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Casimidine, a Fragment of Casimiroedine Containing β-Glucose and N-Methylhistamine

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The molecular and crystal structure of the dihydrochloride of the alkaloid casimidine has been elucidated. A β-glucose residue is established and is shown to be glycosidically linked to an unprotonated N atom not adjacent to the point of attachment of the $CH_2CH_2NHCH_3$ group of N-methylhistamine. The H-bonding scheme suggests that the other two N atoms of the dihydrochloride ($C_{12}H_{21}N_3O_5.2HCl$) are protonated. There are four formula weights in a unit cell of dimensions $a=27{\cdot}81$, $b=10{\cdot}76$ and $c=5{\cdot}76$ Å, and the space group is $P2_12_12_1$. The final value of $R=\Sigma|\,|F_o|-|F_c|\,|/\Sigma|F_o|$ is 0·12 for the 1036 observed reflections.

The alkaloid casimiroedine ($C_{21}H_{27}N_3O_6$), from the seeds of the Mexican fruit *Casimiroa edulis* La Llave & Lejarza (Power & Callan, 1911), is either the *trans* or *cis* cinnamic acid amide of casimidine ($C_{12}H_{21}N_3O_5$), which has been shown to contain D-glucose (Djerassi, 1961) linked to N-methylhistamine (Djerassi, Bankiewicz, Kapoor & Riniker, 1958). However, the organic chemical studies have not established whether the glucose is α or β, whether it is a five or six membered ring, and to which N atom of the histamine ring the sugar is attached.

This X-ray diffraction study establishes these points unambiguously, and furthermore yields the internal and external bond angles of the histamine ring for comparison of the results for the free base when they become available (Andrews, DeCou & Dragsdorf, 1961), and with the more precise values found by Donohue, Lavine & Rollett (1956). In addition, some comparisons are made of the β-glucose residue with the β-glucose (Ferrier, 1960) and α-glucose (McDonald & Beevers, 1952) molecules, and with cellobiose (Jacobson, Wunderlich & Lipscomb, 1961).

This study was carried out on the dihydrochloride of casimidine, $C_{12}H_{21}N_3O_5.2HCl$. After location of Cl^- ions in an array which, distressingly, had more symmetry than $P2_12_12_1$, the only assumptions which were made were atoms of equal scattering power at C, N or O positions of N-methylhistamine and only the six atoms of the ring of the sugar. The structural ambiguities were solved by somewhat intuitive procedures based in part on hydrogen-bonding and other aspects of molecular packing, and in the end all atoms except H were identified by criteria based on the X-ray study. In addition, improved methods of sharpening of Patterson functions were found, and a further development was made for aid in deconvolution of the Patterson function with use of a partial structure, but these techniques will be discussed elsewhere.

Structure determination

A single crystal of casimidine dihydrochloride, grown with some difficulty, was needle-like along the c axis. The reciprocal lattice symmetry of D_{2h}, and extinctions only of $h00$ when h is odd, of $0k0$ when k is odd and of $00l$ when l is odd led to the assignment of $P2_12_12_1$ as the probable space group. Multiple film and timed single film diffraction Weissenberg photographs were taken of the levels hkL for $0\le L\le 4$ with Cu $K\alpha$ radiation ($\lambda=1{\cdot}542$ Å), and timed single film photographs were taken of the $h0l$ and $h1l$ levels with use of a precession camera and Mo $K\alpha$ radiation. Unit cell dimensions are

$$a=27{\cdot}81\pm0{\cdot}05,\ b=10{\cdot}76\pm0{\cdot}04,\ c=5{\cdot}76\pm0{\cdot}02\ \text{Å}\,.$$

The space group requires $4m$ molecules per unit cell, but only choice of $m=1$ yields a reasonable calculated density (1·39 g.cm.$^{-3}$) compared with other organic amine hydrochlorides. Visual estimates with use of a standard scale of timed exposures were made of 1036 observed reflections, which were corrected for Lorentz-polarization factors, correlated, and scaled by Wilson's (1942) method to within about 20% of the final scaling obtained as described below.

In order to achieve the resolution necessary for solution of the structure, derivative sharpening was employed in the calculation of the three-dimensional Patterson function (Jacobson, Wunderlich & Lipscomb, 1961), but about 18% of the unsharpened Patterson function was added at each point to reduce second order negative peaks of the derivative-sharpened function. Inasmuch as these procedures have permitted solutions of structures which were not solved by exponential sharpening, we are currently developing an improvement in the derivative sharpening procedure which involves use of a linear combination of self convolutions of the nth derivatives of the electron density $\varrho(x, y, z)$. A further pos-

Table 1. *Observed and calculated structure amplitudes*

h	k	ℓ	F_o	F_c
0	2	0	28	22
0	4	0	67	66
0	6	0	57	53
0	8	0	41	42
0	10	0	30	36
1	4	0	28	32
1	8	0	21	18
2	1	0	54	54
2	2	0	50	45
2	3	0	37	35
2	4	0	98	103
2	6	0	23	23
2	7	0	32	35
3	1	0	26	19
3	2	0	42	41
3	3	0	9	3
3	4	0	19	20
3	5	0	44	41
4	1	0	28	24
4	2	0	28	28
4	3	0	21	20
4	5	0	17	13
4	8	0	27	31
5	1	0	24	22
5	2	0	51	49
5	3	0	34	36
5	4	0	44	42
5	5	0	24	23
5	6	0	25	21
6	1	0	40	28
6	2	0	110	112
6	3	0	18	14
6	4	0	100	93
6	6	0	23	26
6	8	0	21	24
7	1	0	8	0
7	2	0	13	13
7	4	0	47	42
7	5	0	28	32
8	0	0	45	46
8	1	0	20	20
8	2	0	103	109
8	4	0	76	66
8	6	0	38	34
8	8	0	27	32
9	1	0	47	44
9	3	0	87	88
9	4	0	56	44
9	5	0	35	31
9	6	0	23	25
9	7	0	16	18
9	9	0	18	25
10	0	0	52	40
10	1	0	12	10
10	2	0	66	63
10	3	0	15	11
10	4	0	80	55
10	5	0	20	22
10	6	0	14	13
10	8	0	18	23
11	1	0	61	57
11	2	0	11	13
11	3	0	21	21
11	5	0	13	12
11	6	0	22	22
11	7	0	36	41
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12	4	0	59	52
12	5	0	24	21
12	6	0	41	45
13	2	0	13	11
13	3	0	41	33
13	4	0	30	28
13	6	0	25	21
13	8	0	16	18
14	0	0	22	18
14	1	0	33	31
14	2	0	75	66
14	4	0	56	47
14	6	0	27	28
15	1	0	50	48
15	3	0	54	47
15	4	0	26	23
15	5	0	28	30
15	6	0	18	23
15	7	0	26	31
17	1	0	56	56
17	2	0	15	17
17	3	0	51	44
17	4	0	30	32
17	5	0	33	28
17	6	0	30	27
17	7	0	26	25
18	0	0	32	25
18	2	0	31	30
19	5	0	16	15
20	0	0	31	28
20	4	0	17	17
21	3	0	36	36
22	0	0	48	40
22	3	0	18	11
22	4	0	22	11
22	7	0	17	3*
23	1	0	57	53
23	3	0	29	28
23	5	0	21	20
24	0	0	59	57
24	1	0	21	17
24	4	0	20	11
24	7	0	23	2*
25	3	0	21	20
25	4	0	25	12
26	0	0	34	31
26	2	0	19	22
28	0	0	23	23
30	7	0	13	6
31	1	0	15	16
32	0	0	13	11
0	2	1	108	121
0	3	1	86	80
0	4	1	28	22
0	5	1	14	16
0	6	1	22	23
0	9	1	30	30
0	13	1	11	10
1	2	1	21	19
1	3	1	35	34
1	4	1	26	31
1	5	1	23	21
1	6	1	48	52
1	7	1	12	14
1	11	1	14	17
2	1	1	45	38
2	2	1	93	105
2	3	1	20	21
2	4	1	35	36
2	5	1	17	16
2	8	1	19	20
3	0	1	34	25
3	1	1	66	63
3	2	1	160	177
3	3	1	9	7
3	4	1	59	58
3	5	1	10	9
3	6	1	48	49
3	7	1	9	10
3	8	1	33	35
3	10	1	23	24
3	12	1	14	17
4	0	1	19	25
4	1	1	24	23
4	2	1	51	47
4	3	1	27	24
4	4	1	11	16
4	5	1	20	15
4	6	1	39	41
4	7	1	12	12
4	8	1	10	13
5	0	1	70	66
5	1	1	21	25
5	2	1	63	66
5	3	1	71	65
5	4	1	27	24
5	5	1	19	17
5	6	1	35	39
5	8	1	23	23
5	10	1	17	21
6	0	1	33	30
6	2	1	49	46
6	3	1	44	45
6	4	1	30	33
6	5	1	29	27
6	6	1	24	25
6	7	1	25	25
6	8	1	10	10
6	9	1	17	17
6	1	1	71	64
7	0	1	41	35
7	1	1	71	72
7	2	1	21	18
7	3	1	21	23
7	4	1	23	23
7	5	1	30	28
7	6	1	16	12
7	7	1	13	15
7	8	1	10	10
8	0	1	32	31
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9	1	1	42	34
9	2	1	51	49
9	3	1	13	12
9	4	1	35	30
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9	6	1	8	8
9	7	1	13	14
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9	10	1	14	17
10	0	1	14	10
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11	0	1	74	69
11	1	1	43	35
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11	4	1	55	53
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11	6	1	50	50
11	7	1	19	20
11	8	1	17	18
11	9	1	20	22
11	10	1	14	14
12	1	1	60	58
12	2	1	46	39
12	3	1	36	31
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13	1	1	24	20
13	3	1	62	61
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14	7	1	20	17
14	8	1	20	20
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15	0	1	25	18
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15	2	1	16	14
15	3	1	12	9
15	4	1	17	14
15	6	1	17	19
15	7	1	14	12
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16	0	1	21	20
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17	0	1	27	20
17	1	1	21	18
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19	9	1	9	7
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20	9	1	21	19
21	1	1	36	31
21	2	1	10	7
21	[illegible]	[illegible]	[illegible]	14
21	7	1	14	10
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22	4	1	17	20
22	7	1	14	11
23	0	1	22	19
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23	2	1	29	26
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25	5	1	14	10
26	0	1	10	4
26	1	1	30	25
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26	3	1	28	23
26	5	1	13	9
27	0	1	14	11
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27	5	1	13	5
28	3	1	16	14
28	5	1	12	7
29	2	1	18	14
29	4	1	17	15
29	5	1	11	10
30	0	1	18	15
31	0	1	12	10
31	2	1	12	12
0	2	2	81	77
0	3	2	26	33
0	4	2	56	52
0	6	2	17	18
0	8	2	29	35
0	10	2	29	30
0	11	2	13	14
0	12	2	10	13
0	13	2	6	6
1	0	2	60	55
1	1	2	44	49
1	2	2	89	82
1	3	2	64	64
1	4	2	61	66
1	5	2	24	16
1	6	2	31	26
1	7	2	11	10
1	8	2	26	32
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1	10	2	12	17
1	11	2	9	9
1	12	2	9	12
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2	1	2	32	38
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2	3	2	13	14
2	4	2	27	26
2	5	2	26	28
2	6	2	17	16
2	8	2	8	11
2	9	2	17	20
2	11	2	11	14
3	0	2	39	46
3	2	2	25	22
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3	4	2	35	37
3	5	2	12	10
3	7	2	19	18
3	8	2	10	9
4	0	2	10	9
4	1	2	10	11
4	2	2	34	33
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4	4	2	7	9
4	7	2	12	15
4	8	2	8	6
4	9	2	8	10
5	0	2	71	68
5	1	2	28	27
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5	3	2	42	39
5	4	2	13	14
5	5	2	15	14
5	6	2	36	36
5	7	2	21	20
5	8	2	8	7
5	10	2	14	17
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6	2	2	11	3
6	3	2	13	13
6	4	2	39	35
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6	8	2	8	6
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7	2	2	11	9
7	3	2	16	16
7	4	2	57	56
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7	11	2	9	7
8	0	2	16	12
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8	2	2	34	30
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8	7	2	8	11
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9	0	2	33	31
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9	2	2	11	9
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12	7	2	21	21
12	9	2	14	15
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13	0	2	12	0
13	1	2	25	20
13	2	2	35	34
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21	6	2	8	6
21	7	2	11	10
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22	5	2	18	16
22	6	2	6	5
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23	1	2	23	20
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23	6	2	17	12
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30	2	2	5	7
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31	2	2	11	10
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33	0	2	9	7
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0	5	3	5	1
0	6	3	11	8
0	7	3	12	13
0	8	3	14	16
1	0	3	33	33
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1	2	3	11	7
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1	4	3	44	43
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1	6	3	5	4
1	7	3	8	10
1	8	3	11	12
1	9	3	12	11
1	10	3	12	10
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2	3	3	37	34
2	4	3	41	41
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2	10	3	12	15
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6	6	3	19	16
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6	8	3	6	8
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6	10	3	11	14
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7	1	3	37	34
7	2	3	9	9
7	3	3	32	28
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9	2	3	27	26
9	3	3	17	17
9	4	3	20	20
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9	6	3	20	15
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10	8	3	16	18
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11	5	3	23	21
11	7	3	6	5
11	8	3	15	16
11	9	3	17	17
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12	2	3	23	24
12	3	3	26	26
12	4	3	19	16
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12	8	3	6	6
12	9	3	8	10
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15	2	3	26	23
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15	4	3	13	14
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15	8	3	8	6
15	9	3	9	7
15	10	3	6	0
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16	2	3	6	6
16	4	3	18	19
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16	6	3	9	5
16	7	3	12	11
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18	2	3	8	6
18	3	3	12	13
18	4	3	15	14
18	5	3	11	10
18	7	3	13	13
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12	4	4	19	20
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3	0	5	26	23
4	0	5	21	16
6	0	5	26	22
7	0	5	25	26
10	0	5	28	26

* The following errors in indexing were discovered after completion of the study. Reflection 22.7.0 (F_o=17) is actually 21.7.0 (F_c=19), and reflection 24.7.0 (F_o=23) is actually 23.7.0 (F_c=24).

sibility of improving convergence by extending the list of observations by use of exact or statistical relations to unobserved reflections below the observational limit or beyond the sphere of reflection is also being studied in connection with these sharpening problems.

The 8 Cl atoms were readily located as one of two possible sets by choosing the set which gave the lower value of

$$R=\Sigma||F_o|-|F_c||/\Sigma|F_o|=0{\cdot}50\;.$$

These Cl atoms formed an array of symmetry *Pcmn*, which is centrosymmetric, and hence a false mirror plane is present perpendicular to *b*. However, the known optical activity of the molecule clearly implied removal of this mirror plane by the organic residue. A second difficulty is that the Cl atoms constitute only 19% of the scattering power of the crystal and hence even the phase information supplied to the real part of the structure factor is unreliable. Nevertheless, a three-dimensional electron density map, somewhat sharpened, was computed from the Cl phases (signs). On a relative scale the Cl peaks at 900 units occurred along with 120 peaks over 50 units and over 200 peaks under 50 units in the asymmetric unit. The problem was to sort out the peaks corresponding to the presence of 20 correct C, N and O atoms and the enantiomorph which arises because of the symmetry of the Cl arrangement. Many trial structures which incorporated a five or six membered sugar ring and N-methylhistamine residues were tested, but none refined below $R=0{\cdot}32$. At this stage detailed comparisons were made with the centrosymmetric *c*-axis (5·76 Å) projection for which signs from Cl leave an ambiguity of sign of *y* coordinates, and the *b* axis (10·76 Å) projection which has large overlap but no ambiguity. The first clue was the $CH_2CH_2NH_2^+CH_3$ group, anticipated to be near the two Cl atoms at $y=0$ and $y=\frac{1}{2}$ for electrostatic and hydrogen bond reasons. Then the rough location of the histamine ring followed, and finally the six membered ring of the sugar was found after many unsuccessful attempts to locate a five membered sugar ring. The OH and CH_2OH bonded to the sugar were then readily located, without chemical assumptions as to the nature of the sugar, in the *hk*0 projection with aid from the *h*0*l* projection and especially from the three-dimensional Cl-phased Fourier map. Neither the histamine ring nor the $NH_2^+CH_3$ region of its attached group had been correctly placed, but a further series of refinements of the *hk*0 data with the aid of model building reduced the value of *R* to 0·12 for these *hk*0 data. The *z* coordinates were then located, first for the O atoms of the sugar residue, by assumption of reasonable hydrogen bonding and van der Waals contacts in the model, and by checking against the peaks of the three-dimensional Cl-phased Fourier map. Eventually all C, N and O atoms were found after alternate three-dimensional least-squares refinement and Fourier maps at every stage of which atoms with higher temperature factors or lower than expected electron density were eliminated or moved, and consistency of the partial structure with the Cl-phased Fourier map and chemical reasonableness of stereochemistry were used as important criteria. In the final stage, the end $CH_2NH_2^+CH_3$ group had to be located from a Fourier series in which all other atoms except H were correctly included. We were greatly surprised that this group

Table 2. *Values of* $R=\Sigma||F_o|-|F_c||/\Sigma|F_o|$

Class	R	Range of $\sin\theta$	R
All *hkl*	0·116	0·00 to 0·40	0·09
h odd	0·11	0·40 to 0·50	0·10
k odd	0·11	0·50 to 0·60	0·09
l odd	0·11	0·60 to 0·65	0·11
$h+k$ odd	0·11	0·65 to 0·70	0·12
$h+l$ odd	0·11	0·70 to 0·75	0·12
$k+l$ odd	0·11	0·75 to 0·80	0·17
$h+k+l$ odd	0·11	0·80 to 0·85	0·23
		0·85 to 0·90	0·16
		0·90 to 1·00	0·19

Table 3. *Atomic parameters*

Atom	10^4x	10^4y	10^4z	$10^4\Delta x$	$10^4\Delta y$	$10^4\Delta z$	B
1	1610	−0102	8169	2	4	9	4·0
2	1867	5036	−1865	1	4	8	3·4
3	2908	2602	−1580	8	20	38	4·5
4	2018	2458	3568	7	17	32	3·4
5	2366	1994	1808	7	17	39	4·0
6	1548	2960	2652	6	15	28	2·9
7	1253	3900	3220	5	14	31	2·6
8	0870	2845	0405	6	14	29	2·8
9	0469	4774	1684	5	12	27	2·1
10	−0822	3925	1717	6	15	32	3·1
11	−0409	4781	1557	6	14	29	2·7
12	−0401	5597	−0732	5	13	26	2·1
13	0079	6373	−0878	6	15	28	2·7
14	0512	5519	−0475	5	14	29	2·4
15	0838	3873	1846	5	12	25	2·8
16	1287	2283	0998	5	12	22	2·7
17	2544	3035	0251	5	13	25	3·5
18	0035	4062	1693	4	9	20	3·0
19	−0869	3160	−0262	4	10	18	2·5
20	−0784	6389	−0775	5	12	23	3·6
21	0068	6863	−3244	5	11	24	4·0
22	0916	6333	−0411	5	13	25	4·0

was not in the expected extended form, the assumption of which gave the first correct start on the trial structure. After inclusion of this group, and use of proper form factors (Berghuis *et al.*, 1955) for all C and O atoms and for N of NH$_2^+$, but use of form factors for C for all five atoms of the histamine ring, we found $R=0{\cdot}18$. A considerable decrease of temperature factor constants for two atoms of the histamine ring during least-squares refinement, and the presence of excess electron density at these two atoms in a three-dimensional difference electron density map, led to their identification as N atoms without chemical assumption. In addition, reasonable, but not reliable, positions were found for all 23 H atoms, which were then included as fixed atoms in the final leasts-quares refinements. The final value of $R=0{\cdot}12$ was obtained for all observed reflections, whether or not (111) is included, for which the observed value of 70 compares poorly with the calculated value of 127, and which was omitted in the final refinement.

Observed and calculated structure factors are in Table 1, values of R for different classes are in Table 2, and final position and thermal parameters and their standard deviations are in Table 3. The final results and rate of convergence were found to be sensitive to the weighting scheme, for which we finally chose constant weights for $|F|^2 \leq 50$, and weights inversely proportional to $|F|^2$ for $|F|^2 \geq 50$. Individual thermal parameters were varied isotropically only, since the difference maps showed no evidence of anisotropic thermal motion, and least-squares refinement was stopped when the largest parameter shift was 0·009 Å.

Discussion of the structure

This study establishes the point of attachment of the sugar to the histamine ring (Figs. 1 and 2), that the sugar is a six-membered ring rather than a five-membered ring, that the stereochemistry of attachment of the sugar is β rather than α, that the N to which the sugar is attached forms coplanar bonds, that the other two N atoms of N-methylhistamine are protonated and H bonded to Cl, and that the CH$_2$CH$_2$NH$_2^+$CH$_3$ group is not in the extended form even though the single bonds are all in the staggered configurations. In addition to these conclusions, none of which have been reached from the chemical studies, we have proved independently without chemical assumptions that the sugar residue is glucose, but have not established its absolute configuration, now known to be D, as is reasonable (Raman, Reddy, Lipscomb, Kapoor & Djerassi, 1962). These conclusions complete the main purpose of the investigation.

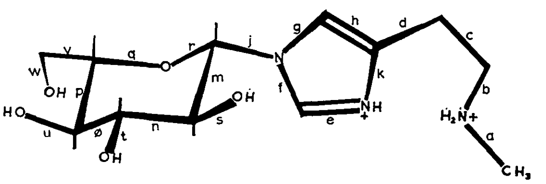

Fig. 1. The dipositive casimidine ion, showing the stereochemistry of the β-glucose residue and point of attachment to the histamine ring.

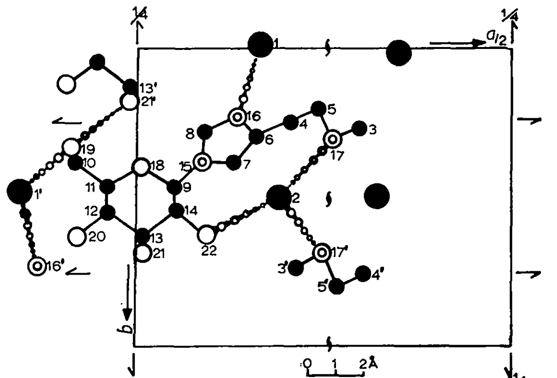

Fig. 2. The relation of a casimidine molecule to Cl ions and to fragments of other, symmetry-related molecules. Hydrogen bonds are shown as chains, and the symmetry elements of the space group $P2_12_12_1$ are also indicated.

Table 4. *Intramolecular bonds*

Bond lengths		Bond angles	
a	1·53 ± 0·03 Å	*ab*	113 ± 1°
b	1·52 ± 0·02	*bc*	112 ± 1
c	1·49 ± 0·03	*cd*	116 ± 2
d	1·51 ± 0·02	*dh*	136 ± 2
e	1·35 ± 0·02	*dk*	120 ± 1
f	1·39 ± 0·02	*hk*	104 ± 1
g	1·40 ± 0·02	*gh*	111 ± 1
h	1·34 ± 0·02	*gf*	108 ± 1
j	1·41 ± 0·02	*fe*	105 ± 1
k	1·40 ± 0·02	*ek*	113 ± 1
m	1·48 ± 0·02	*fj*	124 ± 1
n	1·53 ± 0·02	*gj*	128 ± 1
φ	1·58 ± 0·02	*jm*	111 ± 1
p	1·58 ± 0·02	*jr*	104 ± 1
q	1·46 ± 0·02	*ms*	112 ± 1
r	1·43 ± 0·02	*sr*	105 ± 1
s	1·43 ± 0·02	*nm*	113 ± 1
t	1·46 ± 0·02	*nt*	112 ± 1
u	1·37 ± 0·02	*tφ*	103 ± 1
v	1·48 ± 0·02	*φn*	110 ± 1
w	1·41 ± 0·02	*φu*	109 ± 1
		up	111 ± 1
		pφ	110 ± 1
		pv	114 ± 1
		vq	109 ± 1
		pq	109 ± 1
		qr	115 ± 1
		mr	111 ± 1
		vw	113 ± 1

The bond distances and angles are not highly precisely determined. They are given in Tables 4 and 5 along with standard deviations computed only from the variance elements of the variance-covariance matrix. These deviations are consistent with average deviations based upon chemical assump-

Table 5. *Intermolecular hydrogen bonding*

	Bond lengths		Bond angles	
1.	2,17	3·11 ± 0·02 Å	3,17,2	110 ± 1°
2.	2,17′	3·12 ± 0·02	5,17,2	123 ± 1
3.	2,22	3·11 ± 0·01	3′,17′,2	103 ± 1
4.	1′,16′	3·17 ± 0·01	5′,17′,2	110 ± 1
5.	1′,19	3·25 ± 0·01	6,16,1	129 ± 1
6.	19,21′	2·77 ± 0·02	8,16,1	118 ± 1
7*a*.	1′,20	3·12 ± 0·01	14,22,2	113 ± 1
7*b*.	16′,20	3·23 ± 0·02	22,2,17′	107 ± 1
			17,2,17′	111 ± 1
			22,2,17	136 ± 1
			13′,21′,19	117 ± 1
			10,19,21′	118 ± 1
			1′,19,21′	130 ± 1
			10,19,1′	98 ± 1
			12,20,1′	105 ± 1
			12,20,16′	119 ± 1
			1′,16′,20	109 ± 1
			16′,1′,19	123 ± 1
			20,1′,19	67 ± 0
			16′,1′,20	67 ± 0

tions such as all C–O distances are the same for this molecule, and hence are not expected to be more than roughly 30% low because of neglect of covariance terms in a structure of this complexity. For example, the six C–O distances average to 1·43 ± 0·02 Å, the seven C–C bonds to 1·52 + 0·04 Å, the C=C is 1·34, the two partly conjugated CN bonds of the histamine ring are 1·35 and 1·39 Å in length and the other C–N distances average to 1·45 ± 0·06 Å, but really are two separate sets with two C–NH_2^+ at 1·52 and 1·53 (see Donohue, Lavine & Rollett, 1956) and three C–N at 1·40, 1·40 and 1·41 Å. If we accept two or three times the standard deviation of the X-ray results as a reasonable level of significance, differences within the above sets are of doubtful significance, but the above classifications of distances seem to be reasonable, and consistent with known or expected distances from other related molecules. We do not then feel that the abnormally short C_4–O_4 bond at 1·37 Å is a significant deviation from the mean of 1·34 Å, any more than the same types of deviations that were observed in cellobiose (Jacobson, Wunderlich & Lipscomb, 1961), in α-glucose (McDonald & Beevers, 1957), or not found in the preliminary study of β-glucose (Ferrier, 1960). The average C–O distance compares favorably with the value of 1·43 summarized for ethers and alcohols (Allen & Sutton, 1950).

The most striking bond angle situation is that of 136° between bonds *h* and *d* as compared with the 120° bond angle between bonds *d* and *k*. One might have expected that the 104° bond angle between bonds *h* and *k*, which is only slightly less than the 108° average for the ring would distort the C=C 'ethylene-like' residue in the ring in such a way as to distribute the 360 − 104° = 256° more nearly equally so that these bond angles external to the ring would each be about 128°. Even allowing for the new value of 117° for HCH in C_2H_4 (Dowling & Stoicheff, 1959), these external angles would be perhaps close to 130° (*h*, *d*) and 126° (*d*, *k*). Values of 132° and 122°, respectively, have been found for the histamine-like ring in histidine hydrochloride monohydrate (Donohue, Lavine & Rollett, 1956), a study which is more precise than our own. No such difference of bond angle occurs external to the histamine ring at the N atom attached to the sugar residue.

Seven H atoms are available for hydrogen bonding, four from OH groups, one from NH^+ of the ring and two from the NH_2^+ group attached to CH_3. These bonds are shown in Fig. 2, and their lengths and angles are shown in Table 5. The Cl^-(2) ion receives a hydrogen bond from NH_2^+(17) at 3·11 Å, one from NH_2^+(17′) at 3·12 Å and a third from OH (22) at 3·11 Å, for which the C–NH · · · Cl^- or C–OH · · · Cl^- angles are nearly tetrahedral, as are the angles about Cl^-. The Cl^-(1′) ion receives one hydrogen bond of length 3·25 from OH (19) and one of 3·23 Å from NH^+ of the ring, again at nearly tetrahedral angles. The sixth hydrogen bond is O–H · · · O of 2·77 Å length between atoms 21 and 19′, with C–OH · · · O essentially tetrahedral, but with the angle at oxygen 19 at 130°. The seventh hydrogen bond, formed by OH (20), is nearly tetrahedral at the donor end if it goes either to Cl (1′), for which the length is 3·12 Å but there are two acute angles of about 67° at Cl (1′), or to N (16′), for which the bond angles are reasonable but the length is a bit long, at 3·23 Å. When one finds a situation of this kind which is not completely understandable one usually says that the bond is bifurcated.

No hydrogen bonds are donated to the N atom (15) glycosidically linked to the sugar residue, or to the O atom (18) of the sugar ring. Moreover, the relevant van der Waals contacts are satisfactory in that no C, N or O contacts with one another are shorter than 3·0 Å, and no contacts between these atoms and Cl^- are under 3·5 Å, unless a hydrogen bond is present.

Note added June 4, 1962.—The following remarks on points raised by the Editor and Referee are of additional interest. The small size of the crystal, its needle-like character and the short *c* axis led only to fairly satisfactory Weissenberg photographs with use of Cu *Kα* radiation, and only to relatively faintly exposed precession photographs of the *h*0*l* and *h*1*l* levels. Of the 00*l* relections only 002 was observed ($F_o \sim 50$, $F_c = 45$), but it was given zero weight along with 011 which also was rather unreliably estimated ($F_o \sim 40$ to 60, $F_c = 50$). Some 50 additional reflections for which $F_c \geq 10$ were reliably indexed after the study was completed, but two further cycles of three-dimensional least squares refinement with inclusion of all off-diagonal terms led to $R = 0{\cdot}12$, and, in summary, to six C–O at 1·45 ± 0·03 Å, seven C–C at 1·50 ± 0·05 Å, C=C at 1·42 Å, the two CN at 1·36 Å, the two C–NH_2 at 1·49 Å and 1·54, the three C–N at 1·36, 1·36 and 1·41 Å, and to bond angles different by only 1·4° on the average from those reported in Table 4. However, these additional intensities were not as reliable as those in Table 1, and hence the detailed results have not been revised. Finally, values of F_c for the 548 reflections which are 'unobserved' but which are in the range covered by the photographs were all less (usually much less) than twice the observational limit, but these absent reflections were not included in the least squares refinements.

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The Crystal Structure of Beta Plutonium Metal*

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β–Pu is monoclinic body-centered with 34 atoms per unit cell. At 190 °C

$$a=9{\cdot}284,\ b=10{\cdot}463,\ c=7{\cdot}859\ \text{Å},\ \beta=92{\cdot}13^\circ$$

and the density is 17·70 g.cm.$^{-3}$.

The space group is $I2/m$. One set of two plutonium atoms are in centers of symmetry. Three sets of four plutonium atoms are in reflection planes, one set of four on twofold axes and two sets of eight in general positions. The thirteen parameters in the structure have been determined to an accuracy of about 0·04 Å.

The seven different kinds of plutonium atoms in the structure have coordination number of twelve, thirteen or fourteen. The mean atomic radius is 1·60 Å for coordination number twelve.

Introduction

Plutonium metal can be prepared in six allotropic forms. The various transition temperatures on heating have been reported as follows (Jette, 1955)

Transformation	Temperature	Transformation	Temperature
$\alpha \to \beta$	122 ± 2 °C.	$\delta \to \delta'$	451 ± 4 °C.
$\beta \to \gamma$	206 ± 3	$\delta' \to \varepsilon$	476 ± 5
$\gamma \to \delta$	319 ± 5	$\varepsilon \to$ liq.	640 ± 2

Complete crystal structures are known for all but the β-form. α-Plutonium is monoclinic with 16 atoms to the unit cell, eight different kinds of atoms and 16 degrees of freedom (Zachariasen & Ellinger, 1957). The γ-phase is orthorhombic face-centered with eight equivalent atoms per unit cell and there are no position parameters (Zachariasen & Ellinger, 1955). The three high temperature forms have simple structures: δ-plutonium is cubic face-centered, ε-plutonium cubic body-centered, while δ′-plutonium is tetragonal body-centered (Ellinger, 1956).

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