peak of height 0·71 e Å^{-3} in the difference Fourier map [O—H = 1·23, O···O = 2·454 (4) Å]. Similarly the hydroxyl groups of molecules *B* are H-bonded through an inversion centre (HSM2) at 0,0·5,0·5 with a peak of height 0·52 e Å^{-3} in the difference Fourier map [O···O = 2·457 (4), O—H = 1·23 Å] indicating the presence of symmetric hydrogen bonds (Table 3, Fig. 2). The hydroxyl H atoms are shared between molecules, and hence only one hydroxyl H is associated per molecule, the other protonating the ammonium group. Short O···O hydrogen bonds (~ 2·45 Å) have been observed in the crystal structure of acid salts of mono- and dicarboxylic acids (Speakman, 1972; Novak, 1974). The two independent molecules *A* and *B* have similar bond lengths, bond angles and torsion angles.

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Structure of 2-Methylnaphthimidazolium Chloride Dihydrate

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Abstract. $C_{12}H_{11}N_2^+.Cl^-.2H_2O$, $M_r = 254·72$, triclinic, $P\bar{1}$, $a = 7·326$ (2), $b = 9·634$ (3), $c = 10·927$ (2) Å, $\alpha = 62·42$ (2), $\beta = 74·77$ (2), $\gamma = 72·89$ (2)°, $V = 646·0$ (3) Å^3, $Z = 2$, $D_x = 1·309$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·46$ cm^{-1}, $T = 298$ K, $F(000) = 268$, final $R = 0·035$ for 1410 unique reflections. 2-Methylnaphthimidazolium chloride dihydrate was prepared by reacting 2,3-diaminonaphthalene with glacial acetic acid and hydrochloric acid. The composition of the compound was suggested by ^{1}H NMR spectra. Crystallographic analysis confirmed the complete structure. The cation is essentially planar excluding methyl H atoms. The planar rings have considerable aromatic character. The cations are linked by hydrogen-bonding bridges of the form N—H···Cl$^-$···H—O···H—O···H—N.

Introduction. There have been several reports of the structure of imidazole, including those by Martínez-Carrera (1966), Will (1969) and Craven, McMullan, Bell & Freeman (1977). The latter authors used neutron diffraction and data collected at 293 and 123 K. However, we are not aware of any reports of the structure of an imidazolium salt.

2-Methylnaphthimidazolium chloride dihydrate was synthesized as an intermediate as part of a program to prepare and characterize substituted naphthalenes as potential components of donor–acceptor complexes.

Experimental. 2,3-Diaminonaphthalene (0·5 g) was dissolved in glacial acetic acid (10 ml) and the reaction mixture was heated to the reflux temperature. Hydrochloric acid (37·5%, 3 ml) was added after 5 min and the reaction mixture was refluxed continuously for 1 h. The reaction solution was poured into a 100 ml beaker, covered and left standing at room temperature for 1–2 days. It yielded a crude product, which was filtered and dissolved in H_2O by heating to make a saturated solution. After filtering the hot saturated solution, a clear yellow solution was obtained. It gave yellowish needle-shaped crystals by slow evaporation of solvent at room temperature.

A transparent needle crystal with dimensions 0·58 × 0·18 × 0·25 mm was chosen for study by X-ray diffraction. A modified Picker FACS-1 diffractometer was used for data collection, with graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan technique. Unit-cell parameters from 40 reflections, $27 < 2\theta < 37°$.

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Three standard reflections were monitored after every 47 reflections. Standard reflections did not show significant deviations from mean intensities. 1680 unique reflections were measured in the range $3 \leq 2\theta \leq 45°$ with $0 \leq h \leq 7$, $-9 \leq k \leq 10$, $-10 \leq l \leq 11$ at 293 (2) K. Of these, 1410 reflections were considered observed with $I \geq 2\sigma(I)$. Lorentz and polarization corrections were calculated. Absorption corrections were not applied. The structure was solved by direct methods and Fourier syntheses and refined on F by full-matrix least squares with $\sigma^{-2}(F)$ weights using the *XTAL* system of crystallographic programs (Stewart & Hall, 1984). The $\sigma(F)$ were determined from counting statistics plus a contribution calculated from the excess scatter of the standard reflections. The E map obtained from direct methods gave all of the 17 non-hydrogen atoms except for the water oxygen atoms. These 15 non-hydrogen atoms were refined for several cycles of full-matrix least squares with isotropic temperature factors. Difference Fourier syntheses then gave the two O atoms. After several more cycles of full-matrix least-squares anisotropic refinement with all non-hydrogen atoms, all of the H atoms were located on a difference map. Refinement was continued with the H atoms refined isotropically. For the final cycle of least squares $R = 0{\cdot}040$, $wR = 0{\cdot}035$, $(\Delta/\sigma)_{\max} = 0{\cdot}33$, $(\Delta/\sigma)_{\rm av} = 0{\cdot}016$. In the final difference Fourier map $\Delta\rho = 0{\cdot}26$ e Å^{-3}. Atomic scattering factors were from Cromer & Mann (1968). All computations were carried out with a VAX8600 computer and the *XTAL* system of crystallographic programs.

Discussion. The atomic coordinates of the non-hydrogen atoms and equivalent isotropic temperature factors are listed in Table 1. Bond lengths and angles are presented in Table 2. A view of the molecule with the numbering scheme is shown in Fig. 1.*

The proton nuclear magnetic resonance spectrum shows a sharp singlet absorption at $\delta = 2{\cdot}60$, representing three methyl H atoms, and a broad doublet absorption centered approximately at $\delta = 7{\cdot}42$, representing six aromatic H atoms. The N—H proton signals do not appear in the ^{1}H spectra because of the rapid exchange of the NH proton with deuterium in D_2O. Crystallographic analysis confirms the complete structure.

The bond lengths N(1)—C(11) and N(2)—C(11) are 1·337 (4) and 1·327 (6) Å respectively. Craven *et al.* (1977) found 1·337 (3) Å for the bond length to the protonated N atom and 1·316 (2) Å for the double bond to the unprotonated N atom. The partial double-bond character of the bonds between N and C(11) is based on interaction between the positive charge at C(11) and the electron pairs on the two N

Table 1. *Fractional atomic coordinates with e.s.d.'s in parentheses and equivalent isotropic thermal parameters* (Å^2 × 10^2)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j a_i a_j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cl(1)	0·4216 (1)	0·0564 (1)	0·2723 (1)	7·1
O(1)	−0·0122 (5)	0·2492 (3)	0·2320 (3)	6·6
O(2)	−0·2250 (5)	0·0248 (4)	0·4076 (4)	8·8
C(1)	−0·1487 (5)	0·3226 (4)	0·5618 (4)	4·7
C(2)	−0·2069 (4)	0·4598 (4)	0·4491 (3)	4·3
C(3)	−0·3053 (4)	0·6027 (4)	0·4616 (3)	4·4
C(4)	−0·3455 (5)	0·6170 (4)	0·5848 (4)	4·9
C(5)	−0·3156 (5)	0·4834 (5)	0·8359 (4)	5·5
C(6)	−0·2614 (6)	0·3497 (5)	0·9498 (4)	6·3
C(7)	−0·1771 (5)	0·2027 (5)	0·9405 (4)	6·4
C(8)	−0·1408 (5)	0·1934 (4)	0·8162 (4)	5·9
C(9)	−0·1913 (4)	0·3318 (4)	0·6920 (3)	4·4
C(10)	−0·2843 (4)	0·479 (4)	0·7030 (3)	4·5
C(11)	−0·2719 (5)	0·6438 (4)	0·2394 (4)	5·0
C(12)	−0·2844 (8)	0·7225 (6)	0·0868 (4)	6·8
N(1)	−0·1867 (4)	0·4919 (3)	0·3081 (3)	4·9
N(2)	−0·3434 (4)	0·7139 (3)	0·3266 (3)	5·0

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

C(1)—C(2)	1·372 (4)	C(1)—C(9)	1·413 (6)
C(2)—C(3)	1·404 (5)	C(2)—N(1)	1·400 (5)
C(3)—C(4)	1·363 (6)	C(3)—N(2)	1·407 (4)
C(4)—C(10)	1·414 (4)	C(5)—C(6)	1·356 (5)
C(5)—C(10)	1·426 (6)	C(6)—C(7)	1·408 (7)
C(7)—C(8)	1·354 (7)	C(8)—C(9)	1·434 (4)
C(9)—C(10)	1·429 (5)	N(1)—C(11)	1·337 (4)
N(2)—C(11)	1·327 (6)	C(11)—C(12)	1·494 (6)
C(2)—C(1)—C(9)	116·7 (3)	C(1)—C(2)—C(3)	122·1 (4)
C(1)—C(2)—N(1)	131·3 (4)	C(3)—C(2)—N(1)	106·7 (3)
C(2)—C(3)—C(4)	122·9 (3)	C(2)—C(3)—N(2)	105·6 (3)
C(4)—C(3)—N(2)	131·6 (3)	C(3)—C(4)—C(10)	116·9 (3)
C(6)—C(5)—C(10)	120·8 (4)	C(5)—C(6)—C(7)	120·8 (5)
C(6)—C(7)—C(8)	120·6 (3)	C(7)—C(8)—C(9)	120·8 (4)
C(8)—C(9)—C(1)	120·7 (3)	C(8)—C(9)—C(10)	118·4 (4)
C(1)—C(9)—C(10)	120·9 (3)	C(5)—C(10)—C(9)	118·5 (3)
C(4)—C(10)—C(9)	120·5 (4)	C(4)—C(10)—C(5)	121·0 (4)
C(2)—N(1)—C(11)	108·6 (3)	C(3)—N(2)—C(11)	109·2 (3)
N(1)—C(11)—C(12)	124·7 (4)	N(1)—C(11)—N(2)	110·1 (3)
N(2)—C(11)—C(12)	125·2 (3)		

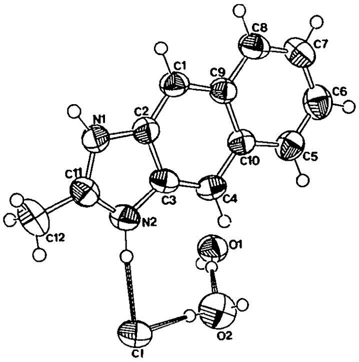

Fig. 1. *ORTEP* view (Johnson, 1976) of the compound illustrating atom labelling. Thermal ellipsoids scaled to 50% probability.

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* Lists of structure factors, anisotropic temperature factors and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44758 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

atoms which causes a dispersion of charge through N(1)—C(11)—N(2). As a consequence the N(1)—C(2) and N(2)—C(3) bonds are longer at 1·400 (5) and 1·407 (4) Å respectively.

The naphthalene rings, imidazolium ring and the methyl C atom are all coplanar, with a maximum deviation from the mean plane of 0·054 (5) Å, indicating that the entire cation is a completely delocalized π system. The Cl^- anion is 2·314 (6) Å removed from the plane of the cation. The anion is the receptor of two strong hydrogen bonds, one from water, to form a complex bridge between pairs of cations of the form N—H···Cl^-···H—O···H—O···H—N. The remaining two water H atoms form weaker hydrogen bonds with the Cl^- anion. A stereo packing diagram of the molecules is shown in Fig. 2. The molecules form stacks along the **a** direction. The two independent interplanar separations between adjacent molecules are 3·57 (3) and 3·49 (3) Å. The bond lengths and angles of the hydrogen bonds are given in Table 3. Other bond lengths and angles fall in the normal ranges.

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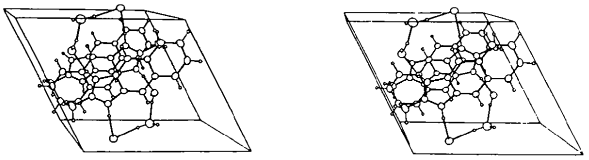

Fig. 2. Stereo packing diagram of the molecules along the *a* axis. The *a* axis is into the page, the *b* axis is vertical and the *c* axis is horizontal.

Table 3. *Hydrogen-bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

X—H···*Y*	*X*—H (Å)	H···*Y* (Å)	*X*···*Y* (Å)	*X*—H···*Y* (°)
N(2)—H(O2)···Cl	1·03 (4)	2·07 (4)	3·096 (3)	176 (3)
O(2)—H(O22)···Cl	1·05 (7)	2·18 (6)	3·179 (5)	150 (2)
O(1)—H(O12)···O(2)	0·95 (4)	1·77 (4)	2·712 (5)	169 (5)
N(1)—H(O1)···O(1)	0·94 (4)	1·78 (4)	2·698 (5)	167 (2)
O(1)—H(O11)···Cl	0·85 (4)	2·36 (4)	3·202 (4)	172 (4)
O(2)—H(O21)···Cl	0·82 (5)	2·36 (5)	3·226 (4)	176 (4)

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Structures of Two Isomeric Thiodisaccharides

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Abstract. Two isomeric thiodisaccharides of formula $C_{27}H_{38}O_{16}S$, $M_r = 650·62$, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 1·5–1·6$ cm^{-1}, $T \sim 298$ K. (1) 3,5-Di-*O*-acetyl-6-*S*-[2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranosyl]-6-deoxy-1,2-*O*-isopropylidene-6-thio-α-D-glucofuranose, triclinic, $P1$, $a = 8·018$ (1), $b = 9·834$ (2), $c = 11·240$ (2) Å, $\alpha = 78·51$ (1), $\beta = 80·64$ (1), $\gamma = 85·68$ (1)°, $V = 856·04$ (25) $Å^3$, $Z = 1$, $D_x = 1·26$ g cm^{-3}, $F(000) = 344$, $R = 0·050$ for 3126 unique observed reflections with $I > \sigma(I)$. (2) 3,5-Di-*O*-acetyl-6-*S*-[2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyl]-6-deoxy-1,2-*O*-isopropylidene-6-thio-α-D-glucofuranose, monoclinic, $P2_1$, $a = 5·589$ (1), $b = 20·161$ (6), $c = 14·983$ (3) Å, $\beta = 94·52$ (2)°, $V = 1682·95$ (64) $Å^3$, $Z = 2$, $D_x = 1·28$ g cm^{-3}, $F(000) = 688$, $R = 0·053$ for 2909 unique observed reflections with $I > \sigma(I)$. The molecules have the same connectivity, and differ only in the gluco–galacto configurations; the consequence is that the galacto isomer is more extended than the gluco. The r.m.s. difference for 46 equivalent bonds in the two isomers is 0·016 Å, which is about three times the least-squares estimate.

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