Apart from the hydrogen bonds, many close van der Waals contacts are found among the molecules within the double layer. The packing of the double layers, on the other hand, is surprisingly loose. The molecules of the neighbouring double layers are separated by a large distance. Only the shortest contacts between the double layers occur for Br···C(29) (3·92 Å) and C(30)···C(19) (3·92 Å), and large columnar holes are left between the double layers. The holes are filled by the molecules of benzene which plays a role as solvent of crystallization.

The authors are grateful to Professor S. Shibata of this Faculty for his encouragement throughout this work and to Dr A. Yoshino for her assistance at the early stage of the X-ray analysis. Thanks are due to the staff of C. Itoh Electronic Computing Service Co. Ltd. for making the CDC 3600 computer available to us. This work was supported by grants from the Hohansha and Takeda Science Foundation.

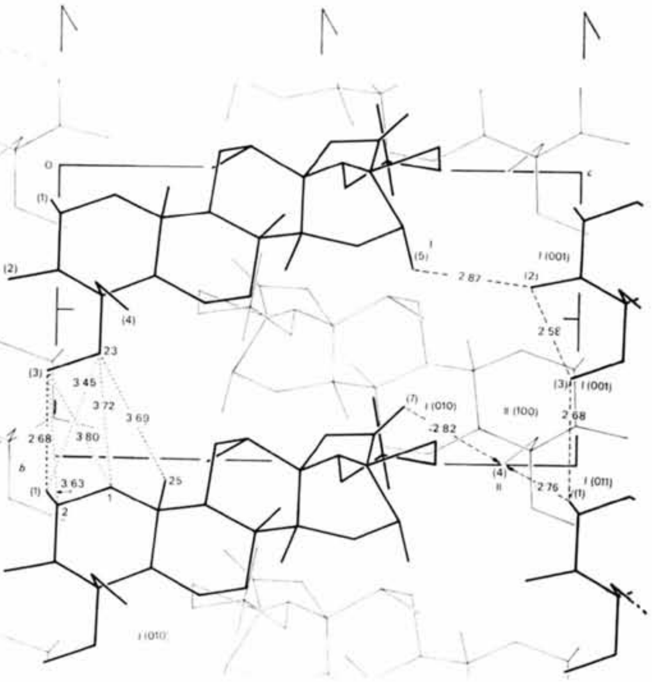


Fig.6. The crystal structure projected along the *a* axis. To represent the correct absolute configuration the positive direction of the *a* axis should be downwards away from the observer. Benzene molecules are omitted for the sake of simplicity. Hydrogen bonds are shown by broken lines.

References

AKIYAMA, T., IITAKA, Y. & TANAKA, O. (1968). *Tetrahedron Letters*, 5577.

AKIYAMA, T., TANAKA, O. & SHIBATA, S. (1968). *Chem. Pharm. Bull.* (*Tokyo*), **16**, 2300.

BERGHUIS, J., HAANAPPEL, IJ. M., POTTERS, M., LOOPSTRA, B. O., MACGILLAVRY, C. H. & VEENENDAAL, A. L. (1955). *Acta Cryst.* **8**, 478.

BIJVOET, J. M., PEERDEMAN, A. F. & VAN BOMMEL, A. J. (1951). *Nature, Lond.* **168**, 271.

BJÅMER, K., FERGUSON, G. & MELVILLE, R. D. (1968). *Acta Cryst.* B**24**, 855.

COOPER, A., GOPALAKRISHNA, E. M. & NORTON, D. A. (1968). *Acta Cryst.* B**24**, 935.

DAUBEN, C. H. & TEMPLETON, D. H. (1955). *Acta Cryst.* **8**, 841.

HALL, S. R. & MASLEN, E. N. (1965). *Acta Cryst.* **18**, 265.

KUBOTA, T. & KITATANI, H. (1968). *Chem. Comm.* p. 1005.

OKAYA, T. & ASHIDA, T. (1967). *HBLS* IV, *The Universal Crystallographic Computing System* (I), p. 65. Japanese Crystallographic Association.

RONDEST, J. & POLONSKY, J. (1963). *Bull. Soc. Chim. France*, p. 1253.

THOMAS, L. H. & UMEDA, K. (1957). *J. Chem. Phys.* **26**, 293.

TSUJIMOTO, M. (1940). *Nippon Nogeikagaku Kaishi*, **16**, 613.

Acta Cryst. (1970). B**26**, 171

The Crystal and Molecular Structure of Verticinone Methyl Bromide*

BY FRANÇOIS BRISSE†

Division of Biochemistry and Molecular Biology, National Research Council of Canada, Ottawa 7, Canada

(*Received* 20 *December* 1968)

The structure of verticinone methyl bromide, $C_{27}H_{43}O_3N.CH_3Br$, was solved by the heavy-atom method. Anisotropic least-squares refinement proceeded to a final R value of 0·091 for 2450 observed reflexions. The crystals are monoclinic, space group $P2_1$, with two molecules in a unit cell of dimensions: $a = 12{\cdot}548$, $b = 12{\cdot}046$, $c = 9{\cdot}161$ Å, $\beta = 92{\cdot}07°$. The structure found for verticinone methyl bromide differs from that proposed by Itô for verticinone in that two six-membered rings are *cis*-fused and not *trans*. A configurational inversion at the nitrogen atom probably takes place on formation of the methyl bromide derivative.

Introduction

Verticine is the main alkaloid of *Fritillaria verticillata* var. *Thumbergii*. As a result of chemical degradation studies Itô, Kato, Shibata & Nozoe (1961, 1963) proposed the structure shown in Fig. 1(*a*). The stereochemistry at positions C(8), C(9), C(12), C(14), C(16) and C(17) was not unequivocally established but was derived by analogy with the steroid series and from biogenetic considerations.

---

* Issued as NRCC No. 11159.

† National Research Council of Canada Postdoctorate Fellow, 1967–1969. Present address: Département de chimie, Université de Montréal, Canada.

In order to confirm the structure Dr Shô Itô suggested that the current investigation be carried out and provided crystals of a heavy atom derivative, verticinone methyl bromide. Its schematic formula, as solved by X-rays, is given in Fig. 1(*b*). The numbering of the atoms adopted is that of the framework of cevane, which was assumed to be present in verticine.

Experimental

The crystals of verticinone methyl bromide were colourless, tabular rhombs. Under polarized light the longest diagonal of the rhomb is parallel to the extinction direction. The crystals were monoclinic, systematic absences: $0k0$ reflexions with k odd, whence the space group is $P2_1$. The $hk0$ and $h0l$ precession photographs were calibrated by concurrent exposure to the $hk0$ zone of protopine, whose cell dimensions are accurately known (Hall & Ahmed, 1968). The wavelength of the Cu radiation was chosen as $\lambda K\bar{\alpha} = 1{\cdot}54178$ Å.

Crystal data

$C_{27}H_{43}O_3N.CH_3Br$, M.W. 526·6

$a = 12{\cdot}548 \pm 0{\cdot}016$, $b = 12{\cdot}046 \pm 0{\cdot}015$, $c = 9{\cdot}161 \pm 0{\cdot}007$ Å, $\beta = 92{\cdot}07 \pm 0{\cdot}04°$, $V = 1383{\cdot}8$ Å^3, $D_m = 1{\cdot}28$ g.cm^{-3} (measured by flotation), $D_x = 1{\cdot}263$ g.cm^{-3}, $Z = 2$, space group $P2_1$ (C_2^2, No. 4),

$\mu = 24{\cdot}82$ cm^{-1}, $F(000) = 560$.

The crystal used in this investigation was ground into a cylinder terminated by a hemisphere (diameter 0·22, length 0·30 mm). Three-dimensional intensity data were visually estimated from equi-inclination Weissenberg photographs with the multiple-film technique. Layers from $k = 0$ to 9 and $h = 0$ to 4 were obtained and the intensity data were scaled by the method of Hamilton, Rollett & Sparks (1965). A total of 2450 independent reflexions were observed out of 2790 accessible reflexions within the Cu sphere. The unobserved reflexions were given an intensity equal to $\frac{2}{3}$ of the minimum observable intensity. Since μR was small (0·27), the absorption correction factor was almost constant with θ and was included in the scale factor. The correction for secondary extinction was not introduced until the structure had been solved. The X-ray scattering factors were obtained from Cromer & Waber (1965) for C, N, O, Br$^-$ and from Stewart, Davidson & Simpson (1965) for H. The real part of the dispersion correction, included in the scattering factor of Br$^-$, was taken from Cromer (1965).

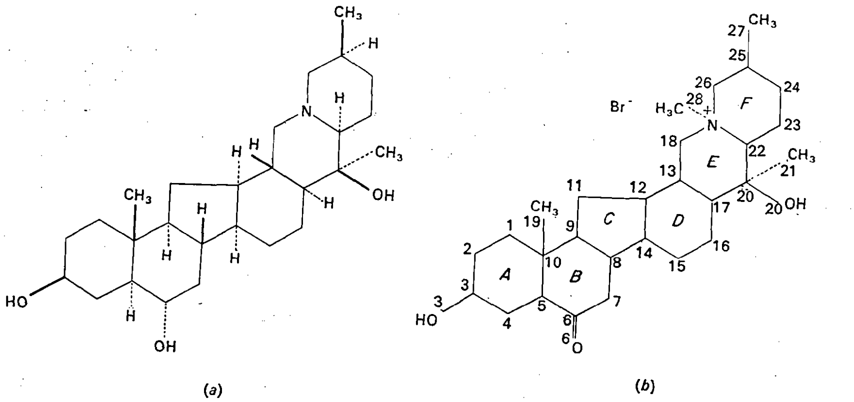
Structure determination

A three-dimensional Patterson synthesis was computed and the coordinates of the Br atom were easily deduced from the Harker section. The set of structure factors calculated using the coordinates of the Br alone ($x = 0{\cdot}159$; $y = 0{\cdot}250$; $z = 0{\cdot}120$) had a reliability index R, defined as $\Sigma||F_o| - |F_c||/\Sigma|F_o|$, of 0·40.

The Br atom at $y = \frac{1}{4}$ gave false symmetry $P2_1/m$ on the first electron density map but this symmetry was destroyed by the identification of seven carbon atoms. All the non-hydrogen atoms were located after three successive structure factor and Fourier synthesis calculations. The reliability index was then 0·29.

The refinement was carried out using Ahmed's (Ahmed, Hall, Pippy & Huber, 1966) block-diagonal least-squares programs for the IBM/360 system. The coefficients $P1$ and $P2$ of the weighting function $1/w = 1 + [(|F_o| - P2)/P1]^2$ were taken as $30e$ and $15e$ respectively, so that $(\Sigma w\Delta^2)/N$ remained constant with $|F_o|$. After eight cycles of refinement with isotropic temperature factors for all atoms, R was brought to 0·16.

A number of the strongest reflexions seemed to suffer from secondary extinction; the plots of the ratio I_c/I_o *vs.* I_c for the two sets of intensity data, taken with the crystal rotating about the b and c axes, appeared nearly linear, thus indicating that this was indeed the case. The extinction corrections, derived by a least-squares fit to a straight line of I_c/I_o *vs.* I_c, were applied to each set of the original data. The different layers



(*a*) (*b*)

Fig. 1. (*a*) Proposed structure for verticine. (*b*) Numbering of the atoms for verticinone methyl bromide.

were then rescaled as above. The corrected $|F_o|$ values, new weighting parameters ($P1=13{\cdot}5e$ and $P2=10e$), and anisotropic temperature factors were used in six additional refinement cycles. The R value came down to 0·11. Eighteen peaks, with heights varying between 0·3 and 0·7 e.Å^{-3} were recognized as hydrogen atoms from a difference Fourier synthesis computed at this stage. The coordinates of twenty-two more hydrogen atoms could be calculated from the C positions, assuming a C–H distance of 1·00 Å and tetrahedral angles, and were also included in the next structure factor calculation. An additional difference electron density map showed the remaining six hydrogen atoms. While the estimated standard deviation of the residual electron density was $\pm 0{\cdot}16$ e.Å^{-3}, there remained around the bromine atom some diffraction ripples as high as 1e.Å^{-3}. The hydrogen atoms were included in the final structure factor calculations but their coordinates were not refined. All H atoms were given an isotropic temperature factor of 4·5 Å^2. The refinement was ended after two more cycles when the average shifts in the coordinates were less than $0{\cdot}3\sigma$. The final R value was 0·091. A summary of the agreement between observed and calculated structure amplitudes is given in Table 1, the complete list is shown in Table 2. The atomic coordinates, the anisotropic vibrational parameters and their standard deviations are listed in Table 3. The vibrational parameters are the coefficients U_{ij} for the expression $T=\exp[-2\pi^2(U_{11}a^{*2}h^2\ldots+2U_{23}b^*c^*kl\ldots)]$. The coordinates of the hydrogen atoms used in the structure factor calculations are given in Table 4.

Table 1. *Agreement summary between the observed and calculated structure factors at the end of the refinement*

$\lvert F_o\rvert$ ranges	N	$\Sigma\lvert F_o\rvert$	$\Sigma\lvert F_c\rvert$	$\Sigma\lvert\Delta\rvert$	$(\Sigma w\Delta^2)/N$	R
0–10	778	5612	5417	801	1·66	14·3%
10–20	984	14158	13686	1321	2·42	9·3
20–30	392	9528	9341	706	2·41	7·4
30–40	185	6370	6519	371	1·52	5·8
40–50	57	2508	2693	212	2·56	8·5
50–70	38	2202	2442	247	4·19	11·2
70–90	10	773	836	69	2·58	9·0
90–120	6	600	652	51	1·89	8·6

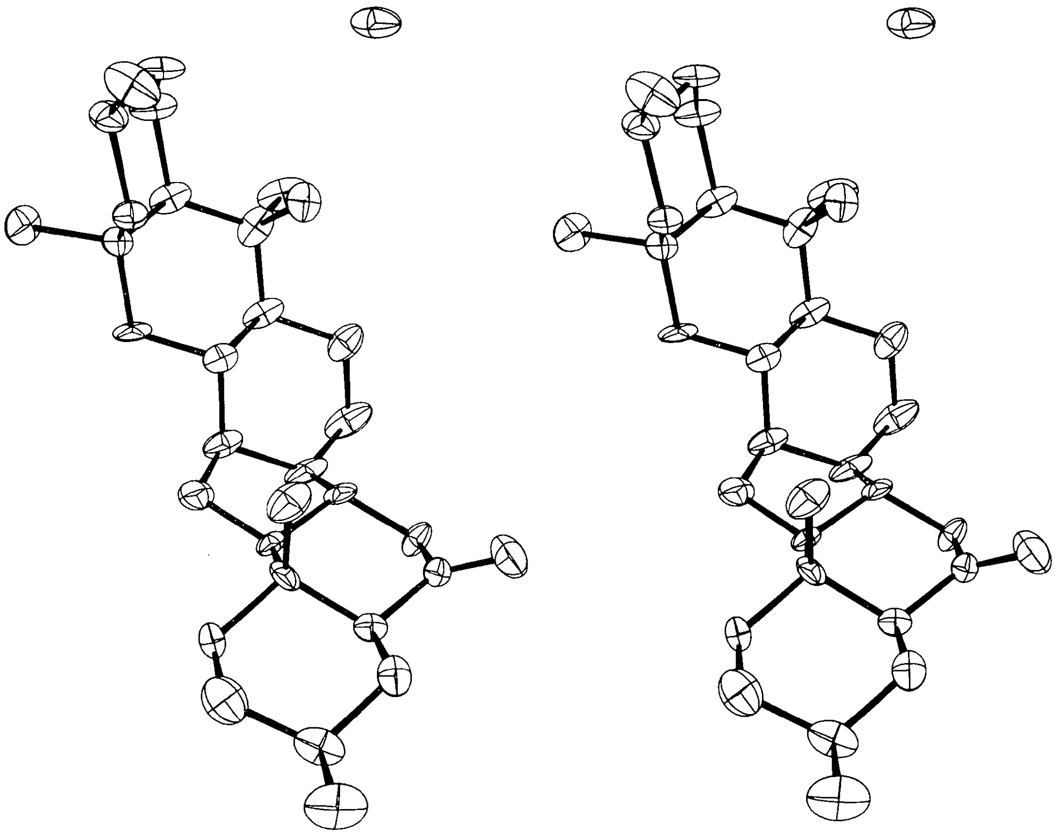


Fig. 2. Stereoscopic pair of drawings showing the structure of verticinone methyl bromide.

Table 2. *Observed and calculated structure amplitudes* (×10)

Unobserved reflexions are indicated by an asterisk.

K	FO	FC
H= 0, L= 0		
2	682	708
4	814	872
6	222	217
8	395	441
10	225	184
12	150	127
14	102	94
H= 1, L= 0		
2	1075	1182
3	898	960
4	437	497
5	164	174
6	211	228
7	304	357
8	232	224
9	321	338
10	218	195
11	142	136
12	154	146
13	146	137
14	96	89
15	53	50
H= 2, L= 0		
0	677	716
1	697	826
2	660	752
3	577	629
4	107	109
5	589	696
6	281	288
7	159	172
8	140	118
9	291	251
10	179	161
11	197	201
12	90	61
13	75	67
14	28*	27
15	44	46
H= 3, L= 0		
0	961	1085
1	269	237
2	613	720
3	224	241
4	279	285
5	272	276
6	214	233
7	209	194
8	354	355
9	58	65
10	216	238
11	131	132
12	144	133
13	69	74
14	113	131
15	66	51
H= 4, L= 0		
0	546	551
1	135	136
2	480	557
3	379	369
4	566	673
5	337	344
6	182	185
7	105	86
8	160	139
9	306	285
10	190	180
11	124	142
12	123	136
13	50	53
14	76	68
H= 5, L= 0		
0	30*	18
1	316	337
2	268	285
3	391	431
4	60	65
5	404	414
6	191	161
7	220	226
8	140	118
9	285	244
H= 6, L= 0		
0	234	211
1	259	232
2	251	261
3	237	231
4	132	140
5	198	171
6	183	196
7	216	196
8	173	149
9	132	109
H= 7, L= 0		
0	255	251
1	221	211
2	305	328
3	172	153
4	171	137
5	162	146
6	81	95
7	199	186
8	315	345
9	101	89
H= 8, L= 0		
0	46	68
1	277	264
2	117	115
3	67	46
4	231	208
5	474	528
6	156	157
7	237	234
8	144	133
9	157	151
H= 9, L= 0		
0	258	253
1	209	199
2	208	204
3	241	224
4	235	232
5	144	128
6	195	172
7	156	145
8	164	160
9	35*	86
H= 10, L= 0		
0	275	282
1	116	89
2	52	47
3	168	138
4	128	120
5	101	89
6	150	171
7	189	174
8	172	158
9	34*	68
H= 11, L= 0		
0	24	27
1	169	153
2	44	41
3	263	255
4	170	168
5	178	175
6	110	123
7	133	132
8	60	41
9	124	125
H= 12, L= 0		
0	116	107
1	145	155
2	62	62
3	105	97
4	150	150
5	188	203
6	50	58
7	20*	21
8	77	61
9	146	156
H= 13, L= 0		
0	90	93
1	93	74
2	75	81
3	35*	40
4	118	116
5	27*	10
6	105	106
7	91	75
8	109	119
H= 14, L= 0		
0	32	38
1	29*	8
2	39	32
3	52	51
4	65	79
5	150	154
6	85	87
7	25	29
H= 15, L= 0		
0	48	63
1	33*	35
2	42	45
3	110	116
4	29*	6
5	66	72
H= 0, L= 1		
0	226	248
1	781	752
2	753	896
3	354	384
4	275	316
5	397	457
6	317	334
7	276	319
8	70	59
9	244	268
10	281	245
11	195	189
12	169	164
13	37*	48
14	95	72
15	80	83
H= 1, L= 1		
0	180	177
1	440	511
2	462	540
3	922	990
4	224	250
5	392	479
6	101	111
7	284	329
8	210	199
9	93	132
10	219	187
11	230	209
12	159	154
13	36	33
14	28*	19
15	105	118
H= -1, L= 1		
0	479	458
1	938	989
2	411	415
3	47	60
4	264	323
5	180	203
6	409	435
7	62	61
8	253	289
9	117	95
H= 2, L= 1		
0	33*	25
1	257	292
2	518	637
3	196	187
4	195	201
5	188	170
6	515	625
7	283	293
8	82	62
9	258	266
10	211	191
11	141	140
12	108	113
13	108	105
14	117	134
15	75	62
H= -2, L= 1		
0	471	479
1	245	277
2	113	100
3	553	631
4	177	169
5	221	231
6	302	294
7	311	348
8	173	176
9	181	215
H= 3, L= 1		
0	168	166
1	218	178
2	424	477
3	500	544
4	395	428
5	395	425
6	418	496
7	325	339
8	104	116
9	151	141
10	254	278
11	103	83
12	60	63
13	66	70
14	108	124
15	50*	56
H= -3, L= 1		
0	560	588
1	167	132
2	430	408
3	381	425
4	320	310
5	318	355
6	354	341
7	411	458
8	273	288
9	137	153
H= 4, L= 1		
0	32*	21
1	404	425
2	283	264
3	559	656
4	432	432
5	536	609
6	82	68
7	418	478
8	306	323
9	101	99
10	72	60
11	124	125
12	49	55
13	96	106
14	65	61
H= -4, L= 1		
0	993	1116
1	286	283
2	457	516
3	148	141
4	468	548
5	86	78
6	269	281
7	169	112
8	258	285
9	204	187
H= 5, L= 1		
0	292	268
1	641	719
2	398	480
3	388	416
4	351	330
5	269	267
6	194	198
7	281	254
8	196	175
9	113	97
H= -5, L= 1		
0	132	134
1	401	442
2	260	255
3	388	388
4	323	328
5	185	211
6	255	207
7	366	353
8	144	132
9	171	172
H= 6, L= 1		
0	235	220
1	94	73
2	353	378
3	104	74
4	297	275
5	128	117
6	235	266
7	137	116
8	251	252
9	134	123
H= -6, L= 1		
0	524	533
1	210	161
2	189	145
3	408	439
4	178	129
5	373	372
6	236	218
7	272	271
8	223	230
9	50	52
H= 7, L= 1		
0	132	133
1	380	381
2	177	147
3	341	319
4	185	142
5	162	153
6	87	79
7	299	288
8	191	189
9	148	150
H= -7, L= 1		
0	200	186
1	276	253
2	624	645
3	171	158
4	298	293
5	191	172
6	257	261
7	247	247
8	181	211
9	141	153
H= 8, L= 1		
0	162	138
1	293	310
2	125	114
3	216	209
4	327	300
5	348	382
6	119	132
7	75	62
8	228	211
9	245	221
H= -8, L= 1		
0	363	380
1	99	71
2	299	329
3	355	378
4	349	354
5	214	191
6	206	190
7	369	410
8	105	88
9	64	54
H= 9, L= 1		
0	154	136
1	62	57
2	281	313
3	302	302
4	269	258
5	196	170
6	134	146
7	110	92
8	225	204
9	164	139
H= -9, L= 1		
0	34*	12
1	220	248
2	200	194
3	307	320
4	318	293
5	378	390
6	113	83
7	197	203
8	122	132
9	140	159
H= 10, L= 1		
0	152	115
1	197	174
2	157	158
3	107	83
4	164	135
5	140	125
6	170	178
7	101	108
8	86	84
9	56	55
H= -10, L= 1		
0	76	50
1	265	270
2	260	315
3	147	101
4	37*	57
5	36*	15
6	104	107
7	180	187
8	109	118
9	90	112
H= 11, L= 1		
0	159	145
1	134	128
2	142	144
3	195	194
4	57*	21
5	160	155
6	121	125
7	108	106
8	33*	52
9	136	127
H= -11, L= 1		
0	116	110
1	215	208
2	226	253
3	113	102
4	69	66
5	48*	17
6	167	161
7	167	178
8	85	88
9	35*	69
H= 12, L= 1		
0	262	278
1	35*	27
2	134	126
3	103	99
4	79	80
5	87	73
6	94	106
7	134	129
8	114	121
9	125	133
H= -12, L= 1		
0	79	70
1	35*	61
2	64	47
3	128	129
4	121	111
5	121	138
6	85	77
7	126	127
8	35*	21
9	89	103
H= 13, L= 1		
0	162	158
1	56	54
2	116	106
3	93	73
4	77	68
5	30*	42
6	133	136
7	55	65
8	49	54
H= -13, L= 1		
0	35*	21
1	102	108
2	77	86
3	96	88
4	102	104
5	64	49
6	50*	51
7	37*	27
8	38*	62
9	148	160
H= 14, L= 1		
0	31*	2
1	103	103
2	26*	44
3	89	85
4	34	36
5	25*	31
6	83	66
7	73	72
H= -14, L= 1		
0	79	62
1	29*	22
2	55	61
3	88	70
4	28*	9
5	66	48
6	75	80
7	84	117
H= 15, L= 1		
0	54	48
1	23*	31
2	72	76
3	46*	33
4	32	41
5	106	105
H= -15, L= 1		
0	25*	23
1	33	24
2	65	57
3	81	84
4	77	80
5	48	59
H= 0, L= 2		
0	194	247
1	707	805
2	265	264
3	504	557
4	199	191
5	462	512
6	226	194
7	176	197
8	112	98
9	367	375
10	51*	17
11	225	224
12	133	126
13	45*	30
14	77	67
15	60	56
H= 1, L= 2		
0	192	253
1	669	710
2	506	608
3	475	543
4	358	376
5	284	300
6	304	384
7	371	405
8	126	118
9	193	191
10	251	256
11	105	96
12	113	108
13	112	115
14	64	60
15	71	73
H= -1, L= 2		
0	145	140
1	487	508
2	551	603
3	400	388
4	438	488
5	196	156
6	433	441
7	104	92
8	98	94
9	208	219
H= 2, L= 2		
0	315	317
1	294	278
2	389	461
3	276	271
4	617	668
5	457	498
6	332	343
7	151	124
8	328	341
9	195	188
10	217	223
11	111	95
12	169	173
13	77	51
14	35*	41
15	55	53
H= -2, L= 2		
0	699	778
1	376	379
2	293	322
3	316	317
4	324	326
5	319	334
6	324	304
7	178	162
8	212	227
9	54	67
H= 3, L= 2		
0	193	196
1	572	679
2	312	340
3	343	324
4	310	289
5	382	397
6	185	168
7	99	92
8	302	326
9	314	355
10	47*	27
11	157	150
12	69	62
13	64	61
14	98	107
H= -3, L= 2		
0	649	612
1	815	867
2	261	245
3	445	516
4	56	57
5	340	348
6	96	69
7	275	307
8	31*	8
9	195	202
H= 4, L= 2		
0	294	273
1	418	432
2	281	264
3	372	368
4	317	285
5	384	435
6	224	233
7	355	368
8	257	250
9	207	224
10	98	102
11	121	111
12	82	71
13	84	69
14	38*	11
H= -4, L= 2		
0	441	426
1	573	585
2	327	300
3	365	356
4	243	223
5	202	191
6	261	232
7	209	185
8	136	139
9	245	237
H= 5, L= 2		
0	512	554
1	176	145
2	288	318
3	468	481
4	475	499
5	181	139
6	238	263
7	123	95
8	307	311
9	41*	10
H= -5, L= 2		
0	819	949
1	342	335
2	34*	63
3	281	277
4	333	353
5	72	78
6	217	223
7	158	114
8	234	260
9	111	111
H= 6, L= 2		
0	284	295
1	363	366
2	294	321
3	88	77
4	283	291
5	154	157
6	194	211
7	63	70
8	152	140
9	131	105
H= -6, L= 2		
0	411	398
1	430	463
2	294	318
3	150	144
4	238	215
5	247	256
6	140	97
7	229	196
8	173	167
9	247	247
H= 7, L= 2		
0	33*	25
1	231	234
2	45	35
3	262	279
4	101	105
5	252	253
6	161	170
7	223	228
8	86	86
9	161	154
H= -7, L= 2		
0	185	166
1	330	332
2	168	158
3	203	175
4	259	254
5	324	308
6	36*	11
7	148	127
8	228	241
9	209	208
H= 8, L= 2		
0	200	192
1	68	80
2	271	276
3	183	179
4	337	315
5	124	107
6	223	257
7	252	265
8	126	125
9	46*	11
H= -8, L= 2		
0	167	158
1	148	121
2	206	231
3	117	115
4	324	325
5	81	73
6	181	181
7	130	116
8	221	219
9	117	121
H= 9, L= 2		
0	236	215
1	127	98
2	306	337
3	88	82
4	138	114
5	130	127
6	229	237
7	124	131
8	147	134
9	35*	23
H= -9, L= 2		
0	162	129
1	217	187
2	182	161
3	155	129
4	261	246
5	209	210
6	159	151
7	162	141
8	41*	38
9	141	150
H= 10, L= 2		
0	233	223
1	168	173
2	171	152
3	197	196
4	41*	36
5	100	110
6	112	102
7	108	107
8	93	72
9	108	110
H= -10, L= 2		
0	173	155
1	231	185
2	99	75
3	216	218
4	198	196
5	76	69
6	124	102
7	126	130
8	81	98
9	101	103
H= 11, L= 2		
0	101	78
1	210	222
2	134	130
3	90	65
4	103	97
5	115	123
6	112	125
7	115	117
8	93	86
9	140	135
H= -11, L= 2		
0	229	232
1	108	109
2	95	70
3	132	113
4	173	185
5	106	114
6	108	113
7	95	95
8	69	93
9	38*	58
H= 12, L= 2		
0	173	168
1	135	124
2	147	137
3	52*	114
4	54	60
5	72	71
6	86	66
7	36*	67
8	31*	70
9	64	57
H= -12, L= 2		
0	109	106
1	144	146
2	92	97
3	36*	7
4	100	89
5	103	99
6	58	57
7	89	107
8	73	78
9	63	66
H= 13, L= 2		
0	38*	28
1	37	41
2	102	87
3	134	142
4	74	68
5	70	65
6	75	67
7	80	93
8	63	56
H= -13, L= 2		
0	39*	35
1	108	100
2	82	73
3	59	54
4	103	108
5	124	144
6	66	67
7	68	81
8	25*	16
H= 14, L= 2		
0	36*	18
1	64	57
2	31*	22
3	88	78
4	32*	19
5	27*	35
6	81	78
H= -14, L= 2		
0	99	97
1	35*	54
2	52	56
3	50	53
4	91	119
5	28*	21
6	42	42
H= 15, L= 2		
0	71	45
1	26*	24
2	23*	37
3	26*	30
4	29*	43
H= -15, L= 2		
0	74	74
1	27*	42
2	71	55
3	64	65
4	60	66
H= 0, L= 3		
0	121	127
1	440	428
2	279	225
3	269	264
4	261	269
5	300	331
6	330	310
7	301	311
8	138	133
9	292	336
10	73	70
11	124	121
12	55	51
13	91	78
14	85	79
H= 1, L= 3		
0	1108	1149
1	375	370
2	439	453
3	189	153
4	421	457
5	111	102
6	327	336
7	148	138
8	339	365
9	105	99
10	47*	21
11	150	130
12	159	168
13	70	71
14	40	58
H= -1, L= 3		
0	720	714
1	371	407
2	318	365
3	356	360
4	236	193
5	339	341
6	38*	26
7	210	211
8	29*	33
9	83	92
H= 2, L= 3		
0	601	709
1	443	448
2	424	453
3	303	308
4	414	455
5	121	110
6	165	155
7	231	223
8	164	141
9	96	67
10	196	168
11	52*	51
12	125	108
13	106	90
14	62	57
H= -2, L= 3		
0	246	225
1	152	157
2	466	552
3	139	128
4	424	453
5	53	36
6	347	302
7	107	100
8	259	282
9	181	200
H= 3, L= 3		
0	235	218
1	413	474
2	212	194
3	195	175
4	265	228
5	334	346
6	195	169
7	226	231
8	217	214
9	231	261
10	98	88
11	130	111
12	47*	47
13	83	86
14	63	42
H= -3, L= 3		
0	141	128
1	355	367
2	349	360
3	424	445
4	381	386
5	331	321
6	324	285
7	213	228
8	196	197
9	165	173
H= 4, L= 3		
0	316	283
1	52	41
2	395	452
3	276	285
4	369	330
5	169	172
6	237	270
7	92	94
8	235	222
9	182	175
10	123	125
11	64	58
12	93	97
13	35*	39
14	83	100
H= -4, L= 3		
0	184	123
1	474	498
2	207	189
3	390	379
4	113	92
5	295	306
6	242	202
7	253	255
8	104	106
9	201	221
H= 5, L= 3		
0	324	354
1	200	165
2	440	512
3	279	270
4	288	293
5	148	153
6	213	221
7	79	66
8	63	61
9	170	162
H= -5, L= 3		
0	187	204
1	179	155
2	539	642
3	211	189
4	232	227
5	73	85
6	316	285
7	180	168
8	142	131
9	135	140
H= 6, L= 3		
0	223	206
1	271	272
2	157	133
3	289	303
4	96	73
5	313	312
6	40*	31
7	118	102
8	66	53
9	229	194
H= -6, L= 3		
0	310	327
1	351	353
2	317	331
3	309	276
4	230	203
5	248	252
6	377	374
7	189	183
8	195	224
9	187	209
H= 7, L= 3		
0	43*	30
1	97	89
2	255	256
3	209	197
4	140	106
5	189	195
6	150	155
7	200	190
8	165	156
9	164	152
H= -7, L= 3		
0	36*	22
1	397	447
2	34*	25
3	355	388
4	224	192
5	114	101
6	180	152
7	351	400
8	127	124
9	80	79
H= 8, L= 3		
0	383	398
1	145	115
2	204	189
3	145	113
4	173	164
5	173	178
6	123	122
7	95	92
8	174	171
9	135	105
H= -8, L= 3		
0	196	200
1	214	202
2	165	161
3	217	188
4	189	170
5	195	207
6	117	91
7	136	116
8	170	181
9	50*	40
H= 9, L= 3		
0	74	59
1	236	231
2	39*	22
3	158	152
4	203	202
5	194	217
6	88	64
7	232	193
8	120	97
9	143	128
H= -9, L= 3		
0	211	185
1	41*	41
2	200	199
3	122	102
4	148	131
5	62	61
6	235	232
7	121	136
8	137	149
9	96	119
H= 10, L= 3		
0	112	102
1	178	175
2	214	242
3	175	180
4	102	81
5	109	100
6	44*	22
7	170	165
8	128	132
9	146	129
H= -10, L= 3		
0	29*	39
1	180	175
2	147	120
3	208	209
4	109	99
5	130	144
6	142	134
7	118	138
8	40*	50
9	45*	76
H= 11, L= 3		
0	370	422
1	85	45
2	74	73
3	134	144
4	202	210
5	99	90
6	110	123
7	90	80
8	73	85
H= -11, L= 3		
0	46*	33
1	158	148
2	69	69
3	130	133
4	31*	41
5	133	141
6	93	79
7	61	73
8	82	92
H= 12, L= 3		
0	36	26
1	171	156
2	103	100
3	181	168
4	195	188
5	126	122
6	63	38
7	114	111
8	30*	39
9	87	84
H= -12, L= 3		
0	43*	42
1	128	101
2	104	116
3	69	51
4	107	117
5	80	77
6	55	71
7	58	49
8	62	77
9	31*	40
H= 13, L= 3		
0	40*	39
1	130	124
2	84	79
3	91	90
4	154	153
5	175	204
6	82	64
7	47*	58
H= -13, L= 3		
0	81	75
1	41*	10
2	60	51
3	91	93
4	40*	57
5	39*	7
6	36*	60
7	84	91
8	91	94
H= 14, L= 3		
0	100	94
1	44	53
2	67	75
3	36*	45
4	101	99
5	76	74
6	138	142
H= -14, L= 3		
0	38*	6
1	105	89
2	61	55
3	34*	86
4	47	43
5	33	33
6	26*	55
H= 15, L= 3		
0	59	52
1	23*	10
2	51	53
3	58*	44
H= -15, L= 3		
0	70	64
1	27*	25
2	83	65
3	36*	32
4	39*	50
H= 0, L= 4		
0	714	752
1	435	457
2	353	340
3	273	229
4	296	280
5	185	153
6	249	234
7	127	121
8	236	247
9	103	87
10	166	164
11	39*	25
12	97	83
13	35*	14
14	94	85
H= 1, L= 4		
0	71	104
1	532	573
2	637	776
3	190	130
4	144	131
5	294	298
6	346	333
7	231	196
8	191	188
9	200	192
10	137	106
11	96	77
12	158	150
13	82	74
14	32	33
H= -1, L= 4		
0	251	243
1	331	331
2	213	176
3	377	382
4	254	221
5	421	398
6	62	40
7	273	283
8	191	209
9	176	211
H= 2, L= 4		
0	32	50
1	297	312
2	327	345
3	530	581
4	365	366
5	566	607
6	185	161
7	262	214
8	129	100
9	140	142
10	140	106
11	161	147
12	82	67
13	65	64
14	112	119
H= -2, L= 4		
0	515	518
1	382	380
2	385	398
3	246	250
4	296	271
5	269	238
6	243	209
7	154	142
8	188	198
9	145	169
H= 3, L= 4		
0	322	318
1	186	176
2	363	380
3	345	353
4	89	88
5	150	129
6	358	387
7	422	438
8	91	70
9	141	126
10	154	160
11	103	104
12	43*	36
13	77	81
H= -3, L= 4		
0	391	395
1	23	22
2	407	433
3	73	95
4	418	394
5	68	71
6	298	259
7	138	118
8	169	187
9	142	158
H= 4, L= 4		
0	371	386
1	153	169
2	381	396
3	274	242
4	329	312
5	258	252
6	273	294
7	272	251
8	178	167
9	34*	26
10	208	220
11	42*	43
12	43	59
13	81	94
H= -4, L= 4		
0	202	183
1	203	176
2	193	171
3	255	242
4	246	205
5	303	294
6	99	85
7	240	265
8	134	124
9	88	118
H= 5, L= 4		
0	72	87
1	308	290
2	157	132
3	463	521
4	38*	58
5	310	340
6	225	235
7	339	334
8	151	130
9	144	160
H= -5, L= 4		
0	298	220
1	222	208
2	167	155
3	297	281
4	259	251
5	298	262
6	171	153
7	110	107
8	104	105
9	70	69
H= 6, L= 4		
0	208	200
1	393	430
2	192	182
3	281	292
4	168	134
5	212	203
6	175	190
7	337	340
8	128	114
9	105	75
H= -6, L= 4		
0	238	221
1	194	170
2	274	286
3	85	41
4	299	281
5	40*	5
6	210	191
7	50	61
8	125	153
9	173	188
H= 7, L= 4		
0	287	292
1	126	116
2	305	347
3	119	103
4	241	219
5	170	163
6	154	169
7	62	38
8	214	220
9	47*	8
H= -7, L= 4		
0	275	299
1	106	96
2	206	167
3	180	161
4	125	101
5	237	229
6	170	139
7	189	208
8	197	207
9	92	94
H= 8, L= 4		
0	213	219
1	167	154
2	166	139
3	169	140
4	252	217
5	186	211
6	90	102
7	186	158
8	132	120
9	84	118
H= -8, L= 4		
0	133	115
1	166	164
2	141	122
3	172	161
4	113	96
5	252	255
6	142	119
7	110	130
8	47*	18
9	133	155
H= 9, L= 4		
0	152	121
1	193	190
2	40*	11
3	277	314
4	149	142
5	120	124
6	69	43
7	207	213
8	98	95
9	45*	62
H= -9, L= 4		
0	209	199
1	144	118
2	163	159
3	48*	25
4	150	144
5	44*	27
6	76	78
7	137	149
8	153	155
9	127	141
H= 10, L= 4		
0	177	159
1	164	158
2	154	165
3	141	139
4	111	103
5	153	131
6	77	86
7	134	128
8	148	143
9	40	53
H= -10, L= 4		
0	65	54
1	121	109
2	111	101
3	121	122
4	160	130
5	123	134
6	168	154
7	107	112
8	116	127
9	70	89
H= 11, L= 4		
0	45*	10
1	59	61
2	179	204
3	69	51
4	80	79
5	62	65
6	87	109
7	61	60
8	33*	41
9	32*	37
H= -11, L= 4		
0	46*	10
1	123	129
2	106	90
3	144	148
4	58	61
5	103	97
6	98	71
7	85	74
8	74	83
9	92	93
H= 12, L= 4		
0	35	46
1	85	76
2	82	73
3	82	81
4	27	29
5	124	126
6	49	57
7	35*	50
8	62	58
H= -12, L= 4		
0	150	136
1	40*	37
2	84	83
3	91	78
4	44*	52
5	70	72
6	80	79
7	38*	49
8	28*	43
9	115	127
H= 13, L= 4		
0	100	87
1	79	71
2	101	101
3	42*	53
4	75	46
5	88	74
6	71	77
H= -13, L= 4		
0	42*	50
1	39*	21
2	60	45
3	30*	47
4	105	113
5	35*	54
6	73	62
H= 14, L= 4		
0	63	56
1	50	41
2	73	57
3	30*	22
4	33*	17
5	37*	56
H= -14, L= 4		
0	35*	13
1	98	113
2	54	67
3	72	76
4	34*	23
5	105	115
H= 15, L= 4		
0	42*	24
H= -15, L= 4		
0	95	116
1	54	77
H= 0, L= 5		
0	626	659
1	320	315
2	332	318
3	203	174
4	291	268
5	160	132
6	328	330
7	101	93
8	83	68
9	154	152
10	212	211
11	35*	31
12	42*	126
H= 1, L= 5		
0	233	252
1	291	248
2	145	140
3	590	673
4	65	61
5	254	204
6	169	138
7	183	174
8	153	127
9	186	196
10	158	147
11	150	160
12	39*	26
13	82	71

Table 2 (*cont.*)

K	FO	FC
H= -1, L= 5		
0	389	378
1	354	391
2	230	233
3	386	388
4	265	229
5	261	223
6	189	157
7	195	155
8	182	176
9	191	217
H= 2, L= 5		
0	171	123
1	496	563
2	172	181
3	332	318
4	165	128
5	375	418
6	252	249
7	213	169
8	89	75
9	148	118
10	154	155
11	105	104
12	80	69
13	52	57
H= -2, L= 5		
0	247	229
1	553	605
2	111	93
3	350	339
4	62	42
5	325	293
6	167	130
7	245	253
8	65	59
9	160	167
H= 3, L= 5		
0	469	498
1	199	154
2	251	248
3	133	110
4	311	282
5	247	231
6	85	83
7	178	142
8	335	363
9	176	147
10	123	118
11	48	42
12	76	78
13	34*	5
H= -3, L= 5		
0	365	348
1	297	271
2	331	339
3	125	132
4	205	159
5	245	226
6	243	199
7	162	153
8	180	209
9	151	133
H= 4, L= 5		
0	309	284
1	197	152
2	123	99
3	160	129
4	260	270
5	287	273
6	74	36
7	79	61
8	259	272
9	171	174
10	145	154
11	152	164
12	85	72
13	104	100
H= -4, L= 5		
0	499	542
1	188	150
2	280	270
3	69	72
4	179	159
5	129	97
6	186	180
7	59	80
8	187	216
9	132	131
H= 5, L= 5		
0	132	95
1	339	356
2	270	300
3	225	255
4	36*	4
5	206	168
6	67	58
7	241	243
8	103	90
9	148	129
H= -5, L= 5		
0	243	200
1	267	267
2	190	153
3	272	253
4	171	138
5	231	212
6	103	70
7	163	169
8	62	72
9	215	251
H= 6, L= 5		
0	413	469
1	168	133
2	57	73
3	236	245
4	252	234
5	128	116
6	136	174
7	35*	5
8	222	217
9	96	67
H= -6, L= 5		
0	379	391
1	246	242
2	202	154
3	184	169
4	218	205
5	289	263
6	91	83
7	76	71
8	113	109
9	112	113
H= 7, L= 5		
0	190	188
1	260	237
2	144	143
3	178	161
4	283	314
5	33*	2
6	64	79
7	65	71
8	152	147
9	40*	26
H= -7, L= 5		
0	39*	29
1	37	27
2	326	375
3	80	78
4	196	178
5	80	71
6	196	185
7	124	113
8	198	242
9	94	106
H= 8, L= 5		
0	126	105
1	226	229
2	73	65
3	104	106
4	186	189
5	243	262
6	39*	29
7	108	117
8	122	112
9	130	94
H= -8, L= 5		
0	94	84
1	190	189
2	150	143
3	199	182
4	221	207
5	203	212
6	178	156
7	113	125
8	106	97
9	42*	54
H= 9, L= 5		
0	106	90
1	42*	39
2	186	218
3	148	173
4	211	227
5	105	97
6	129	138
7	96	85
8	113	106
9	44*	22
H= -9, L= 5		
0	61	43
1	156	150
2	112	100
3	148	124
4	195	192
5	119	110
6	82	80
7	86	83
8	44*	47
9	87	103
H= 10, L= 5		
0	169	148
1	164	171
2	141	146
3	104	86
4	153	156
5	113	116
6	166	208
7	95	86
8	87*	48
H= -10, L= 5		
0	154	168
1	46*	56
2	98	84
3	137	122
4	122	142
5	80	69
6	96	103
7	84	102
8	124	140
H= 11, L= 5		
0	212	212
1	111	128
2	83	52
3	148	165
4	66	68
5	153	154
6	38*	50
7	37*	47
8	31*	68
H= -11, L= 5		
0	136	130
1	44*	63
2	54	49
3	98	74
4	76	73
5	61	40
6	111	115
7	81	97
8	64	58
H= 12, L= 5		
0	129	99
1	92	91
2	35*	25
3	104	111
4	115	111
5	79	87
6	77	75
7	18*	49
H= -12, L= 5		
0	44*	4
1	56	63
2	38*	34
3	72	79
4	52	41
5	37*	37
6	40*	64
8	61	59
H= 13, L= 5		
0	102	95
1	32*	14
2	87	98
3	27	36
4	80	90
5	54	36
H= -13, L= 5		
0	68	63
1	47	44
2	51	49
3	66	45
4	33*	41
5	30*	43
H= 14, L= 5		
0	60	43
1	68	53
2	62	77
H= -14, L= 5		
0	30*	7
1	66	59
2	39*	47
3	41*	63
H= 0, L= 6		
0	159	130
1	208	165
2	158	126
3	303	288
4	144	125
5	221	183
6	192	176
7	196	204
8	112	99
9	104	89
10	111	83
11	115	114
12	59	62
13	73	79
H= 1, L= 6		
0	90	92
1	335	310
2	210	167
3	320	309
4	198	183
5	34	32
6	319	334
7	349	321
8	127	105
9	109	98
10	58	33
11	109	105
12	30	30
13	54	62
H= -1, L= 6		
0	121	107
1	39*	18
2	220	227
3	105	83
4	362	374
5	94	83
6	291	338
7	123	112
8	203	206
9	154	145
H= 2, L= 6		
0	337	310
1	141	124
2	250	231
3	175	132
4	373	376
5	284	286
6	350	392
7	109	83
8	267	249
9	70	53
10	69	90
11	97	76
12	79	70
13	57	66
H= -2, L= 6		
0	96	84
1	226	218
2	201	209
3	360	370
4	161	140
5	197	183
6	287	266
7	164	142
8	130	137
9	127	114
H= 3, L= 6		
0	245	228
1	328	325
2	298	298
3	342	304
4	373	342
5	163	147
6	161	153
7	54	42
8	200	204
9	123	121
10	86	86
11	74	70
12	63	54
H= -3, L= 6		
0	43*	23
1	218	214
2	196	184
3	334	296
4	135	99
5	208	205
6	157	136
7	162	163
8	118	116
9	133	148
H= 4, L= 6		
0	58	51
1	361	382
2	151	127
3	264	258
4	219	189
5	163	156
6	48*	32
7	298	311
8	169	151
9	125	111
10	102	92
11	111	110
H= -4, L= 6		
0	69	61
1	85	78
2	316	346
3	85	53
4	178	178
5	200	169
6	316	325
7	175	192
8	79	71
9	139	134
H= 5, L= 6		
0	235	201
1	176	177
2	285	308
3	214	190
4	314	311
5	178	187
6	198	237
7	67	62
8	126	110
9	44*	10
H= -5, L= 6		
0	225	225
1	259	290
2	160	167
3	321	332
4	257	255
5	133	106
6	167	138
7	147	151
8	125	159
9	114	105
H= 6, L= 6		
0	34*	29
1	208	189
2	356	439
3	184	163
4	253	251
5	101	87
6	182	212
7	88	71
8	105	116
9	65	52
H= -6, L= 6		
0	245	210
1	287	303
2	89	101
3	274	289
4	107	84
5	157	149
6	144	121
7	170	189
8	160	193
9	104	98
H= 7, L= 6		
0	188	195
1	280	327
2	196	213
3	246	250
4	156	125
5	188	208
6	69	66
7	117	102
8	63	51
9	133	118
H= -7, L= 6		
0	40	44
1	217	208
2	150	145
3	95	90
4	186	177
5	149	122
6	194	197
7	101	105
8	122	138
9	92	89
H= 8, L= 6		
0	41*	15
1	159	147
2	140	141
3	177	182
4	94	88
5	105	125
6	87	99
7	156	147
8	99	98
9	76	48
H= -8, L= 6		
0	96	70
1	161	137
2	224	279
3	98	77
4	93	99
5	45*	4
6	172	171
7	172	189
8	96	112
9	45*	52
H= 9, L= 6		
0	48*	14
1	127	108
2	217	255
3	50*	52
4	98	96
5	44*	23
6	159	165
7	42*	47
8	101	107
9	81	73
H= -9, L= 6		
0	88	89
1	171	179
2	81	72
3	91	83
4	108	110
5	145	157
6	63	60
7	86	79
8	69	74
9	88	97
H= 10, L= 6		
0	236	232
1	184	219
2	74	48
3	110	87
4	48	55
5	40*	33
6	108	116
7	144	139
8	58	58
H= -10, L= 6		
0	48*	21
1	115	117
2	85	84
3	111	100
4	139	153
5	70	70
6	83	85
7	40*	45
8	69	90
9	50	71
H= 11, L= 6		
0	42*	17
1	120	135
2	65	51
3	141	157
4	37*	31
5	104	101
6	26	19
7	82	82
H= -11, L= 6		
0	44*	36
1	31*	17
1	58	17
2	90	104
3	42	46
4	104	113
5	54	59
6	59	64
7	34*	46
8	44*	107
H= 12, L= 6		
0	154	150
1	31	25
2	83	70
3	115	110
4	77	70
5	77	75
6	35*	69
H= -12, L= 6		
0	83	77
1	75	80
2	55	41
3	40*	32
4	35*	8
5	57	77
6	65	79
H= 13, L= 6		
0	41*	19
1	58	61
2	63	76
3	92	90
H= -13, L= 6		
0	40*	34
1	55	68
2	72	83
3	72	69
4	70	72
5	38*	47
H= -14, L= 6		
0	84	84
1	51	48
H= 0, L= 7		
0	61	60
1	358	348
2	81	85
3	272	253
4	198	167
5	192	169
6	173	147
7	182	165
8	46	41
9	204	198
10	61	59
11	90	79
12	48	50
H= 1, L= 7		
0	313	290
1	208	160
2	201	175
3	196	148
4	237	218
5	181	176
6	153	160
7	105	83
8	183	168
9	105	95
10	134	135
11	34*	21
12	65	70
H= -1, L= 7		
0	387	388
1	254	258
2	269	262
3	223	200
4	250	227
5	128	127
6	201	203
7	67	47
8	174	182
9	146	141
H= 2, L= 7		
0	275	264
1	211	162
2	236	243
3	163	160
4	368	360
5	30	26
6	180	175
7	82	50
8	175	152
9	70	56
10	124	131
11	63	61
12	50	59
H= -2, L= 7		
0	325	314
1	163	127
2	203	209
3	86	56
4	321	331
5	226	221
6	181	179
7	36*	22
8	200	214
9	124	121
H= 3, L= 7		
0	168	141
1	333	349
2	289	303
3	199	170
4	173	157
5	191	201
6	160	135
7	166	157
8	47*	29
9	168	158
10	43*	32
11	92	92
H= -3, L= 7		
0	174	152
1	310	306
2	155	144
3	276	270
4	68	44
5	207	200
6	190	194
7	124	135
8	78	62
9	131	133
H= 4, L= 7		
0	49*	12
1	236	209
2	196	182
3	160	140
4	128	105
5	207	228
6	94	81
7	180	190
8	76	67
9	77	64
10	92	86
11	110	110
H= -4, L= 7		
0	354	362
1	244	213
2	69	66
3	279	289
4	186	180
5	81	69
6	172	158
7	122	134
8	64	75
9	125	110
H= 5, L= 7		
0	69	47
1	136	108
2	210	244
3	53*	54
4	226	243
5	62	60
6	141	160
7	49*	31
8	113	93
9	80	62
H= -5, L= 7		
0	183	188
1	207	200
2	173	181
3	200	185
4	251	252
5	164	164
6	204	216
7	112	91
8	113	121
9	49	81
H= 6, L= 7		
0	261	265
1	243	246
2	43*	12
3	144	142
4	112	121
5	134	141
6	102	110
7	114	119
8	123	108
9	80	82
H= -6, L= 7		
0	161	150
1	186	197
2	149	153
3	139	108
4	136	106
5	126	141
6	109	101
7	82	47
8	171	185
9	144	154
H= 7, L= 7		
0	62	45
1	168	176
2	108	103
3	162	178
4	116	118
5	111	126
6	69	70
7	170	166
8	87	80
9	120	102
H= -7, L= 7		
0	51*	17
1	225	229
2	44*	65
2	97	65
3	106	83
4	60	59
5	106	119
6	46*	15
7	85	103
8	55	72
9	98	102
H= 8, L= 7		
0	168	165
1	52	42
2	116	139
3	50*	53
4	151	152
5	84	74
6	83	111
7	55	44
8	60	53
9	76	72
H= -8, L= 7		
0	61	64
1	45*	39
2	90	100
3	77	62
4	101	94
5	78	63
6	75	79
7	82	91
8	106	121
9	37*	2
H= 9, L= 7		
0	108	109
1	49	87
2	82	94
3	98	96
4	116	107
5	77	74
6	65	48
7	76	87
8	106	127
H= -9, L= 7		
0	62	70
1	88	82
2	84	85
3	93	88
4	105	93
5	41*	24
6	99	98
7	39*	31
8	52	64
9	36*	57
H= 10, L= 7		
0	41*	27
1	154	161
2	85	70
3	113	117
4	36*	31
5	136	146
6	79	83
7	38*	12
H= -10, L= 7		
0	82	73
1	102	119
2	89	87
3	77	83
4	64	35
5	37*	46
6	35*	42
7	46	36
H= 11, L= 7		
0	114	116
1	33*	50
2	30*	40
3	112	121
4	121	143
5	65	47
H= -11, L= 7		
0	39*	5
1	46	36
2	35*	29
3	54	63
4	70	70
5	65	76
H= 12, L= 7		
0	32*	52
1	37*	52
2	125	116
3	38*	8
H= -12, L= 7		
0	34*	80
1	85	84
2	107	118
3	62	55
4	55	51
5	46	63
H= -13, L= 7		
0	53	44
1	106	117
2	41	49
H= 0, L= 8		
0	197	190
1	177	155
2	94	84
3	211	177
4	192	171
5	137	137
6	111	117
7	132	121
8	69	75
9	80	76
10	73	42
11	96	102
H= 1, L= 8		
0	201	178
1	103	76
2	247	251
3	146	114
4	160	143
5	49*	33
6	156	153
7	87	61
8	104	101
9	48*	7
10	111	102
11	60	47
H= -1, L= 8		
0	38*	11
1	246	258
2	149	137
3	184	164
4	120	83
5	138	134
6	50*	45
7	218	219
8	71	58
9	49*	57
H= 2, L= 8		
0	51	50
1	208	206
2	148	127
3	254	243
4	95	67
5	201	202
6	79	72
7	112	101
8	61	29
9	67	66
10	94	90
H= -2, L= 8		
0	164	144
1	83	74
2	158	174
3	151	127
4	133	131
5	117	122
6	127	138
7	51*	64
8	133	138
9	48*	47
H= 3, L= 8		
0	114	104
1	249	239
2	102	93
3	212	210
4	165	140
5	165	156
6	89	69
7	142	146
8	44*	49
9	60	58
10	81	71
H= -3, L= 8		
0	136	155
1	161	168
2	104	103
3	184	168
4	162	157
5	106	96
6	136	164
7	63	69
8	108	108
9	99	93
H= 4, L= 8		
0	177	163
1	168	152
2	253	298
3	83	79
4	163	161
5	48*	34
6	101	100
7	60	28
8	180	169
9	105	101
10	61	61
H= -4, L= 8		
0	90	51
1	171	174
2	65	47
3	178	179
4	95	67
5	106	111
6	115	111
7	149	143
8	71	71
9	96	119
H= 5, L= 8		
0	158	158
1	128	112
2	99	97
3	79	64
4	149	159
5	149	159
6	75	71
7	108	103
8	105	95
9	89	66
H= -5, L= 8		
0	164	145
1	48*	30
2	163	187
3	184	200
4	102	86
5	125	137
6	142	163
7	48*	24
8	83	91
9	80	73
H= 6, L= 8		
0	32	32
1	216	219
2	88	96
3	151	164
4	58	65
5	106	117
6	43*	14
7	95	102
8	79	62
9	133	157
H= -6, L= 8		
0	83	68
1	60	66
2	138	150
3	100	78
4	121	125
5	97	83
6	159	159
7	45*	39
8	91	110
9	80	71
H= 7, L= 8		
0	211	217
1	92	84
2	144	156
3	102	104
4	122	114
5	70	59
6	103	125
7	72	73
8	125	129
H= -7, L= 8		
0	39	37
1	105	111
2	43*	24
3	157	157
4	91	70
5	149	160
6	68	49
7	79	62
8	96	114
9	48	58
H= 8, L= 8		
0	143	149
1	74	53
2	109	122
3	65	48
4	133	145
5	66	54
6	96	110
7	41	43
H= -8, L= 8		
0	105	100
1	84	88
2	91	88
3	118	122
4	92	81
5	40*	56
6	76	81
7	81	103
8	137	127
H= 9, L= 8		
0	40*	7
1	111	106
2	76	73
3	106	106
4	51	53
5	88	98
6	80	95
H= -9, L= 8		
0	100	119
1	67	40
2	87	85
3	56	32
4	94	110
5	48	54
6	98	107
7	61	67
H= 10, L= 8		
0	109	113
1	64	64
2	74	78
3	74	72
4	92	107
5	89	92
H= -10, L= 8		
0	39*	49
1	50	79
2	63	68
3	94	107
4	73	67
5	30*	27
H= 11, L= 8		
0	76	88
1	35*	33
2	106	115
H= -11, L= 8		
0	33*	10
1	94	106
2	35*	21
H= 0, L= 9		
0	132	112
1	113	88
2	202	226
3	115	82
4	168	219
5	123	112
6	151	152
7	43*	18
8	105	107
9	50	46
H= 1, L= 9		
0	148	128
1	133	104
2	102	86
3	124	116
4	150	145
5	125	120
6	152	159
7	97	96
8	130	124
9	77	78
H= -1, L= 9		
0	148	168
1	159	168
2	79	83
3	172	164
4	66	49
5	186	205
6	76	71
7	111	96
8	55	46
9	81	80
H= 2, L= 9		
0	52*	21
1	196	185
2	83	76
3	153	158
4	49*	22
5	181	186
6	67	60
7	111	107
8	73	59
9	79	77
H= -2, L= 9		
0	52*	45
1	143	134
2	99	100
3	172	181
4	130	130
5	109	121
6	115	119
7	124	116
8	39*	35
9	80	96
H= 3, L= 9		
0	195	184
1	96	87
2	166	180
3	118	95
4	140	152
5	101	79
6	126	148
7	119	96
8	132	134
9	34*	53
H= -3, L= 9		
0	165	176
1	48*	35
2	132	146
3	50*	60
4	106	155
5	64	61
6	111	121
7	95	69
8	95	102
9	50	51
H= 4, L= 9		
0	218	219
1	95	76
2	144	141
3	82	50
4	156	154
5	80	70
6	134	146
7	77	64
8	110	121
H= -4, L= 9		
0	186	217
1	100	117
2	121	124
3	127	120
4	95	74
5	121	135
6	49	52
7	76	67
8	90	86
9	48	49
H= 5, L= 9		
0	48*	3
1	150	145
2	45	35
3	160	165
4	43*	18
5	132	149
6	84	91
7	97	94
8	59	48
H= -5, L= 9		
0	50*	5
1	104	131
2	55	29
3	139	151
4	45*	18
5	102	106
6	36	50
7	101	109
8	47	68
H= 6, L= 9		
0	148	142
1	106	119
2	73	86
3	130	124
4	101	92
5	90	89
6	69	71
7	40*	29
8	40*	93
H= -6, L= 9		
0	115	111
1	81	73
2	72	74
3	49*	55
4	78	73
5	80	69
6	77	93
7	43	54
8	81	82
H= 7, L= 9		
0	42*	62
1	39*	16
2	165	199
3	54	41
4	112	112
5	66	57
6	142	136
H= -7, L= 9		
0	72	72
1	56	61
2	78	99
3	45*	40
4	66	67
5	57	60
6	76	71
H= 8, L= 9		
0	62	62
1	104	113
2	94	100
3	90	102
4	83	58
5	108	122
H= -8, L= 9		
0	41*	55
1	93	69
2	35*	18
3	72	95
4	36*	12
5	55	67
H= 9, L= 9		
0	36*	5
1	87	104
2	51	50
3	107	124
H= -9, L= 9		
0	99	81
1	57	57
2	36*	11
3	36*	25
4	62	72
H= -10, L= 9		
0	38*	21
1	42*	47
2	47	58
H= 0, L= 10		
0	208	189
1	44	55
2	148	144
3	85	78
4	103	86
5	39*	29
6	124	142
7	62	70
8	58	75
H= 1, L= 10		
0	71	52
1	183	183
2	43*	24
3	149	144
4	41	40
5	63	58
6	41	13
7	72	74
H= -1, L= 10		
0	134	139
1	96	81
2	104	128
3	86	75
4	116	131
5	50	42
6	61	66
7	35*	25
H= 2, L= 10		
0	126	115
1	164	137
2	66	72
3	100	73
4	85	70
5	86	94
6	100	116
7	64	61
H= -2, L= 10		
0	67	53
1	152	166
2	76	81
3	106	118
4	41*	41
5	109	106
6	36*	43
7	48	63
H= 3, L= 10		
0	110	117
1	90	69
2	130	139
3	98	81
4	139	139
5	34*	27
6	60	64
7	34*	40
H= -3, L= 10		
0	133	119
1	58	73
2	72	67
3	85	86
4	78	81
5	53	56
6	70	71
7	53	71
H= 4, L= 10		
0	77	47
1	164	166
2	89	83
3	96	92
4	60	43
5	78	85
6	59	47
H= -4, L= 10		
0	112	119
1	67	62
2	107	140
3	83	67
4	81	88
5	99	112
6	90	117
7	50	56
H= 5, L= 10		
0	39*	20
1	110	114
2	60	62
3	87	109
4	53	58
5	99	98
H= -5, L= 10		
0	42*	45
1	110	125
2	55	39
3	92	95
4	96	103
5	31	51
H= 6, L= 10		
0	53	67
1	42	35
2	78	103
3	71	80
4	85	102
H= -6, L= 10		
0	56	47
1	105	113
2	29	25
3	50	50
4	54	67
5	80	91
H= 7, L= 10		
0	130	141
1	87	99
2	46	38
3	135	122
H= -7, L= 10		
0	59	77
1	33	34
2	78	90
3	87	106
4	81	91
5	46	44
H= -8, L= 10		
0	36*	14
1	88	99
2	97	98
H= 0, L= 11		
0	63	55
1	102	106
2	36	40
3	120	138
4	36*	21
5	69	75
H= 1, L= 11		
0	109	114
1	112	107
2	44	32
3	132	137
4	92	82
5	83	89
H= -1, L= 11		
0	78	90
1	53	64
2	81	92
3	35*	7
4	106	85
5	91	86
H= 2, L= 11		
0	35*	34
1	69	53
2	89	94
3	32*	37
4	116	138
H= -2, L= 11		
0	50	84
1	72	71
2	58	63
3	74	75
4	90	109
5	61	75
H= 3, L= 11		
0	32*	23
1	87	100
2	148	155
3	103	115
4	37*	77
H= -3, L= 11		
0	30*	2
1	100	97
2	44	30
3	77	90
4	31	30
H= 4, L= 11		
0	42*	14
1	129	161
2	60	59
3	38*	102
H= -4, L= 11		
0	49	50
1	44*	36
2	46	62
3	59	73
4	61	78
H= -5, L= 11		
0	42*	22
1	45*	3
2	59	73

Table 3. *The atomic coordinates, anisotropic vibrational parameters and their standard deviations (all quantities* × 10⁴)

	x/a	y/b	z/c	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{13}$	$2U_{12}$
Br	8493 (1)	7488 (2)	8924 (1)	618 (6)	546 (7)	329 (4)	172 (12)	−164 (9)	−673 (15)
O(3)	9882 (7)	7242 (6)	−2036 (10)	592 (49)	186 (43)	542 (45)	288 (65)	−373 (76)	68 (75)
O(6)	1763 (8)	6777 (11)	3567 (10)	625 (56)	829 (80)	402 (46)	−550 (100)	−97 (80)	412 (116)
O(20)	7238 (6)	6239 (7)	6072 (8)	389 (38)	290 (44)	399 (39)	120 (67)	55 (61)	191 (72)
N	8323 (6)	5337 (8)	3250 (10)	194 (36)	274 (51)	449 (47)	−359 (79)	36 (64)	159 (70)
C(1)	3343 (8)	5529 (11)	−805 (10)	312 (50)	477 (77)	229 (43)	−189 (88)	184 (74)	−14 (99)
C(2)	2662 (11)	6330 (13)	−1737 (13)	593 (45)	399 (57)	337 (52)	76 (105)	76 (86)	4 (83)
C(3)	1548 (10)	6389 (10)	−1161 (12)	543 (46)	155 (57)	321 (48)	130 (99)	−289 (84)	109 (82)
C(4)	1592 (8)	6714 (10)	434 (12)	366 (52)	204 (54)	389 (52)	−124 (88)	249 (83)	−184 (95)
C(5)	2218 (8)	5833 (9)	1289 (11)	269 (43)	152 (49)	280 (43)	15 (74)	−46 (69)	−54 (80)
C(6)	2202 (8)	5945 (10)	2931 (11)	241 (44)	378 (65)	264 (44)	−130 (83)	131 (71)	−104 (90)
C(7)	2727 (8)	5005 (11)	3793 (11)	194 (39)	459 (73)	308 (45)	310 (94)	155 (69)	39 (90)
C(8)	3885 (7)	4899 (9)	3340 (10)	168 (38)	362 (64)	270 (43)	105 (83)	−20 (65)	−307 (83)
C(9)	3926 (7)	4775 (9)	1658 (11)	130 (35)	174 (50)	380 (48)	−137 (80)	9 (66)	−47 (73)
C(10)	3405 (8)	5758 (9)	832 (10)	364 (50)	249 (57)	196 (39)	−232 (76)	86 (70)	−175 (92)
C(11)	5085 (9)	4458 (11)	1438 (13)	292 (49)	368 (70)	479 (62)	−410 (105)	−59 (89)	88 (100)
C(12)	5535 (8)	3988 (10)	2866 (14)	209 (44)	203 (57)	656 (71)	−70 (103)	−127 (88)	53 (87)
C(13)	6414 (8)	4722 (9)	3603 (12)	243 (43)	143 (50)	434 (58)	41 (83)	−45 (78)	70 (82)
C(14)	4534 (9)	3903 (11)	3840 (16)	231 (47)	210 (62)	859 (92)	−33 (120)	−74 (104)	−246 (93)
C(15)	4800 (10)	3746 (14)	5496 (17)	339 (61)	588 (96)	720 (88)	795 (155)	−61 (111)	−294 (128)
C(16)	5657 (9)	4532 (12)	6090 (14)	321 (53)	517 (85)	468 (64)	405 (115)	154 (94)	−36 (111)
C(17)	6697 (8)	4435 (10)	5214 (14)	225 (42)	204 (57)	575 (67)	406 (98)	−143 (86)	−14 (85)
C(18)	7398 (8)	4613 (9)	2696 (14)	206 (42)	189 (56)	638 (71)	−453 (101)	−38 (88)	−178 (86)
C(19)	3987 (9)	6871 (11)	1107 (15)	292 (50)	284 (65)	593 (71)	110 (108)	93 (94)	−81 (100)
C(20)	7626 (8)	5150 (11)	5868 (12)	313 (48)	370 (68)	414 (54)	573 (102)	70 (82)	143 (100)
C(21)	8001 (11)	4649 (13)	7362 (16)	484 (68)	415 (79)	671 (84)	692 (135)	−438 (124)	−278 (127)
C(22)	8589 (8)	5116 (9)	4911 (13)	246 (44)	169 (52)	543 (62)	329 (96)	−138 (85)	41 (86)
C(23)	9509 (9)	5917 (10)	5350 (15)	265 (47)	212 (56)	586 (63)	175 (98)	−349 (91)	−21 (89)
C(24)	9359 (8)	7135 (9)	4932 (13)	291 (47)	190 (56)	469 (57)	41 (83)	−310 (83)	−189 (82)
C(25)	9011 (8)	7282 (9)	3333 (11)	313 (45)	210 (63)	354 (45)	−75 (77)	12 (72)	−72 (86)
C(26)	8089 (7)	6545 (9)	2897 (10)	194 (38)	233 (56)	280 (43)	44 (75)	−113 (65)	61 (76)
C(27)	8651 (12)	8497 (10)	3019 (13)	634 (75)	163 (56)	416 (57)	94 (92)	−168 (104)	73 (113)
C(28)	9258 (9)	4968 (11)	2324 (15)	331 (53)	266 (66)	661 (75)	−321 (115)	326 (102)	147 (103)

Table 4. *Coordinates of hydrogen atoms used in the structure factor calculations*

All quantities × 10³. The hydrogen atoms are referred to the C or O atom to which they are attached.

	x/a	y/b	z/c
O(3)	80	668	−309
O(20)	770	664	691
C(1)	301	490	−116
C(1)	419	541	−87
C(2)	298	706	−170
C(2)	262	607	−273
C(3)	119	569	−127
C(4)	88	676	79
C(4)	194	742	55
C(5)	158	516	102
C(7)	231	448	325
C(7)	245	478	496
C(8)	415	573	354
C(9)	350	412	169
C(11)	534	513	109
C(11)	516	390	71
C(12)	590	317	265
C(13)	621	561	356
C(14)	398	322	336
C(15)	507	290	587
C(15)	416	387	602
C(16)	579	470	728
C(16)	546	546	592
C(17)	693	368	526
C(18)	734	476	152
C(18)	741	385	295
C(19)	423	705	220
C(19)	463	662	60
C(19)	345	744	75
C(21)	829	523	798
C(21)	860	404	719
C(21)	741	431	782
C(22)	889	433	500
C(23)	1014	566	490
C(23)	999	550	587
C(24)	1002	752	511
C(24)	883	753	565
C(25)	961	710	275
C(26)	798	664	172
C(26)	760	657	365
C(27)	897	898	374
C(27)	894	873	200
C(27)	777	857	310
C(28)	984	544	262
C(28)	955	427	228
C(28)	903	514	136

Discussion of the structure

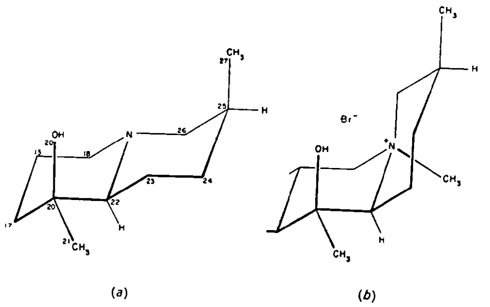
The stereochemical structure found for verticinone methyl bromide is shown in Fig. 2. All the six-membered rings of the skeleton of verticinone methyl bromide were found to have the chair conformation. The ring junctions *A–B*, *B–C*, *D–E* are *trans*-fused while the rings *C–D* and *E–F* are *cis*-fused. The *cis*-fusion of the *E* and *F* rings constitutes the only difference between the skeleton of verticinone methyl bromide and that suggested for verticine by Itô *et al.* (1963). The stereochemistry of the *E* and *F* rings in the two compounds is compared in Fig. 3. A configurational inversion at the nitrogen atom probably takes place on quaternization, hence the configuration of the methyl bromide derivative is not necessarily that of the original base. On hydrogen bonding and on conformational grounds the *trans*-quinolizidine arrangement appears favoured for verticine.

In agreement with Itô's proposal, the OH groups are found in equatorial position for O(3) and in axial position for O(20). Also two methyl groups, C(19)H$_3$ and C(21)H$_3$, are found attached to C(10) and C(20), in axial and equatorial positions respectively. The C(27) methyl group is attached to C(25) in the equatorial position, whereas Itô found from measurements of the chemical shifts by nuclear magnetic resonance that, for verticinone and other verticine derivatives, it was in the axial position. This difference, however, is to be expected on quaternization of the nitrogen atom which changes the *E–F* fusion from *trans* to *cis*. Since the difference in the chemical shifts of a methyl group in the equatorial or axial position is small, and not always a reliable indicator of its configuration, it seemed useful to examine the nuclear magnetic resonance spectrum of verticinone methyl bromide in order to compare it with spectra of compounds with known configuration of the C(27) methyl group.

As is shown in Table 5, the orientation of the C(27) methyl group can be assigned from the chemical shift for compounds with the cevane skeleton. In addition, the value of the chemical shift of the C(28) methyl group is $\delta = 3{\cdot}23$ p.p.m., thus confirming that this methyl group is attached to the nitrogen atom.

Bond distances and angles

The interatomic distances and angles are summarized in Fig. 4. These distances calculated from the final refined coordinates, by the expression of Ahmed & Cruickshank (1953), are not corrected for thermal motion. The e.s.d.'s of distances between the non-hydrogen atoms are in the range of 0·013 to 0·021 Å, and the angles have standard deviations of about 1°. The average distances in verticinone methyl bromide are compared below with the values given by Sutton (1965).



(*a*) (*b*)

Fig. 3. Comparison of (*a*) verticine and (*b*) verticinone methyl bromide.

Bond type	Average distance	Sutton's values
C sp^3–C sp^3	1·534 Å	1·537 ± 0·005 Å
C sp^3–C sp^2	1·515	1·506 ± 0·005
C sp^3–OH	1·440	1·426 ± 0·005

All bond distances are within 2σ of the expected average, with the exception of the C=O bond which is 1·292 Å and is therefore significantly longer than the value of 1·215 Å given by Sutton. The average C sp^3–N$^+$ sp^3 bond length of 1·537 Å is also longer than the average value of 1·478 Å reported by Sutton. The latter, however, seems to be underestimated, as Hamilton, Hamor, Robertson & Sim (1962) give a list of ten alkaloids for which the C sp^3–N$^+$ sp^3 average bond length is 1·52 Å. Using the more reliable bond distances of that list (e.s.d.'s $\leq 0{\cdot}03$ Å) and including the values of 1·54 Å (Przybylska, 1965), 1·55 Å, (Camerman, Camerman & Trotter, 1965) and 1·54 Å, (Nilsson, 1968), an average of 1·527 Å was obtained which agrees well with the mean for C–N bonds in verticinone methyl bromide. It is of interest to note that the C sp^3–N$^+$ sp^3 and C sp^3–C sp^3 bonds have comparable lengths.

For clarity, the values of the following angles were not indicated in Fig. 4(*b*): C(8)–N–C(26) = 111·0(± 0·8), C(22)–N–C(28) = 110·3(± 0·8), C(1)–C(10)–C(9) = 110·5(± 0·8), C(5)–C(10)–C(19) = 110·9(± 0·9), C(17)–C(20)–C(21) = 108·9(± 1·0), O(20)–C(20)–C(22) = 112·7° (± 0·9). All rings are somewhat distorted with angles varying from 105·0 to 116·8°. Although C(6)–O(6) is a double bond, ring *B* retains the chair conformation. The C(5)–C(6)–C(7) angle has a value of 115·5°, halfway between sp^2 and sp^3 bond angles. The angles C(5)–C(6)–O(6) and C(7)–C(6)–O(6) of 122·6 and 121·9° respectively are quite close to the theoretical value of 120°. The angles around the nitrogen atom vary between 103·8 and 113·7° in keeping with the tetrahedral coordination of this atom.

Mean planes

The planes through different groups of atoms of the

Table 5. *Orientation and chemical shift of the* C(27) *methyl group for compounds with the cevane skeleton measured at* 60 MHz

Compound	Orientation of the C(27) methyl group	Chemical shift δ(p.p.m.)	Reference
Verticinone methyl bromide*	Equatorial	0·85	This work
Dehydrocevine derivatives†	Equatorial	0·84–0·85	Itô *et al.* (1964)
Cevine derivatives†	Axial	1·04–1·09	Itô *et al.* (1964)
Verticine derivatives†	Axial	1·03–1·09	Itô *et al.* (1963)

* In dimethyl sulphoxide-d_6.
† In chloroform-d_3.

alkaloid are listed in Table 6. The average deviation of the atoms from the least-squares planes is about 0·03 Å with the exception of C(14) and C(15) which are at 0·19 and −0·12 Å respectively from plane 7. The dihedral angles formed between these planes are shown in Fig. 5. They vary between 122·3 and 137·3° and their average, 130·7°, is slightly larger than the theoretical angle of 125·2°. This flattening of the ring system has already been observed for a number of steroid molecules (Norton, 1965).

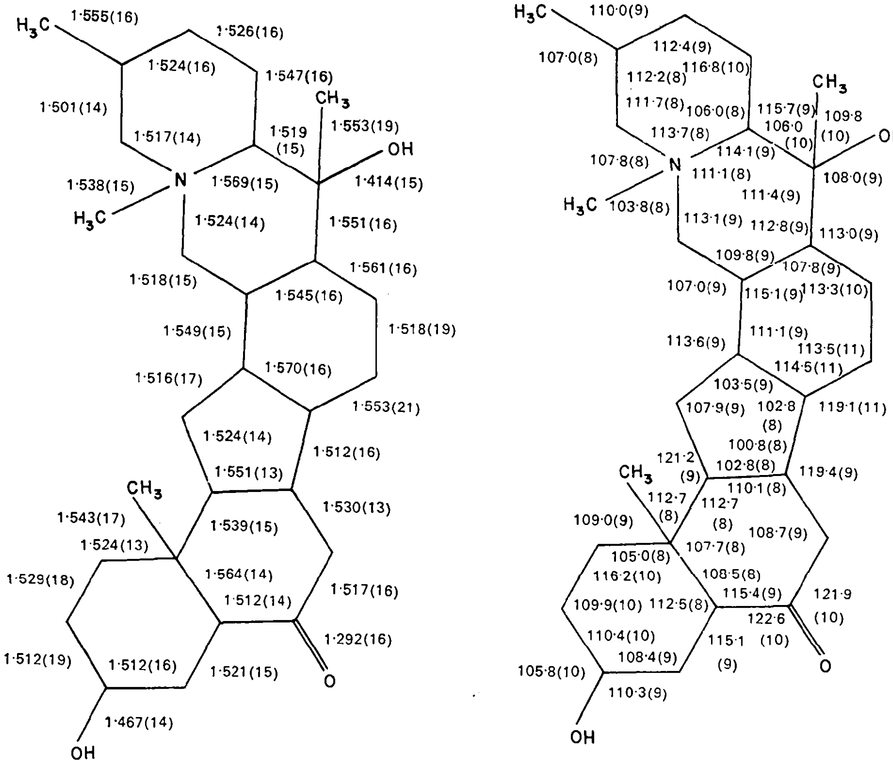
Packing of the molecules

The molecular packing as seen along the *b* and *c* axes is shown in Fig. 6(*a*) and (*b*) respectively. The intermolecular contacts less than 4·00 Å between pairs of non-

Table 6. *Planes*

The planes are expressed by $lX+mY+nZ-p=0$ with XYZ and p in Å. The orthogonal system of axes has $X \| a$-axis, Y in the a,b-plane, and Z along the c^* axis. All planes defined by more than three atoms were obtained by least-squares.

Plane	Atoms	*l*	*m*	*n*	*p*
1	C(2), C(3), C(4)	−0·0485	0·9646	−0·2591	7·6029
2	C(1), C(2), C(4), C(5)	−0·6169	−0·7262	−0·3035	−7·1838
3	C(1), C(5), C(6), C(10)	0·0171	0·9903	−0·1376	6·8043
4	C(6), C(7), C(9), C(10)	−0·6369	−0·6362	−0·4355	−7·4353
5	C(7), C(8), C(9)	0·0611	0·9939	−0·0918	5·8754
6	C(8), C(9), C(14)	−0·8316	−0·5554	−0·0044	−7·2523
7	C(9), C(11), C(12), C(14), C(15)	−0·2635	−0·9254	−0·2723	−6·9683
8	C(12), C(13), C(15), C(16)	−0·5855	0·7933	−0·1668	−0·6586
9	C(13), C(16), C(17), C(18)	−0·2063	−0·9613	−0·1827	−7·6977
10	N, C(17), C(18), C(20)	−0·5510	0·8212	−0·1485	−0·8631
11	N, C(20), C(22)	−0·1362	−0·9797	−0·1470	−8·1437
12	N, C(22), C(23)	0·6758	−0·6948	−0·2459	1·7867
13	N, C(23), C(24), C(26)	0·7932	−0·0231	−0·6085	6·2097
14	C(24), C(25), C(26)	0·6476	−0·7175	−0·2565	0·1734



(*a*) (*b*)

Fig. 4. (*a*) Bond distances (e.s.d.'s in parentheses ×10³). (*b*) Bond angles (e.s.d.'s in parentheses ×10).

hydrogen atoms are listed in Table 7. None of the intermolecular contacts is shorter than 3·30 Å. The *N*-methyl verticinone cations are packed in 'layers' with the cations in two successive 'layers' related to each other by a twofold screw axis. As indicated in Fig. 6, the bromide ion is connected to the cation by an almost linear hydrogen bond. The angle Br. . . H–O(20) is 172°, the Br. . . H distance is 2·30 Å and the H–O(20) bond length is 1·07 Å. The bromide ion is also at 3·30 Å

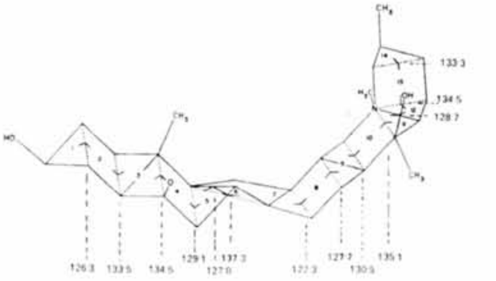


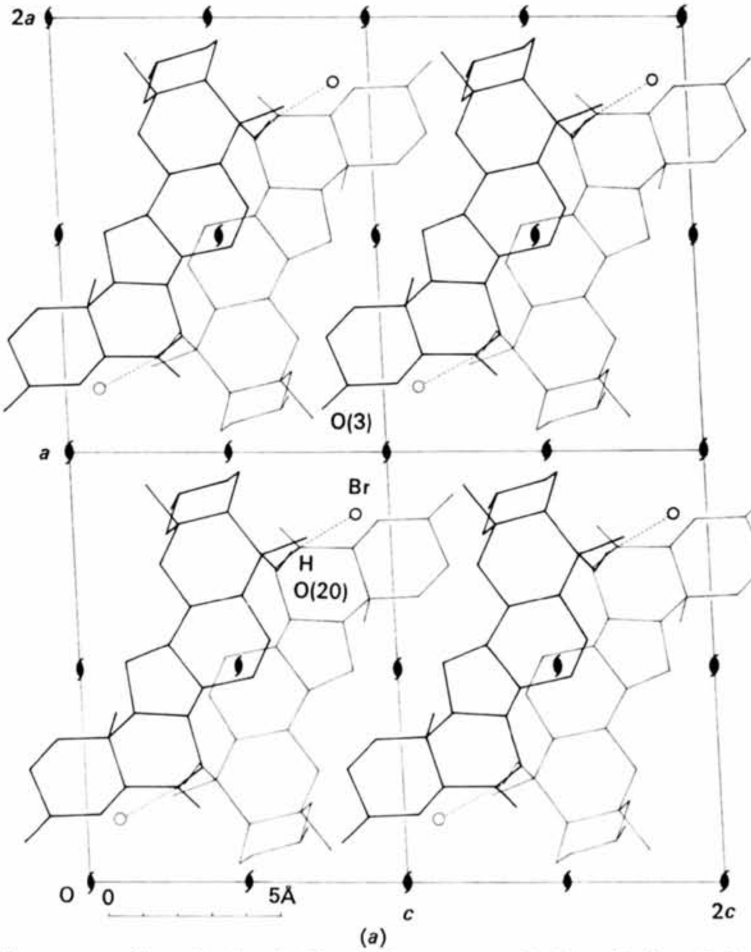
Fig. 5. Dihedral angles between mean planes.

Table 7. *Intermolecular contacts shorter than* 4·00 Å

Average e.s.d.'s: 0·018 Å.

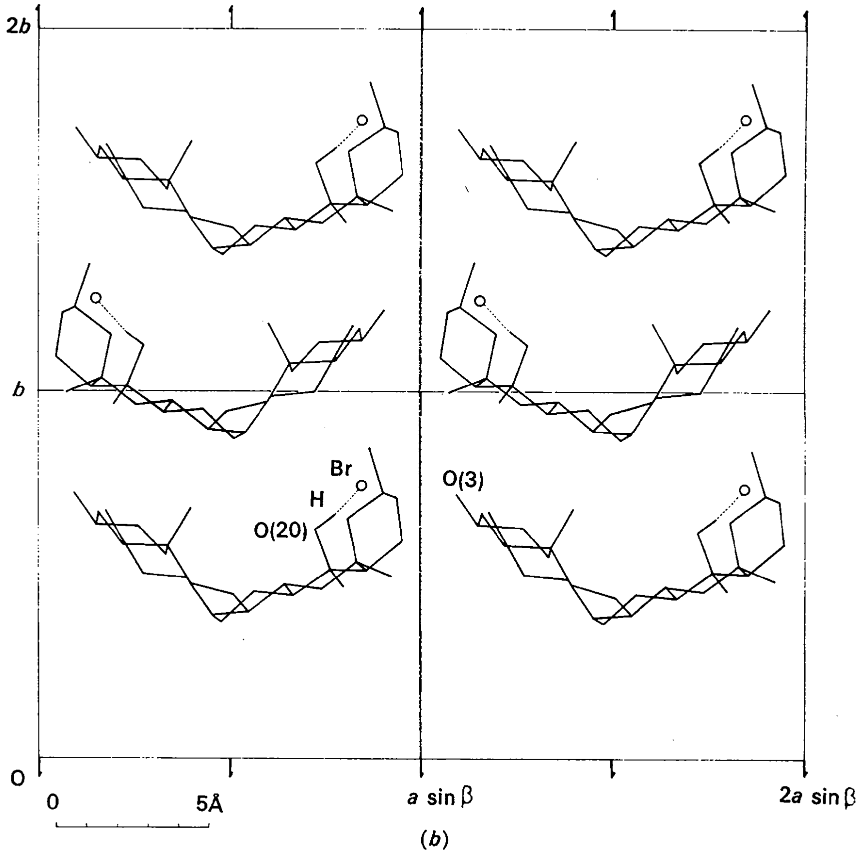
From atom	To atom	Position*	Distance
Br	O	1, $+a+c$	3·296
	C(26)	1, $+c$	3·865
	C(27)	1, $+c$	3·942
O(3)	N	2, $+c$	3·995
	C(18)	2, $+c$	3·566
	C(23)	1, $-a-c$	3·377
	C(24)	1, $-a-c$	3·391
	C(28)	2, $+c$	3·309
O(6)	C(17)	2, $-b$	3·882
	C(21)	2	3·577
	C(23)	1, $-a$	3·476
	C(24)	1, $-a$	3·336
	C(25)	1, $-a$	3·506
	C(28)	1, $-a$	3·959
O(20)	C(14)	2	3·907
C(1)	C(27)	2, $-b+c$	4·000
C(2)	C(27)	2, $-b+c$	3·950
C(3)	C(27)	2, $-b+c$	3·881
C(7)	C(27)	2, $-b$	3·897
C(23)	C(27)	2, $+a-b$	3·976
C(28)	C(6)	1, $+a$	3·899

* First symbol: equivalent positions 1 (x,y,z)
2 $(1-x,\frac{1}{2}+y,1-z)$.
Second symbol: cell translations.



(*a*)

Fig. 6. (*a*) Molecular packing as seen along the *b* axis. The circles represent the bromide ion. Hydrogen bonds are shown by broken lines.



(b)

Fig. 6 (*cont.*) (*b*) Molecular packing as seen along the *c* axis. The circles represent the bromine ion. Hydrogen bonds are shown by broken lines.

from the oxygen atom, O(3), of another cation translated by +**a**, +**c**. The difference Fourier synthesis, however, did not show any significant peak between Br and O(3), and the hydrogen attached to O(3) is at 3·62 Å from the bromine atom.

The author expresses his thanks to Dr Shô Itô for suggesting the problem and providing the crystals, and to Dr M. Przybylska for her encouragement and helpful comments in reviewing the manuscript of this paper. Dr C.P. Huber, of this laboratory, is gratefully acknowledged for help with the computations, and Mr J.L.M. Lesage for taking the n.m.r. spectrum. The stereoscopic drawing (Fig. 2) was made with the *ORTEP* program written by Dr C.K. Johnson.

While this paper was being prepared for publication, a note on the X-ray structure analysis of verticinone methobromide, by Itô, Fukazawa, Okuda and Iitaka (1968) came to our attention. Their results, as reported in the note, agree with ours.

References

Ahmed, F. R. & Cruickshank, D. W. J. (1953). *Acta Cryst.* **6**, 385.

Ahmed, F. R., Hall, S. R., Pippy, M. E. & Huber, C. P. (1966). *NRC Crystallographic Programs for the IBM* 360 *System. World List of Crystallographic Programs.* 2nd ed. Appendix, page 52.

Camerman, A., Camerman, N. & Trotter, J. (1965). *Acta Cryst.* **19**, 314.

Cromer, D. T. (1965). *Acta Cryst.* **18**, 17.

Cromer, D. T. & Waber, J. T. (1965). *Acta Cryst.* **18**, 104.

Hall, S. R. & Ahmed, F. R. (1968). *Acta Cryst.* B**24**, 337.

Hamilton, J. A., Hamor, T. A., Robertson, J. M. & Sim, G. A. (1962). *J. Chem. Soc.* p. 5061.

Hamilton, W. C., Rollett, J. S. & Sparks, R. A. (1965). *Acta Cryst.* **17**, 1040.

Ito, S., Kato, M., Shibata, K. & Nozoe, T. (1961). *Chem. Pharm. Bull.* **9**, 253.

Ito, S., Kato, M., Shibata, K. & Nozoe, T. (1963). *Chem. Pharm. Bull.* **11**, 1337.

Ito, S., Stothers, J. B. & Kupchan, S. M. (1964). *Tetrahedron,* **20**, 913.

Ito, S., Fukazawa, Y., Okuda, T. & Iitaka, Y. (1968). *Tetrahedron Letters,* **51**, 5373.

Nilsson, B. (1968). *Acta Cryst.* B**24**, 252.

Norton, D. A. (1965). *Biophys. J.* **5**, 425.

Przybylska, M. (1965). *Acta Cryst.* **18**, 536.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175.

Sutton, L. E. (1965). *Tables of Interatomic Distances and Configuration in Molecules and Ions.* Supplement 1956–1959. London: The Chemical Society.