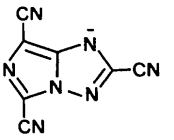
Table 2. *Selected bond lengths* (Å) *and angles* (°)

N(1)–C(2)	1·108 (8)	C(2)–C(3)	1·409 (9)
C(3)–N(4)	1·360 (7)	C(3)–N(4′)	1·367 (8)
N(4)–C(5)	1·351 (8)	N(4′)–C(5′)	1·312 (6)
C(5)–C(5′)	1·344 (8)	C(5)–C(6)	1·351 (8)
C(5′)–C(6′)	1·400 (8)	C(6)–N(7)	1·387 (11)
C(6)–C(8)	1·445 (9)	C(6′)–N(7)	1·312 (7)
C(6′)–C(8′)	1·421 (12)	C(8)–N(9)	1·134 (8)
C(8′)–N(9′)	1·164 (13)	P(1)–N(11)	1·572 (5)
P(1)–C(11)	1·797 (6)	P(1)–C(21)	1·791 (5)
P(1)–C(31)	1·790 (4)	P(2)–N(11)	1·573 (5)
P(2)–C(41)	1·799 (6)	P(2)–C(51)	1·790 (5)
P(2)–C(61)	1·786 (6)		

N(1)–C(2)–C(3)	177·5 (6)	C(2)–C(3)–N(4)	122·2 (6)
C(2)–C(3)–N(4′)	120·7 (5)	N(4)–C(3)–N(4′)	117·1 (5)
C(3)–N(4)–C(5)	99·7 (5)	C(3)–N(4′)–C(5′)	99·9 (4)
N(4)–C(5)–C(5′)	110·5 (5)	N(4)–C(5)–C(6)	141·5 (6)
C(5′)–C(5)–C(6)	108·0 (6)	N(4′)–C(5′)–C(5)	112·8 (5)
N(4′)–C(5′)–C(6′)	140·9 (5)	C(5)–C(5′)–C(6′)	106·2 (5)
C(5)–C(6)–N(7)	109·5 (6)	C(5)–C(6)–C(8)	126·5 (7)
N(7)–C(6)–C(8)	124·0 (6)	C(5′)–C(6′)–N(7)	110·5 (6)
C(5′)–C(6′)–C(8′)	124·6 (5)	N(7)–C(6′)–C(8′)	124·9 (6)
C(6)–N(7)–C(6′)	105·8 (5)	C(6)–C(8)–N(9)	179·0 (9)
C(6′)–C(8′)–N(9′)	176·5 (7)	N(11)–P(1)–C(11)	113·4 (2)
N(11)–P(1)–C(21)	107·3 (2)	C(11)–P(1)–C(21)	106·9 (3)
N(11)–P(1)–C(31)	113·7 (3)	C(11)–P(1)–C(31)	106·5 (2)
C(21)–P(1)–C(31)	108·8 (2)	N(11)–P(2)–C(41)	114·2 (3)
N(11)–P(2)–C(51)	108·2 (2)	C(41)–P(2)–C(51)	108·0 (3)
N(11)–P(2)–C(61)	110·5 (3)	C(41)–P(2)–C(61)	106·9 (3)
C(51)–P(2)–C(61)	108·9 (3)	P(1)–N(11)–P(2)	146·0 (3)

crystal. The C–N moiety common to both rings of the anion is disordered and both atoms were therefore refined as C with site occupation factor $1\frac{1}{12}$. All non-H atoms anisotropic; H atoms included using a riding model with C–H 0·96 Å, $U(H) = 1{\cdot}2\ U_{eq}(C)$; weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}0003F^2$; 478 parameters; $S = 1{\cdot}56$; max. Δ/σ 0·04; max. features in final $\Delta\rho$ map 0·45, −0·35 e Å^{-3}. Atom scattering factors from *SHELXTL*. Final atomic coordinates are given in Table 1, and selected bond lengths and angles in Table 2.* Fig. 1 shows the atom-numbering scheme.

Related literature. Synthesis of other salts of the same anion (1) and correct suggestion of its structure: Wiley, Webster & Blanchard (1976).



(1)

We thank the Verband der Chemischen Industrie for financial support, and Professor H. W. Roesky and Mr H. Hofmann for providing the crystals.

* Lists of structure factors, H-atom coordinates, bond lengths and angles, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43070 (35 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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α-[2′,5′-Bis(methoxymethyl)phenyl]-3,4,5-trimethoxyphenylacetonitrile

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Abstract. $C_{21}H_{25}NO_5$, $M_r = 371{\cdot}43$, triclinic, $P\bar{1}$, $a = 7{\cdot}876\ (4)$, $b = 10{\cdot}435\ (5)$, $c = 12{\cdot}242\ (4)$ Å, $\alpha = 84{\cdot}87\ (3)$, $\beta = 83{\cdot}44\ (3)$, $\gamma = 106{\cdot}65\ (3)°$, $V = 949{\cdot}7\ (6)$ Å^3, $Z = 2$, $D_x = 1{\cdot}299$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 0·86 cm^{-1}, $F(000) = 396$, $T = 295$ K, final $R = 0{\cdot}043$ for 2556 observed reflections. There are no unusual bond lengths or angles.

Experimental. Colorless rectangular plates, unit-cell parameters by least-squares fit of 15 reflections in the range $17 < 2\theta < 25°$. Crystal 0·54 × 0·33 × 0·18 mm, automatic Syntex $P2_1$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode, 3363 independent reflections in the range $3 < 2\theta < 50°$, hkl range h −9→9, k −12→12, l 0→14, 2556 observed reflections with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics; two standard reflections, 100 and 022, remeasured after every 100 reflections did not show any significant change in intensity; Lorentz–polarization correction, no absorption or extinction correction. Direct-methods *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refinement by full-matrix least squares using *SHELX*76 (Sheldrick, 1976), anisotropic, H atoms located in difference Fourier map, H

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Table 1. *Fractional atomic coordinates with equivalent isotropic thermal parameters for the non-H and isotropic for H atoms (e.s.d.'s in parentheses)*

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}/U(Å^2)
C(1)	0·0977 (2)	0·2110 (1)	0·6448 (1)	0·0330 (3)
C(2)	0·0557 (2)	0·2578 (1)	0·7462 (1)	0·0360 (3)
C(3)	−0·0793 (2)	0·3187 (2)	0·7549 (1)	0·0427 (3)
C(4)	−0·1712 (2)	0·3369 (2)	0·6673 (1)	0·0462 (3)
C(5)	−0·1290 (2)	0·2925 (2)	0·5678 (1)	0·0420 (3)
C(6)	0·0040 (2)	0·2291 (2)	0·5588 (1)	0·0395 (3)
C(7)	0·1511 (2)	0·2451 (2)	0·8443 (1)	0·0405 (3)
O(8)	0·3345 (2)	0·3202 (1)	0·8170 (1)	0·0466 (2)
C(9)	0·4311 (3)	0·3097 (2)	0·9063 (2)	0·0586 (4)
C(10)	−0·2180 (3)	0·3128 (2)	0·4675 (2)	0·0609 (5)
O(11)	−0·3588 (2)	0·3660 (1)	0·4934 (1)	0·0588 (3)
C(12)	−0·4205 (3)	0·4097 (2)	0·3963 (2)	0·0657 (5)
C(13)	0·2499 (2)	0·1464 (2)	0·6292 (1)	0·0355 (3)
C(14)	0·2737 (2)	0·1078 (2)	0·5163 (1)	0·0406 (3)
N(15)	0·2919 (2)	0·0763 (2)	0·4298 (1)	0·0592 (4)
C(16)	0·2318 (2)	0·0232 (2)	0·7123 (1)	0·0350 (3)
C(17)	0·0709 (2)	−0·0804 (2)	0·7366 (1)	0·0369 (3)
C(18)	0·0581 (2)	−0·1894 (2)	0·8147 (1)	0·0361 (3)
O(19)	−0·0926 (2)	−0·2968 (1)	0·8460 (1)	0·0477 (2)
C(20)	−0·2296 (2)	−0·3141 (2)	0·7775 (2)	0·0511 (4)
C(21)	0·2039 (2)	−0·1941 (2)	0·8682 (1)	0·0366 (3)
O(22)	0·1862 (2)	−0·2973 (1)	0·9511 (1)	0·0440 (2)
C(23)	0·2232 (3)	−0·4123 (2)	0·9128 (2)	0·0548 (4)
C(24)	0·3658 (2)	−0·0921 (2)	0·8393 (1)	0·0388 (3)
O(25)	0·5031 (2)	−0·1075 (1)	0·8926 (1)	0·0484 (2)
C(26)	0·6685 (2)	−0·0015 (2)	0·8689 (2)	0·0532 (4)
C(27)	0·3796 (2)	0·0169 (2)	0·7617 (1)	0·0383 (3)
H(3)	−0·107 (2)	0·351 (2)	0·825 (1)	0·044 (5)
H(4)	−0·265 (2)	0·380 (2)	0·677 (2)	0·056 (5)
H(6)	0·030 (2)	0·199 (2)	0·488 (1)	0·039 (4)
H(71)	0·142 (2)	0·150 (2)	0·870 (1)	0·041 (5)
H(72)	0·092 (2)	0·275 (2)	0·909 (1)	0·044 (5)
H(91)	0·560 (3)	0·358 (2)	0·879 (2)	0·080 (7)
H(92)	0·423 (3)	0·217 (2)	0·929 (2)	0·076 (7)
H(93)	0·386 (3)	0·348 (2)	0·965 (2)	0·080 (7)
H(101)	−0·256 (4)	0·231 (3)	0·428 (2)	0·096 (8)
H(102)	−0·130 (3)	0·371 (3)	0·401 (2)	0·088 (8)
H(121)	−0·521 (4)	0·440 (3)	0·424 (2)	0·097 (8)
H(122)	−0·454 (4)	0·332 (3)	0·345 (2)	0·112 (10)
H(123)	−0·319 (4)	0·481 (3)	0·347 (2)	0·105 (9)
H(13)	0·358 (2)	0·214 (2)	0·636 (1)	0·040 (4)
H(17)	−0·030 (2)	−0·072 (2)	0·700 (1)	0·046 (5)
H(201)	−0·316 (3)	−0·398 (2)	0·809 (2)	0·058 (5)
H(202)	−0·188 (3)	−0·324 (2)	0·708 (2)	0·081 (7)
H(203)	−0·300 (4)	−0·252 (3)	0·785 (2)	0·096 (8)
H(231)	0·186 (3)	−0·481 (2)	0·976 (2)	0·075 (7)
H(232)	0·356 (3)	−0·386 (3)	0·878 (2)	0·088 (8)
H(233)	0·139 (3)	−0·451 (2)	0·860 (2)	0·069 (6)
H(261)	0·742 (3)	−0·037 (2)	0·911 (2)	0·073 (6)
H(262)	0·655 (3)	0·084 (2)	0·894 (2)	0·062 (6)
H(263)	0·721 (3)	0·016 (2)	0·788 (2)	0·062 (6)
H(27)	0·491 (2)	0·086 (2)	0·742 (2)	0·050 (5)

isotropic, $w = 1/(\sigma_F^2 + 0{\cdot}002913F^2)$, $\sum w(|F_o| - |F_c|)^2$ minimized, $R = 0{\cdot}043$, $wR = 0{\cdot}051$; $S = 1{\cdot}08$; $(\Delta/\sigma)_{max} = 0{\cdot}11$; $\Delta\rho_{max,min} = 0{\cdot}21$, $-0{\cdot}24$ e Å^{-3} in final difference Fourier map. Atomic scattering factors for C, H, N and O used were those stored in *SHELX*76. The final atomic parameters are given in Table 1.* Fig. 1 shows the molecule and the numbering scheme adopted, Fig. 2 the packing of the molecules in the cell.

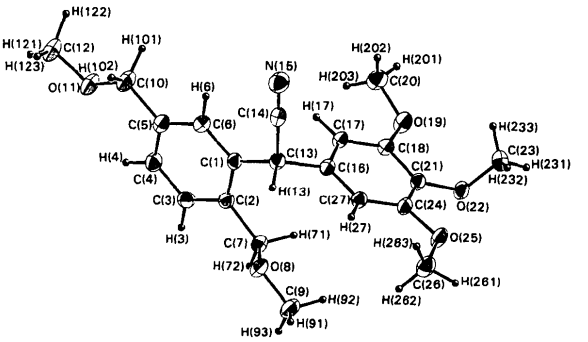

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule. Thermal ellipsoids at the 50% probability level. H atoms represented as spheres of arbitrary radii.

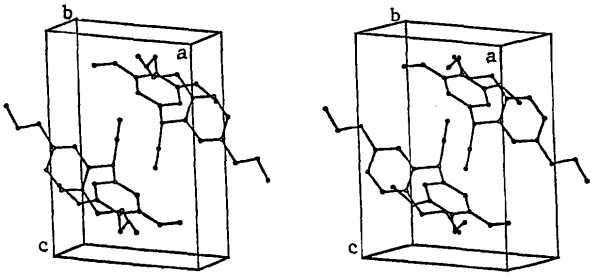

Fig. 2. Stereoscopic view of the packing of the molecules in the cell.

Related literature. The title compound is representative of a class of compounds which are hydrolyzed readily to isochroman-3-ones. The latter lactones have been shown (Spangler, Beckmann & Kim, 1977) to be useful precursors for the preparation of benzocyclobutenes which are of increasing importance as intermediates in the synthesis of natural products (Kametani & Fukumoto, 1975; Oppolzer, 1978). During our studies of the synthetic applications of the aryne reaction, we have recently developed a simple method for the synthesis of the title compound and a variety of structrally-related nitriles (Khanapure & Biehl, 1986).

This work was supported by the Robert E. Welch Foundation.

* Lists of structure factors, anisotropic thermal parameters, bond lengths and bond angles, least-squares planes, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43354 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of the Tri-*O*-isopropylidene Derivative of D-Glucosone Hydrate

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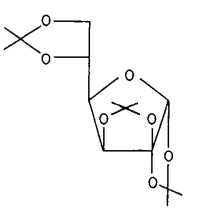
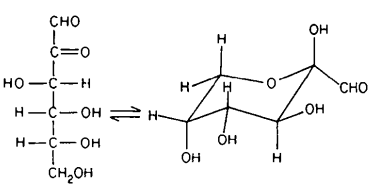
Abstract. 1,2;2,3;5,6-Tri-*O*-isopropylidene-D-*arabino*-hexos-2-ulose hydrate (1,2;2,3;5,6-tri-*O*-isopropylidene-D-glucosone hydrate) (I), $C_{15}H_{24}O_7$, $M_r = 316{\cdot}35$, m.p. 398 K, $[\alpha]_D^{15°C} = -7°$ (c, 2 g dm^{-3} in methanol), monoclinic, $P2_1$, $a = 8{\cdot}743$ (1), $b = 10{\cdot}358$ (2), $c = 9{\cdot}526$ (1) Å, $\beta = 107{\cdot}05$ (1)°, $V = 824{\cdot}7$ Å^3, $Z = 2$, $D_x = 1{\cdot}274$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 0·63 mm^{-1}, $F(000) = 340$, $T = 298$ K, final $R = 0{\cdot}055$, $wR = 0{\cdot}056$ for 1495 unique observed ($I > 0$) reflections. The structure determination was undertaken to confirm the structure and stereochemistry of the title compound. The furanoid ring and the 2,3-*O*- and 5,6-*O*-isopropylidene rings have conformations closest to envelopes with C(4), C(10) and C(13), respectively, out of the planes formed by the rest of the rings. The 1,2-*O*-isopropylidene ring has a twist conformation with C(7) and O(2) out of plane.

Experimental. The material (I) was synthesized from D-glucose *via* D-glucosone (II) with retention of chirality following the method of Bayne, Collie & Fewster (1952) and crystallized (colourless prisms) from ether–hexane. Crystal dimensions approximately 0·5 × 0·3 × 0·1 mm. Lattice parameters refined using 25 reflections in the range $10 < \theta < 15°$. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Intensity data collected with $\omega/2\theta$ scan technique ($2 < \theta < 25°$) on 1637 reflections [1495 unique reflections ($I > 0$), $-10 \le h \le 10$, $k \le 12$, $l \le 11$]. Two standard reflections (324 and $\bar{2}16$) showed no decay. The data were corrected for Lorentz and polarization effects; no corrections for absorption or extinction. The structure was solved by direct methods using *SHELXS*84 (Sheldrick, 1983) (default setting). Scattering factors from *International Tables for X-ray Crystallography* (1974). The structure was refined by full-matrix least-squares procedure minimizing the function $\sum w(|F_o| - |F_c|)^2$ with $w = [\sigma(F)]^{-2}$. The full-matrix least-squares program *SHELX*76 (Sheldrick, 1976) was used. Since a difference Fourier synthesis did not reveal the positions of all the hydrogen atoms, all H atoms were included in ideal calculated positions in a riding model (all C–H = 1·08 Å). A common isotropic temperature factor refined to 0·070 (5) Å^2. The methyl groups in the isopropylidene rings were refined as rigid groups free to rotate. Refinement with non-hydrogen atoms treated anisotropically converged at $R = 0{\cdot}055$ and $wR = 0{\cdot}056$. When the refinement was terminated all shift/e.s.d. ratios were less than 0·02, except those of the rotation parameters of the methyl groups, of which the highest was 0·08. A final difference Fourier synthesis showed $\Delta\rho = \pm 0{\cdot}3$ e Å^{-3}.



(I)



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