The Structure of L-Mimosine, an L-DOPA Analogue

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The crystal structure of L-mimosine, β-N-(3-hydroxy-4-pyridone)-
α-amino propionic acid, has been determined by X-ray methods using
1729 observed reflections collected by counter methods. The crystals
are orthorhombic, space group $P2\,2_12_1$, with unit cell dimensions
$a = 20.45_3$ Å; $b = 9.48_5$ Å; $c = 8.50_0$ Å. The asymmetric unit consists
of two independent molecules. The refinements yielded a conventional
R-factor of 0.064; estimated standard deviations are about 0.005 Å
in bond lengths and 0.3° in angles. The conformation of the two non-
equivalent molecules is nearly identical and is similar to that found in
L-DOPA. The pyridone ring has a pronounced quinoid character.

Mimosine, β-N-(3-hydroxy-4-pyridone)-α-amino propionic acid, leucenol, is
a naturally occurring amino acid found in certain plants.[1] Its notable bio-
logical properties have been described by several authors and summarized
by Owen.[2] Hegarthy et al.[3] point out that inhibition of mitosis in the hair fol-
licle is the reason for hair loss in sheep by mimosine intoxication, Hylin and
Lichton [4] report the loss of fertility in female rats by ingestion of mimosine,
and Prabhakaran et al.[5] found that injection of the compound inhibit the
development of melanoma in mice.

Thompson et al.[1] have reviewed various theories for the mechanisms of the
toxic action of mimosine in animals. One of these depends on the structural
similarity between mimosine and DOPA; mimosine may thus interact with
the enzymes concerned with the DOPA metabolism.

The chemical formula for mimosine is usually written as I.
Several authors [6—8] have, however, indicated II as an important contributor
to the electronic structure. It may be seen that III, the protonized form of II,
is isoelectronic with DOPA.

The absolute configuration of L-mimosine has been investigated by chem-
ical methods [9] and shown to belong to the S-series.

After the publication of the X-ray structure analysis of L-DOPA,[10] Dr.
Hegarthy pointed out for us the analogy between this compound and mimosine
and suggested a structure analysis of the latter compound; he also kindly
supplied a sample of pure L-mimosine.

I   II   III   DOPA
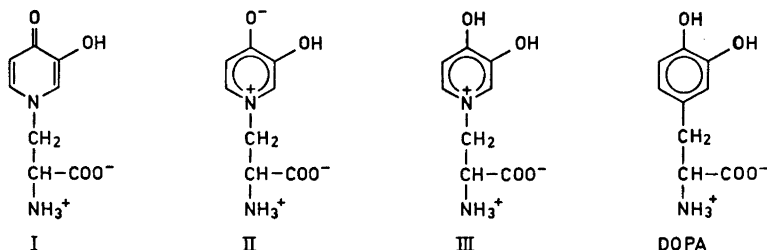
EXPERIMENTAL

A hot saturated solution of L-mimosine was cooled to room temperature during a period of 2 – 3 days. Single crystals in the form of thin, needle-formed very brittle plates separated; a crystal roughly cut to the dimensions $0.13 \times 0.21 \times 0.06$ mm^3 was used for the X-ray experiments.

Oscillation and Weissenberg photographs indicated orthorhombic symmetry; systematically absent reflections determined the space group to be $P2_12_12_1$.

Unit cell parameters were determined from diffractometer measurements on 62 reflections using a manual Picker diffractometer with a copper tube ($\lambda_\beta = 1.3922$ Å, $\lambda_{\alpha_1 + \alpha_2} = 1.5418$ Å). The take-off angle was 1.0°. The computer program employed in the least-squares calculation of cell parameters as well as the programs applied during the structure investigation are described in Ref. 11.

Three-dimensional intensity data were recorded on an automatic Picker four-angle diffractometer using graphite crystal monochromated MoK radiation. The take-off angle was 4°. Intensities of 2640 reflections with $2\theta < 60°$ were measured using the $\omega - 2\theta$ scanning mode with a 2θ scanning speed of 1° min^{-1}. Background counts were taken for 20 sec at each of the scan range limits. Three standard reflections measured after every 100 reflections showed a variation of up to 10 % during the run; the intensity data were accordingly adjusted. Estimated standard deviations in the intensities were taken as the square root of the total counts with a 2.5 % addition to allow for the uncertainty in the adjustments.

The 1729 reflections with intensity larger than 2σ (I) were considered to be observed; the rest were regarded as unobserved and excluded from the refinement procedure. Lorentz and polarization corrections were applied to the intensity data.

Atomic form factors used were those of Hanson et al.[12] for oxygen, nitrogen, and carbon, and of Stewart et al.[13] for hydrogen.

CRYSTAL DATA

S-β-N-(3-hydroxy-4-pyridone)-α-amino propionic acid (L-mimosine, leucenol), $C_8H_{10}N_2O_4$, orthorhombic. $a = 20.453(0.002)$ Å, $b = 9.485(0.001)$ Å; $c = 8.500(0.001)$ Å. Figures in parentheses are estimated standard deviations. $V = 1648.9$ Å^3; $M = 198.18$; F (000) $= 832$; $Z = 8$; $D_{obs} = 1.59$ g cm^{-3} (flotation); $D_{calc} = 1.595$ g cm^{-3}. Absent reflections: ($h00$) for h odd, ($0k0$) for k odd, ($00l$) for l odd; space group $P2_12_12_1$.

STRUCTURE DETERMINATION

The data were put on an absolute scale by Wilson's statistical method and normalized structure amplitudes were calculated. Eight sets of phases for 250 structure amplitudes were obtained by the application of the symbolic

Table 1. Observed and calculated structure factors. The columns are h, k, l, $10\,F_o$, and $10|F_c|$.

h	k	l	10Fo	10Fc
2	0	0	257	266
4	0	0	666	675
6	0	0	787	813
8	0	0	655	657
10	0	0	275	273
12	0	0	610	626
14	0	0	384	395
16	0	0	506	540
18	0	0	1045	1137
20	0	0	89	92
22	0	0	183	185
24	0	0	176	166
26	0	0	143	173
28	0	0	172	177
27	1	0	68	52
22	1	0	126	115
20	1	0	162	170
19	1	0	221	225
17	1	0	155	158
16	1	0	213	235
15	1	0	292	312
14	1	0	95	105
13	1	0	166	177
12	1	0	181	180
10	1	0	382	381
8	1	0	303	321
7	1	0	87	83
6	1	0	65	81
5	1	0	48	29
4	1	0	292	313
2	1	0	223	228
1	1	0	83	124
0	2	0	1298	1383
1	2	0	52	50
2	2	0	337	366
3	2	0	69	63
4	2	0	129	133
5	2	0	44	34
6	2	0	81	70
7	2	0	90	88
8	2	0	336	341
9	2	0	44	56
10	2	0	354	359
11	2	0	64	70
12	2	0	70	84
13	2	0	84	91
14	2	0	126	127
15	2	0	163	173
16	2	0	214	232
18	2	0	350	364
19	2	0	185	200
20	2	0	76	73
21	2	0	79	90
22	2	0	104	114
23	2	0	93	74
26	2	0	100	101
27	2	0	100	76
26	3	0	175	164
24	3	0	72	65
21	3	0	154	157
19	3	0	359	381
18	3	0	100	104
17	3	0	194	207
15	3	0	261	272
14	3	0	318	338
13	3	0	64	56
12	3	0	298	305
11	3	0	332	351
10	3	0	548	531
9	3	0	246	256
8	3	0	546	552
7	3	0	107	107
6	3	0	683	688
5	3	0	160	164
4	3	0	152	150
3	3	0	473	487
2	3	0	467	480
1	3	0	593	591
0	4	0	92	99
1	4	0	45	39
2	4	0	569	583
3	4	0	146	143
4	4	0	388	391
5	4	0	231	226
6	4	0	261	270
7	4	0	210	218
8	4	0	129	123
9	4	0	376	391
10	4	0	157	159
11	4	0	150	148
12	4	0	112	111
13	4	0	163	166
14	4	0	309	330
15	4	0	49	80
16	4	0	320	339
17	4	0	85	103
18	4	0	129	132
20	4	0	140	141
21	4	0	71	88
22	4	0	71	61
23	4	0	61	47
25	4	0	75	58
26	4	0	124	125
27	4	0	90	75
25	5	0	94	95
23	5	0	76	65
21	5	0	104	122
17	5	0	175	186
16	5	0	69	57
15	5	0	86	83
14	5	0	164	180
12	5	0	68	84
11	5	0	75	81
10	5	0	334	345
9	5	0	46	61
8	5	0	84	81
7	5	0	216	228
6	5	0	223	232
4	5	0	257	261
3	5	0	357	365
2	5	0	297	313
1	5	0	502	531
0	6	0	924	993
2	6	0	380	388
3	6	0	255	270
5	6	0	146	151
6	6	0	194	206
7	6	0	57	41
8	6	0	100	108
9	6	0	142	145
10	6	0	105	113
11	6	0	145	167
12	6	0	129	143
13	6	0	153	168
15	6	0	145	156
16	6	0	253	267
18	6	0	204	220
19	6	0	124	121
20	6	0	119	128
24	6	0	107	105
20	7	0	61	101
19	7	0	181	182
16	7	0	87	91
15	7	0	161	166
14	7	0	65	67
13	7	0	150	158
12	7	0	75	91
11	7	0	126	137
10	7	0	58	49
9	7	0	93	101
8	7	0	83	90
7	7	0	53	62
6	7	0	57	62
5	7	0	83	78
4	7	0	53	56
3	7	0	215	214
2	7	0	63	61
1	7	0	379	404
0	8	0	70	81
2	8	0	62	59
4	8	0	94	97
6	8	0	109	117
7	8	0	94	83
9	8	0	56	49
8	8	0	101	105
12	8	0	88	89
14	8	0	62	63
16	8	0	91	98
17	8	0	58	62
18	8	0	140	127
20	9	0	70	44
18	9	0	83	84
17	9	0	122	115
14	9	0	87	88
12	9	0	179	180
11	9	0	60	55
10	9	0	311	326
9	9	0	83	104
8	9	0	133	131
7	9	0	66	37
6	9	0	296	305
3	9	0	66	72
2	9	0	119	113
1	9	0	162	173
0	10	0	71	85
2	10	0	63	75
6	10	0	110	98
8	10	0	58	39
9	10	0	157	160
10	10	0	141	156
13	10	0	115	122
15	10	0	61	77
18	10	0	79	94
15	11	0	92	87
13	11	0	104	93
8	11	0	63	57
7	11	1	59	71
5	11	1	91	89
3	11	1	78	78
1	11	1	95	115
0	12	0	107	90
6	12	0	96	88
9	12	0	71	58
4	13	0	101	88
11	12	1	141	133
10	12	1	81	52
9	12	1	115	115
7	12	1	88	97
6	12	1	110	116
0	12	1	73	53
2	11	1	84	76
3	11	1	97	76
6	11	1	115	132
7	11	1	84	88
8	11	1	274	286
10	11	1	148	198
11	11	1	130	129
15	11	1	69	61
16	10	1	113	110
11	10	1	126	125
10	10	1	82	83
9	10	1	131	140
8	10	1	121	103
7	10	1	158	167
6	10	1	120	131
4	10	1	76	83
0	10	1	56	48
1	9	1	135	131
2	9	1	261	267
2	9	1	123	123
3	9	1	165	161
4	9	1	136	130
5	9	1	89	81
6	9	1	136	136
7	9	1	123	116
8	9	1	277	292
10	9	1	147	153
12	9	1	79	77
13	9	1	75	63
14	9	1	81	69
15	9	1	66	72
16	9	1	66	80
17	9	1	119	105
18	9	1	82	78
19	9	1	88	71
22	8	1	79	77
18	8	1	115	120
17	8	1	90	101
16	8	1	177	185
15	8	1	91	86
14	8	1	75	62
13	8	1	62	78
12	8	1	52	41
11	8	1	143	154
9	8	1	87	99
8	8	1	64	63
7	8	1	80	84
6	8	1	103	106
5	8	1	93	97
3	8	1	147	145
2	8	1	50	67
1	8	1	125	132
0	8	1	383	405
0	7	1	46	41
1	7	1	192	200
2	7	1	134	142
3	7	1	73	63
4	7	1	84	93
6	7	1	93	97
7	7	1	74	67
8	7	1	213	218
9	7	1	145	147
10	7	1	163	168
11	7	1	90	90
12	7	1	103	105
13	7	1	83	96
14	7	1	76	72
15	7	1	86	97
16	7	1	123	134
17	7	1	63	65
25	6	1	114	95
24	6	1	71	78
21	6	1	78	73
20	6	1	127	125
19	6	1	74	72
18	6	1	185	185
17	6	1	86	85
16	6	1	94	96
15	6	1	121	128
14	6	1	55	66
13	6	1	83	88
12	6	1	96	97
11	6	1	246	252
10	6	1	159	163
9	6	1	156	158
7	6	1	282	289
6	6	1	113	124
5	6	1	124	133
4	6	1	63	62
3	6	1	100	95
2	6	1	66	68
1	6	1	207	210
0	6	1	303	317
0	5	1	92	95
1	5	1	232	233
2	5	1	124	141
3	5	1	162	157
4	5	1	210	223
6	5	1	175	175
7	5	1	138	143
8	5	1	658	667
9	5	1	155	157
10	5	1	353	363
12	5	1	188	185
13	5	1	85	75
14	5	1	100	92
15	5	1	143	137
16	5	1	241	252
17	5	1	183	183
18	5	1	142	144
20	5	1	56	65
22	5	1	70	48
24	5	1	118	108
26	5	1	76	58
27	4	1	75	44
28	4	1	108	105
25	4	1	103	111
24	4	1	59	52
23	4	1	68	65
22	4	1	54	48
21	4	1	77	91
20	4	1	144	151
18	4	1	72	77
17	4	1	253	268
15	4	1	97	106
14	4	1	80	84
13	4	1	146	145
11	4	1	105	114
10	4	1	57	64
9	4	1	351	351
8	4	1	173	173
7	4	1	176	184
6	4	1	262	261
4	4	1	144	148
3	4	1	261	259
2	4	1	301	297
1	4	1	178	174
0	4	1	181	172
0	3	1	304	312
1	3	1	232	224
1	3	1	1529	1477
2	3	1	274	273
3	3	1	168	164
4	3	1	218	218
5	3	1	276	275
6	3	1	142	145
7	3	1	425	422
8	3	1	348	348
9	3	1	176	180
10	3	1	347	346
11	3	1	56	58
12	3	1	157	161
13	3	1	236	246
14	3	1	76	86
16	3	1	291	303
17	3	1	182	194
18	3	1	286	299
19	3	1	160	166
20	3	1	257	265
21	3	1	72	84
22	3	1	86	92
23	3	1	144	135
25	3	1	88	81
26	2	1	73	71
27	2	1	97	93
28	2	1	141	153
27	2	1	95	84
25	2	1	81	84
21	2	1	61	68
19	2	1	144	138
18	2	1	164	161
16	2	1	144	147
15	2	1	526	544
13	2	1	293	300
12	2	1	88	76
11	2	1	181	186
9	2	1	145	146
8	2	1	214	221
7	2	1	113	103
6	2	1	508	491
5	2	1	266	255
4	2	1	530	508
6	2	1	318	310
5	2	1	479	475
4	2	1	440	432
3	2	1	675	641
2	2	1	1556	1525
1	2	1	354	344
0	2	1	116	112
0	1	1	116	119
1	1	1	430	416
2	1	1	494	515
3	1	1	104	110
4	1	1	341	354
5	1	1	270	270
6	1	1	482	463
7	1	1	520	508
8	1	1	419	403
9	1	1	441	445
10	1	1	316	310
11	1	1	225	221
12	1	1	100	112
13	1	1	142	145
16	1	1	288	306
17	1	1	248	268
18	1	1	345	363
19	1	1	113	116
20	1	1	88	97
21	1	1	66	62
22	1	1	122	124
24	1	1	133	146
26	1	1	64	63
27	1	1	142	124
28	1	1	122	113
25	0	1	80	82
24	0	1	63	78
23	0	1	110	92
22	0	1	62	50
21	0	1	63	66
20	0	1	90	104
18	0	1	221	225
17	0	1	194	195
16	0	1	84	86
14	0	1	189	197
12	0	1	154	152
11	0	1	89	99
9	0	1	279	284
7	0	1	248	246
6	0	1	135	122
5	0	1	188	190
4	0	1	51	38
3	0	1	53	60
2	0	1	94	89
1	0	1	52	77
0	0	2	1494	1615
1	0	2	533	536
2	0	2	86	73
3	0	2	500	524
4	0	2	378	385
5	0	2	442	444
6	0	2	524	501
7	0	2	251	248
8	0	2	652	637
9	0	2	839	808
10	0	2	606	604
13	0	2	130	130
15	0	2	49	51
16	0	2	227	224
18	0	2	247	257
19	0	2	99	92
20	0	2	63	73
21	0	2	92	96
23	0	2	69	45
25	0	2	151	141
26	0	2	107	103
27	0	2	87	67
28	0	2	152	153
27	1	2	72	62
26	1	2	83	55
25	1	2	96	110
23	1	2	176	169
21	1	2	112	122
19	1	2	105	120
17	1	2	318	322
16	1	2	187	192
15	1	2	92	97
14	1	2	85	95
13	1	2	70	77
12	1	2	83	86
11	1	2	252	262
10	1	2	349	335
9	1	2	99	105
8	1	2	647	623
7	1	2	151	145
6	1	2	264	258
5	1	2	667	642
4	1	2	408	391
3	1	2	817	817
2	1	2	219	222
1	1	2	844	850

Table 1. Continued.

The data are listed in five parallel columns, each giving h, k, l, $|F_o|$, $|F_c|$.

Column 1

h	k	l	$|F_o|$	$|F_c|$
0	1	2	209	219
0	2	2	2085	2099
1	2	2	251	253
2	2	2	726	722
3	2	2	332	328
4	2	2	270	255
5	2	2	463	435
6	2	2	549	537
7	2	2	625	592
8	2	2	293	285
9	2	2	536	521
10	2	2	275	256
11	2	2	228	225
12	2	2	163	168
13	2	2	148	151
14	2	2	172	172
15	2	2	77	75
16	2	2	415	420
17	2	2	215	222
18	2	2	487	497
19	2	2	161	166
20	2	2	101	99
21	2	2	120	134
23	2	2	65	81
24	2	2	77	81
26	2	2	101	92
27	2	2	104	108
26	3	2	122	110
25	3	2	162	161
24	3	2	95	85
23	3	2	100	93
21	3	2	104	107
20	3	2	68	67
19	3	2	83	76
18	3	2	154	160
17	3	2	272	270
16	3	2	74	80
15	3	2	142	145
14	3	2	209	205
13	3	2	175	174
12	3	2	110	115
11	3	2	232	230
10	3	2	172	170
9	3	2	298	303
8	3	2	199	192
7	3	2	289	287
6	3	2	185	178
5	3	2	121	115
4	3	2	225	210
3	3	2	303	298
2	3	2	417	413
1	3	2	221	223
0	3	2	104	106
0	4	2	841	826
1	4	2	95	96
2	4	2	432	425
3	4	2	74	85
4	4	2	149	141
5	4	2	331	340
6	4	2	258	259
7	4	2	207	206
8	4	2	86	91
9	4	2	62	45
10	4	2	152	152
11	4	2	110	115
12	4	2	176	171
13	4	2	217	224
14	4	2	54	66
15	4	2	237	242
16	4	2	260	265
17	4	2	164	172
18	4	2	204	215
19	4	2	142	142
20	4	2	64	65
21	4	2	83	84
22	4	2	116	124
23	4	2	86	67
24	4	2	119	117
26	4	2	67	63
24	5	2	82	73
20	5	2	89	96
18	5	2	77	73
17	5	2	186	185
16	5	2	170	178
15	5	2	181	185
14	5	2	136	146
13	5	2	61	65
12	5	2	177	173
11	5	2	148	153
10	5	2	134	137
9	5	2	173	176
8	5	2	140	142
7	5	2	251	250
6	5	2	298	291
5	5	2	265	267
4	5	2	222	225
3	5	2	140	133
2	5	2	159	162
1	5	2	454	447
0	5	2	36	45
0	6	2	201	208
1	6	2	65	60
2	6	2	148	145
3	6	2	144	147
4	6	2	131	135
5	6	2	101	95

Column 2

h	k	l	$|F_o|$	$|F_c|$
6	6	2	247	248
7	6	2	109	118
8	6	2	54	57
9	6	2	134	131
10	6	2	46	45
11	6	2	146	147
12	6	2	115	116
13	6	2	64	42
14	6	2	93	82
16	6	2	54	51
17	6	2	57	44
18	6	2	97	103
22	6	2	59	68
24	6	2	80	76
21	7	2	65	41
20	7	2	55	51
19	7	2	60	66
18	7	2	79	77
16	7	2	59	48
15	7	2	90	98
14	7	2	89	92
13	7	2	79	77
12	7	2	155	149
11	7	2	110	105
10	7	2	356	365
9	7	2	143	148
8	7	2	256	261
7	7	2	217	215
6	7	2	294	297
5	7	2	108	106
4	7	2	201	207
3	7	2	111	114
2	7	2	134	140
1	7	2	150	147
0	8	2	99	93
1	8	2	44	28
2	8	2	115	117
4	8	2	54	58
7	8	2	315	326
8	8	2	138	132
9	8	2	310	316
11	8	2	133	144
12	8	2	110	102
18	8	2	85	84
20	8	2	73	80
19	9	2	96	94
18	9	2	80	74
15	9	2	79	90
10	9	2	127	128
9	9	2	90	92
8	9	2	90	86
0	10	2	98	100
14	11	2	76	70
13	11	2	81	74
10	11	2	70	63
8	11	2	82	90
4	11	2	62	84
3	11	2	75	65
2	11	2	67	58
1	11	2	69	63
0	12	2	88	95
8	12	2	62	38
6	12	2	75	29
3	12	2	65	33
20	8	3	64	64
19	8	3	126	131
18	8	3	169	165
17	8	3	114	117
16	8	3	116	108
15	8	3	65	43
14	8	3	68	64
13	8	3	73	68
12	8	3	67	56
11	8	3	58	66
10	8	3	68	77
9	8	3	130	118
8	8	3	72	63
7	8	3	71	68
6	8	3	96	103
5	8	3	130	129
4	8	3	281	284
3	8	3	178	179
10	8	3	86	77
12	8	3	90	74
13	8	3	70	68
15	8	3	58	60
16	8	3	74	70
9	8	3	155	160
8	8	3	84	82
7	8	3	114	115
6	8	3	150	160
5	8	3	69	65
4	8	3	86	87
3	8	3	82	101
2	8	3	83	86

Column 3

h	k	l	$|F_o|$	$|F_c|$
1	8	3	137	144
0	8	3	47	37
0	7	3	74	77
1	7	3	130	116
3	7	3	75	84
4	7	3	68	70
5	7	3	74	71
6	7	3	174	183
7	7	3	49	51
8	7	3	339	342
9	7	3	180	191
10	7	3	251	258
11	7	3	76	59
13	7	3	111	113
15	7	3	59	71
16	7	3	105	117
18	7	3	60	64
19	7	3	84	56
22	7	3	117	83
24	6	3	89	85
20	6	3	66	58
19	6	3	61	77
18	6	3	143	146
17	6	3	144	150
16	6	3	162	175
15	6	3	99	102
14	6	3	139	122
13	6	3	74	69
11	6	3	127	135
10	6	3	135	132
9	6	3	99	100
8	6	3	101	105
7	6	3	132	130
5	6	3	154	154
4	6	3	144	145
3	6	3	93	97
2	6	3	184	177
1	6	3	257	256
0	6	3	343	335
0	5	3	156	164
1	5	3	475	491
3	5	3	196	210
4	5	3	148	154
5	5	3	137	129
7	5	3	149	155
8	5	3	162	159
9	5	3	80	68
10	5	3	70	85
11	5	3	66	67
12	5	3	138	144
13	5	3	70	72
14	5	3	87	73
15	5	3	165	170
16	5	3	56	67
17	5	3	241	240
18	5	3	83	84
19	5	3	199	196
22	5	3	71	64
23	5	3	69	80
25	5	3	137	127
26	4	3	136	125
23	4	3	75	85
20	4	3	71	55
18	4	3	86	75
17	4	3	230	224
16	4	3	95	105
15	4	3	378	388
14	4	3	226	234
13	4	3	124	118
12	4	3	115	125
11	4	3	149	157
10	4	3	98	95
9	4	3	156	155
8	4	3	297	309
7	4	3	142	145
6	4	3	156	161
4	4	3	189	193
3	4	3	117	117
2	4	3	248	256
1	4	3	248	243
0	4	3	802	777
0	3	3	163	167
1	3	3	719	709
3	3	3	63	58
4	3	3	481	490
5	3	3	147	153
6	3	3	138	134
7	3	3	128	125
8	3	3	219	216
9	3	3	161	161
10	3	3	141	138
11	3	3	161	157
12	3	3	170	169
13	3	3	175	170
14	3	3	192	202
15	3	3	198	195
16	3	3	258	262
17	3	3	225	235
18	3	3	102	93
19	3	3	169	171
24	3	3	230	235
25	3	3	99	93
26	3	3	100	92
26	2	3	101	88
23	2	3	107	118
21	2	3	63	64

Column 4

h	k	l	$|F_o|$	$|F_c|$
20	2	3	58	69
19	2	3	51	36
18	2	3	162	173
17	2	3	123	144
16	2	3	91	106
14	2	3	89	97
13	2	3	113	111
12	2	3	113	102
11	2	3	136	132
10	2	3	121	121
9	2	3	386	382
8	2	3	367	373
7	2	3	440	414
6	2	3	187	185
5	2	3	87	85
4	2	3	95	90
3	2	3	96	102
2	2	3	361	366
1	2	3	60	60
0	2	3	572	557
0	1	3	111	118
1	1	3	514	514
3	1	3	261	268
4	1	3	313	308
5	1	3	511	507
6	1	3	511	499
7	1	3	208	210
8	1	3	180	175
9	1	3	434	426
10	1	3	232	229
11	1	3	54	51
13	1	3	292	299
14	1	3	158	162
15	1	3	151	153
16	1	3	195	200
17	1	3	154	158
18	1	3	80	76
19	1	3	133	135
20	1	3	114	118
21	1	3	126	127
23	1	3	137	139
24	1	3	98	91
27	1	3	245	226
26	0	3	168	169
25	0	3	118	105
23	0	3	120	111
16	0	3	112	111
15	0	3	80	89
12	0	3	154	158
11	0	3	48	35
10	0	3	63	64
9	0	3	61	55
8	0	3	70	62
7	0	3	141	142
6	0	3	133	135
5	0	3	434	439
3	0	3	319	327
2	0	3	278	292
1	0	3	257	270
0	0	4	184	183
2	0	4	267	272
1	0	4	226	248
3	0	4	615	648
5	0	4	49	63
7	0	4	68	57
8	0	4	622	630
9	0	4	189	190
11	0	4	631	638
10	0	4	212	210
12	0	4	54	56
13	0	4	104	116
15	0	4	93	77
17	0	4	142	143
19	0	4	222	227
21	0	4	153	161
25	0	4	150	144
26	1	4	74	77
25	1	4	102	97
19	1	4	79	60
18	1	4	102	84
17	1	4	162	164
15	1	4	137	127
14	1	4	117	126
13	1	4	111	112
11	1	4	63	65
12	1	4	186	184
11	1	4	99	100
10	1	4	109	105
9	1	4	216	219
8	1	4	270	265
7	1	4	111	114
6	1	4	183	195
5	1	4	67	56
3	1	4	146	150
2	1	4	511	524
1	1	4	165	168
0	1	4	512	524
1	2	4	358	368
3	2	4	215	217
4	2	4	253	258
5	2	4	112	113

Column 5

h	k	l	$|F_o|$	$|F_c|$
5	2	4	207	211
6	2	4	181	187
7	2	4	339	342
8	2	4	160	165
9	2	4	255	255
10	2	4	280	279
12	2	4	44	43
13	2	4	94	105
14	2	4	128	138
15	2	4	95	97
16	2	4	113	114
17	2	4	141	148
18	2	4	187	196
19	2	4	65	79
20	2	4	65	63
21	2	4	136	131
23	2	4	68	82
24	2	4	79	67
25	2	4	119	111
23	3	4	102	101
22	3	4	81	75
21	3	4	109	98
20	3	4	57	49
19	3	4	63	56
18	3	4	92	86
17	3	4	121	125
16	3	4	104	99
13	3	4	50	63
11	3	4	157	157
10	3	4	92	96
9	3	4	67	73
8	3	4	222	224
7	3	4	72	56
6	3	4	74	80
5	3	4	122	127
4	3	4	190	190
3	3	4	193	200
2	3	4	333	331
1	3	4	176	186
0	3	4	416	425
0	4	4	374	371
1	4	4	166	153
2	4	4	378	369
3	4	4	111	111
4	4	4	88	89
5	4	4	84	75
6	4	4	72	66
7	4	4	222	223
8	4	4	81	83
9	4	4	304	303
10	4	4	174	176
11	4	4	193	191
12	4	4	45	47
13	4	4	143	159
14	4	4	122	110
15	4	4	126	125
16	4	4	199	196
17	4	4	201	203
18	4	4	143	145
19	4	4	74	91
20	4	4	192	185
21	4	4	82	56
25	4	4	77	82
24	5	4	203	177
23	5	4	85	84
21	5	4	112	117
20	5	4	91	94
19	5	4	112	109
18	5	4	197	184
17	5	4	132	118
16	5	4	72	53
14	5	4	112	125
12	5	4	61	172
11	5	4	84	84
10	5	4	445	455
8	5	4	452	472
7	5	4	204	202
6	5	4	297	295
5	5	4	125	123
4	5	4	208	213
3	5	4	238	236
1	5	4	147	151
0	5	4	250	256
0	6	4	66	53
1	6	4	185	192
2	6	4	99	104
3	6	4	100	77
4	6	4	119	133
5	6	4	46	56
7	6	4	338	342
8	6	4	141	145
9	6	4	304	303
10	6	4	114	116
11	6	4	252	244
12	6	4	72	69
13	6	4	101	97
15	6	4	111	108
16	6	4	113	115
17	6	4	143	145
19	6	4	88	80
20	6	4	60	65
22	6	4	64	66
17	7	4	123	115
16	7	4	85	101
15	7	4	73	92
11	7	4	50	31

Table 1. Continued.

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 10  7  4  149  148    5  5  5  129  137    6  0  5  122  128    12  5  6  153  141    14  4  7  103   84
  9  7  4  162  160    6  5  5  162  160    5  0  5  141  143    10  5  6  221  217    13  4  7  115   98
  8  7  4  141  139    8  5  5  182  177    4  0  5  163  175     9  5  6   57   64    12  4  7   69   67
  7  7  4   81   78    9  5  5  100  101    3  0  5  122  125     8  5  6  290  292    11  4  7  111  115
  6  7  4  168  163   10  5  5   76   80    2  0  5  260  265     6  5  6  125  128     9  4  7   86   80
  5  7  4   66   51   11  5  5  127  133    0  0  6  190  197     5  5  6   93   94     8  4  7   60   67
  4  7  4   96   98   12  5  5   94  101    1  0  6  123  125     4  5  6  113  115     7  4  7  147  158
  2  7  4   47   30   13  5  5  111  102    2  0  6  340  342     3  5  6  131  140     6  4  7  255  257
  1  7  4  207  206   14  5  5  154  154    3  0  6  107  105     1  5  6   49   65     5  4  7  125  137
  0  8  4  258  274   15  5  5   82   93    4  0  6   44   46     0  6  6   78   88     4  4  7  118  129
  1  8  4  180  181   16  5  5   75   61    5  0  6  142  145     3  6  6   99  107     2  4  7  128  135
  2  8  4  141  148   17  5  5   78   75    6  0  6   78   89     4  6  6   88   94     1  4  7   67   58
  3  8  4   81   70   19  5  5  107   99    7  0  6   83   89     6  6  6   73   60     0  4  7  188  203
  4  8  4   66   54   21  5  5   63   48    8  0  6   49   46     8  6  6  118  106     0  3  7   89   92
  5  8  4   77   71   20  4  5   71   60    9  0  6  355  378     9  6  6  119  131     1  3  7  173  166
  6  8  4   66   48   19  4  5   70   63   10  0  6  344  363    12  6  6   56   45     3  3  7  160  171
  7  8  4   78   79   17  4  5   85   81   11  0  6  183  186    13  6  6   78   50     5  3  7  141  155
  9  8  4  100   95   16  4  5   69   73   12  0  6  119  115    15  6  6   67   42     6  3  7  138  139
 10  8  4  109  102   15  4  5   82   69   13  0  6   75   70    16  6  6   71   71     7  3  7  109  115
 14  8  4   62   47   14  4  5   92   90   14  0  6   70   74    18  6  6  128  122     8  3  7  153  161
 16  8  4  160  144   12  4  5   64   46   16  0  6   91   96    19  6  6   72   55     9  3  7   79   79
 18  8  4  160  150   11  4  5  167  175   17  0  6   66   43    19  7  6   67   81    10  3  7  121  127
 19  8  4  101   71   10  4  5  106  108   18  0  6  145  148    18  7  6   69   45    11  3  7   51   49
 12  9  4   68   40    9  4  5  116  114   19  0  6   96  107    17  7  6   80   88    12  3  7  105  101
 10  9  4   67   42    8  4  5  166  169   20  0  6  100   93    16  7  6   96   94    13  3  7   64   56
  7  9  4  123  113    7  4  5  127  127   22  0  6  118  108    14  7  6   63   36    14  3  7   54   43
  6  9  4   85   94    6  4  5   84   92   23  0  6  104   86    13  7  6  110   79    15  3  7   57   55
  4  9  4   72   43    5  4  5   48   45   24  0  6   91   80    12  7  6   68   56    16  3  7   86   78
  2  9  4   62   51    4  4  5  127  128   24  1  6   81   75    10  7  6   93   78    17  3  7  136  125
  1  9  4   75   85    3  4  5   60   50   19  1  6  108  111    10  7  6   85   70    18  3  7   70   75
  4 10  4   67   55    2  4  5   63   53   17  1  6  103   88     9  7  6  114  101    21  3  7   82   70
  7 10  4  109  104    0  4  5  131  135   16  1  6   78   69     8  7  6  107  106    20  2  7  113   93
  9 10  4   74   64    0  3  5  139  144   15  1  6   67   68     7  7  6  108   94    15  2  7   88  100
 11 10  4  132  131    1  3  5   64   73   13  1  6   72   73     3  7  6  142  152    14  2  7   84   85
 15 10  4   67   54    2  3  5  221  227   12  1  6   96  108     1  7  6  157  179    13  2  7  126  122
 10 11  4  100   75    3  3  5   71   69   11  1  6   97   96     0  7  6   87  102    12  2  7   88   91
  7 11  4  104   99    4  3  5   93  104   10  1  6  246  254     0  8  6  213  218    11  2  7  149  142
  3 11  4   89   65    5  3  5  169  172    8  1  6  207  214     1  8  6   84   71    10  2  7   66   73
  6 11  5   81   64    6  3  5  236  244    7  1  6  157  166     2  8  6   74   75     8  2  7   58   71
  8 11  5   82   74    7  3  5   84   73    6  1  6  129  142     6  8  6  143  134     7  2  7  250  253
 10 11  5   94   82    8  3  5  636  647    5  1  6   78   73     7  8  6  205  192     6  2  7   47   54
 13 10  5   66   41    9  3  5  257  260    4  1  6   92   99    11  8  6  176  152     5  2  7  165  174
 11 10  5  102   88   10  3  5  467  473    3  1  6   42   52    12  8  6   70   42     2  2  7  105  106
 10 10  5   70   47   12  3  5  179  188    1  1  6   58   60    16  8  6  101   95     2  2  7  208  215
  8 10  5   71   78   13  3  5  151  159    0  1  6   81   83    17  8  6   90   62     1  2  7   64   68
  7 10  5   86   99   16  3  5  133  130    0  2  6  116  132    14  9  6   84   92     1  1  7   71   76
  4 10  5   85   79   18  3  5  126  120    1  2  6   81   78     8  9  6  177  166     2  1  7  103  104
  2 10  5   99   95   19  3  5   61   49    2  2  6   69   61     5  9  6   59   57     3  1  7  105  120
  0 10  5  151  148   20  3  5  111   93    3  2  6  196  205     2  9  6  129  130     4  1  7   64   60
  0  9  5   94   96   21  3  5  129  123    4  2  6  133  136     2  9  6   84   94     5  1  7  100  115
  1  9  5  126  127   23  3  5   66   56    5  2  6   55   54     0  9  6  111  102     6  1  7  216  227
  4  9  5   66   50   24  3  5  148  116    6  2  6   67   56     9  9  6   72   63     8  1  7  592  621
  5  9  5   99   95   25  2  5  104  110    7  2  6  260  278    10 10  6  152  150    10  1  7  338  347
  8  9  5   57   53   24  2  5   68   87    8  2  6   61   57     2 10  6  112  103    12  1  7  121  126
  9  9  5   60   42   22  2  5   69   72    9  2  6  390  406     7 10  6   66   81    13  1  7   96   86
  6  9  5   60   58   20  2  5  103   95   10  2  6  177  186     9 10  6   85   56    14  1  7   82   71
 12  9  5   67   60   19  2  5   79   94   11  2  6  188  188    11 10  6   79   66    15  1  7   56   75
 13  9  5   85   73   18  2  5  134  122   12  2  6   52   43     2 10  7   70   70    16  1  7  115   98
 19  8  5  111  107   17  2  5  145  147   13  2  6   65   84     2 10  7  161  142    19  1  7   72   74
 15  8  5   85   78   15  2  5   50   55   14  2  6   85   76     1  9  7   84  107    20  1  7  115  112
 14  8  5   71   68   14  2  5  117  133   15  2  6  101  107     2  9  7   68   74    22  0  7   77   78
  9  8  5  106   91   12  2  5   98   85   17  2  6  140  142     3  9  7   77   82    20  0  7   62   50
  4  8  5   79   91   11  2  5   67   69   18  2  6   60   36     7  9  7   62   47    18  0  7   84   90
  3  8  5   78   66   10  2  5  176  188   19  2  6   55   40     9  9  7   75   70    12  0  7   80   71
  2  8  5  103  106    9  2  5   55   40   21  2  6   61   36    15  8  7   82   90    11  0  7   71   65
  1  8  5   75   83    8  2  5  103  106   24  2  6   69   46    13  8  7   70   70     9  0  7   89   96
  0  7  5  162  174    7  2  5  225  223   18  3  6  101   80    10  8  7  114   90     7  0  7   63   71
  1  7  5  329  338    5  2  5  147  137   17  3  6   98   87     9  8  7   75   64     6  0  7   61   69
  2  7  5  145  154    4  2  5  125  124   16  3  6   89   75     7  8  7   79   64     5  0  7   65   95
  3  7  5   77   83    2  2  5  202  209   15  3  6   55   52     6  8  7   87   76     3  0  7   53   72
  6  7  5   62   60    1  2  5  145  140   12  3  6   57   42     3  8  7   62   66     2  0  7   79   75
  7  7  5   66   67    0  2  5  324  346   10  3  6  251  251     2  8  7  114  124     0  0  8   46   42
  8  7  5  156  155    7  1  5  169  172    9  3  6  147  157     1  8  7   58   55     0  0  8   60   70
  9  7  5   95   98    8  1  5  145  156    8  3  6  234  244     1  7  7  149  169     4  0  8   95  114
 10  7  5   86   98    9  1  5  206  205    7  3  6  153  164     4  7  7   76   60     5  0  8  139  151
 11  7  5   92   77   10  1  5  146  151    6  3  6  301  304     6  7  7   98   98     6  0  8   85   92
 12  7  5   73   90   11  1  5  118  123    5  3  6  138  139     8  7  7   65   56     7  0  8   87   90
 15  7  5  107  118   12  1  5  383  389    4  3  6  138  141     9  7  7   80   74     8  0  8  156  155
 16  7  5   83   84   13  1  5  521  537    3  3  6  180  172    10  7  7  119  118     9  0  8  168  177
 17  7  5  164  173   15  1  5  500  496    2  3  6  125  142    14  6  7  148  144    10  0  8  126  148
 18  7  5   84   84   16  1  5  322  340    1  3  6   84   90    13  6  7   63   51    11  0  8  180  195
 19  7  5  175  173   17  1  5  185  191    0  3  6  119  120    12  6  7   74   54    13  0  8  146  157
 20  7  5   74   57   18  1  5   82   87    0  4  6  174  174    11  6  7  109   98    14  0  8   95   93
 22  6  5   99   75   19  1  5  225  244    1  4  6  115  120    10  6  7   76   72    15  0  8   96   84
 20  6  5  134  128   20  1  5  121  125    2  4  6   79   95     7  6  7  160  166    16  0  8   75   51
 19  6  5  111   95   22  1  5  288  300    3  4  6  147  143     6  6  7   51   47    19  1  8   91   57
 18  6  5  137  129   20  0  5  148  147    4  4  6  114  117     5  6  7   80   67    18  1  8  122   95
 17  6  5   87   86   19  0  5   49   44    5  4  6  178  186     1  6  7   51   54    15  1  8   66   63
 16  6  5  213  200   18  0  5   62   66    6  4  6  126  126     0  5  7   99   94    14  1  8   77   74
 14  6  5  127  118   17  0  5  121  127    7  4  6  120  121     1  5  7   93  103    13  1  8  133  137
 13  6  5   73   67   16  0  5   55   76    8  4  6  182  173     2  5  7   85   97    12  1  8   65   71
 12  6  5   64   56   14  0  5  181  164    9  4  6  507  523     4  5  7  118  113    10  1  8  209  210
 11  6  5   80   93   13  0  5   79   79   10  4  6   95   91     6  5  7   97   99     9  1  8   97  116
 10  6  5  159  155   12  0  5  175  179   11  4  6   95   92     7  5  7   51   50     8  1  8  228  244
  9  6  5   75   61   10  0  5   86   73   13  4  6   67   83     8  5  7  191  191     6  1  8   86   91
  8  6  5  103   97    9  0  5  184  181   14  4  6   85   91    10  5  7  142  134     5  1  8  177  184
  7  6  5  306  301    8  0  5  119  131   15  4  6  127  115    12  5  7   59   65     2  1  8  111  112
  5  6  5   51   41    7  0  5  102   85   16  4  6  121  117    14  5  7   81   82     1  1  8  122  118
  3  6  5   49   65    5  0  5   71   64   18  4  6  101   99    15  5  7   67   64     0  1  8  114  116
  2  6  5  330  327    3  0  5   75   64   21  4  6   89   86    16  5  7  113  110     0  1  8  112  142
  1  6  5  329  326    2  0  5   66   59   20  5  6   80   75    18  5  7   97   67     2  2  8  112   99
  0  6  5  229  242    1  0  5  104  118   18  5  6   98   77    20  5  7  101   85     3  2  8   81   90
  0  5  5  111  107    0  0  5  106  101   17  5  6   84   94    18  4  7   94   81     9  2  8   84  101
  1  5  5  179  179    0  1  5   83   82   16  5  6   72   50    15  4  7  136  128
  2  5  5  178  186    1  1  5  110  110
  3  5  5  161  170    2  1  5  179  180
                      3  1  5  153  155
                      4  1  5  146  140
```

Table 1. Continued.

10	2	8	65	93	6	7	9	67	42	4	0	9	54	21	5	5	0	35	29*	14	3	4	46	38*
11	2	8	66	62	13	6	9	65	40	0	0	10	69	54	1	6	0	37	22*	12	3	4	41	25*
12	2	8	53	48	2	6	9	75	56	1	0	10	71	72	4	6	0	37	10*	13	5	4	46	4*
14	2	8	60	46	0	6	9	82	85	2	0	10	125	119	14	6	0	46	9*	9	5	4	43	17*
15	2	8	58	60	1	5	9	81	86	4	0	10	91	97	1	8	0	44	1*	2	6	4	41	36*
19	2	8	63	42	6	5	9	69	49	5	0	10	108	42	3	8	0	44	16*	6	6	4	44	38*
15	3	8	117	105	7	5	9	76	61	7	0	10	87	105	5	8	0	45	2*	3	7	4	45	27*
13	3	8	118	105	8	5	9	127	139	9	0	10	65	50	5	9	0	48	2*	0	7	4	44	24*
11	3	8	84	79	10	5	9	75	55	12	1	10	95	77	4	9	0	47	31*	4	7	5	49	54*
10	3	8	75	74	11	5	9	80	44	10	1	10	70	53	10	8	1	48	41*	5	7	5	49	47*
8	3	8	154	155	13	5	9	75	76	9	1	10	71	60	4	8	1	47	72*	6	6	5	46	25*
6	3	8	101	108	14	4	9	72	59	8	1	10	109	114	5	7	1	40	21*	4	6	5	45	18*
5	3	8	52	40	11	4	9	67	53	2	1	10	61	48	8	6	1	44	45*	4	5	5	42	35*
4	3	8	83	90	10	4	9	86	91	0	1	10	100	98	5	5	1	35	23*	8	5	5	47	25*
3	3	8	88	101	9	4	9	107	108	1	2	10	72	66	11	5	1	39	21*	13	4	5	48	23*
1	3	8	82	104	7	4	9	109	105	2	2	10	98	84	13	4	1	39	5*	4	4	5	38	18*
0	4	8	266	274	6	4	9	73	79	7	2	10	99	85	17	2	1	45	11*	11	3	5	43	30*
1	4	8	102	104	5		9	56	51	8	2	10	61	79	14	1	1	38	50*	14	3	5	48	52*
3	4	8	65	55	2		9	87	86	12	2	10	64	61	15	1	1	40	38*	6	1	5	35	15*
4	4	8	75	80	1	4	9	90	87	8	3	10	86	79	19	0	1	45	9*	10	1	5	40	48*
6	4	8	60	48	0	3	9	91	97	3	3	10	83	64	15	0	1	38	20*	15	0	5	44	6*
7	4	8	53	55	1	3	9	95	99	1	3	10	57	80	13	0	1	36	7*	11	0	5	41	40*
11	4	8	60	46	2	3	9	82	90	6	4	10	82	66	10	0	1	34	27*	8	0	5	35	2*
14	4	8	94	89	4	3	9	66	87	7	4	10	117	125	8	0	1	33	33*	7	0	5	35	12*
15	4	8	57	45	6	3	9	79	72	9	4	10	77	69	11	0	2	35	13*	1	0	5	34	46*
16	4	8	85	78	7	3	9	73	81	10	5	10	109	107	12	0	2	35	2*	2	1	6	37	45*
18	4	8	129	125	8	3	9	114	120	8	5	10	141	136	14	0	2	39	17*	11	3	6	47	43*
14	5	8	106	108	9	3	9	97	90	7	5	10	69	45	17	0	2	44	24*	7	5	6	46	57*
13	5	8	59	63	10	3	9	179	171	6	5	10	95	92	18	1	2	47	51*	2	5	6	47	45*
12	5	8	61	53	12	3	9	103	109	2	5	10	70	53	0	7	2	40	3*	0	5	6	43	24*
7	5	8	87	84	16	3	9	68	73	1	5	10	102	67	3	8	2	43	25*	1	6	6	45	14*
5	5	8	113	115	12	2	9	76	87	0	4	11	74	59	5	8	2	43	21*	2	6	6	46	35*
2	5	8	109	107	11	2	9	172	174	0	3	11	64	61	6	8	2	48	26*	5	6	6	48	20*
0	5	8	69	90	9	2	9	133	145	4	3	11	81	63	0	9	2	46	12*	3	4	7	47	63*
0	6	8	168	173	8	2	9	63	45	5	3	11	81	64	12	6	3	47	25*	2	3	7	43	36*
2	6	8	150	152	7	2	9	167	165	6	3	11	103	118	6	6	3	40	26*	4	3	7	43	18*
4	6	8	79	66	6	2	9	80	66	8	3	11	137	138	2	5	3	37	20*	9	2	7	45	9*
5	6	8	96	86	1	2	9	86	84	8	2	11	78	73	6	5	3	38	24*	0	2	7	41	19*
8	6	8	102	112	0	2	9	191	187	5	2	11	62	39	11	3	3	38	16*	0	1	7	40	7*
9	6	8	78	67	1	1	9	92	111	4	2	11	63	61	15	2	3	43	25*	7	1	7	46	33*
11	6	8	103	88	2	1	9	78	77	1	1	11	114	129	2	1	3	31	10*	9	1	7	47	28*
13	6	8	72	78	5	1	9	106	101	6	1	11	105	79	12	1	3	37	20*	10	0	7	47	35*
15	6	8	69	47	6	1	9	88	107	9	1	11	82	61	18	0	3	48	14*	8	0	7	45	5*
16	6	8	111	116	8	1	9	273	282	18	1	0	49	25*	17	0	3	42	9*	2	0	7	42	30*
10	7	8	112	111	10	1	9	155	163	11	1	0	35	27*	13	0	3	37	8*	1	0	7	40	32*
8	7	8	181	163	14	1	9	63	52	9	1	0	33	10*	4	0	4	32	41*	1	0	8	45	23*
6	7	8	84	78	16	1	9	89	70	3	1	0	32	1*	14	0	4	43	43*	5	1	8	46	41*
2	9	8	69	45	17	1	9	135	113	17	2	0	47	21*	16	0	4	48	61*	4	1	8	48	40*
1	9	8	87	93	18	1	9	68	58	16	3	0	45	50*	11	2	4	39	33*	0	2	8	45	10*
2	8	9	93	76	11	0	9	71	88	13	5	0	41	19*	15	3	4	45	37*	4	2	8	47	42*
1	7	9	71	75	5	0	9	88	72															

addition procedure and the tangent formula.[14,15] In one of the corresponding eight E-maps the atoms of the two non-equivalent pyridone rings and also the atoms bonded directly to the rings could be located. A Fourier map based on phases calculated from these atomic positions including all observed structure factors yielded information on the positions of all non-hydrogen atoms.

Preliminary refinements were carried out by the minimum residual method[16] using the 300 largest structure factors. A couple of full-matrix least-squares refinement cycles with isotropic thermal parameters yielded a conventional R-factor of 0.125; introduction of anisotropic thermal parameters followed by some cycles of block-diagonal least-squares refinement gave an R-factor of 0.084. At this point a difference Fourier map was calculated and from this it was possible to establish the position of the hydrogen atoms. New series of block-diagonal least-squares calculations with refinement of positional parameters of all atoms and anisotropic thermal parameters of the nonhydrogen atoms (the isotropic thermal parameters for the hydrogen atoms were kept constant at 2.2 Å^2) resulted in a final R-value of 0.064 ($R_W = 0.047$). The overdetermination ratio was only 5.5, 313 parameters being refined on the basis of 1729 observed reflections with $\sin \theta/\lambda < 0.7$ Å^{-1}. No rigid-body thermal analysis was carried out.

A comparison of observed and calculated structure factors is given in Table 1; the final parameters for non-hydrogen atoms are listed in Table 2 and for

Table 2. Fractional atomic coordinates and thermal parameters with estimated standard deviations ($\times 10^5$). The temperature factor is given by
$$\exp-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl).$$

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
O1	− 7615	17176	45274	119	894	1001	142	189	− 59
	11	29	31	7	38	46	26	30	76
O2	− 7809	− 13723	20360	81	1251	1364	− 70	19	− 968
	11	30	35	6	42	49	28	31	85
O3	25216	− 14133	− 1473	131	1201	902	− 157	204	− 408
	12	31	31	7	41	42	30	32	81
O4	30325	− 11327	21311	85	1729	1122	− 162	− 11	43
	12	34	33	6	50	47	30	33	91
N1	9439	4982	24697	86	504	836	78	− 6	− 121
	14	31	35	7	36	50	27	33	81
N2	19650	− 12695	38617	108	895	986	32	62	218
	15	36	39	7	45	52	34	37	94
C1	3591	− 446	19485	101	493	702	36	− 15	− 155
	16	38	44	8	45	56	33	40	91
C2	− 2112	3666	26290	96	487	711	− 70	− 33	308
	16	38	43	8	46	57	33	41	95
C3	− 2284	13308	39214	108	504	714	39	95	203
	17	41	43	9	45	58	35	41	97
C4	3945	17919	44271	140	609	810	− 11	− 107	− 375
	18	42	46	9	51	64	38	43	102
C5	9514	14018	36823	98	545	1254	36	− 152	− 171
	18	41	50	9	46	70	37	45	106
C6	15559	1541	16361	87	814	799	18	56	− 29
	17	41	45	8	54	63	38	41	105
C7	18674	− 12397	21462	74	691	839	− 30	1	16
	16	41	46	8	48	60	35	40	103
C8	25402	− 12829	13021	96	552	918	15	206	6
	17	41	46	9	46	58	37	42	99
O1*	− 8862	32200	93725	102	939	1014	− 153	253	− 271
	12	28	31	6	39	46	27	29	76
O2*	− 8171	50870	68492	87	1325	1044	165	46	376
	11	29	32	6	44	45	30	30	85
O3*	29816	47919	69112	74	1116	2469	212	111	114
	12	31	41	6	44	65	28	39	103
O4*	26465	68867	59996	161	868	1360	− 94	273	449
	13	30	34	7	39	51	27	35	82

Table 2. Continued.

N1*	8818	43544	75614	66	546	918	−32	12	−299
	14	31	36	7	36	51	27	33	80
N2*	16380	69677	81156	90	708	768	−116	81	−368
	13	32	37	7	40	50	30	35	85
C1*	3193	49011	69395	98	502	699	25	−24	133
	16	38	44	8	45	56	33	40	91
C2*	−2749	45594	75628	89	560	768	45	−23	−34
	16	39	43	9	46	59	34	41	98
C3*	−3261	36160	88501	116	514	765	−34	81	−471
	17	41	44	9	46	59	37	42	98
C4*	2738	31479	94992	135	574	983	−101	−31	95
	18	42	48	10	49	67	38	47	104
C5*	8554	35107	88238	111	401	1068	−23	−142	273
	17	38	49	9	44	66	36	44	99
C6*	15116	46747	67810	84	623	899	−19	172	−352
	17	41	47	8	48	63	34	41	106
C7*	19556	55957	77694	77	638	676	2	53	−83
	17	40	42	8	46	56	34	39	97
C8*	25944	58040	68153	74	880	903	−107	−41	−368
	17	42	46	8	55	61	36	43	107

hydrogen atoms in Table 3. Standard deviations were calculated from the correlation matrix from the block-diagonal least-squares calculations ignoring the standard deviations in cell parameters.

Table 3. Fractional atomic coordinates ($\times 10^4$) with estimated standard deviations for hydrogen atoms.

Atom	x	y	z	Atom	x	y	z
H1	408	−773	1224	H1*	351	5508	6099
	16	36	40		16	37	40
H4	426	2424	5319	H4*	276	2533	10496
	16	37	42		16	36	39
H5	1353	1658	3913	H5*	1310	3243	9242
	16	36	40		16	36	38
H61	1844	924	1915	H61*	1394	5129	5742
	16	34	40		16	34	40
H62	1461	56	610	H62*	1716	3772	6593
	16	33	40		16	36	40

Table 3. Continued.

H7	1600	− 2043	1891	H7*	2071	5108	8759
	15	34	41		16	34	39
H1N	2147	− 308	4183	H1N*	1285	6796	8961
	16	36	38		16	35	39
H2N	1479	− 1352	4436	H2N*	1459	7400	6248
	16	36	40		16	34	40
H3N	2259	− 2108	4037	H3N*	1947	7601	9609
	16	36	40		17	34	41
HO2	− 1098	78	2530	HO2*	− 1126	5073	7544
	16	35	38		16	35	38

DISCUSSION

A drawing of the two molecules (I and II) in the asymmetric unit is presented in Fig. 1; the numbering of the atoms is indicated, as are the bond lengths and angles arrived at in the structure analysis. Interatomic distances and bond angles are also given in Table 4 together with their standard deviations. The latter values might be somewhat underestimated, however, since possible correlations between positional parameters of different atoms are disregarded in the computing procedure.

The crystals of L-mimosine offer the opportunity to study two non-equivalent molecules in different environment. However, the different surroundings seem to have only minor influence on the molecular structure as the bond lengths and angles as well as the conformation of the two molecules are nearly equal.

The molecular conformation. Both of the molecules in the asymmetric unit have a conformation similar to the one found in L-DOPA.[10] The dihedral angle C1 − N − C6 − C7 is 275° and C1*N* − C6* − C7* 113°, the side chain is thus situated on opposite sides of the ring plane in the two molecules, and the molecule II (*) is the one corresponding to L-DOPA.

The carboxyl groups are in *trans* position relative to the phenyl rings with respect to the $C\alpha - C\beta$ bond; the dihedral angles N − C6 − C7 − C8 and N* − C6* − C7* − C8* are 172° and 178°, respectively, as compared to 175° for the corresponding angle in L-DOPA.

The alanine part. The zwitterionic character of the molecule is apparent in the present structure as usual for such compounds. The four C − O bonds of the carboxy groups are equal, mean bond length 1.242 Å, with a calculated standard deviation equal to the estimated standard deviation of each bond, 0.005 Å. The $C - NH_3^+$ bonds, 1.472 and 1.484 Å, are possibly on the short side of the expected value 1.49 Å, as found in L-DOPA [10] and L-tyrosine.[17] The C6 − C7 bonds (1.530 and 1.515) are found to be slightly shorter than the C7 − C8 bonds (1.552 and 1.550 Å). The α carbon atom is within the experimental accuracy situated in the plane of the carboxy group in both molecules. The dihedral angle N2 − C7 − C8 − O4 is 350.1° (in clock-wise direction) and the N2* − C7* − C8* − O4* angle is 334.8° (e.s.d. 0.5°), both being within the limits

Table 4. Interatomic distances (Å) and bond angles (°). Estimated standard deviations are 0.005 Å and 0.3°, respectively.

Bond	Molecule I	Molecule II(*)	Bond angle	Molecule I	Molecule II
O1−C3	1.261	1.285	O1−C3−C4	124.4	123.1
O2−C2	1.357	1.360	O1−C3−C2	121.4	121.2
O3−C8	1.239	1.247	O2−C2−C3	119.3	121.1
O4−C8	1.237	1.244	O2−C2−C1	118.5	117.8
N2−C7	1.472	1.484	C1−C2−C3	122.1	121.0
N1−C1	1.375	1.368	C2−C3−C4	114.2	115.7
N1−C5	1.341	1.340	C3−C4−C5	122.1	120.9
N1−C6	1.475	1.480	C4−C5−N1	121.6	121.5
C1−C2	1.359	1.365	C5−N1−C1	119.8	120.1
C2−C3	1.430	1.417	N1−C1−C2	120.1	120.6
C3−C4	1.414	1.416	C1−N1−C6	120.0	118.7
C4−C5	1.355	1.364	C5−N1−C6	120.1	121.1
C6−C7	1.530	1.515	N1−C6−C7	114.1	113.1
C7−C8	1.552	1.550	C6−C7−N2	110.7	110.7
			C6−C7−C8	105.1	106.8
			N2−C7−C8	109.7	111.2
			C7−C8−O3	115.8	113.8
			C7−C8−O4	117.1	118.0
			O3−C8−O4	127.1	128.2

Hydrogen bond lengths

O1−N2*(−x, −½+y, ½−z)	2.699
O2−O3*(½−+x, ½−y, 1−z)	2.704
O3−N2 (½−x, −y, −½+z)	2.880
O3−N2*(x, −1+y, −1+z)	2.793
O4−O2*(½+x, ½−y, 1−z)	2.696
N2−O1*(−x, −½+y, ½−z)	2.713
N2−O4*(x, −1+y, z)	2.882
O1*−N2*(−x, −½+y, ½−z)	2.872

Other intermolecular distances (*cf.* Fig. 2)

O1−N1*	3.348
O1−C1*	3.578
O1−C5*	3.355
O1*−N1	3.448
O1*−C1	3.694
O1*−C5	3.690

reported for amino acids.[18] In both molecules the hydrogen atoms are tetrahedrally arranged about the nitrogen atom; their positions correspond to a $10-15°$ rotation about the C−N bond relative to a strictly staggered conformation.

The hydroxypyridone part. The six-membered rings are nearly planar, the deviations of the atoms from a least-squares plane being less than 0.03 Å. The non-hydrogen atoms attached to the rings are situated from 0.05 to 0.12 Å out of the ring planes.

Except for the C3−O1 and the C3*−O1* bond lengths the two hydroxypyridone moieties are identical. An estimate of the relative contribution to the structure from the formulae I and II indicated in the introduction may be based on several features in the geometry. The mean value of the C1−C2, C4−C5, C1*−C2*, and the C4*−C5* bonds is 1.361 Å (e.s.d. 0.005 Å) as compared to the mean value for C2−C3, C3−C4, C2*−C3* and C3*−C4* of 1.419 Å (e.s.d. 0.007 Å). The corresponding bond lengths in quinone are 1.322 Å and 1.477 Å,[19] and in quinone in the quinone-resorcinal complex 1.330 Å and 1.468 Å,[20] respectively. The C−C bond length to be expected for
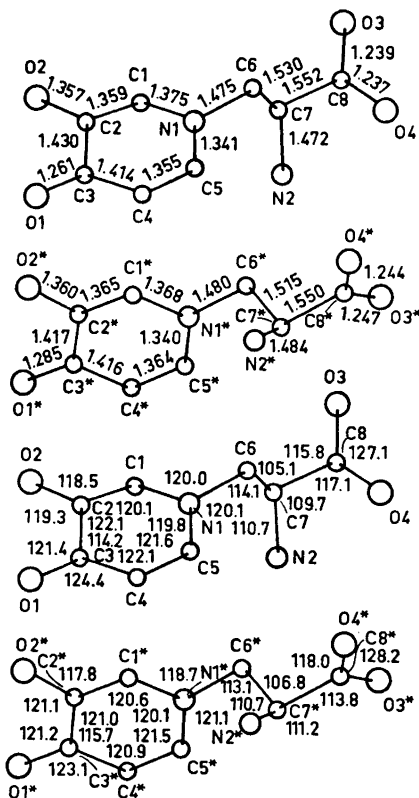
Fig. 1. Bond lengths (Å) and angles (°) observed in L-mimosine.
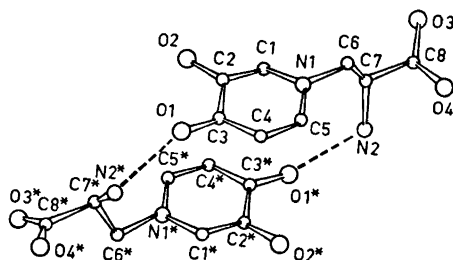
Fig. 2. The dimeric arrangement of the two crystallographically independent molecules of L-mimosine.

an aromatic model is 1.397 Å; the contributions from the two extreme structures appear thus to be about equal. This feature is also indicated by the C3 − O1 and C3* − O1* bonds of 1.261 Å and 1.285 Å, these being close to the mean length of the quinone C=O bond (1.22 Å) and the phenolic C − O bond (1.36 − 1.38 Å).

From these results one might expect the C − N bond lengths to be somewhere between the values reported for pyrrol, 1.374 [21] ("quinoid" case) and for pyridine, 1.339 [22] ("aromatic" case). In the present structure determination both of these values were found for the C − N bonds, the C1 − N1 bond being the longer in both molecules. The difference between the two C − N bonds in each of the two independent molecules is significant according to normal standards of structure analysis; we find no obvious explanation for this difference, however.

The two phenolic C − O bonds are equal in the two molecules, 1.357 and 1.360 Å, respectively.

The bond between the hydroxypyridone and the alanine moieties, $N1 - C6$, is found equal to 1.475 and 1.480 Å. The $N_{sp^3} - C_{sp^3}$ bond length to be expected is somewhat shorter than the aliphatic $N - C$ of 1.47 Å; the relative lengthening in the present case may be attributed to the positive charge on the nitrogen atom in analogy with the bond length in $C_{sp^3} - \overset{+}{N}H_3$ of 1.49 Å.

The crystal structure. Packing and hydrogen bonding. The high values for the melting point (decomp. 227 – 228°C) and density (1.59 g cm^{-3}) indicate strong intermolecular forces and a close packing of the molecules in the crystal. The packing coefficient [23] estimated from data given by Bondi [24] is as high as 0.82 as compared with the one calculated for L-DOPA of 0.79 and L-tyrosine of 0.75, whereas a normal value for benzene derivatives is given by Kitaigorodskii [23] to be 0.69. It seems reasonable to relate the high values to the zwitterionic character of the molecules, especially for L-mimosine. From the partial quinoid structure it may be deduced that the nitrogen atom in the ring is positively and the keto ozygen atom negatively charged. This also seems reasonable from the way pairs of crystallographically independent molecules are forming dimers in the crystals. The asymmetric unit may be represented as visualized in Fig. 2. The two molecules are linked together by two hydrogen bonds, $O1 - N2*$ (2.699 Å) and $O1* - N2$ (2.872 Å). The planes of the pyridone moieties are nearly parallel, the interplanar separation being 3.4 Å. The shortest distances between atoms from each molecule apart from the hydrogen bonded atoms are $N1 - O1*$ of 3.448 Å and $N1* - O1$ of 3.348 Å. This may indicate
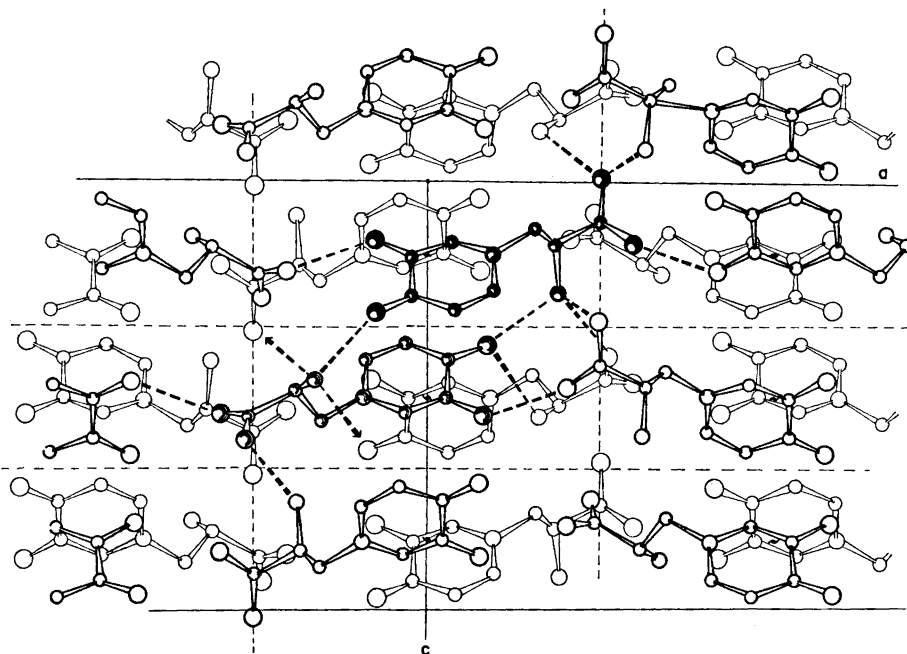
Fig. 3. The crystal structure as seen along the *b*-axis. Hydrogen bonds from one dimer (shaded) are indicated by broken lines.

an electrostatic interaction between the two ring systems owing to their zwitterionic character, and results in a pseudo centre of symmetry between the planar parts of the molecules.

The hydrogen bond system in the crystal is shown in Fig. 3, the hydrogen bond lengths are listed in Table 4. All hydrogen atoms bonded to hetero atoms are engaged in hydrogen bonds, each molecule being involved in eight hydrogen bonds to seven different neighbouring molecules. There is some difference in hydrogen bonding for the two crystallographically independent molecules in that the carbonyl oxygen atom (O1) in the pyridone part is hydrogen acceptor for only one ammonium group, whereas the corresponding oxygen atom (O1*) of the other molecule act as an acceptor of two such hydrogen bonds. Correspondingly, one of the oxygen atoms of the carboxyl group (O3) is acceptor for two ammonium hydrogen atoms and O3* for only one. The nitrogen atom N2 is engaged in hydrogen bonds to two carboxyl oxygen atoms (O3 and O4*) and one carbonyl oxygen atom (O1*); N2* is hydrogen bonded to one carboxyl (O3) and two carbonyl (O1 and O1*) oxygen atoms.

The molecules of type II (*) related by a screw axis in the b direction are connected through the hydrogen bonds between O1* and N2*. Similarly, molecules of type I related by a screw axis along a are forming chains by means of hydrogen bonds between N2 and O3. Both types of molecules are involved in the hydrogen bonded helix about the screw axis along the c direction through the hydrogen bonds O2 − O3* and O4* − N2.

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