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The Crystal Structure of Solaphyllidine, $C_{29}H_{47}NO_5$, a New Steroidal Alkaloid

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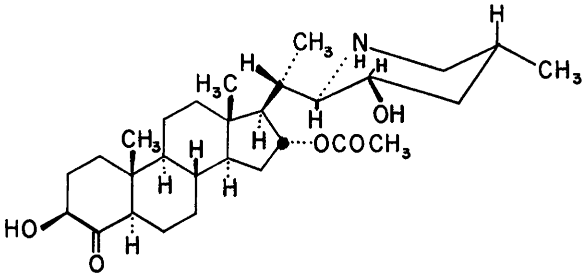
The structural formula of solaphyllidine, a new steroidal alkaloid, isolated from the leaves and green berries of *Solanum hypomalacophyllum* Bitter, a tree native to the Venezuelan Andes, has been established to be



The molecule is completely extended with ring junctions *A*/*B*, *B*/*C* and *C*/*D* all *trans*. The average planes of the *O*-acetyl group and the 3-hydroxy-5-methylpiperidyl group are nearly perpendicular to the average plane of the steroid nucleus. Rings *A*, *B*, *C* and *E* have the chair conformation while ring *D* has the β-envelope conformation with atom C(13) 0·68 Å out of the plane of the other four atoms in the ring. Asymmetric centers in the piperidyl group are 22*R*, 23*S*(*eq*) and 25*R*(*eq*). The steroidal alkaloid crystallizes with CH_3OH in a 1:1 ratio. A layered structure is formed by four independent hydrogen bonds between steroid molecules and the CH_3OH group. The material crystallizes in space group $P2_12_12_1$ with $a=15{\cdot}24\pm0{\cdot}03$, $b=10{\cdot}32\pm0{\cdot}02$ and $c=17{\cdot}50\pm0{\cdot}03$ Å and $Z=4$. The structure was solved by the application of the symbolic addition procedure for phase determination in noncentrosymmetric space groups.

Introduction

Steroidal alkaloids have been isolated from both animal and plant sources. Complete crystal structure analyses, which determined the structural formulae as well as the stereochemistry, have been performed on batrochotoxinin A, a very lethal venom from the Colombian poison arrow frog (Karle & Karle, 1969*a*; see also Tokuyama, Daly & Witkop, 1969), samandarine, the venom from the European fire salamander (Wölfel, Schöpf, Weitz & Habermehl, 1961) and veratrobasine, from the European hellebore, a perennial plant (Reeke, Vincent & Lipscomb, 1968). Structural studies by X-ray diffraction have also been made on tomatidine.HBr (Kennard, Riva di Sanseverino & Rollett, 1967) and the isomorphous tomatidine.HI (Höhne, Ripperger & Schreiber, 1967), a steroidal alkaloid from the *Solanum* group. A new steroidal alkaloid has been isolated from the leaves and green berries of *Solanum hypomalacophyllum* Bitter, a tree native to the Venezuelan Andes. The present X-ray analysis has confirmed and supplemented chemical observations in establishing the structural formula of solaphyllidine. A preliminary note describing the chemistry and structure of solaphyllidine has been published (Usubillaga, Seelkopf, Karle, Daly & Witkop, 1969).

Structure analysis

Colorless, opaque, diamond-shaped tabular crystals of solaphyllidine grown from methanol were made available through the cooperative efforts of Drs B. Witkop and J. Daly of the National Institutes of Health, Bethesda, Maryland, and Drs A. Usubillaga and A. Seelkopf of the Universidad de los Andes, Merida, Venezuela. The non-transparent nature of the crystals made it difficult to observe optical extinctions under crossed Nicol prisms. The *a* and *b* axes of the orthorhombic unit-cell corresponded to the diagonals of the diamond face. Cell parameters and space group were obtained from precession photographs. These values are listed in Table 1. Intensity data were collected by the multiple-film, equi-inclination Weissenberg technique and read by visual comparison with a calibrated film strip. Layers 0–11 along the *a* axis and 0–1 along the *b* axis were recorded. Lorentz, polarization and spot-size corrections were made, the data from the two axes were cross-correlated and put on the same scale, and normalized structure factors $|E|$ as well as structure factors $|F|$ were derived.

The atoms in this steroid of partially unknown structural formula and containing no heavy atoms were located by means of an *E* map computed with phase angles derived directly from the structure factor magnitudes using the symbolic addition procedure (Karle & Karle, 1964; 1966) for noncentrosymmetric crystals. The phase determination was initiated by applying the relationship

$$\varphi_{\mathbf{h}} \simeq \langle \varphi_{\mathbf{k}} + \varphi_{\mathbf{h}-\mathbf{k}} \rangle_{\mathbf{k}r} \, . \qquad (1)$$

This relationship is contained in the first nontrivial inequality (34) relating structure factor magnitudes in the complete set of inequalities based on the non-negativity principle (Karle & Hauptman, 1950). To implement equation (1), the origin was specified (Hauptman & Karle, 1956) by assigning the phase values $+\pi/2$, $+\pi/2$ and 0 to the reflections 3,0,13, 052 and 506 respectively, and, in addition, the reflections 022, 020, 819 and 919 were assigned the symbols a, b, p, and q to represent the phase angles. In the course of applying equation (1), it became apparent that $b=\pi$, a probably was equal to zero, and p and q were near $\pm\pi/2$. To specify an enantiomorph in space group $P2_12_12_1$, the symbol p was chosen to have the value $+\pi/2$.* Accordingly, two possibilities remained since q could be either $+\pi/2$ or $-\pi/2$. In addition to the assigned phases, phases for 43 additional reflections had been derived with equation (1). These 50 phases, now assigned numerical values, were used as input into the tangent formula (Karle & Hauptman, 1956):

$$\tan \phi_{\mathbf{h}} = \frac{\sum\limits_{\mathbf{k}} E_{\mathbf{k}} \, E_{\mathbf{h}-\mathbf{k}} \sin (\phi_{\mathbf{k}} + \phi_{\mathbf{h}-\mathbf{k}})}{\sum\limits_{\mathbf{k}} E_{\mathbf{k}} \, E_{\mathbf{h}-\mathbf{k}} \cos (\phi_{\mathbf{k}} + \phi_{\mathbf{h}-\mathbf{k}})} \, . \qquad (2)$$

The input phases were refined by one iteration, and then phases for additional reflections were obtained by the use of (2). A total of 425 reflections with $|E| > 1\cdot0$ were used to compute E maps. The twelve strongest peaks of the E map based on $q=-\pi/2$ could be identified with atoms in the four rings of a steroid nucleus. Phases based on these twelve atom positions for reflections with $|E| > 1\cdot5$ and $|F_{\text{calc}}| > 0\cdot3|F_{\text{obs}}|$ were used as input in equation (2), in order to compute phases for additional reflections with $|E| > 1\cdot0$ (Karle, 1968). The resulting E map showed the location of 30 atoms in the molecule. The remaining five atoms in the steroid and a CH_3OH molecule cocrystallized with the steroid were found in a difference map.

A full-matrix least-squares refinement on the coordinates and thermal parameters was performed utilizing the computer program of Busing, Martin & Levy (1962). Several cycles of refinement with anisotropic thermal factors, based on the minimization of the function $\sum w(|F_o|-|F_c|)^2$ where $w=0\cdot5$ for $F_o=0$, $w=1$ for

Table 1. *Physical data for solaphyllidine*

Molecular formula		$C_{29}H_{47}NO_5 . CH_3OH$
Molecular weight		489·7 + 32·0
Melting point		165–170 °C
Habit		Opaque diamond tablets (001)
Crystal size		~ 1·0 × 1·0 × 0·3 mm
Space group		$P2_12_12_1$
a		15·24 ± 0·03 Å
b		10·32 ± 0·02 Å
c		17·50 ± 0·03 Å
V		2889 $Å^3$
Z		4 molecules per unit cell
Density (calc)		1·199 $g.cm^{-3}$
Density (obs. by flotation)		1·178 $g.cm^{-3}$
Radiation		Cu $K\alpha$, 1·5418 Å
Number of independent reflections		2603
$\langle \lvert E \rvert \rangle$	for *hkl*	0·837
$\langle \lvert \lvert E \rvert^2 - 1 \rvert \rangle$		0·788
$\langle \lvert E \rvert \rangle$	for *hk*0, 0*kl*, *h*0*l*	0·797
$\langle \lvert \lvert E \rvert^2 - 1 \rvert \rangle$		1·023

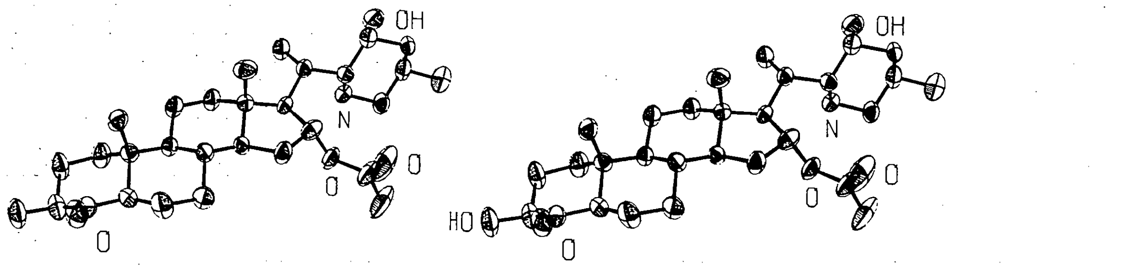


Fig. 1. Stereodrawing of the solaphyllidine molecule, drawn by computer from a program prepared by Johnson (1965).

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* The values appearing in Table 2 correspond to $p=-\pi/2$. After the structure determination was completed, the enantiomorph was changed to agree with the absolute configuration of cholesterol.

$|F_o| < 30$ and $w = 30/|F_o|$ for $|F_o| > 30$ and using the scattering factors listed in *International Tables for X-ray Crystallography* (1962), reduced the R index to 13·2%. A difference map revealed the location of 32 hydrogen atoms (out of 51). Inclusion of the 32 hydrogen atoms as constant parameters and further refinement on the

Table 2. *Observed and calculated structure factors for solaphyllidine*

The columns are the index l, $10|F_o|$, $10|F_c|$, and the phase in cycles (× 100).

0	0	L	
2	2184	2086	50
4	328	283	50
6	518	475	0
8	156	123	0
10	414	431	0
12	147	143	50
14	357	352	0
16	0	37	0
18	141	141	50
20	102	110	0
22	43	54	0
0	1	L	
2	459	415	74
3	359	393	74
4	458	346	25
5	1043	995	74
6	263	252	25
7	325	305	74
8	437	416	25
9	369	328	74
10	214	209	74
11	78	80	74
12	22	16	74
13	77	64	74
14	0	9	25
15	180	188	74
16	104	79	74
17	173	157	25
18	51	58	25
19	27	33	25
20	79	67	25
21	132	153	74
0	2	L	
0	3150	3218	50
1	347	420	50
2	1798	1828	0
3	1027	1124	50
4	175	67	0
5	167	170	0
6	150	170	50
7	60	91	0
8	386	337	50
9	20	65	50
10	53	43	0
11	243	254	0
12	64	68	50
13	168	169	50
14	110	105	50
15	80	69	50
16	76	73	0
17	50	59	0
18	0	8	50
19	31	33	0
20	24	41	50
21	0	11	0
22	25	53	50
0	3	L	
1	428	427	74
2	472	500	74
3	148	173	25
4	202	193	25
5	307	340	25
6	465	435	25
7	141	74	25
8	186	161	74
9	78	68	25
10	261	252	25
11	251	252	74
12	133	128	25
13	128	115	25
14	79	74	25
15	141	127	25
16	0	2	74
17	0	9	25
18	0	9	74
19	64	56	25
0	4	L	
0	1199	1214	0
1	375	385	0
2	243	279	50
3	213	237	0
4	168	200	0
5	83	77	50
6	201	217	0
7	83	78	0
8	287	273	0
9	78	83	0
10	305	299	50
11	0	46	0
12	96	112	0
13	142	139	0
14	177	181	0
15	0	23	0
16	129	120	0
17	54	55	50
18	0	10	0
19	110	98	0
0	5	L	
1	337	294	74
2	681	724	25
3	203	186	25
4	150	158	25
5	375	402	74
6	114	102	74
7	180	215	74
8	21	40	25
9	94	79	74
10	46	92	25
11	34	31	74
12	0	24	74
13	107	85	74
14	25	9	74
15	254	255	74
16	0	15	25
17	98	60	25
18	21	18	74
19	0	8	25
20	70	70	25
0	6	L	
0	833	830	50
1	532	570	50
2	538	543	0
3	34	23	50
4	45	69	50
5	129	98	0
6	220	188	50
7	38	39	50
8	134	137	50
9	100	84	0
10	54	47	50
11	25	11	0
12	0	32	0
13	249	253	0
14	112	115	50
15	85	75	0
16	80	73	50
17	0	8	0
18	62	74	0
19	53	47	50
20	50	71	50
0	7	L	
1	97	112	25
2	173	186	74
3	0	19	74
4	31	13	74
5	78	72	74
6	23	39	25
7	125	126	25
8	24	17	74
9	231	218	74
10	87	68	74
11	95	88	25
12	134	132	74
13	8	13	25
14	0	2	25
15	46	59	25
16	38	20	25
17	8	2	74
18	0	5	74
19	38	43	25
0	8	L	
0	272	274	0
1	0	20	50
2	138	119	50
3	82	70	50
4	59	46	0
5	91	75	50
6	58	36	50
7	35	40	50
8	172	177	0
9	88	71	0
10	25	12	0
11	8	6	50
12	0	6	0
13	48	32	50
14	39	49	0
15	8	18	0
16	46	36	50
17	48	51	50
0	9	L	
1	124	120	74
2	0	10	74
3	115	108	74
4	0	7	74
5	155	148	25
6	51	45	25
7	268	273	74
8	0	13	25
9	43	43	25
10	8	27	74
11	63	53	74
12	86	84	25
13	0	20	74
14	84	54	74
0	10	L	
0	95	77	50
1	0	40	0
2	102	91	50
3	67	54	50
4	76	41	50
5	0	6	50
6	50	37	0
7	92	81	0
8	128	110	50
9	24	34	50
10	0	18	0
11	0	22	0
12	98	68	50
13	32	22	0
14	39	37	50
0	11	L	
1	55	47	25
2	112	90	25
3	0	24	25
4	0	26	74
5	0	2	74
6	0	7	74
7	90	81	25
8	0	16	74
9	67	54	74
10	58	28	74
11	48	38	25
12	0	0	25
13	74	61	74
0	12	L	
10	69	47	50
11	59	50	0
0	13	L	
7	73	67	74
1	0	L	
2	1259	1101	0
3	449	353	74
4	293	263	0
5	63	21	74
6	125	101	0
7	420	394	74
8	558	446	50
9	337	284	25
10	209	198	0
11	74	78	74
12	99	98	0
13	83	77	25
14	99	109	50
15	23	32	25
16	0	38	0
17	115	124	74
18	80	55	50
19	132	150	25
20	91	75	50
21	84	105	74
22	26	47	0
1	1	L	
0	259	370	25
1	207	246	79
2	901	834	54
3	835	766	36
4	489	447	8
5	200	145	45
6	276	256	24
7	371	276	17
8	179	168	37
9	504	469	76
10	176	175	25
11	158	119	29
12	88	86	60
13	124	125	82
14	187	189	91
15	93	73	80
16	175	174	56
17	121	108	44
18	32	27	2
19	74	63	45
20	0	26	97
21	27	24	2
22	25	51	60
1	2	L	
0	358	366	74
1	[illegible]	1051	80
2	681	749	46
3	320	313	93
4	411	348	31
5	52	62	5
6	181	124	39
7	516	495	55
8	253	257	84
9	316	300	96
10	290	222	33
11	79	92	10
12	78	68	16
13	193	183	30
14	40	47	56
15	99	107	46
16	128	120	52
17	58	53	65
18	31	35	19
19	20	41	69
20	44	44	96
21	69	62	23
22	18	23	40
1	3	L	
0	520	630	74
1	250	274	44
2	587	599	37
3	113	154	63
4	231	235	92
5	127	124	42
6	81	64	63
7	325	302	81
8	242	203	3
9	464	454	20
10	116	109	78
11	173	180	77
12	116	124	50
13	114	115	69
14	23	28	19
15	62	62	26
16	125	123	29
17	32	42	28
18	37	44	59
19	0	16	16
20	73	68	1
21	59	63	63
1	4	L	
0	300	314	74
1	302	335	40
2	83	68	88
3	454	491	69
4	332	378	40
5	240	234	85
6	117	90	29
7	351	347	8
8	216	206	13
9	195	188	48
10	109	88	46
11	74	65	12
12	239	249	41
13	0	33	48
14	125	130	62
15	0	17	13
16	51	38	2
17	0	15	52
18	54	60	49
19	49	39	10
20	34	43	35
21	33	34	77
1	5	L	
0	189	164	25
1	150	172	92
2	397	424	70
3	479	491	35
4	438	437	9
5	150	150	93
6	261	236	23
7	193	193	20
8	39	44	58
9	92	102	82
10	74	82	49
11	77	77	14
12	23	33	17
13	205	215	30
14	70	93	10
15	93	84	11
17	65	39	92
18	19	25	3
19	83	83	48
1	6	L	
0	222	229	74
1	0	26	20
2	107	102	33
3	68	65	32
4	184	181	99
5	171	194	98
6	146	146	8
7	177	184	47
8	156	145	42
9	189	187	84
10	101	98	13
11	159	163	47
12	41	48	85
13	47	56	32
14	23	39	2
15	50	45	2
16	37	57	61
17	62	80	14
18	112	99	14
19	81	96	74
20	45	60	73
1	7	L	
0	73	76	25
1	117	97	38
2	224	233	19
3	140	155	99
4	141	135	56
5	190	191	24
6	96	84	88
7	130	138	70
8	90	94	17
9	79	82	1
10	62	68	12
11	0	18	15
12	23	30	46
13	73	69	91
14	39	55	47
15	42	42	48
16	34	41	79
17	30	42	40
18	45	36	43
19	48	59	98
1	8	L	
0	0	33	74
1	149	154	14
2	160	169	98
3	110	110	92
4	0	22	62
5	121	122	57
6	111	125	57
7	159	189	1
8	139	134	18
9	80	77	50
10	94	101	69
11	93	97	9
12	91	96	23
13	44	47	66
14	46	45	99
15	0	7	12
16	63	54	91
17	0	17	52
18	81	95	57
1	9	L	
0	86	57	25
1	80	48	39
2	86	61	98
3	113	91	81
4	57	75	61
5	40	37	64
6	132	140	93
7	173	166	10
8	33	31	48
9	80	85	63
10	85	74	45
11	0	3	40
12	79	69	9
13	0	15	2
14	77	55	56
15	28	42	37
16	47	45	23
17	37	56	96
1	10	L	
0	47	36	74
1	52	39	82
2	99	89	62
3	47	33	22
4	70	55	11
5	46	42	17
6	65	56	15
7	45	45	66
8	99	95	67
9	22	42	7
10	108	105	23
11	0	11	54
12	84	64	72
13	46	36	4
14	18	18	43
15	29	43	59
1	11	L	
0	110	77	74
1	32	21	58
2	39	21	43
3	54	39	5
4	38	40	3
5	0	11	10
6	0	14	18
7	29	32	48
8	0	8	84
9	86	60	21
10	37	26	77
11	0	11	37
12	53	38	43
1	12	L	
8	73	57	0
10	55	42	61
1	13	L	
3	59	33	27
2	0	L	
0	285	402	50
1	154	72	25
2	695	660	50
3	411	341	25
4	671	537	50
5	1010	989	25
6	350	319	0
7	469	432	74
8	518	488	50
9	400	366	25
10	159	172	50
11	334	331	25
12	151	135	50
13	336	302	74
14	119	126	50
15	88	74	25
16	288	303	0
17	90	111	25
18	133	145	50
19	0	31	25
20	39	37	0
2	1	L	
0	54	69	0
1	57	176	22
2	149	132	29
3	1131	1111	34
4	707	676	1
5	713	669	75
6	669	600	18
7	727	696	21
8	520	440	62
9	246	267	42
10	204	211	89
11	163	160	14
12	212	205	6
13	172	168	21
14	200	203	48
15	140	135	69
16	158	157	91
17	38	51	32
18	0	34	69
19	36	41	57
20	23	28	99
2	2	L	
0	805	830	0
1	80	45	20
2	320	340	5
3	366	425	95
4	213	198	84
5	392	396	17
6	498	444	23
7	412	396	40
8	295	272	83
9	224	189	78
10	109	92	66
11	237	229	74
12	114	114	61
13	243	242	9
14	235	243	9
15	0	27	27
16	55	52	19
17	98	79	66
18	88	87	89
19	61	53	28
20	22	38	8
2	3	L	
0	141	152	0
1	142	176	59
2	365	344	89
3	164	182	31
4	551	545	36
5	428	454	62
6	253	212	67
7	470	421	67
8	320	337	9
9	210	187	93
10	204	209	84
11	109	77	90
12	229	252	65
13	111	109	56
14	105	111	78
15	179	186	73
16	24	34	65
17	32	47	52
18	43	46	77
19	0	5	15
20	83	75	38
2	4	L	
0	304	322	0
1	306	314	36
2	118	110	41
3	310	332	33
4	280	273	41
5	462	442	62
6	432	416	63
7	149	154	2
8	189	187	35
9	263	270	45
10	55	73	24
11	194	220	22
12	85	77	51
13	121	111	84
14	106	104	46
15	63	55	53
16	80	86	39
17	133	141	21
18	29	30	33
19	45	49	73
20	27	37	73
2	5	L	
0	85	90	50
1	36	14	3
2	205	218	59
3	268	266	22
4	304	332	97
5	98	117	59
6	327	330	0
7	196	178	23
8	81	53	29
9	133	116	79
10	38	60	90
11	60	113	54
12	70	87	80
13	118	113	13
14	41	44	40
15	117	110	35
16	109	115	86
17	47	55	8
18	94	83	26
19	62	53	32
20	82	108	92
2	6	L	
0	226	224	50
1	97	88	42
2	138	147	94
3	349	342	98
4	116	97	13
5	144	167	98
6	64	63	37
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10	86	87	49
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13	131	136	38
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17	121	121	64
18	64	60	8
19	44	37	24
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3	160	163	89
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7	297	312	71
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9	23	10	96
10	106	107	8
11	53	54	92
12	53	54	37
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15	95	110	91
16	83	99	36
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18	89	91	77
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6	123	126	65
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6	101	95	17
7	95	107	23
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11	99	89	33
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13	48	43	83
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15	39	54	26
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6	123	122	15
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8	32	33	62
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6	394	367	0
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8	287	234	50
9	69	34	25
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11	260	251	74
12	141	151	0
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14	113	115	50
15	291	281	74
16	102	103	0
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6	87	86	13
7	517	475	89
8	153	115	81
9	186	171	18
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12	260	260	59
13	131	144	32
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15	169	201	26
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19	78	77	67
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6	322	296	15
7	523	472	48
8	261	253	97
9	178	165	61
10	221	209	18
11	140	129	6
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13	311	321	69
14	112	109	58
15	340	348	20
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17	144	148	73
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19	27	38	79
3	3	L	
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6	323	296	58
7	238	239	68
8	75	71	27
9	147	134	5
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11	160	123	40
12	127	104	16
13	60	44	85
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15	93	96	83
16	120	126	78
17	153	161	51
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19	65	67	16
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6	174	186	88
7	132	129	81
8	114	102	54
9	178	169	68
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11	201	213	58
12	130	130	21
13	270	281	24
14	183	176	4
15	215	215	70
16	59	70	84
17	91	99	15
18	74	90	55
19	37	34	78
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2	175	157	51
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5	213	220	71
6	237	247	3
7	123	106	4
8	149	148	52
9	83	72	30
10	75	43	17
11	297	306	61
12	134	149	55
13	135	152	65
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15	60	65	42
16	106	100	51
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18	0	24	21
19	99	90	57
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3	295	294	67
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6	117	128	47
7	220	234	39
8	119	135	98
9	66	54	81
10	135	136	35
11	255	278	12
12	46	63	36
13	224	225	62
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15	75	72	19
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1	184	167	68
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3	177	184	77
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5	227	249	24
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9	86	98	71
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6	60	45	57
7	92	90	99
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13	104	101	11
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15	39	38	84
16	52	51	57
17	33	28	17
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3	155	161	23
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6	114	110	94
7	46	49	27
8	69	65	39
9	68	42	77
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6	39	27	17
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5	63	62	22
6	35	32	33
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9	44	36	81
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11	26	16	22
12	28	26	30
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5	46	36	70
6	52	46	88
4	0	L	
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2	216	181	0
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6	174	165	0
7	374	365	74
8	75	96	50
9	341	336	74
10	246	226	0
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12	111	131	50
13	258	280	74
14	205	215	50
15	119	89	25
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18	0	6	0
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21	42	34	25
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7	139	134	74
8	471	414	48
9	317	320	70
10	184	180	89
11	84	88	78
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13	275	280	82
14	160	172	68
15	169	179	26
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17	70	49	13
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4	2	L	
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5	253	257	32
6	526	489	72
7	310	288	97
8	448	413	23
9	204	189	45
10	172	154	51
11	148	153	54
12	85	85	58
13	161	161	22
14	139	149	13
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16	139	149	53
17	62	49	95
4	3	L	
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3	217	246	30
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5	138	116	91
6	208	174	42
7	72	68	38
8	101	120	91
9	75	38	26
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11	167	175	88
12	104	112	51
13	312	333	26
14	258	277	19
15	91	94	91
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6	225	233	4
7	144	161	78
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12	34	19	95
13	166	158	72
14	49	42	80
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16	134	139	99
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3	93	101	45
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6	136	154	13
7	122	115	79
8	175	158	56
9	118	106	73
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11	146	163	16
12	129	128	13
13	85	73	77
14	73	81	56
15	58	71	82
4	6	L	
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5	120	97	84
6	219	214	58
7	115	97	43
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9	70	67	54
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9	149	145	8
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12	120	110	78
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13	82	71	2
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7	74	40	92
4	13	L	
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8	202	193	50
9	42	11	25
10	104	93	50
11	295	301	74
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13	208	220	25
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2	74	70	99
3	302	283	87
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7	502	487	17
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11	346	349	73
12	119	110	49
13	93	61	14
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15	124	124	60
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6	323	307	34
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8	313	306	71
9	98	82	47
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13	37	57	72
14	131	138	46
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6	345	336	11
7	270	267	90
8	237	218	54
9	128	128	15
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13	179	179	74
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15	151	136	9
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6	153	198	85
7	208	219	54
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9	76	87	87
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12	62	68	77
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16	49	40	13
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2	126	110	96
3	93	82	55
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6	141	154	37
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9	152	155	57
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15	57	62	77
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8	237	259	45
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15	78	90	97
16	29	38	79
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2	114	107	57
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7	127	127	73
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6	72	70	8
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7	75	56	75
8	40	34	3
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6	459	500	50
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8	207	183	54
9	226	202	31
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12	107	117	12
13	194	208	29
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21	33	47	23
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6	109	80	99
7	192	191	44
8	107	100	13
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10	129	131	61
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13	112	110	83
14	112	105	65
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17	65	66	82
18	86	82	46
19	45	59	29
20	37	46	18
6	4	L	
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2	65	33	90
3	122	136	28
4	91	87	91
5	226	256	47
6	332	351	52
7	233	249	14
8	193	191	95
9	109	124	63
10	122	133	29
11	45	66	57
12	94	77	15
13	120	121	12
14	92	93	39
15	67	57	42
16	0	21	77
17	88	92	23
18	31	34	29
6	5	L	
0	245	265	0
1	110	84	59
2	0	26	14
3	153	143	45
4	286	294	75
5	242	242	18
6	249	234	26
7	62	74	89
8	118	129	63
9	107	96	46
10	282	283	2
11	86	104	71
12	73	72	75
13	65	60	21
14	76	80	13
15	0	21	26
16	0	8	18
17	76	52	32
6	6	L	
0	55	60	0
1	48	53	12
2	130	126	85
3	174	178	39
4	300	308	45
5	205	223	80
6	274	280	96
7	86	78	58
8	187	184	21
9	60	52	0
10	87	91	61
11	0	15	7
12	33	36	29
13	109	110	50
14	105	118	11
15	41	51	75
16	0	24	33
17	29	27	70
6	7	L	
0	0	3	50
1	30	24	75
2	184	196	67
3	31	49	3
4	382	406	23
5	111	117	47
6	221	233	77
7	51	56	33
8	86	74	7
9	147	160	91
10	104	107	54
11	91	102	18
12	70	59	30
13	91	93	74
14	75	79	20
15	0	5	5
16	70	70	73
6	8	L	
0	0	3	50
1	192	198	49
2	189	184	59
3	40	53	94
4	265	274	98
5	103	108	23
6	98	97	52
7	40	43	6
8	52	68	60
9	40	41	43
10	55	65	13
11	43	45	66
12	62	52	51
13	101	104	1
14	0	6	35
15	44	50	44
16	46	44	89
6	9	L	
0	46	46	50
1	46	46	7
2	164	176	15
3	87	70	34
4	143	130	73
5	61	49	6
6	61	54	26
7	96	90	72
8	83	77	92
9	81	87	37
10	55	57	5
11	28	22	99
12	0	17	84
13	0	12	19
6	10	L	
0	0	3	50
1	55	50	89
2	109	86	16
3	55	38	73
4	83	83	59
5	53	91	80
6	140	122	1
7	72	60	47
8	40	31	47
9	0	20	12
10	0	19	92
6	11	L	
0	29	9	50
1	54	53	58
2	29	28	62
3	28	19	5
4	0	25	32
5	27	25	58
6	64	39	73
7	0	7	45
8	62	33	22
9	81	67	90
10	59	46	51
11	39	36	37
6	12	L	
0	30	9	0
1	36	20	50
2	41	41	60
3	40	24	47
4	73	61	0
5	59	39	14
6	93	78	43
7	32	27	96
8	25	23	82
7	0	L	
1	239	222	25
2	639	581	50
3	243	226	74
4	62	66	50
5	214	182	74
6	216	182	0
7	72	66	25
8	272	251	0
9	71	58	25
10	183	182	50
11	67	65	25
12	85	73	0
13	34	35	74
14	164	148	50
15	102	107	25
16	0	3	0
17	0	2	25
18	29	33	0
19	100	91	74
20	25	26	0
7	1	L	
0	253	310	74
1	162	126	19
2	94	91	5
3	291	300	79
4	271	242	99
5	375	346	49
6	177	162	40
7	147	123	15
8	87	92	7
9	135	123	78
10	208	193	95
11	148	149	29
12	141	136	46
13	139	147	92
14	79	65	22
15	87	86	29
16	0	5	20
17	82	86	80
18	0	21	88
19	30	46	27
20	0	21	40
7	2	L	
0	245	300	25
1	177	166	65
2	514	540	98
3	99	66	56
4	179	166	70
5	147	139	20
6	207	190	20
7	192	202	99
8	116	108	61
9	142	133	62
10	137	139	16
11	95	90	51
12	148	146	52
13	41	39	37
14	41	39	74
15	114	110	86
16	0	20	88
17	65	74	84
18	118	94	70
19	29	25	31
20	39	49	38
7	3	L	
0	178	199	25
1	174	183	86
2	197	230	68
3	240	264	25
4	215	211	48
5	187	181	16
6	117	121	6
7	163	170	71
8	129	121	17
9	79	59	36
10	146	140	31
11	140	117	73
12	99	91	0
13	200	197	30
14	82	69	68
15	40	52	90
16	0	14	58
17	0	26	96
7	4	L	
0	63	50	25
1	209	208	86
2	212	212	64
3	145	146	83
4	337	328	6
5	92	97	62
6	39	43	56
7	151	141	40
8	0	38	65
9	86	97	95
10	0	49	67
11	149	137	55
12	137	130	91
13	82	76	93
14	0	23	57
15	39	52	77
16	30	50	39
7	5	L	
0	0	29	74
1	130	146	80
2	149	181	20
3	175	180	49
4	137	132	77
5	0	9	74
6	134	125	58
7	92	93	19
8	95	81	36
9	32	28	94
10	74	59	19
11	24	27	57
12	82	77	52
13	129	122	77
14	0	5	39
15	0	27	5
16	0	19	19
17	75	56	49
7	6	L	
0	104	106	74
1	120	133	18
2	154	164	91
3	0	38	44
4	91	88	44
5	98	110	15
6	120	111	33
7	40	48	71

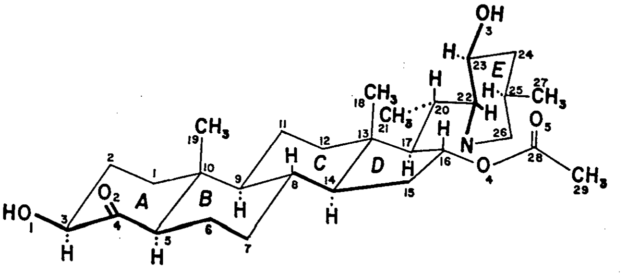
Table 2 (*cont.*)

8	113	113	75
9	100	93	61
10	104	115	15
11	123	136	5
12	93	97	54
13	71	58	38
14	0	13	92
7	7	L	
0	77	97	25
1	142	165	23
2	202	226	67
3	101	113	58
4	215	204	38
5	0	33	2
6	244	243	2
7	47	61	48
8	53	60	64
9	53	61	39
10	52	60	49
11	32	28	0
12	77	79	95
13	67	67	43
14	68	64	40
15	54	44	94
7	8	L	
0	94	68	74
1	126	133	72
2	178	179	41
3	71	96	11
4	88	91	12
5	75	65	41
6	131	127	87
7	0	7	60
8	99	99	26
9	40	40	18
10	63	70	71
11	57	54	63
12	58	65	28
13	0	10	1
14	42	35	72
7	9	L	
0	163	170	74
1	47	55	57
2	150	145	30
3	53	67	14
4	99	98	90
5	40	25	1
6	80	81	46
7	32	35	16
8	49	45	15
9	43	39	72
10	41	45	73
11	47	40	38
12	53	46	20
13	39	35	74
7	10	L	
0	118	97	74
1	55	59	17
2	78	80	10
3	0	24	45
4	0	21	29
5	48	53	92
6	67	77	44
7	0	33	72
8	83	83	89
9	45	39	41
10	79	54	37
11	33	31	81
7	11	L	
0	64	56	25
1	57	40	22
7	12	L	
0	60	55	25
1	56	37	83
2	44	32	69
3	50	39	24
8	0	L	
0	476	415	0
1	45	24	25
2	74	88	0
3	231	246	74
4	270	274	50
5	110	124	74
6	207	196	50
7	82	80	25
8	45	12	0
9	205	204	74
10	143	114	50
11	342	338	74
12	242	245	0
13	117	116	25
14	83	78	50
15	75	50	25
16	75	63	50
17	69	73	74
18	76	66	50
19	124	115	25
20	29	34	0
8	1	L	
0	84	89	0
1	69	80	40
2	390	404	47
3	82	71	17
4	335	331	73
5	210	201	98
6	303	295	28
7	253	228	34
8	93	89	60
9	530	515	80
10	165	141	90
11	370	367	24
12	47	55	38
13	38	37	80
14	48	54	67
15	22	31	82
16	31	58	30
17	32	26	10
18	45	55	72
19	36	24	81
20	0	24	58
8	2	L	
0	259	299	50
1	68	49	45
2	85	85	17
3	108	94	48
4	115	97	54
5	184	192	45
6	49	38	6
7	97	92	96
8	166	149	80
9	100	107	9
10	30	32	10
11	153	148	17
12	273	239	37
13	187	183	65
14	23	35	71
15	108	115	45
16	42	43	94
17	134	163	12
18	47	66	15
19	87	94	65
20	63	82	53
8	3	L	
0	30	59	50
1	92	100	71
2	161	202	75
3	138	127	38
4	177	168	23
5	66	72	14
6	194	199	84
7	176	166	5
8	50	45	73
9	222	219	23
10	131	132	30
11	265	271	78
12	64	53	19
13	79	54	26
14	71	72	3
15	65	66	15
16	81	87	74
17	0	18	30
18	0	25	29
19	0	13	95
8	4	L	
0	118	125	0
1	77	63	95
2	158	149	50
3	44	47	43
4	37	41	6
5	66	74	90
6	48	40	85
7	123	129	95
8	37	45	24
9	140	140	55
10	114	101	59
11	145	137	67
12	91	96	73
13	111	105	10
14	88	70	99
15	63	92	84
16	0	14	26
17	105	95	63
18	74	78	57
19	32	51	20
8	5	L	
0	0	15	0
1	93	80	14
2	101	109	53
3	87	99	57
4	116	109	30
5	82	83	24
6	84	63	81
7	80	73	56
8	88	81	21
9	32	38	65
10	136	140	72
11	194	201	21
12	45	45	41
13	116	103	86
14	79	87	36
15	0	13	74
16	0	16	81
17	0	33	85
18	36	37	71
8	6	L	
0	41	58	0
1	111	136	96
2	72	55	65
3	123	111	32
4	105	93	80
5	0	12	73
6	38	37	22
7	55	68	68
8	107	110	73
9	56	67	92
10	45	73	79
11	139	138	6
12	38	43	26
13	30	48	55
14	0	24	99
15	0	5	35
16	34	44	54
17	50	62	19
8	7	L	
0	118	123	50
1	70	67	49
2	104	93	83
3	31	45	85
4	55	64	41
5	145	141	25
6	0	18	52
7	123	133	80
8	85	90	93
9	122	121	42
10	77	77	16
11	0	10	5
12	46	49	69
13	0	18	27
14	0	11	34
15	34	40	50
16	23	27	82
8	8	L	
0	0	0	50
1	158	161	47
2	68	71	98
3	85	82	67
4	112	118	46
5	79	84	92
6	39	37	83
7	32	20	32
8	66	61	7
9	0	8	4
10	42	48	12
11	56	57	55
12	56	51	74
13	36	42	2
14	65	55	36
8	9	L	
0	0	25	50
1	101	112	93
2	64	46	50
3	77	63	26
4	38	36	78
5	44	54	75
6	53	47	42
7	63	58	38
8	29	18	34
9	79	76	99
10	40	34	47
11	30	27	45
8	10	L	
0	0	22	0
1	30	28	23
2	94	95	53
3	51	48	23
4	100	108	2
5	0	1	79
6	61	39	81
7	0	1	80
8	53	49	46
9	46	33	3
8	11	L	
0	0	1	50
1	95	78	56
2	59	40	23
3	87	55	8
4	0	5	45
5	0	25	41
6	56	37	96
8	12	L	
0	49	47	50
1	60	45	43
2	59	51	5
3	69	59	83
4	47	51	63
5	32	25	22
9	0	L	
1	223	201	74
2	257	232	0
3	71	73	25
4	209	195	0
5	133	107	25
6	193	194	50
7	27	17	74
8	28	20	0
9	190	187	25
10	31	33	50
11	233	230	74
12	25	48	50
13	35	20	74
14	67	65	0
15	0	24	74
16	21	32	50
17	87	92	25
18	62	64	0
19	81	100	74
9	1	L	
0	71	55	74
1	220	258	77
2	134	142	99
3	191	180	96
4	113	112	65
5	251	261	29
6	161	139	44
7	171	177	17
8	256	238	35
9	395	396	74
10	155	161	91
11	177	175	29
12	93	94	47
13	42	50	4
14	85	71	1
15	48	54	66
16	74	83	75
17	38	26	13
18	33	44	32
19	60	67	28
9	2	L	
0	25	23	25
1	145	164	14
2	172	180	60
3	63	49	93
4	145	165	35
5	173	157	44
6	113	118	97
7	94	78	37
8	251	266	75
9	262	281	81
10	129	125	16
11	230	211	27
12	46	47	85
13	56	65	10
14	78	71	36
15	37	39	47
16	29	25	15
17	64	64	71
18	68	83	56
19	59	89	23
9	3	L	
0	114	122	74
1	89	76	95
2	177	204	51
3	183	203	52
4	67	71	28
5	42	31	33
6	156	151	0
7	112	103	89
8	95	103	89
9	239	223	20
10	175	194	44
11	183	184	69
12	69	83	92
13	0	22	69
14	38	32	59
15	56	54	8
16	73	80	21
17	0	20	41
18	58	64	66
19	44	49	65
9	4	L	
0	26	39	25
1	37	54	81
2	89	89	90
3	71	58	58
4	73	73	59
5	70	71	40
6	62	48	55
7	210	226	4
8	109	108	9
9	169	162	31
10	23	35	5
11	140	134	68
12	79	80	42
13	63	44	32
14	61	56	63
15	54	50	20
16	39	28	61
17	28	25	24
18	61	75	98
9	5	L	
0	63	51	25
1	75	58	22
2	142	149	88
3	71	75	7
4	21	23	98
5	96	78	21
6	76	74	9
7	22	40	35
8	124	138	45
9	173	185	67
10	130	135	99
11	92	96	21
12	32	36	73
13	31	45	40
14	41	44	99
15	0	15	2
16	0	23	86
17	71	78	87
18	22	33	17
9	6	L	
0	53	44	25
1	76	78	50
2	116	132	51
3	82	97	96
4	168	157	4
5	108	109	18
6	32	43	10
7	0	7	34
8	122	130	67
9	173	186	71
10	46	60	32
11	89	92	33
12	53	55	89
13	20	12	91
14	0	13	98
15	47	40	71
16	0	15	41
17	28	51	89
9	7	L	
0	0	24	74
1	113	115	10
2	64	45	37
3	138	121	43
4	46	40	65
5	40	48	94
6	65	63	46
7	0	29	41
8	113	111	91
9	71	59	98
10	109	109	42
11	56	42	70
12	88	94	2
13	32	29	59
14	58	51	68
15	56	60	96
16	28	41	26
9	8	L	
0	140	130	74
1	40	40	89
2	140	149	9
3	40	63	33
4	40	51	66
5	125	109	84
6	112	112	67
7	121	123	57
8	87	85	23
9	150	166	19
10	48	59	70
11	94	82	78
12	68	55	18
9	9	L	
0	46	43	25
1	67	59	60
2	0	23	57
3	38	42	15
4	0	10	37
5	0	24	1
6	99	92	92
7	28	36	62
8	79	75	54
9	80	63	38
10	31	30	5
11	56	43	89
12	30	37	47
9	10	L	
0	29	14	25
1	61	36	85
2	50	51	68
3	35	27	52
4	74	60	30
5	72	57	34
6	111	90	9
7	43	41	4
8	77	59	70
9	27	33	63
9	11	L	
0	21	20	25
1	0	15	17
2	72	42	56
3	35	31	76
4	34	29	0
5	33	28	63
6	25	28	50
7	81	62	30
8	54	42	9
10	0	L	
0	42	31	0
1	44	41	74
2	135	138	50
3	210	212	25
4	82	51	0
5	181	159	74
6	149	135	0
7	107	96	25
8	65	56	0
9	101	98	25
10	91	78	0
11	36	6	25
12	199	183	50
13	0	2	25
14	46	58	0
15	35	31	74
16	0	22	0
17	64	58	74
18	0	12	0
10	1	L	
0	54	51	0
1	104	113	28
2	142	136	89
3	162	190	85
4	82	75	25
5	113	107	59
6	111	101	9
7	104	81	41
8	101	87	83
9	165	146	7
10	114	142	61
11	83	78	55
12	119	121	22
13	44	39	93
14	32	45	13
15	22	23	91
16	40	45	56
17	0	15	48
18	74	82	3
10	2	L	
0	84	70	0
1	214	255	5
2	76	65	36
3	180	201	73
4	118	108	59
5	111	87	11
6	125	122	16
7	60	64	39
8	132	126	99
9	150	140	64
10	228	221	66
11	90	96	81
12	96	92	21
13	39	32	88
14	61	56	74
15	0	22	6
16	32	42	95
17	29	45	27
18	29	38	38
10	3	L	
0	153	176	0
1	85	84	52
2	126	131	45
3	122	118	45
4	117	107	65
5	77	69	28
6	70	75	31
7	214	227	70
8	0	22	42
9	156	166	31
10	84	83	0
11	121	109	4
12	71	61	65
13	69	65	59
14	0	21	40
15	0	7	30
16	76	70	26
17	0	7	32
18	83	96	58
10	4	L	
0	80	76	50
1	0	33	3
2	135	117	90
3	78	76	25
4	49	30	64
5	71	57	73
6	37	28	39
7	116	128	87
8	77	84	75
9	154	155	45
10	118	116	19
11	121	130	8
12	66	61	98
13	0	20	47
14	0	28	18
15	0	19	46
16	77	88	44
10	5	L	
0	220	228	50
1	134	146	99
2	0	45	93
3	42	36	53
4	0	9	61
5	53	54	68
6	0	24	99
7	146	136	15
8	121	118	94
9	160	173	74
10	90	99	41
11	77	60	38
12	0	13	36
13	35	50	22
14	0	19	13
15	37	42	50
16	32	50	71
10	6	L	
0	65	70	50
1	120	130	56
2	76	85	20
3	49	48	40
4	67	69	74
5	100	105	6
6	50	69	24
7	39	45	34
8	39	61	18
9	109	108	5
10	76	72	75
11	150	152	59
12	86	85	24
13	33	40	97
14	73	76	60
15	0	15	87
16	68	85	0
10	7	L	
0	0	45	0
1	96	93	65
2	39	46	78
3	71	65	26
4	85	58	34
5	101	86	14
6	96	92	72
7	127	127	74
8	44	61	36
9	80	97	38
10	0	19	29
11	74	74	4
12	39	48	90
13	0	18	83
14	26	37	44
15	21	28	85
10	8	L	
0	128	101	0
1	78	58	28
2	95	79	54
3	67	54	97
4	70	62	13
5	0	38	34
6	61	50	84
7	73	75	93
8	65	64	64
9	84	100	57
10	40	53	19
11	107	82	24
12	104	102	69
13	34	33	87
14	90	103	10
10	9	L	
0	60	40	50
1	52	42	7
2	21	7	88
3	36	39	71
4	46	35	96
5	61	53	58
6	0	28	32
7	57	52	20
8	63	45	1
9	78	64	82
10	67	59	67
11	49	33	35
12	29	41	40
10	10	L	
0	60	29	0
1	0	14	80
2	69	41	76
3	0	17	67
4	38	30	39
5	43	47	91
6	58	31	68
7	78	66	48
8	51	47	27
9	46	41	12
10	27	28	87
11	0	L	
1	55	31	25
2	401	388	0
3	201	233	74
4	92	86	50
5	304	264	25
6	161	147	50
7	109	111	74
8	49	54	0
9	43	35	74
10	88	99	0
11	0	29	74
12	87	84	50
13	146	141	25
14	0	21	50
15	36	26	74
16	33	16	0
17	106	83	74
11	1	L	
0	124	115	74
1	215	215	29
2	282	289	44
3	136	142	27
4	390	366	99
5	52	34	69
6	46	31	44
7	190	195	83
8	130	111	33
9	62	65	42
10	54	62	81
11	50	52	37
12	0	4	2
13	0	29	11
14	59	51	18
15	29	29	76
16	73	68	51
11	2	L	
0	0	20	74
1	97	108	75
2	212	230	54
3	196	216	18
4	160	158	17
5	139	144	60
6	92	76	8
7	204	193	7
8	79	84	72
9	108	105	59
10	97	104	33
11	52	41	39
12	0	11	89
13	100	105	82
11	3	L	
0	186	207	25
1	154	148	93
2	157	147	94
3	75	86	70
4	156	150	56
5	66	65	45
6	83	86	92
7	93	107	26
8	62	43	29
9	97	93	84
10	74	96	42
11	90	88	41
11	4	L	
0	0	18	74
1	120	139	29
2	171	189	10
3	174	165	84
4	68	54	93
5	129	152	22
6	122	113	61
7	72	76	58
8	73	63	70
9	104	96	84
10	0	18	70
11	95	87	66
12	49	50	27
13	88	85	7
11	5	L	
0	138	156	74
1	0	38	60
2	203	218	45
3	0	33	50
4	71	90	4
5	62	47	14
11	6	L	
0	0	17	25
1	190	207	86
2	103	102	58
3	103	110	37
4	0	36	19
5	137	150	63
6	0	35	97
7	90	95	14
11	7	L	
0	164	185	25
1	0	25	14
2	181	194	88
12	0	L	
0	270	283	0
1	0	24	74
2	375	364	50
3	144	185	25
4	155	147	0
5	255	239	74
6	0	20	0
7	0	11	74
8	36	35	0
9	50	34	74
10	0	2	0
11	91	74	74
12	82	83	50
13	100	76	25
14	59	66	0
12	1	L	
0	135	120	50
1	171	184	74
2	198	205	85
3	155	174	28
4	380	389	50
5	65	52	16
6	257	272	6
7	94	98	30
8	50	68	94
9	87	73	78
10	133	112	53
11	42	39	62
12	0	25	28
13	0	20	40
14	46	53	98
15	46	44	98
13	0	L	
1	111	108	74
2	226	197	0
3	93	104	25
4	214	211	50
5	50	53	25
6	50	33	0
11	65	52	74
13	1	L	
0	35	20	74
1	130	140	93
2	195	208	42
3	106	130	41
4	94	76	77
5	190	223	72
6	106	100	75
7	122	119	20
8	93	78	28
9	91	90	25
10	24	16	96
14	0	L	
0	178	180	0
1	100	111	74
2	0	15	50
3	100	86	25
4	94	96	50
5	0	25	25
6	129	125	0
7	0	46	25
8	0	30	50
9	93	49	74
10	39	45	50
11	56	56	25
14	1	L	
0	122	111	0
1	131	111	62
2	0	39	2
3	150	189	21
4	0	16	60
5	158	162	78
6	99	92	48
7	24	41	40
8	48	54	85
9	46	41	86
10	89	75	5
11	42	26	26
12	126	99	48
13	38	34	14
14	39	30	30
15	93	101	66
15	0	L	
1	208	194	74
2	49	46	50
3	0	0	25
4	0	33	50
5	42	43	25
6	58	68	0
7	0	28	74
8	50	39	0
9	0	10	74
10	43	45	50
11	33	38	74
12	113	93	0
13	151	140	25
14	76	70	50
15	1	L	
0	74	50	74
1	49	49	92
2	49	50	0
3	60	70	41
4	34	38	19
5	0	19	67
6	0	6	45
7	0	93	73
8	31	30	3
9	30	35	91
10	35	6	6
11	72	38	34
12	65	51	57
13	57	48	69
14	49	51	3
16	0	L	
0	173	162	0
1	0	13	25
2	104	81	50
10	113	87	0
11	54	26	74
16	1	L	
0	33	38	50
1	0	8	96
2	61	46	57
3	0	18	38
4	64	72	7
5	0	6	18
6	64	45	80
7	62	73	71
8	144	120	46
9	72	72	27
10	134	125	97
11	27	25	7
17	0	L	
1	64	37	25
2	0	1	0
3	0	18	74
4	35	23	50
5	48	52	25
6	61	53	0
7	70	42	74
8	111	85	50
9	70	46	25
10	28	30	50
17	1	L	
0	21	19	74
1	21	23	76
2	0	2	50
3	0	13	89
4	0	19	19
5	0	19	43
6	102	67	51
7	0	17	15
8	144	121	96
9	49	46	79
10	151	143	47
18	0	L	
7	77	56	74
18	1	L	
0	44	23	50

heavy atoms resulted in an R index of 9·9 %. Observed and calculated structure factors are listed in Table 2.

Discussion

The X-ray analysis has shown the structural formula and the stereoconfiguration of solaphyllidine to be



A three-dimensional view of the molecule may be obtained from the stereodrawings in Fig. 1. The absolute configuration has not been established (the presence of a heavy atom is normally required to determine the absolute configuration from anomalous dispersion studies) but is assumed to be the same as that determined for cholesterol (see *e.g.* Fieser & Fieser, 1959).

The molecule is completely extended. The *A*/*B*, *B*/*C* and *C*/*D* ring junctions are all *trans* and the C(3)· · · C(16) distance, a measure of the length of the steroid nucleus, is 9·11 Å. The C(3)· · ·C(16) distance is 9·02 Å in androsterone (High & Kraut, 1966), a steroid with a comparable nucleus, *i.e.* with saturated rings and all *trans* ring-junctions, but the distance is only 7·25 Å in digitoxigenin (Karle & Karle, 1969*b*), where the *A*/*B* and *C*/*D* ring junctions are *cis*. Rings *A*, *B*, *C* and *E* are in the chair conformation even though ring *A* is a substituted cyclohexanone. Ring *D* has the *β*-envelope con-

Table 3. *Least-squares planes*

		Plane	Maximum deviation
(I)	C(1)–C(17)	$1{\cdot}1465x+10{\cdot}5069y-4{\cdot}0338z=1{\cdot}5699$	0·40 Å
(II)	C(14)–C(17)	$1{\cdot}7998x+10{\cdot}2167y-5{\cdot}4257z=1{\cdot}0134$	0·06
(III)	C(2), C(3), C(5), C(10), C(7), C(8)	$-2{\cdot}6137x+10{\cdot}6592y-0{\cdot}7682z=2{\cdot}7330$	0·11
(IV)	C(5), C(6), C(8), C(9), C(12), C(13)	$-0{\cdot}1673x+9{\cdot}4536y-8{\cdot}5357z=0{\cdot}4323$	0·02
(V)	C(22), C(24), C(25), N	$-5{\cdot}4512x-0{\cdot}5474y+16{\cdot}3198z=2{\cdot}5625$	0·02
(VI)	C(2), C(1), C(10), C(8), C(14), C(15)	$6{\cdot}5160x+7{\cdot}7035y-9{\cdot}7666z=-0{\cdot}3746$	0·18

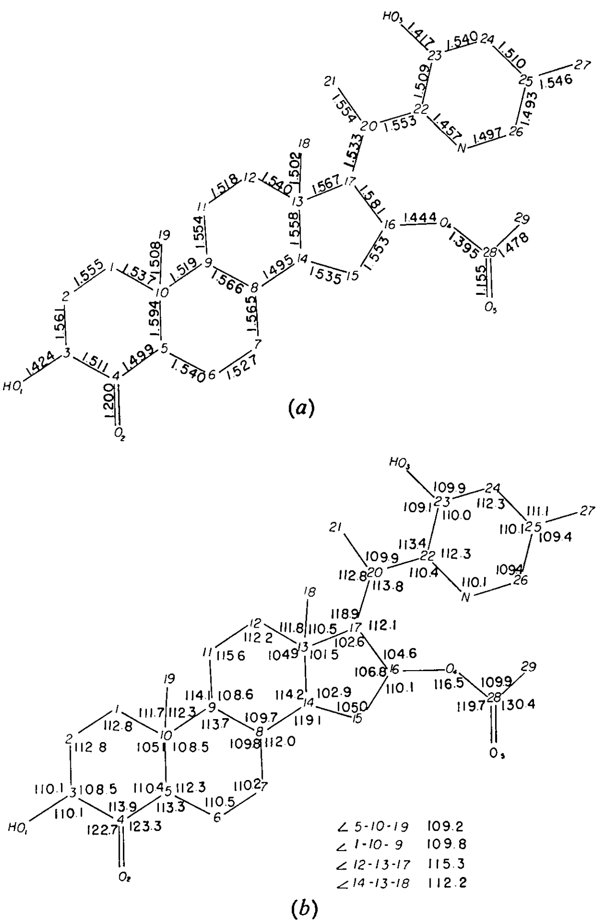


Fig. 2. (*a*) Bond lengths; (*b*) bond angles.

Fig. 3. Packing diagram for solaphyllidine. Hydrogen bonds are indicated by the lighter lines. The axes are *a* ↑, *c* → and *b* up out of the plane of the paper.

formation with atom C(13) 0·68 Å out of the plane of the other four atoms. The torsion angle about the C(13)–C(14) bond with respect to ring *D* is 46° 20′, comparable with an average value of ∼45° for various steroids with *trans C*/*D* ring junctions (Brutcher & Leopold, 1966).

The plane of the acetyl group at 16α is oriented approximately perpendicular, 79°, to the average plane (Table 3) through the steroid nucleus. Ring *E* is also nearly perpendicular, 73°, to the steroid nucleus and oriented so that the OH group is directed upwards and the N atom downwards.

Least-squares planes computed for various portions of the molecule are listed in Table 3. Coordinates from Table 4 may be substituted directly into the equations in Table 3 to obtain the deviations of atoms from these planes. The values on the right side of the equations are the origin-to-plane distances in Å units. Deviations of atoms C(1), C(4), C(6) and C(9) from plane III, atoms C(7), C(10), C(11) and C(14) from plane V, and atoms C(23) and C(26) from plane V are all in the range of 0·60 to 0·73 Å. These values along with the torsional angles listed in Table 5 indicate that rings *A*, *B*, *C* and *E* have nearly ideal chair conformations. Plane VI characterizes seven atoms forming a zigzag chain from ring *A* to ring *D*. The systematic deviations of atoms from this plane indicate that the steroid nucleus is bowed slightly with an upward bulge near atom C(10).

Fractional coordinates and thermal parameters are listed in Table 4. Values for the bond lengths and angles are shown in Fig. 2. The standard deviations based solely on the least-squares refinement are 0·011–0·013 Å for the bond lengths, except for the CH_3OH molecule, C–O = 1·376 Å, where the standard deviation is 0·017 Å. The standard deviations for the angles are of the order of 0·7°. There are no unusual bond lengths. The average of 28 saturated C–C single bonds is 1·540 Å.

The packing of the molecules in a unit-cell is illustrated in Fig. 3. The average plane of the steroid nucleus is nearly *parallel* to the *ac* plane of the cell. However, hydrogen bonding between molecules creates a layered structure with the layers *perpendicular* to the *ac* plane. Four independent hydrogen bonds, O(1)H···O(M) at 2·72 Å, O(M)H···O(3) at 2·77 Å, O(3)H···N at 2·90 Å, and NH···O(2) at 3·03 Å, labelled *A*, *B*, *C*, *D* respectively in Fig. 4, tie together four different steroid molecules and one molecule of methanol, which has cocrystallized with the steroid. A repetition of the hydro-

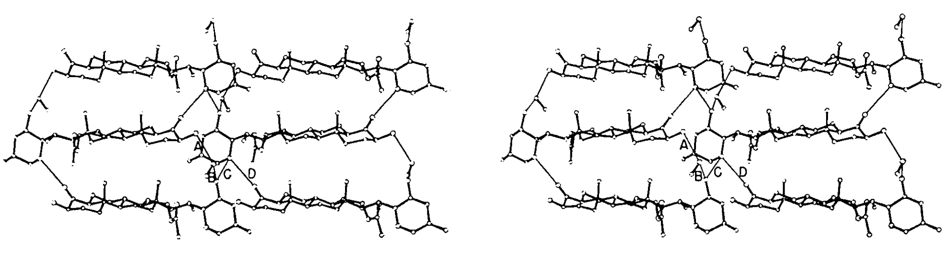


Fig. 4. A section of an infinite sheet formed by steroid and CH_3OH molecules linked together by four independent hydrogen bonds, labelled *A*, *B*, *C* and *D*. The molecules can be referred to the axes *a* →, *b* ↑ and *c* up.

gen bonding in each unit-cell forms infinite sheets of molecules.

There is no hydrogen bonding between layers. The nearest approaches between atoms in different layers are O(5)···C(27) at 3·26 and C(12)···C(29) at 3·48 Å where O(5) and C(29) are part of the acetyl group. Within the same layer, the nearest intermolecular approach, O(5)···C(M) at 3·07 Å also involves the keto oxygen atom of the acetyl group. Except for O(M)···C(3) at 3·31 Å and O(3)···C(26) at 3·36 Å, both of which are adjacent to hydrogen bonds, all other intermolecular separations are greater than 3·4 Å for C···O approaches and greater than 3·6 Å for C···C approaches.

In the natural steroidal alkaloid, it is interesting to note that the substituents at C(3), C(16), C(17), C(23) and C(25) are all in the equatorial positions.

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Table 4. *Fractional atomic coordinates and thermal parameters for* $C_{29}H_{47}NO_5.CH_3OH$

Thermal parameters are of the form

$$T=\exp\left[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)\;(\times 10^{-4})\right].$$

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	B*
O(1)	−0·3788	0·2398	0·0935	38	164	54	23	−13	1	5·8
O(2)	−0·3046	0·3603	0·2097	41	131	44	24	1	−3	4·8
O(3)	0·4866	0·4024	0·3066	66	91	27	−14	−3	2	4·2
O(4)	0·2528	0·1327	0·4016	69	108	17	−10	−8	2	3·9
O(5)	0·2797	0·2513	0·5016	170	132	25	−4	−20	−19	6·7
C(1)	−0·1335	0·1650	0·0854	52	126	30	10	−5	−16	4·5
C(2)	−0·2275	0·1942	0·0560	33	164	38	21	−6	−19	5·1
C(3)	−0·2974	0·1934	0·1212	38	99	45	3	−12	−1	4·5
C(4)	−0·2644	0·2732	0·1859	39	79	37	14	3	11	4·1
C(5)	−0·1757	0·2369	0·2151	30	89	30	−1	1	3	3·5
C(6)	−0·1497	0·3043	0·2892	34	166	33	12	7	−23	4·8
C(7)	−0·0618	0·2565	0·3186	30	174	30	17	10	0	4·2
C(8)	0·0112	0·2760	0·2570	32	92	21	−4	2	−9	3·0
C(9)	−0·0158	0·2102	0·1808	32	75	22	7	0	−3	3·0
C(10)	−0·1039	0·2523	0·1496	32	77	24	0	2	−4	2·9
C(11)	0·0611	0·2216	0·1230	36	138	20	17	4	−3	3·6
C(12)	0·1507	0·1850	0·1530	36	129	18	12	3	−12	3·5
C(13)	0·1750	0·2552	0·2266	31	54	23	0	1	−1	2·7
C(14)	0·0983	0·2285	0·2830	29	76	19	−4	0	−7	2·7
C(15)	0·1351	0·2723	0·3600	39	139	18	9	0	−6	3·7
C(16)	0·2345	0·2416	0·3567	57	75	20	7	5	−3	3·4
C(17)	0·2553	0·2021	0·2716	39	68	15	2	−2	−2	2·6
C(18)	0·1867	0·3910	0·2121	45	80	48	6	4	23	4·3
C(19)	−0·1022	0·3849	0·1235	36	127	34	18	3	18	4·1
C(20)	0·3481	0·2397	0·2472	34	88	20	−3	−1	−3	3·0
C(12)	0·3727	0·1918	0·1662	34	148	24	7	2	−9	4·2
C(22)	0·4205	0·2029	0·3054	25	100	24	0	2	1	3·2
C(23)	0·5044	0·2752	0·2960	39	95	27	4	−4	−2	3·5
C(24)	0·5727	0·2316	0·3550	32	127	25	−17	−8	2	3·3
C(25)	0·5827	0·0930	0·3559	36	145	25	10	−7	−6	3·9
C(26)	0·4954	0·0331	0·3672	37	109	33	9	−10	7	3·8
N	0·4352	0·0700	0·3036	43	85	23	0	−2	4	3·4
C(27)	0·6458	0·0514	0·4201	73	143	38	12	−15	−3	5·7
C(28)	0·2761	0·1519	0·4778	94	114	18	−6	−6	0	5·4
C(29)	0·2886	0·0319	0·5165	170	144	28	−11	−30	21	7·1
C(M)	0·4272	0·4447	0·4944	163	189	47	−38	16	−12	8·2
O(M)	0·4634	0·5266	0·4432	136	161	38	23	3	2	7·4
Standard deviations										
O(1–5)	0·0004	0·0007	0·0004	4	8	3	6	2	4	0·3
C(1–29)	0·0006	0·0009	0·0005	5	8	3	6	3	4	0·3
O(M)	0·0007	0·0008	0·0004	8	10	3	8	4	5	0·5
C(M)	0·0012	0·0014	0·0007	14	18	5	15	7	8	0·5

* Values of the thermal parameters from the isotropic refinement.

Table 5. *Torsional angles in the rings**

Ring *A*		Ring *B*		Ring *C*		Ring *D*		Ring *E*	
Bond	φ_{A-B}	Bond	φ_{A-B}	Bond	φ_{A-B}	Bond	φ_{A-B}	Bond	φ_{A-B}
1—2	55·6°	5—6	58·1°	8—9	51·8°	13–14	46·3°	N–22	60·3°
2—3	50·1	6—7	58·8	9–11	49·8	14–15	34·7	22–23	53·1
3—4	55·2	7—8	57·0	11–12	54·3	15–16	9·9	23–24	49·8
4—5	62·8	8—9	56·4	12–13	55·7	16–17	18·5	24–25	54·0
5–10	60·6	9–10	54·0	13–14	62·7	17–13	39·2	25–26	59·6
10—1	57·7	10—5	54·6	14—8	62·7			26–N	63·0

* φ_{A-B} is the torsional angle about the *A*-*B* bond, in which the other two atoms required to define the bond are those attached to either end of the bond and are in the ring in question.

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The Crystal Structure of Hexabarium 17-Titanate

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The crystal structure and the chemical composition of $Ba_6Ti_{17}O_{40}$, a new compound in the system $BaO–TiO_2$, have been established by single-crystal methods. Hexabarium 17-titanate crystallizes in space group $C2/c$ with $a=9·883$, $b=17·08$, $c=18·92$ Å, $\beta=98°42'$, $Z=4$. The 2094 observed F_{hkl} were used in a combination of Patterson and Fourier methods to determine the crystal structure and to refine it to $R=0·06$. The structure can be described as a three-dimensional framework of TiO_6 coordination octahedra. The most prominent features of this framework are ribbons of octahedra of composition Ti_3O_{10} which extend in the $[\bar{1}10]$ direction at height $z=0$, and in the [110] direction at $z=\frac{1}{2}$. The ribbons are joined by clusters of octahedra of composition $Ti_{11}O_{40}$. The voids in this framework are filled by the Ba atoms. An alternate way of looking at the crystal structure is to regard it as a hexagonally close-packed array of oxygen and barium atoms with the titanium atoms filling the energetically most favorable octahedral voids. The direction of packing is [103] and the stacking sequence is *ABCACABCBCABABCACABCBCAB*. $Ba_6Ti_{17}O_{40}$ is another example in the short list of titanates which can be described as close-packed arrays, while most belong to the Wadsley–Anderson type phases with one cell edge of about 4 Å. The details of the atomic arrangement show appreciable distortions of the bond lengths and angles. The individual bond distances Ti–O vary from 1·77 to 2·43 Å. The mean of all Ti–O bond lengths formed by one oxygen atom can be predicted by using the expression $d_{Ti-O}=(1·552+0·200\zeta)$ Å, where ζ is the sum of the electrostatic valences received by this oxygen atom. This is in accord with the extended electrostatic valence rule.

Introduction

Compounds in the system $BaO–TiO_2$ have been extensively studied in recent years. So far the crystal structures of Ba_2TiO_4 (Bland, 1961), cubic $BaTiO_3$ (Megaw, 1947), hexagonal $BaTiO_3$ (Burbank & Evans, 1948), $BaTi_2O_5$ (Harrison, 1956), $BaTi_4O_9$ (Lukaszewicz, 1957) and $BaTi_5O_{11}$ (Tillmanns, 1969) have been described. Rase & Roy (1955) report another compound in this system of composition $BaTi_3O_7$ and two more are mentioned by Kwestroo (1960): $Ba_2Ti_5O_{12}$ and $Ba_2Ti_9O_{20}$. The X-ray powder patterns for these three compounds are not indexed and single-crystal work has not been undertaken.