anisotropic temperature factor coefficients* (Å$^2 \times 10^3$)

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}
F	−0·9183 (2)	−0·4847 (2)	−0·7736 (1)	0·073
O(1)	−1·2063 (2)	−0·5111 (3)	−0·5319 (1)	0·066
O(2)	−0·9890 (3)	−0·6677 (2)	−0·5943 (2)	0·063
O(3)	−0·7552 (2)	−0·2816 (2)	−0·6524 (2)	0·055
O(4)	−0·4725 (2)	−0·3205 (3)	−0·6959 (1)	0·077
C(1)	−1·1153 (3)	−0·5611 (3)	−0·6308 (2)	0·061
C(2)	−1·0204 (3)	−0·4301 (3)	−0·6834 (2)	0·055
C(3)	−0·8986 (3)	−0·3588 (2)	−0·5961 (2)	0·044
C(4)	−0·8069 (3)	−0·4704 (2)	−0·5170 (2)	0·043
C(5)	−0·9258 (3)	−0·6025 (2)	−0·4899 (2)	0·049
C(6)	−1·1008 (3)	−0·5545 (3)	−0·4355 (2)	0·053
C(7)	−0·6361 (3)	−0·5043 (3)	−0·5818 (3)	0·056
C(8)	−0·6045 (3)	−0·3649 (3)	−0·6497 (2)	0·053