Fig. 2. The crystal structure viewed along the *c* axis. H atoms omitted.

formation: atoms C(7) and C(10) deviate +0·639 (3) and −0·695 (4) Å, respectively, from the mean plane of the other four atoms [maximum deviation 0·001 (5) Å]. The distance between the axial methyl groups, C(13)··· C(14) = 3·250 (7) Å, is even shorter than the corresponding one in drimenol: 3·359 (6) Å [average of three molecules; Escobar & Wittke (1984)] and also than that in a new derivative of the latter: 3·29 (1) Å (Brianso, Piniella, Germain & Solans, 1987). Closely related to isodrimenin is alantolactone (the lactone ring is *cis* fused to the cyclohexene ring) where the distance under discussion is 3·44 (1) Å, as calculated from the published coordinates (Schmalle, 1986). Among other compounds where this steric arrangement of the cyclohexane methyl groups is observed are forskolin with C···C 3·23 (1) Å (Paulus, 1980) and an *ent*-beyerene diterpene with C···C 3·245 (8) Å (Watson, Tavanaiepour, Lee & Mabry, 1986). Bond lengths and bond angles in the present structure are comparable to those observed in the compounds referenced above.

A view of the crystal packing is given in Fig. 2, where the dashed lines show the most significant intermolecular proximities, as given in Table 2.

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Structure of 2-Amino-3-methyl-5-(D-*arabino*-1,2,3,4-tetrahydroxybutyl)imidazolium Chloride

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Abstract. $C_8H_{16}N_3O_4^+.Cl^-$, $M_r = 253\cdot7$, orthorhombic, $P2_12_12_1$, $a = 10\cdot289$ (5), $b = 15\cdot343$ (8), $c = 7\cdot367$ (4) Å, $V = 1163\cdot0$ (6) Å^3, $Z = 4$, $D_m = 1\cdot45$, $D_x = 1\cdot45$ Mg m^{-3}, $\lambda(Mo\,K\alpha) = 0\cdot71069$ Å, $\mu = 0\cdot32$ mm^{-1}, $F(000) = 536$, room temperature, final $R = 0\cdot08$ for 1372 observed reflections. The imidazole group is involved in a protonation process. The delocalization of the positive charge was evidenced by MNDO calculations. The configuration in the crystals and in the isolated molecule is biplanar (angles between planes 7·1°); also the configuration around the chiral centres C(8), C(7) and C(6) of the sugar chain is *R, R* and *S, i.e.* D-*arabino*. Packing is governed by van der Waals forces and hydrogen bonds. The Cl anion is involved in two H bonds, one with the sugar and the other with the imino group.

Introduction. The title compound has been studied by UV, IR and NMR (Alaiz-Barragan, 1987) at the Organic Chemistry Department of the University of Seville, Spain. The compound was synthesized by reaction with hydrogen chloride of the precipitated picrate (II), obtained by reaction of 1-alkyl 1-methyl-amino-1-deoxy-D-*lyxo*-hexulose (I) with cyanamide and precipitation with picric acid.

(I)   (II)   (III)
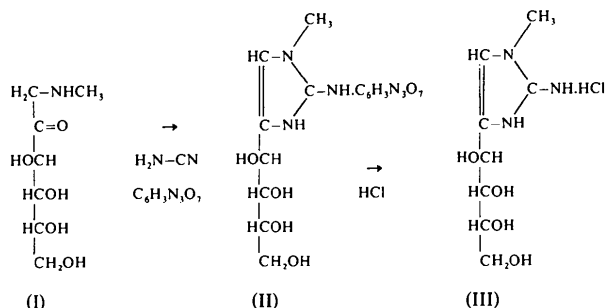
In an attempt to establish possible correlations between UV, IR and NMR spectroscopic data and X-ray single-crystal data, the crystal structure of (III) has been determined. The resolution of the structure confirms the conformation and the presence of a protonated iminoimidazole group as indicated by the spectroscopic data.

Experimental. Crystal size $0.28 \times 0.20 \times 0.40$ mm. D_m by flotation. Enraf–Nonius CAD-4 diffractometer with graphite monochromator and Mo $K\alpha$ radiation. Cell dimensions from 2θ angles for 25 reflections ($3 < \theta < 14°$), $\omega/2\theta$ mode, $2\theta_{max} = 60°$ ($h \leq 14$, $k \leq 21$, $l \leq 10$). 1378 independent reflections measured, $R_{int} = 0.03$ from merging 43 equivalent reflections, $I > 2\sigma(I)$; Lp correction, absorption ignored; two check reflections $(130,\overline{1}30)$ every 5400s showed no unusual variation; direct methods, *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); full-matrix least-squares refinement based on F, anisotropic temperature factors for the non-hydrogen atoms; difference Fourier synthesis revealed H atoms which were included with the isotropic temperature factors for the atoms to which they were bound, but not refined. Further least-squares refinement gave $R = 0.08$ ($wR = 0.09$); weighting scheme based on statistical count criterion $w = 1/\sigma^2(I)$. $(\Delta/\sigma)_{max} = 0.16$, $S = 3.14$ for 145 refined parameters. 11.5 reflections/parameter. Final difference synthesis showed $\Delta\rho = \pm 0.3$ e Å^{-3}. Crystallographic programs of *XRAY*70 system (Stewart, Kundell & Baldwin, 1970) were used. The uncommonly high R factor and e.s.d.'s, as well as the discrepancies between F_o and F_c, seem to be due to absorption problems resulting from the shape and mediocre quality of the crystals. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^4$)

	x	y	z	U_{eq}
Cl	0.2110 (3)	−0.3435 (1)	0.1913 (4)	553 (8)
N(1)	0.3429 (6)	0.0188 (4)	0.1746 (9)	310 (19)
N(2)	0.2567 (7)	−0.1251 (4)	0.1802 (11)	466 (24)
N(3)	0.1327 (6)	0.0058 (4)	0.1943 (10)	368 (21)
C(2)	0.2451 (7)	−0.0388 (4)	0.1849 (10)	308 (20)
C(3)	0.0032 (8)	−0.0328 (6)	0.1959 (14)	503 (32)
C(4)	0.1621 (7)	0.0953 (5)	0.1903 (12)	371 (24)
C(5)	0.2916 (7)	0.1036 (4)	0.1782 (10)	300 (20)
C(6)	0.3773 (7)	0.1821 (5)	0.1647 (9)	291 (20)
C(7)	0.2988 (7)	0.2663 (4)	0.1951 (9)	279 (19)
C(8)	0.3914 (7)	0.3459 (5)	0.2089 (9)	289 (19)
C(9)	0.3110 (7)	0.4282 (5)	0.2261 (11)	356 (24)
O(6)	0.4357 (5)	0.1823 (4)	−0.0097 (7)	381 (18)
O(7)	0.2050 (5)	0.2773 (3)	0.0584 (8)	333 (16)
O(8)	0.4751 (5)	0.3352 (4)	0.3604 (8)	392 (18)
O(9)	0.3969 (6)	0.5006 (4)	0.2481 (11)	487 (23)

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

O(6)–C(6)	1.418 (8)	C(7)–C(8)	1.552 (10)
N(1)–C(5)	1.404 (8)	N(3)–C(4)	1.406 (9)
N(1)–C(2)	1.341 (9)	N(3)–C(2)	1.346 (9)
C(3)–N(3)	1.457 (10)	C(9)–C(8)	1.515 (10)
C(5)–C(6)	1.496 (10)	C(9)–O(9)	1.429 (10)
C(5)–C(4)	1.342 (10)	C(8)–O(8)	1.419 (8)
O(7)–C(7)	1.405 (8)	C(2)–N(2)	1.330 (8)
C(7)–C(6)	1.540 (9)		

C(5)–N(1)–C(2)	109.1 (5)	O(6)–C(6)–C(7)	110.6 (5)
N(1)–C(5)–C(4)	106.7 (5)	O(6)–C(6)–C(5)	108.2 (5)
N(1)–C(5)–C(6)	121.5 (6)	C(5)–C(4)–N(3)	107.9 (6)
C(6)–C(5)–C(4)	131.8 (6)	C(8)–C(9)–O(9)	108.7 (6)
O(7)–C(7)–C(8)	112.0 (5)	C(7)–C(8)–C(9)	109.0 (5)
O(7)–C(7)–C(6)	110.9 (5)	C(9)–C(8)–O(8)	111.2 (6)
C(6)–C(7)–C(8)	110.3 (5)	C(7)–C(8)–O(8)	109.5 (5)
C(3)–N(3)–C(2)	125.5 (6)	N(1)–C(2)–N(3)	108.2 (5)
C(3)–N(3)–C(4)	126.3 (6)	N(3)–C(2)–N(2)	125.8 (6)
C(4)–N(3)–C(2)	108.1 (5)	N(1)–C(2)–N(2)	126.0 (6)
C(5)–C(6)–C(7)	110.9 (5)		

Discussion. Table 1 gives the atomic coordinates and Table 2 the bond lengths and angles.* A view of the molecule with the atom numbering is shown in Fig. 1. Bond distances and angles in the sugar chain which have the expected values will not be discussed. The imidazole part of the molecule is protonated: consequently the three N atoms N(1), N(2) and N(3) are chemically equivalent. The three bond lengths C(2)–N [mean value 1.338 (9) Å] are shorter than N(1)–C(5) = 1.404 (8) and N(3)–C(4) = 1.406 (9) Å; their values are intermediate between a double bond (1.265 Å) and a single bond (1.470 Å). This result indicates a delocalization of the double bond on the imidazole group and a delocalization of the positive charge on the central C atom of this group, C(2), and probably on the
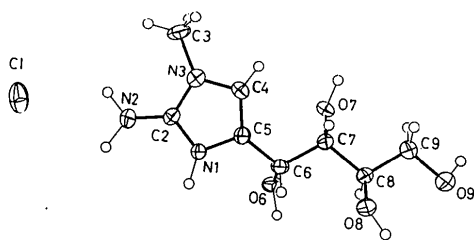
$C_8H_{16}N_3O_4^+.Cl^-$

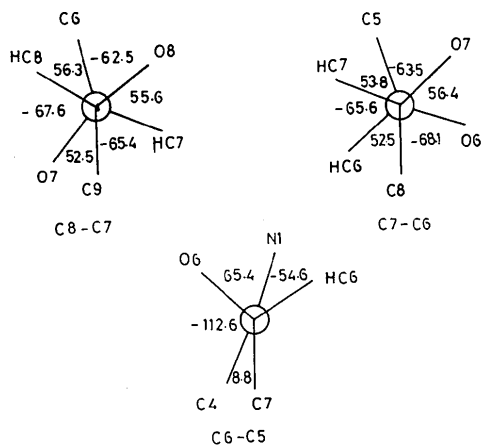Fig. 1. A view of the molecule showing the labelling scheme employed.
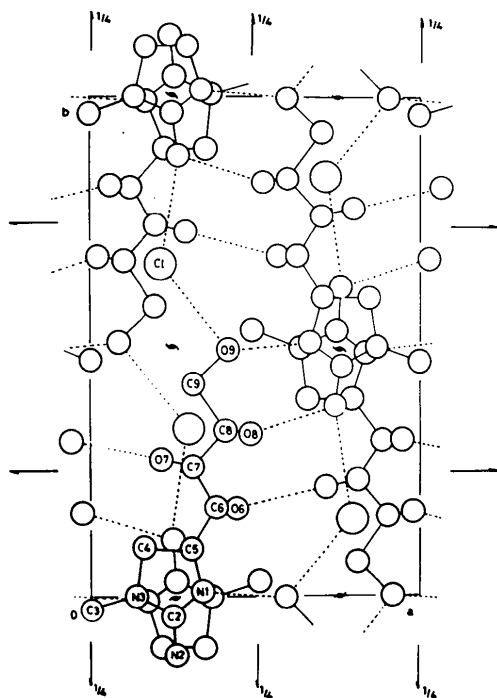

Fig. 2. Some Newman projections (angles in degrees).

Fig. 3. Projection of the unit-cell packing. Dashed lines indicate hydrogen bonds.

Table 3. *Geometry* (Å,°) *of the hydrogen bonds* (*e.s.d.'s in parentheses*)

It is assumed that e.s.d.'s for H atoms in calculated positions are ten times those of the atoms to which they are attached.

Donor—H	Donor⋯ Acceptor	H⋯ Acceptor	Donor—H⋯ Acceptor
N2—H22	N2⋯Cl	H22⋯Cl	N2—H22⋯Cl
1·00 (6)	3·385 (6)	2·70 (6)	125 (5)
O(7)—H(7)	O(7)⋯O(6^i)	H(7)⋯O(6^i)	O(7)—H(7)⋯O(6^i)
0·99 (5)	2·862 (7)	2·06 (5)	135 (4)
O(8)—H(8)	O(8)⋯N(2^{ii})	H(8)⋯N(2^{ii})	O(8)—H(8)⋯N(2^{ii})
0·98 (5)	2·842 (9)	2·01 (5)	140 (5)
O(6)—H(6)	O(6)⋯O(7^{iii})	H(6)⋯O(7^{iii})	O(6)—H(6)⋯O(7^{iii})
0·97 (5)	2·862 (7)	1·99 (5)	147 (4)
O(9)—H(9)	O(9)⋯Cl^{iv}	H(9)⋯Cl^{iv}	O9—H9⋯Cl^{iv}
1·01 (6)	3·091 (7)	2·22 (6)	143 (6)
N(2)—H(21)	N(2)⋯O(8^v)	H(21)⋯O(8^v)	N(2)—H(21)⋯O(8^v)
1·09 (6)	2·842 (9)	1·89 (7)	143 (4)
N(1)—H(11)	N(1)⋯O(9^v)	H(11)⋯O(9^v)	N(1)—H(11)⋯O(9^v)
1·00 (6)	2·751 (9)	1·81 (6)	152 (5)

Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, -z$; (ii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (iii) $x + \frac{1}{2}, -y + \frac{1}{2}, -z$; (iv) $x, y + 1, z$; (v) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$.

H atoms bonded to the three N atoms. In order to verify this assumption, semiempirical MNDO calculations (Dewar & Phiel, 1977) were performed on the protonated molecule. Only results concerning the imidazole groups present some features of interest. The atomic charges for the atoms in this group are:
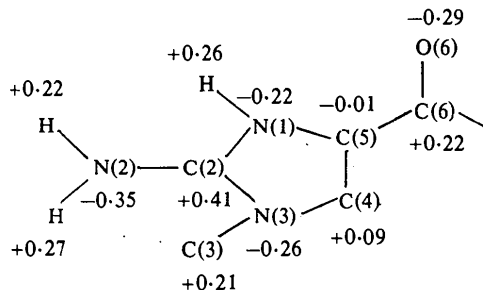
We can observe that the most important positive charge is located on C(2) and the remainder is distributed mainly on the three H atoms of the imidazole group [+0·22 and +0·27 on H atoms attached to N(2) and +0·26 on H atom attached to N(1)]. The sugar chain and the imino group are planar (maximum deviations 0·009 and 0·001 Å respectively). The atoms N(2), C(3) and C(6) are above the plane by 0·035 (9), 0·081 (10) and 0·031 (8) Å. The dihedral angle between the sugar and the imino-group planes is 7·1 (3)°. The Newman projections corresponding to the C—C bonds of the sugar chain are shown in Fig. 2. According to the Klyne & Prelog (1960) rules the configuration around the chiral centres C(8), C(7) and C(6) is *R, R* and *S,* in accordance with the D-*arabino* constitution. The crystal cohesion is ensured by H bonding, which is shown in Fig. 3. The H-bond geometry calculated using unrefined H-atom positions is given in Table 3. In this structure the Cl$^-$ anion is involved in two H bonds, one with the O(9) of

the sugar chain and another with the N(2) of the imino group.

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Structure of (2S,4S)-trans-4-Methylthio-2-phthalimido-γ-butyrolactone

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Abstract. $C_{13}H_{11}NO_4S$, $M_r = 277\cdot29$, monoclinic, $P2_1$, $a = 11\cdot211$ (3), $b = 6\cdot822$ (2), $c = 8\cdot710$ (2) Å, $\beta = 99\cdot90$ (1)°, $V = 656\cdot2$ (3) Å³, $Z = 2$, $D_x = 1\cdot40$ g cm⁻³, $\lambda(\mathrm{Cu}\,K\alpha) = 1\cdot5418$ Å, $\mu = 22\cdot451$ cm⁻¹, $F(000) = 288$, room temperature, $R = 0\cdot038$ for 1301 observed unique reflections. The lactone ring has an envelope conformation with C3 deviating by $0\cdot498$ (3) Å from the least-squares plane formed by the remaining four ring atoms. The absolute configuration of the two chiral carbon atoms was deduced as (2S,4S). The phthalimido and methylthio groups are in pseudo-equatorial and pseudo-axial position respectively, trans to each other.

Introduction. In order to establish the stereochemistry of the different 2,4-disubstituted γ-butyrolactones we report here the crystal and molecular structure of the title compound. Some of the other stereoisomers from this series have already been published (Bruvo, Sikirica & Vicković, 1981; Sikirica, Vicković & Viterbo, 1979).

Experimental. Colorless prismatic crystal, approximately $0\cdot29 \times 0\cdot52 \times 0\cdot19$ mm, Siemens AED single-crystal diffractometer, Ni-filtered Cu $K\alpha$ radiation, cell parameters from least-squares fit of 30 reflections ($\theta > 15°$) accurately measured on the diffractometer, room temperature, one check reflection measured every

50 counts without significant variation, profile analysis with Lehmann & Larsen (1974) procedure modified by Belletti, Ugozzoli, Cantoni & Pasquinelli (1979); $3 < \theta < 70°$, Lp correction, absorption ignored, $-13 < h < 13$, $0 < k < 8$, $0 < l < 10$, 2304 reflections collected, 1306 unique observed at $2\sigma(I)$ level ($R_{int} = 0\cdot012$),

Table 1. *Atomic fractional coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å² × 10⁴)

	x	y	z	U_{eq}*
S	6340 (1)	8204	6918 (1)	588 (2)
O1	5862 (2)	10343 (3)	9313 (2)	536 (6)
O2	6969 (2)	12761 (4)	10572 (3)	688 (7)
O3	7170 (2)	9660 (4)	13413 (2)	592 (6)
O4	10478 (2)	9650 (5)	11003 (2)	731 (8)
N	8640 (2)	9631 (4)	11855 (2)	462 (6)
C1	6895 (2)	11088 (4)	10132 (3)	477 (7)
C2	7856 (2)	9506 (4)	10349 (3)	461 (7)
C3	7127 (3)	7624 (5)	10019 (3)	580 (10)
C4	6039 (2)	8318 (4)	8876 (3)	496 (7)
C5	4921 (4)	9013 (8)	5840 (5)	822 (13)
C6	8229 (2)	9661 (4)	13273 (3)	437 (7)
C7	9335 (2)	9705 (4)	14491 (3)	418 (6)
C8	9456 (3)	9706 (5)	16098 (3)	506 (8)
C9	10628 (3)	9669 (5)	16949 (3)	537 (7)
C10	11620 (2)	9666 (5)	16220 (3)	531 (8)
C11	11506 (2)	9665 (5)	14592 (3)	509 (7)
C12	10333 (2)	9679 (4)	13760 (3)	433 (6)
C13	9909 (2)	9661 (5)	12042 (3)	487 (7)

* Hamilton (1959).