

Table 2. Molecular geometry

Bond distances (Å)			
N1-C2	1.341 (5)	N1-C6	1.353 (5)
N1-C7	1.465 (5)	C2-C3	1.364 (6)
C3-C4	1.363 (7)	C4-C5	1.383 (7)
C5-C6	1.367 (7)	C7-C8	1.312 (6)
C7-C9	1.476 (5)	C9-C10	1.391 (5)
C9-C14	1.401 (6)	C10-C11	1.382 (6)
C11-C12	1.373 (7)	C12-C13	1.389 (7)
C13-C14	1.381 (7)	B-F1	1.352 (6)
B-F2	1.382 (6)	B-F3	1.384 (6)
B-F4	1.336 (6)		
Bond angles (°)			
C6-N1-C7	118.2 (3)	C2-N1-C7	121.5 (3)
C2-N1-C6	120.3 (4)	N1-C2-C3	121.2 (4)
C2-C3-C4	119.2 (4)	C3-C4-C5	119.8 (4)
C4-C5-C6	119.4 (5)	N1-C6-C5	120.1 (4)
N1-C7-C9	114.7 (3)	N1-C7-C8	118.4 (4)
C8-C7-C9	126.9 (4)	C7-C9-C14	119.6 (3)
C7-C9-C10	121.1 (4)	C10-C9-C14	119.2 (3)
C9-C10-C11	120.1 (4)	C10-C11-C12	120.9 (4)
C11-C12-C13	119.4 (4)	C12-C13-C14	120.8 (4)
C9-C14-C13	119.7 (4)	F1-B-F2	110.5 (4)
F1-B-F3	107.0 (4)	F1-B-F4	113.6 (4)
F2-B-F3	108.0 (4)	F2-B-F4	111.4 (4)
F3-B-F4	106.0 (4)		
Some torsion angles (°)			
C6-N1-C7-C8	119.9 (5)	C2-N1-C7-C8	-59.1 (6)
C6-N1-C7-C9	-60.2 (5)	C2-N1-C7-C9	120.8 (4)
N1-C7-C9-C10	-35.1 (5)	N1-C7-C9-C14	147.1 (4)
C8-C7-C9-C10	144.9 (5)	C8-C7-C9-C14	-33.0 (6)
Short contacts involving F atoms (Å)			
C14...F2(x,y,z)	3.419 (5)	C11...F2(-½+x, ½-y, 1-z)	3.127 (6)
C2...F1(x,y,1+z)	3.200 (6)	C12...F2(1-x, -½+y, ½-z)	3.367 (6)
C6...F3(1-x, ½+y, ½-z)	3.216 (6)		

Related literature. No reports on the structure of phenylethenyl derivatives have been found. Previous structure determination of styrylpyridinium salts: Alvarez-Builla, Novella, Galvez, Smith, Florencio, Garcia-Blanco, Bellanato & Santos (1986). The structural geometry of the tetrafluoroborate is comparable

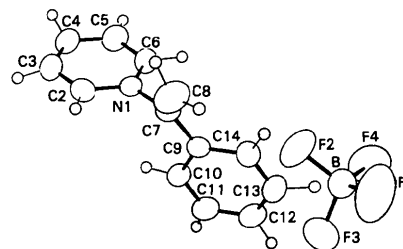


Fig. 1. A view of the molecule with the numbering scheme.

with that of Allman & Waśkowska (1981). Examples of the synthesis of 1-vinylpyridine salts are described by Relles (1973) and Novella & Alvarez-Builla (1985).

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Structure of 1,2:5,6-Di-O-isopropylidene- α -D-gulofuranose

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Abstract. $C_{12}H_{20}O_6$, $M_r = 260.3$, orthorhombic, $P2_12_12_1$, $a = 5.621$ (2), $b = 12.665$ (3), $c = 18.680$ (4) Å, $V = 1329.8$ Å³, $Z = 4$, $D_x = 1.300$ Mg m⁻³, $\lambda(\text{Cu K}\alpha) = 1.5418$ Å, $\mu = 0.78$ mm⁻¹, $F(000) = 560$, $T = 293$ K, $R = 0.039$ for 1145 unique observed reflections [$F > 3\sigma(F)$]. The fused five-

membered rings both have envelope conformations with maximum distances from the best least-squares plane for each ring of -0.215 (2) Å for O4 and 0.132 (2) Å for O1 and an angle between the planes of 122.6 (8)°. The largest deviation from the best plane in the third ring is -0.187 (2) Å for O6. The single hydroxyl group

forms a hydrogen bond, O3 to O6 ($1-x, \frac{1}{2}+y, \frac{1}{2}-z$) with an O...O distance of 2.832 (6) Å, linking the molecules in the z direction.

Experimental. Material supplied by Sigma Ltd, crystallized from 2-propanol/water, tabular, 0.63 × 0.26 × 0.15 mm, Enraf-Nonius CAD-4F diffractometer, Ni-filtered Cu $K\alpha$; cell parameters from 48 θ measurements in the range 27–36°; reflections measured for four octants to $2\theta = 140^\circ$ for ranges of h, k and l of 0 to 6, -15 to 15 and -22 to 22, respectively; 3890 reflections measured plus 504 flagged as <0 ; intensity control (141 measurements): average count of 3639 with a standard deviation (of the distribution) = 77 (2.12%) and no significant trend; no absorption correction; data merged using *SHELX76* (Sheldrick, 1976) to give 1147 unique reflections with $R_{\text{int}} = 0.023$; h, k, l range 0 to 6, 15 and 22; two reflections with high F_o/F_c ratios, possibly due to extinction, removed; structure solved by direct methods with *SHELXS86* (Sheldrick, 1985), non-H atoms refined by least squares (F magnitudes) with anisotropic thermal parameters; ten H atoms found from difference Fourier syntheses, ten in calculated positions, refined with fixed U_{iso} 's; $R = 0.039$, $wR = 0.061$; for final cycle, maximum shift/e.s.d. = 0.283 (H) and 0.157, average = 0.054 (all); $w = 1[\sigma^2(F) + 0.006385F^2]^{-1}$; difference Fourier synthesis showed a maximum value of 0.17 e Å⁻³ and a minimum value of -0.20 e Å⁻³; atom scattering factors from *International Tables for X-ray Crystallography* (1974).

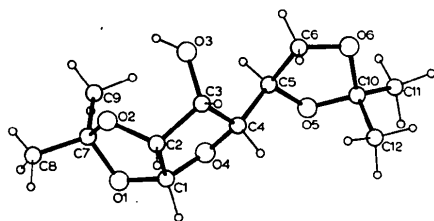


Fig. 1. View of the title compound with atom labels, drawn by the program *PLUTO78* (Motherwell & Clegg, 1978).

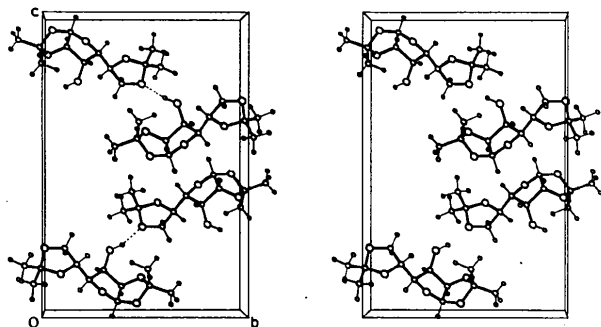


Fig. 2. Stereoscopic view of the crystal structure along a , with b horizontal and c vertical.

Table 1. Atom coordinates ($\times 10^4$) with e.s.d.'s and equivalent U_{iso} 's ($\text{\AA}^2 \times 10^4$)

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

	x	y	z	U_{eq}
C1	3373 (6)	6288 (2)	5334 (1)	509
C2	5554 (6)	6083 (2)	5807 (2)	503
C3	5557 (6)	6991 (2)	6364 (2)	538
C4	3661 (5)	7738 (2)	6073 (1)	441
C5	2329 (6)	8357 (2)	6639 (1)	472
C6	3972 (8)	8966 (3)	7146 (2)	617
C7	2837 (6)	4688 (2)	5889 (2)	538
C8	3119 (11)	3576 (3)	5623 (2)	845
C9	1051 (7)	4802 (4)	6485 (2)	747
C10	1767 (6)	10167 (2)	6513 (1)	525
C11	-246 (8)	10929 (3)	6635 (2)	676
C12	3473 (8)	10543 (3)	5937 (2)	737
O1	2173 (5)	5339 (2)	5295 (1)	589
O2	5114 (4)	5074 (2)	6109 (1)	601
O3	4875 (6)	6669 (2)	7063 (1)	750
O4	2009 (4)	7077 (1)	5693 (1)	502
O5	836 (4)	9156 (1)	6329 (1)	525
O6	2968 (5)	9991 (2)	7173 (1)	613

Table 2. Bond lengths (\AA) and bond angles ($^\circ$)

C1-C2	1.533 (5)	C1-O1	1.381 (4)
C2-C3	1.550 (4)	C7-O1	1.433 (4)
C3-C4	1.525 (4)	C2-O2	1.419 (3)
C4-C5	1.514 (4)	C7-O2	1.430 (4)
C5-C6	1.531 (4)	C3-O3	1.421 (4)
C7-C8	1.502 (4)	C4-O4	1.438 (3)
C7-C9	1.505 (5)	C1-O4	1.427 (4)
C10-C11	1.504 (5)	C5-O5	1.437 (4)
C10-C12	1.518 (5)	C6-O6	1.417 (4)
C10-O5	1.425 (4)	C10-O6	1.424 (3)
O1-C1-C2	105.9 (2)	O1-C7-C8	108.1 (3)
O1-C1-O4	111.8 (3)	C9-C7-C8	113.9 (3)
C2-C1-O4	106.1 (2)	O1-C7-O2	105.0 (2)
O2-C2-C1	104.0 (2)	O2-C7-C9	110.5 (3)
C1-C2-C3	105.2 (2)	O2-C7-C8	108.8 (3)
C3-C2-O2	113.6 (2)	O6-C10-C11	109.0 (2)
C2-C3-O3	113.8 (2)	O6-C10-C12	111.3 (3)
C2-C3-C4	102.7 (2)	C11-C10-C12	112.4 (3)
C4-C3-O3	108.5 (3)	O5-C10-O6	104.1 (2)
C3-C4-O4	105.4 (2)	O5-C10-C11	109.7 (3)
C3-C4-C5	114.7 (2)	O5-C10-C12	110.0 (2)
C5-C4-O4	109.1 (2)	C1-O1-C7	109.4 (2)
C4-C5-C6	113.2 (3)	C2-O2-C7	110.5 (2)
C4-C5-O5	111.9 (2)	C1-O4-C4	107.0 (2)
C6-C5-O5	104.3 (2)	C10-O5-C5	108.7 (2)
O6-C6-C5	104.1 (3)	C6-O6-C10	107.5 (2)
O1-C7-C9	110.1 (3)		

Fig. 1 shows the molecule and numbering scheme and Fig. 2 a stereoscopic view of the unit-cell contents. Table 1* lists atom parameters; Table 2 gives bond distances and angles.

Related literature. The obtuse angle between the fused rings is 122.6 (8)°, calculated as the angle between the

* Lists of structure amplitudes, anisotropic thermal parameters, torsion angles, H-atom parameters and least-squares-planes' details have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44577 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

best planes formed by C1, C2 and the atoms bonded directly to them [cf. 2,3:5,6-di-*O*-isopropylidene- α -D-mannofuranose (Sheldrick, Mackie & Akrigg, 1985)].

Variations of the O...O distances in this and other structures with only one hydrogen bond per molecule seem to indicate that the energy of the hydrogen bond is not sufficient to dominate the crystal packing.

We wish to thank Andrea Whitehead for taking a series of X-ray photographs and the University of Leeds Computing Service for the provision of computing facilities.

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Structure of Dimethyl(picrato)(trifluoromethyl)tellurium

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Abstract. $[\text{Te}(\text{C}_6\text{H}_2\text{N}_3\text{O}_7)(\text{CF}_3)(\text{CH}_3)_2]$, $M_r = 454.78$, monoclinic, $P2_1/c$, $a = 12.468$ (5), $b = 7.239$ (9), $c = 16.346$ (9) Å, $\beta = 103.31$ (5)°, $V = 1436$ (2) Å³, $Z = 4$, $D_x = 2.104$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 2.1$ mm⁻¹, $F(000) = 872$, $T = 291$ (1) K, final $R = 0.033$ for 2255 unique observed [$F \geq 3.0\sigma(F)$] diffractometer data and 221 variables. In the crystalline state $[(\text{CH}_3)_2\text{Te}(\text{CF}_3)][\text{C}_6\text{H}_2\text{O}(\text{NO}_2)_3]$ molecules are linked *via* weak intermolecular Te...O contacts [3.134 (4), 3.138 (3) Å] such that if the free electron pair is taken into account a distorted pseudopentagonal bipyramid around Te is formed with C (trifluoromethyl) [Te–C 2.192 (5) Å] and O (picrate) [Te–O 2.496 (3) Å] in the apical positions and two C (methyl) [Te–C 2.115 (5), 2.111 (5) Å], two O (NO₂) atoms of two neighbouring molecules and the free electron pair in the equatorial positions. In the picrate group the planes through the NO₂ groups form dihedral angles of 46.0 (1), 7.8 (1) and 14.4 (1)° with the plane through the C atoms of the ring. Aromatic C–C bond lengths in the picrate ring range between 1.366 (6) and 1.445 (6) Å and intra-ring C–C–C angles between 111.5 (4) and 126.1 (4)°.

Experimental. Dimethyl(trifluoromethyl)tellurium iodide (Wilkes, 1984) reacts with picric acid to give the hitherto unknown title compound. Yellow single crystals could be obtained by slow evaporation of an ethanol–water solution at room temperature. Crystal

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size $\sim 0.13 \times 0.29 \times 0.19$ mm, $\omega/2\theta$ scan, scan speed 2.0–5.0° min⁻¹ in θ , Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$; lattice parameters from least-squares fit with 25 reflections up to $2\theta = 26.2^\circ$ equally distributed in reciprocal space; six standard reflections recorded every 2.5 h, only random

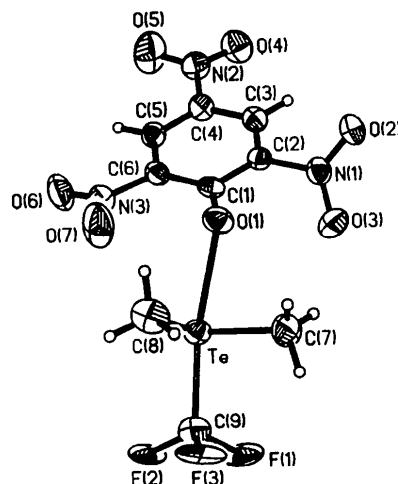


Fig. 1. View (*SHELXTL-Plus* graphic) of the molecule, showing the atom-numbering scheme. The F atoms are disordered and for each F atom two positions have been refined. Only one of these two positions is shown in the figure.