Literatur

BORCHERT, W. (1952). *Miner. Petrogr.* **2**, 124.
BRESLOW, R. (1968). *Angew. Chem.* **80**, 573.
DIETRICH, H. (1953). Diplomarbeit, Heidelberg.
DIETRICH, H. & DIERKS, H. (1968). *Angew. Chem.* **80**, 487; *Angew. Chem. International Ed.* **6**, 465.
DORKO, F. A., HENCHER, J. L. & BAUER, S. H. (1968). *Tetrahedron,* **24**, 2425.
DUNITZ, J. D., FELDMAN, H. G. & SHOEMAKER, V. (1952). *J. Chem. Phys.* **20**, 1708.
DUNITZ, J. D. & MUGNOLI, A. (1966). *Helv. chim. Acta,* **49**, 1680.
HEILBRONNER, E. (1966). *Theor. chim. Acta.* **4**, 64.
I.U.Cr. (1966). *World List of Crystallographic Computer Programs.* 2nd ed. Utrecht: Oosthoek.
KÖBRICH, G. & HEINEMANN, H. (1965). *Angew. Chem.* **77**, 590; *Angew. Chem. International ed.* **4**, 594.
KÖBRICH, G., HEINEMANN, H. & ZÜNDORF, W. (1967). *Tetrahedron,* **23**, 565.
SCHLENK, W., JR (1949). *Liebigs Ann.* **565**, 204.
WELTIN, E., GERSON, F., MURRELL, J. N. & HEILBRONNER, E. (1961). *Helv. chim. Acta,* **44**, 1400.

Acta Cryst. (1970). B**26**, 53

The Crystal and Molecular Structure of 1-(1-Phenylcyclohexyl) piperidine hydrochloride

BY PATRICK ARGOS*, R. E. BARR† AND A. H. WEBER

Physics Department, Saint Louis University, Saint Louis, Missouri 63103, *U.S.A.*

(*Received* 21 *November* 1968)

1-(1-Phenylcyclohexyl)piperidine hydrochloride ($C_{17}H_{25}N.HCl$) is monoclinic with space group $P2_1/c$, a = 8·880, b = 13·866, c = 13·098 Å, β = 104°33′, four molecules per unit cell, and measured density 1·190 g.cm^{-3}. 1-(1-Phenylcyclohexyl)piperidine hydrobromide ($C_{17}H_{25}N.HBr$), investigated also to aid in the elucidation of the chlorine atom position, is monoclinic with space group $P2_1/c$, a = 8·888, b = 14·132, c = 13·396 Å, β = 104°28′, four molecules per unit cell, and measured density 1·322 g.cm^{-3}. For the hydrochloride, 2159 visually estimated reflection intensities corrected for extension, contraction, and absorption yielded Patterson–Harker sections to locate the chlorine atom, and Fourier difference syntheses to find the carbon, nitrogen, and hydrogen atom positions, refined by the block-diagonal least-squares approximation with anisotropic temperature factors. The final R index was 0·11.

The phenyl (1) ring is equatorial and the cyclohexane (2) and piperidine (3) rings are in chair conformations. The angles between normals to the mean planes of (1) and (2), (2) and (3), and (1) and (3) are 110, 15 and 96° respectively. Average bond lengths uncorrected for thermal motion are 1·382, 1·536 and 1·512 Å for C–C of (1), (2), and (3) respectively; 1·508 Å for C–N of (3); 3·088 Å for N–Cl (hydrogen bond); and 1·09 and 1·03 Å for C(sp^3)–H and C(sp^2)–H respectively. Average bond angles are 120·0, 110·9 and 111·1° for (1), (2), and (3) respectively; 119·4° for C–C–H of (1); 108·7° for N–C–H, C–C–H, and H–C–H of (2) and (3).

Introduction

1-(1-Phenylcyclohexyl)piperidine hydrochloride (also termed sernyl or phencyclidine hydrochloride) (SHCl, $C_{17}H_{25}N.HCl$) and sernyl hydrobromide (SHBr, $C_{17}H_{25}N.HBr$), supplied by Parke, Davis & Company, Ann Arbor, Michigan, are compounds with neurotropic effects of interest in chemistry and biology. Single crystals were grown in solutions of ethanol and butyl ether by slow evaporation at room temperature. Only the (y,z) bromine position was determined in SHBr to elucidate the location of the chlorine atom in SHCl.

Crystal data

The crystal data for SHCl and SHBr are shown in Table 1. The unit-cell parameters were determined from zero-layer a and b axis equi-inclination photographs calibrated by an aluminum powder diffraction pattern on the same film (Swanson & Tatge, 1953) with extrapolation correction (Taylor & Sinclair, 1945), excepting the SHBr b-dimension taken from a rotation photograph. The space group $P2_1/c$ (No. 14, *International Tables for X-ray Crystallography*, 1952) was uniquely determined by systematic absences $h0l$ with l odd and $0k0$ with k odd. The crystal densities were the same as thosc of isopropyl bromide at 16·0 °C for SHBr and dimethyl phthalate at 24·5 °C for SHCl. The SHCl crystal selected for analysis had dimensions 0·76, 0·39, and 0·41 ± 0·01 mm; the SHBr crystal was about 0·5 mm on a side.

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* Present address: Washington University School of Medicine; St. Louis, Missouri.

† Present address: University of Missouri; Columbia, Missouri.

Table 1. *Crystallographic data for* SHCl *and* SHBr

Parameter	SHCl	SHBr
a	8·880 ± 0·010 Å	8·888 ± 0·010 Å
b	13·866 ± 0·020	14·132 ± 0·010
c	13·098 ± 0·010	13·396 ± 0·010
β	104°33′ ± 10′	104°28′ ± 10′
Space group	$P2_1/c$	$P2_1/c$
Z	4	4
V	1561·0 Å^3	1629·3 Å^3
Formula weight	279·9	324·3
μ (Cu $K\alpha$)	20·3 cm^{-1}	36·5 cm^{-1}
ϱ_c	1·191 g.cm^{-3}	1·321 g.cm^{-3}
ϱ_o	1·190 ± 0·004 g.cm^{-3}	1·322 ± 0·003 g.cm^{-3}

Intensity data

For SHBr a zero layer a axis Weissenberg equi-inclination photograph was taken at room temperature with nickel-filtered Cu $K\alpha$ radiation. SHCl photographs were taken for the zero to seventh layers about the b axis and the zero to fifth layers about the a axis. Intensities with range 1 to 2500 for SHCl and 1 to 300 for SHBr were measured by visual comparison with a series of timed exposures of an hkl reflection; strong and weak reflections were correlated by the multiple-film technique. For SHBr 154 $0kl$ intensities were observed with maximum sin θ value 0·979; for SHCl the intensity data for 2311 reflections with maximum sin θ value 0·993 were collected from 3950 non-equivalent hkl intensities. Interlayer scaling factors were obtained through common reflections.

Absorption, extension, and contraction (Phillips, 1954, 1956) corrections were applied to the SHCl data. The SHCl a axis intensities were corrected for absorption assuming cylindrical symmetry with the area of the crystalline rhombic face nearly normal to the **a** direction (85° 20′) set equal to the cylindrical cross section (μR = 0·45) (*International Tables for X-ray Crystallography*, 1959). The correction for the b axis intensities employed the absorption integral evaluated by the m-point Gaussian integration formula (Busing & Levy, 1957; Wells, 1960; Kane, 1966). Wilson's (1942) method gave an overall temperature factor of B = 1·5 Å^2 for SHBr and B = 1·3 Å^2 for SHCl with an approximate absolute scale factor.

Structure determination and refinement

The SHCl [100] Patterson map indicated two possible chlorine positions; however, the subsequent SHBr [100] map clearly indicated the position of the bromine atom (y=0·31, z=0·51). The SHCl and SHBr [100] Fourier syntheses proved ambiguous because of overlap; three-dimensional data for SHCl were then collected. Patterson–Harker sections clearly indicated the chlorine position (x= −0·120, y=0·313, z=0·515). The R index $(\sum\limits_{F}|F_o-F_c|/\sum\limits_{F}|F_o|)$ based on the chlorine position was 0·53 with B = 1·3 Å^2. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1959).

A three-dimensional Fourier difference synthesis, computed with 1523 reflections with phases based on the chlorine atom alone and for which $|F_o| \le 2|F_c|$ indicated plausible carbon and nitrogen positions which yielded an R index of 0·33 with B = 1·3 Å^2. Two difference synthesis refinements and several cycles of

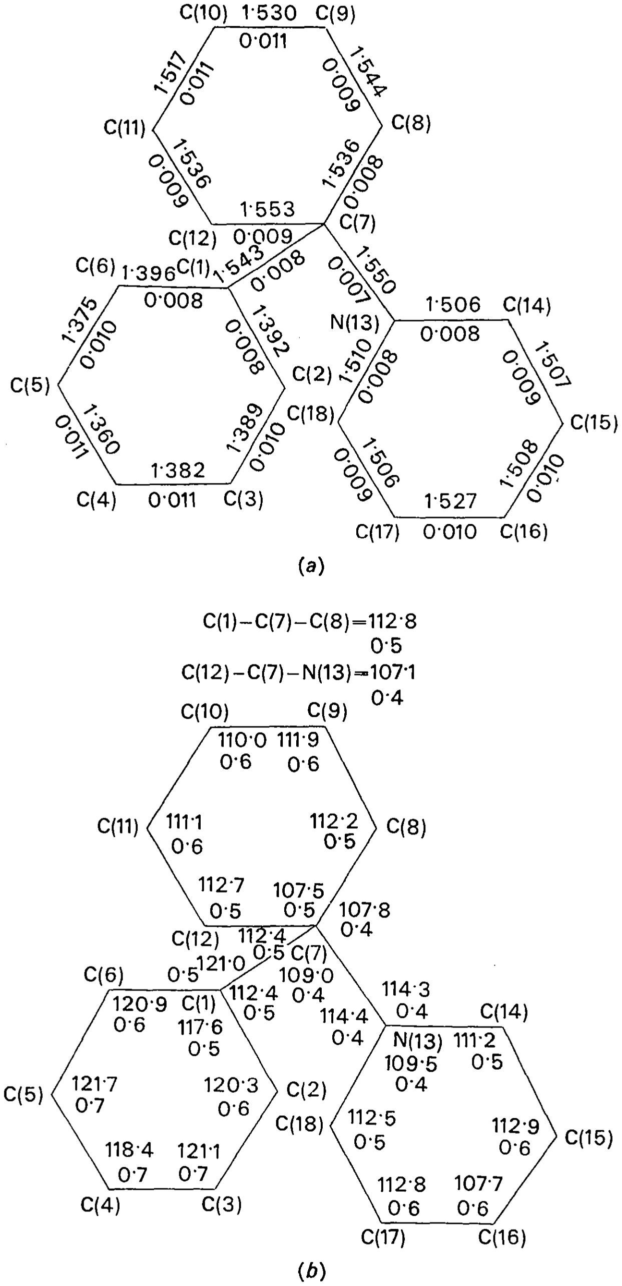

Fig. 1. (*a*) Bond lengths (Å) with estimated standard deviations (Å) given below the bond value. (*b*) Bond angles (degrees) with estimated standard deviations (degrees) given below the bond value.

Table 2. *Observed and unobserved structure factors*

(*a*) Observed (FO) and calculated (FC) structure factors (× 10) and associated Miller indices (*HKL*). An asterisk indicates a reflection deleted from the refinement and *R* factor calculations.

H	FO	FC
K=	0, L=	0
-1	709	874
-2	809	-1201
-3	577	-668
-4	580	-631
-6	41	-44
-7	78	78
-8	138	134
K=	0, L=	2
0	481	-626
1	199	-177
3	151	151
4	205	-217
5	301	-352
6	359	-332
8	46	59
9	128	140
10	199	221
-1	203	-277
-2	701	809
-3	616	-785
-4	109	88
-5	461	447
-7	107	83
-8	209	201
-9	200	191
K=	0, L=	4
0	825	895
1	247	238
2	86	65
3	99	-68
4	267	-266
5	346	313
7	130	-135
8	93	89
-1	198	160
-2	160	-125
-3	468	-541
-4	589	-584
-5	478	-562
-6	113	-108
-7	220	216
-8	64	67
-9	227	159
-10	113	95
-11	127	-79
K=	0, L=	6
0	516	431
1	874	732
2	216	202
3	340	329
4	423	-435
5	606	-622
6	126	-144
8	141	160
9	20	28
-2	157	161
-3	368	-386
-4	194	-184
-5	74	-95
-6	150	149
-7	328	323
-8	109	95
-9	43	-22
-10	167	-133
K=	0, L=	8
0	357	-311
1	182	146
2	249	233
3	113	103
4	169	-196
5	144	-152
7	43	-61
-2	118	-109
-3	440	-437
-4	303	-284
-5	59	-52
-6	236	280
-7	166	178
-8	75	65
-9	122	105
K=	0, L=	10
0	49	27
1	489	435
2	162	156
3	176	181
4	94	98
5	175	-185
6	40*	-49
7	35*	-63
-1	54	44
-2	162	164
-3	167	-168
-4	400	-479
-6	106	133
-7	372	424
-8	123	157
-9	110	-93
-10	103	-111
K=	0, L=	12
1	205	213
2	213	227
3	213	-245
-2	434	-492
-3	143	-142
-4	294	321
-5	137	142
-6	61*	97
-8	110	123
-9	170	135
K=	0, L=	14
1	125	142
2	56	108
3	80	102
-1	213	-233
-2	295	-333
-3	177	-210
-4	176	-191
-5	92	94
-6	237	230
-7	94	66
K=	0, L=	16
0	150	145
-1	52	39
-2	170*	-275
-3	125	-145
-4	122	129
-5	65	88
K=	1, L=	0
4	240*	559
-1	85	73
-2	397	-445
-3	100	-88
-5	123	-112
-6	231	218
-7	169	155
-8	77	90
-9	72	-85
H	FO	FC
-10	54	-76
K=	1, L=	1
0	248	289
1	444	387
2	647	784
3	418	-454
4	557	-738
5	118	-130
6	101	-99
-1	133	-125
-3	541	589
-4	379	380
-5	83	-75
-6	179	-170
-7	43	22
-8	44*	73
-9	42*	70
K=	1, L=	2
0	391	567
1	208	-172
2	642	698
3	306	323
4	95	85
5	156	-150
6	292	-304
7	138	-132
8	76	-68
-1	140	-132
-2	595	-812
-5	309	344
-6	216	234
-7	103	101
-8	62	57
-9	73	-66
-10	51	-55
K=	1, L=	3
0	163	148
1	327	307
2	172	-140
4	240	-217
5	86	84
6	158	124
-1	821	-1053
-2	90	-80
-3	246	-745
-4	301	-329
-5	93	92
-6	52	58
-7	144	140
-8	100	67
K=	1, L=	4
0	182	-170
2	416	383
3	266	268
4	211	206
5	81	-92
6	253	-268
8	90	-99
9	48	52
-1	885	-1038
-2	419	-531
-3	222	-254
-4	55	-26
-5	463	512
-6	168	167
-7	41	56
-9	103	-78
K=	1, L=	5
0	356	301
1	847	774
2	289	273
3	217	-177
4	200	-193
9	42	-52
-1	75	-48
-2	82	-71
-3	54	40
-4	131	115
-5	339	-398
-6	321	-367
-7	142	126
-8	168	167
-9	103	93
-11	76	-76
K=	1, L=	6
0	38*	-8
1	112	-102
2	361	352
3	139	153
4	126	126
5	313	323
6	99	-107
7	89	-112
8	72	-67
9	81	-91
-1	239	-221
-2	207	-209
-3	170	-169
-4	115	117
-5	114	116
-6	38	21
-7	188	189
-9	101	-122
-10	60*	-100
-11	137	-110
K=	1, L=	7
0	176	165
2	130	-137
3	47	40
4	118	-176
5	137	-134
-1	57	-37
-3	133	-156
-4	74	-76
-5	138	139
-6	178	187
-7	143	124
-10	59	-32
-11	39	-19
K=	1, L=	8
0	175	143
2	69	77
3	261	275
4	111	-101
7	58	-90
8	59	-89
-1	519	-501
-2	279	-277
-4	155	132
-5	265	286
-6	309	304
-7	161	165
-9	96	-83
-10	149	-150
K=	1, L=	9
0	267	-215
1	251	253
2	154	136
3	96	98
H	FO	FC
4	131	125
5	41*	-70
-1	247	-216
-2	223	-196
-3	158	-158
-4	122	128
-5	52	-76
-6	101	-117
-9	91	95
-10	30	41
K=	1, L=	10
0	568	-554
1	45	-55
2	155	136
3	317	315
4	192	203
5	65	71
6	65	-79
7	60	-78
-1	577	-514
-2	58	78
-4	249	246
-5	219	219
-7	72	-79
-8	68	-83
-9	35	-47
K=	1, L=	11
0	403	391
2	74	-77
5	98	-108
-1	434	479
-2	122	-98
-3	386	-393
-4	241	-199
-5	219	144
-6	327	287
-8	65	-77
K=	1, L=	12
3	54	74
5	87	109
-2	123	-101
-4	157	161
-5	97	82
-6	239	211
-7	179	145
-8	183	-163
K=	1, L=	13
0	276	-247
3	25*	40
4	70	82
-1	59	-80
-3	149	-133
-5	115	101
-6	206	167
-7	63	77
K=	1, L=	14
0	245	-253
1	41	-57
3	133	164
-1	239	-269
-2	130	-105
-4	234	235
-5	219	265
-6	195	168
-7	29*	-50
K=	1, L=	15
0	50	54
2	26*	48
2	52	48
-1	73	81
-2	109	-114
-3	32*	-56
-4	109	-105
-5	104	-99
-6	81	82
K=	1, L=	16
0	143	-176
-3	43	62
-4	116	124
K=	2, L=	0
0	288	367
1	24	10
-1	92	75
-2	564	-681
-4	603	711
-5	282	288
-6	216	-211
-7	258	-232
-9	126	-96
K=	2, L=	1
0	678*	1189
1	296	265
2	603	745
4	222	-228
5	83	-65
6	104	97
7	112	90
8	102	-103
-1	471	533
-2	537	-635
-3	395	-465
-4	508	-547
-5	237	253
-6	378	398
-9	148	-111
K=	2, L=	2
0	500	-640
1	857	-990
2	375	-375
3	111	98
4	124	135
5	154	141
6	299	283
8	146	-132
-1	605	730
-2	59	45
-3	106	105
-4	230	222
-5	164	152
-7	43	-21
-8	78	-92
K=	2, L=	3
0	207	-224
1	179	157
2	412	266
3	69	-64
4	207	200
5	29	-18
6	435	-396
7	200	-216
8	68	-91
-1	691	-600
-2	420	-441
-3	131	-126
-4	150	122
H	FO	FC
-5	473	492
-6	217	227
-7	128	122
-8	44*	88
-9	105	-52
K=	2, L=	4
0	175	-164
1	97*	35
2	26	14
3	30	15
4	276	258
5	143	159
7	41	-53
8	36	-48
-1	527	-539
-2	364	-379
-3	221	269
-4	51	52
-5	310	344
-6	145	142
-7	240	-273
-8	154	-145
-9	61	-72
K=	2, L=	5
0	210	191
1	513	-458
2	45	44
3	226	211
4	175	151
5	319	340
6	60	41
7	68	-101
8	100	-106
-1	358	-314
-2	470	-533
-5	37	27
-6	66	-70
K=	2, L=	6
0	492	-459
1	610	-494
2	161	-133
3	112	105
4	215	204
5	50	-44
8	73	-67
-1	285	267
-2	531	598
-3	311	321
-4	101	87
-5	78	-60
-6	260	-277
-9	162	-148
K=	2, L=	7
0	35	44
1	448	370
2	271	247
3	206	172
6	95	-128
7	57*	-98
-1	438	-427
-2	48	31
-4	92	99
-5	347	379
-6	154	97
-7	42	46
-9	122	-86
K=	2, L=	8
0	338	-270
1	446	-329
2	84	57
3	123	-119
5	197	194
6	108	142
7	51*	92
-1	74	-40
-2	70	55
-3	188	183
-4	99	85
-5	173	173
-6	187	172
-7	74	-96
-8	220	-243
-9	39	-50
K=	2, L=	9
0	314	-235
1	76	-76
2	89	97
3	75	83
4	39	-43
5	139	125
7	90	-100
-1	432	-432
-2	301	-282
-4	211	219
-5	174	185
-6	193	167
-7	221	193
-8	71	-93
-9	145	-175
K=	2, L=	10
0	180	-151
1	171	-152
2	61	-66
3	117	102
4	42*	71
-1	195	-174
-2	377	376
-3	364	385
-6	196	-177
-9	61	-88
K=	2, L=	11
0	73	-45
1	86	-98
2	117	-113
3	260	284
4	174	173
5	61	62
6	44	61
-1	132	-122
-2	134	111
-8	37	-46
-9	121	-126
K=	2, L=	12
0	51*	-21
1	131	-117
2	153	-168
3	121	-125
5	31	44
-1	89	63
-2	122	102
-3	156	159
-4	347	304
-6	239	-221
-7	224	-185
-8	34	-53
-9	48	60
K=	2, L=	13
H	FO	FC
0	304	-306
1	77	-86
2	80	89
3	124	157
-2	132	136
-3	54	36
-4	59*	208
-5	189	160
-6	95	96
-7	35	42
-8	104	-81
K=	2, L=	14
0	33*	-55
1	74	-94
2	68	-78
-1	65*	-4
-2	121	102
-3	50*	82
-4	196*	56
-6	48	-53
-7	51	-60
-8	50	-63
K=	2, L=	15
0	173	-188
1	124	-116
2	40*	-63
-1	172	-183
-2	94	-84
-4	95	99
-5	137	112
-6	114	113
-7	36	-44
K=	2, L=	16
0	99	114
-1	37	52
-3	103	113
-4	166	171
-5	63	-59
K=	3, L=	0
-1	194	-129
-2	513	685
-3	87	68
-4	364	-396
-5	273	-235
-6	106	-68
-9	56	-79
K=	3, L=	1
0	557	-751
1	220	-222
2	129	-119
3	70	61
4	254	261
5	46	44
6	233	215
7	143	134
8	134	-124
9	112	-108
-1	347	-327
-2	45	-34
-3	451	529
-4	298	337
-5	33	36
-7	265	-248
-8	115	-109
-9	44	-62
-10	111	-114
K=	3, L=	2
0	52	-34
1	424	-388
2	75	-68
3	35	-25
4	200	205
5	42	-52
6	368	-350
8	163	149
-1	678	846
-2	98	80
-3	227	243
-4	336	360
-5	101	-70
-6	157	-147
-7	77	-119
-9	62	88
K=	3, L=	3
0	694	-936
1	391	-473
2	120	90
3	80	86
4	304	297
5	327	336
6	174	160
7	151	-148
8	110*	-114
8	138	-114
9	55	-65
-2	355	392
-3	317	360
-4	212	221
-5	161	167
-6	68	77
-7	62	-48
-8	66	-75
-9	153	-133
K=	3, L=	4
0	272	267
1	359	-343
2	393	-459
3	79	-85
4	180	-189
5	126	106
6	158	166
7	42	-24
8	53	57
-1	44	-31
-2	237	-216
-4	105	-110
-5	83	75
-6	39	59
-7	107	-118
K=	3, L=	5
0	242	-230
1	31*	4
2	70	-81
3	172	-142
4	197	183
7	40*	65
8	58	-83
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-6	68	-74
-7	151	-161
-8	135	-114
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K=	3, L=	6
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H	FO	FC
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3	109	-103
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7	112	125
8	52	72
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-2	101	77
-3	191	203
-5	160	-162
-6	211	-259
-7	168	-198
-8	63	43
K=	3, L=	7
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1	734	-565
2	288	-276
4	213	196
5	292	288
6	138	133
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8	35	-32
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-2	261	246
-3	246	277
-4	194	212
-5	217	247
-6	191	-219
-7	339	-362
-8	108	-125
-9	59*	-92
-10	59*	18
K=	3, L=	8
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2	163	-130
3	159	-158
4	147	-152
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-3	159	152
-4	70	-77
-5	40	-33
-6	104	-124
-7	76*	126
-8	225	249
K=	3, L=	9
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1	132	-100
2	164	-156
3	175	-177
5	150	146
6	79	108
7	23*	52
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-2	41	26
-3	239	232
-4	130	140
-5	42	34
-8	59	-71
K=	3, L=	10
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4	220	-144
5	69	-85
6	103	94
-1	128	-115
-2	44	61
-3	41	56
-5	81	-96
-6	152	-158
-7	75	-106
-8	69	-105
-9	61*	102
-10	174	167
K=	3, L=	11
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6	33	-39
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-4	138	127
-5	74	104
-6	227	-238
-7	342	-326
-8	114	-128
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-4	137	-127
-5	80	-85
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2	151	-161
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4	30*	-63
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-2	304	333
-3	174	181
-5	40*	-76
-6	149	-151
-7	85	-89
-8	87	-95
K=	3, L=	14
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-7	175	141
-8	46	61
K=	3, L=	15
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-3	69	75
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K=	3, L=	16
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-1	101	121
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-3	89	-106
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K=	4, L=	0
H	FC	FC
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-5	72	-64
-6	135	-115
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K=	4, L=	1
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2	84	-71
3	177	-174
4	294	-317
5	128	125
6	213	213
7	168	171
8	75	76
9	89	-132
10	101*	-37
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-6	130	-99
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K=	4, L=	2
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2	105	87
3	141	134
4	41	50
5	122	-121
6	159	162
7	205	253
9	131*	15
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-7	80	-103
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K=	4, L=	3
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4	67	-102
5	131	120
6	203	230
7	43	48
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-2	381	444
-3	170	172
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-6	189	-210
-7	78	-80
-9	171	137
-10	117	103
K=	4, L=	4
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2	67	42
3	134	-120
4	89	89
5	26	25
6	252	-283
7	103	-107
8	110*	-6
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-2	149	-128
-3	293	-287
-5	112	92
-6	235	235
-7	110	117
K=	4, L=	5
0	266	217
1	154	114
2	156	-140
3	345	-362
4	180	-161
5	114	102
6	106	107
7	98	125
8	115	115
-1	252	229
-2	67	-59
-3	76	77
-5	107	-85
-6	160	-157
-7	268	-280
-10	108	90
-11	121	148
K=	4, L=	6
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1	485	-421
2	180	-162
3	114	94
4	43*	-16
5	43	48
6	73	111
9	25	24
-1	235	234
-2	40	31
-3	55*	15
-4	127	-103
-5	225	-221
-7	109	-108
-8	78	-78
-10	35	31
K=	4, L=	7
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2	307	-276
3	228	-230
4	137	-136
6	137	142
7	47	-49
-1	525	454
-2	410	427
-3	192	189
-4	131	-112
-5	318	-319
-6	319	-324
-8	155	109
-9	143	113
-10	189	139
K=	4, L=	8
1	180	145
2	67	56
4	43*	75
6	36*	-67
-1	46	-37
-2	171	146
-3	219	219
-5	33*	56
-7	173	-191
-8	75	82
-9	97	87
-10	31	-26
H	FO	FC
K=	4, L=	9
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1	169	176
2	109	-99
3	352	-337
4	155	-201
5	56	-75
6	45	61
7	127	153
-1	178	150
-2	39	45
-3	108	92
-4	220	-217
-5	226	-217
-10	29*	57
K=	4, L=	10
0	51*	-21
1	39	35
2	44*	77
3	82	-92
4	97	-92
6	45	-63
-1	56	-59
-2	166	-153
-7	168	177
-8	139	124
-9	78	-77
-10	101	58
K=	4, L=	11
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-2	248	255
-3	55	-72
-4	254	-238
-5	246	-273
-6	339	-300
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-8	224	215
-9	52	63
K=	4, L=	12
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4	26*	-60
5	40	54
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-3	73	93
-4	42	-48
-5	42	-49
-6	92	-93
-7	147	-149
K=	4, L=	13
0	105	108
2	46	57
3	36	-49
4	125	-156
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-3	53	-53
-4	191	-202
-5	171	-182
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2	32	45
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-3	134	118
-4	61*	-101
-7	131	132
K=	4, L=	15
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-3	102	-121
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-5	149	-129
-6	65	-68
K=	4, L=	16
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-2	114	-118
-3	94	-98
K=	5, L=	0
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-2	297	308
-3	229	214
-4	42	-24
-5	265	-263
-6	80	-68
-7	256	265
-8	43	41
-9	64	-77
K=	5, L=	1
0	395	399
1	201	-159
2	138	-117
3	200	-173
4	215	-208
5	158	-155
6	68	81
7	238	231
8	59	80
-1	665	800
-2	116	95
-3	353	-418
-4	268	-275
-5	370	-393
-6	233	-201
-7	84	61
-8	83	86
-9	67	72
K=	5, L=	2
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1	54	-55
2	253	-260
3	138	-123
4	62	43
5	161	-156
7	230	241
8	57*	107
9	32*	13
-1	84	-72
-2	186	-199
-3	80	71
-4	112	-74
-5	39*	7
-7	183	-185
-8	144	-121
-9	57	68
K=	5, L=	3
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1	301	313
2	417	425
H	FO	FC
3	126	110
4	191	-170
5	234	-250
6	173	-172
8	38	39
9	73	111
-1	291	309
-2	249	255
-3	213	-200
-4	344	-367
-5	137	-135
-6	246	235
-7	113	90
-9	170	163
K=	5, L=	4
0	57*	25
1	213	-177
2	156	-129
3	43	-25
4	97	89
5	112	95
7	40	-41
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-3	32	33
-5	124	-114
-6	285	-349
-8	181	189
K=	5, L=	5
0	369	385
1	366	341
2	52	-26
3	151	-149
4	103	-102
5	166	-159
6	237	-277
7	39	-52
8	55	78
9	74	96
-1	116	-75
-2	339	-367
-3	238	-248
-4	156	-146
-5	216	-232
-7	177	167
-8	178	187
-9	74	81
K=	5, L=	6
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2	39	19
3	72	-58
4	112	-108
5	143	-124
7	126	119
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-2	79	-70
-3	38	-39
-4	93	76
-5	165	-158
-7	64	62
-8	64	-67
-9	145	136
K=	5, L=	7
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1	267	222
2	170	152
4	115	-112
5	98	-124
7	102	98
8	79	97
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-2	323	-296
-3	416	-457
-4	118	-126
-7	157	-121
-8	63	54
-9	70	66
-10	32	-35
K=	5, L=	8
0	96	60
3	54	-44
4	156	-148
5	40	20
6	50*	90
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-3	111	89
-5	86	-66
-6	173	-169
-7	175	-173
-8	43	58
-9	55	52
K=	5, L=	9
0	249	222
1	331	289
2	169	147
3	45*	-80
4	87	-91
6	130	-121
7	130	-167
-2	175	-165
-3	122	-132
-4	258	-250
-5	56	-60
-6	236	247
-7	255	280
-8	240	250
-10	78	-60
K=	5, L=	10
0	51*	16
1	131	106
3	42	-43
4	69	-85
5	143	-134
6	34	38
-1	172	151
-2	123	139
-3	61	-73
-4	115	-91
-5	156	-134
-7	60	73
-8	35	54
-9	183	141
K=	5, L=	11
0	131	120
1	142	141
2	79	104
3	55	75
5	123	-102
-1	144	-146
-2	298	-290
-3	156	-133
-4	127	-114
-5	89	-96
-6	42	-65
-7	70	91
-8	199	194
K=	5, L=	12
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1	109	132
2	48	64
H	FO	FC
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4	90	-88
5	27	38
-2	77	-102
-3	45	-40
-4	87	-80
-5	44	-56
-6	57	68
-9	29	-40
K=	5, L=	13
1	174	179
2	233	264
3	92	104
4	61	-83
-1	59	-88
-2	90	-112
-3	128	-119
-4	57	-58
-5	153	132
-6	50	65
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-8	55	55
K=	5, L=	14
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2	24*	-37
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-6	87	-88
K=	5, L=	15
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1	92	98
-1	99	-108
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-3	175	-176
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-6	208	249
K=	5, L=	16
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-3	107	-118
-4	92	-105
K=	6, L=	0
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-5	403	-406
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-7	113	98
-8	126	121
-9	109	132
K=	6, L=	1
0	288	-274
1	75	-75
2	302	305
3	166	160
4	81	-70
5	140	-119
6	91	-79
7	132	-120
8	57	-78
9	113	-69
10	35*	76
-1	532	-550
-2	250	-246
-3	187	-177
-4	146	-134
-5	271	254
-6	209	170
-7	48*	138
-8	43*	68
K=	6, L=	2
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1	231	210
2	252	258
3	172	-168
4	196	-204
5	73	-68
7	75	87
8	115	103
10	56*	-23
-1	346	334
-3	123	-137
-4	196	-209
-5	65	-79
K=	6, L=	3
0	48*	-131
1	244	202
2	268	276
3	218	226
4	124	132
6	61	64
8	51	-59
-1	214	-215
-2	212	-208
-3	236	-229
-4	168	169
-5	50	45
-6	132	-99
-7	140*	20
-8	114	-130
K=	6, L=	4
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2	36	-32
4	41	-44
5	226	-238
6	75	-91
9	83	104
-1	188	190
-2	302	-269
-3	231	-218
-4	269	-248
-5	99	-107
-6	151	149
-7	159	151
-8	159	161
-9	125	103
K=	6, L=	5
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1	297	241
2	336	304
3	39	51
4	43	-49
5	62	-72
6	103	-94
7	53	-70
8	105	-116
9	66	-54
-1	273	-257
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-4	36	-51
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H	FO	FC
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-10	124	-114
K=	6, L=	6
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1	187	176
2	207	183
3	85	-96
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5	136	-133
6	200	-200
8	44	43
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-2	110	-110
-3	285	-274
-4	264	-238
-7	45	-58
-8	133	100
-9	97	44
K=	6, L=	7
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1	47*	-42
1	58	-42
2	52	-60
3	134	113
4	229	223
5	92	92
6	37	-42
7	136	-123
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-5	97	78
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-7	135	129
-8	61	-51
K=	6, L=	8
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1	73	34
2	126	124
3	43*	2
4	42	-41
5	117	112
7	117	-135
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-2	173	-169
-3	133	-121
-4	41	-26
-6	45	39
-7	173	166
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K=	6, L=	9
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3	135	128
4	143	166
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6	70	-67
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-2	92	-65
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-6	156	138
-8	69	-80
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K=	6, L=	10
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1	373	325
2	173	168
4	118	-128
5	171	-173
6	100	-104
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-2	367	-368
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-8	52	63
K=	6, L=	11
0	106	-89
1	46	-70
3	140	136
4	83	125
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-2	210	-210
-3	55	-63
-4	161	166
-5	213	215
-6	160	172
-7	93	90
-8	99	72
K=	6, L=	12
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3	40	60
-2	52	68
-3	169	-176
-4	131	-136
-6	37	-20
-7	160	137
-8	119	112
K=	6, L=	13
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1	49*	-89
2	51	70
3	69	90
-1	102	-105
-2	160	-146
-4	91	93
-5	39*	73
-6	66	80
-7	141	-106
-8	105	-128
K=	6, L=	14
1	166	165
2	146	169
-1	146	-181
-2	138	-130
-3	51	-52
-4	33	-37
-6	264	234
-7	200	203
K=	6, L=	15
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-1	24*	-10
-2	26*	54
-3	95	106
-4	36	45
-5	94	112
K=	7, L=	0
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-2	232	-222
-3	717	-234
-4	162	153
-5	377	386
H	FO	FC
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-7	188	205
-8	39	56
-9	45	-63
K=	7, L=	1
0	37*	-15
1	386	-389
2	335	-340
3	178	174
4	156	154
5	38	-27
6	44	35
7	105	-91
-1	456	435
-2	93	79
-4	106	94
-5	82	82
-6	46	-25
-8	58	65
-9	101	-92
K=	7, L=	2
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1	86	-55
2	169	167
3	187	180
4	199	210
5	86	96
6	62	-85
7	102	-129
-1	84	-84
-2	223	-215
-3	300	-319
-5	182	-160
-6	45	52
-7	202	213
-8	42	-22
K=	7, L=	3
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1	127	-113
2	64	77
3	37	33
4	123	101
6	107	98
7	70	88
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-2	140	-127
-3	48	35
-5	264	270
-6	220	257
-7	141	-135
-8	175	-179
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K=	7, L=	4
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1	308	280
2	138	115
3	284	315
4	117	100
5	67	-70
7	121	-144
-1	198	-189
-2	213	-195
-3	62	-49
-4	48	69
-5	58	29
-6	204	215
-7	212	215
-9	127	-129
-10	211	-247
K=	7, L=	5
0	80	67
1	138	-133
2	82	-85
3	29*	8
4	178	184
5	120	131
6	41	-58
7	107	-118
-1	71	93
-2	33	35
-3	102	99
-4	67*	17
-5	53	-43
-6	134	-123
-7	45	52
-9	112	-124
-10	50	51
K=	7, L=	6
0	562	-569
1	124	-95
2	265	241
3	343	371
4	206	227
5	271	-293
6	180	-229
7	56	-70
8	38	-40
-1	264	-241
-2	250	-229
-3	112	-112
-4	211	239
-5	259	288
-6	190	205
-7	45	-33
-8	43	-26
-9	37*	71
K=	7, L=	7
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1	251	-231
2	99	-83
3	45	53
4	45	23
5	59	-70
6	35*	66
7	69	79
-1	37	22
-2	37*	4
-3	57	79
-4	130	134
-5	177	190
-6	44	30
-7	78	-103
-8	103	-95
-10	49	-39
K=	7, L=	8
0	187	-197
2	189	172
3	105	114
4	156	158
5	64	79
6	44	56
-1	182	-175
-2	211	-215
-3	53	-45
-4	56	55
-5	124	113
-6	110	111
-8	99	-98
-9	144	-140
-10	85	-91

Table 2 (*cont.*)

H	FO	FC
K= 7, L= 9		
0	167	-152
3	82	-82
4	49	59
5	174	192
-1	208	-210
-2	71	87
-3	104	86
-4	43*	3
-5	99	89
-8	53*	-87
-9	74	-96
K= 7, L= 10		
0	134	-101
2	131	123
3	49	64
4	120	135
6	79	-79
-1	42	-24
-2	211	-198
-4	206	205
-5	278	306
-6	243	282
-8	146	-125
-9	78	-84
K= 7, L= 11		
1	48	-45
2	48	-61
-2	54	68
-3	203	211
-4	183	192
-5	165	-173
-6	249	-217
-8	30	24
K= 7, L= 12		
0	182	-182
1	173	-170
3	32	44
4	87	97
-1	281	-281
-2	182	-181
-3	175	163
-4	111	101
-5	97	116
-6	87	70
-7	83	-69
K= 7, L= 13		
0	85	-90
1	38	-45
-1	82	-92
-4	100	111
-5	159	152
-6	75	58
-7	76	-89
K= 7, L= 14		
0	144	-133
1	23	-14
-1	167	-151
-3	158	127
-4	176	174
-5	139	155
-6	58	-51
K= 7, L= 15		
0	85	-101
-2	126	128
-3	105	108
-4	37*	60
-5	90	-104
K= 8, L= 0		
0	40*	-7
1	45	-61
2	152	144
3	261	242
4	69	55
K= 8, L= 1		
0	99	-49
2	112	-115
3	187	178
4	224	-204
5	144	-130
-1	246	-211
-2	40	31
-3	162	137
-4	32	40
-5	250	-239
K= 8, L= 2		
0	451	-475
1	404	-354
2	115	-111
3	80	53
4	41	41
5	184	185
-1	684	-571
-2	107	96
-3	318	270
-4	285	269
-5	399	431
K= 8, L= 3		
0	233	-214
1	226	204
3	83	76
4	61	75
5	43	-41
-1	43*	9
-2	132	114
-3	154	-145
-4	125	112
-5	69	66
K= 8, L= 4		
0	433	-412
1	283	-248
2	118	-112
3	179	175
4	340	337
5	302	328
-1	283	-258
-2	210	182
-3	249	265
-4	283	265
-5	149	-139
K= 8, L= 5		
0	160	-159
1	46	35
2	115	114
3	63	-38
4	169	-157
5	44	-50
-1	150	144
-2	61	62
-3	46	37
-4	49	-54
-5	50	-63
K= 8, L= 6		
0	167	189
1	50	-42
2	210	-182
3	92	-77
4	143	-179
-3	205	187
-4	322	326
-5	216	233
K= 8, L= 7		
0	49	-31
2	161	-177
3	47	-55
4	169	169
5	43	-47
-2	67	-90
K= 8, L= 8		
0	415	-409
1	272	-253
2	276	-274
3	47	44
4	191	182
5	138	117
-1	199	-217
-2	213	196
-3	176	160
-4	256	250
-5	163	154
K= 8, L= 9		
0	51	-50
1	202	-188
4	150	144
5	61	83
-1	230	257
-2	86	82
-3	88	102
K= 8, L= 10		
1	101	-110
2	198	-199
3	131	-120
4	117	130
5	166	169
-1	243	229
-2	190	144
-3	160	136
-4	135	157
K= 8, L= 11		
0	83	100
1	46	63
3	81	-96
-1	101	-106
-2	183	-149
-4	97	89
-5	172	180
K= 8, L= 12		
0	61	-91
1	192	-193
2	118	-138
3	165*	-34
-1	114	135
-2	194	203
-3	154	157
-4	109	114
-5	71	80
K= 8, L= 13		
0	37	-70
1	170	-125
2	110*	3
-1	39*	62
-3	37	55
-5	106	-97
K= 8, L= 14		
0	182	-202
1	104	-113
-2	116	132
-3	152	173
-5	72	-85
K= 9, L= 0		
1	99	-105
2	203	-187
3	275	-265
4	120	-109
5	168	155
K= 9, L= 1		
0	312	269
1	62	-77
2	173	-142
3	83	91
4	107	-88
5	96	-85
-1	139	-118
-2	61	72
-4	244	241
-5	380	415
K= 9, L= 2		
0	190	153
1	143	-120
2	217	-198
3	226	-225
4	208	-197
-1	418	290
-2	317	292
-3	139	137
-4	157	-139
-5	199	-189
K= 9, L= 3		
0	339	-322
1	353	-351
3	108	106
4	45	46
5	235	242
-1	281	-264
-4	166	159
-5	50	53
K= 9, L= 4		
2	217	-201
3	151	-154
-2	270	256
-3	341	325
-4	100	102
-5	154	-155
K= 9, L= 5		
0	351	350
1	85	87
2	286	-273
3	46*	81
5	110	112
-1	174	154
-2	339	323
-3	97	-102
-4	224	-215
-5	113	-101
K= 9, L= 6		
0	132	116
1	268	-262
2	181	-162
3	133	-122
4	45	-61
-1	585	559
-2	278	278
-3	141	-145
-4	157	-158
-5	171	-152
K= 9, L= 7		
0	443	-483
1	173	-140
3	66	80
4	63	-67
5	141	-102
-2	265	250
-3	143	140
-4	311	345
-5	55	49
K= 9, L= 8		
0	238	199
2	65	-64
3	100	-96
4	223	-226
5	122	-97
-1	121	89
-2	111	108
-3	125	133
-4	53*	-94
-5	124	-136
K= 9, L= 9		
0	102	-112
1	50	-57
2	110	-109
4	38	40
5	94	89
-2	86	79
-3	71	75
-4	87	-119
-5	216	-221
K= 9, L= 10		
0	139	126
2	187	-172
3	114	-139
4	45	35
-1	237	227
-2	184	161
-3	83	-101
-4	68*	-107
-5	32	-37
K= 9, L= 11		
1	182	180
3	97	-85
-1	146	143
-2	175	139
-3	64*	104
-4	77*	124
-5	43*	-90
K= 9, L= 12		
0	191	201
1	37	45
-1	225	228
-2	40*	75
-4	92	-116
-5	211	-235
K= 9, L= 13		
0	66	-49
1	155	-142
-1	82	103
K= 9, L= 14		
0	86	132
-4	105	-121
K= 10, L= 0		
0	163	138
1	271	262
2	182	168
3	92	-80
5	127	-156
K= 10, L= 1		
0	141	123
1	66	-46
3	205	-193
-1	243	237
-2	256	248
-3	240	239
-4	180	-168
-5	200	-186
K= 10, L= 2		
0	263	276
1	137	-123
2	63	-64
3	171	-172
4	349	-369
-1	132	126
-3	48	-35
-4	202	-186
-5	89	-89
K= 10, L= 3		
0	187	202
1	227	-182
2	101	-86
3	233	-230
4	200	-192
-1	184	169
-5	146	130
K= 10, L= 4		
1	546	511
2	206	180
3	32*	55
4	48	-31
5	107	-105
-1	463	-389
-4	157	-152
-5	200	-206
K= 10, L= 5		
0	152	-133
1	269	-269
3	141*	28
5	105	85
-2	399	410
-3	209	209
-4	272	-278
-5	249	-254
K= 10, L= 6		
0	268	323
1	202	204
2	46	54
3	81	-89
4	193	-183
5	122	-113
-1	185	186
-2	85	-114
-3	71	-83
-4	89	111
-5	50	-68
K= 10, L= 7		
0	448	467
1	202	163
2	140	-140
3	209	-234
4	206	-205
-1	230	216
-2	99	95
-3	88	-101
-4	104	-104
-5	49	-59
K= 10, L= 8		
1	89	119
4	97	-114
5	105	-97
-2	48	-60
-3	313	-317
-4	218	-205
K= 10, L= 9		
4	104	-114
-2	97	97
-3	48	-60
K= 10, L= 10		
0	140	140
1	176	145
2	115	106
3	58	83
4	32*	53
-1	85	115
-2	136	-116
-3	161	-154
-4	113	-120
K= 10, L= 11		
0	199	213
1	70	98
2	158	-141
3	71	-75
-1	136	132
-2	42*	87
-3	59	-66
-4	103	-107
-5	210	-197
K= 10, L= 12		
0	36*	56
-1	78	111
-3	153	-168
-4	62*	-98
-5	118	90
K= 10, L= 13		
0	129	172
1	129	177
-1	67	99
-3	72	-95
-4	56*	-96
-5	69	-105
K= 11, L= 0		
1	79	77
2	187	176
4	91	87
5	47	45
K= 11, L= 1		
0	245	267
1	151	114
2	235	-240
3	92	-108
5	149	-153
-1	272	257
-2	33*	87
-3	71	-58
-4	140	-152
-5	88	-98
K= 11, L= 2		
0	70	72
1	51	45
2	198	162
5	110	-91
-1	103	94
-4	73	-83
-5	88	80
K= 11, L= 3		
0	197	215
1	347	322
2	162	146
3	248	-244
4	162	-145
5	61	-79
-1	140	131
-2	290	-287
-3	279	-289
-4	157	-163
K= 11, L= 4		
0	88	-116
1	51	-68
2	162	151
3	32	-18
5	103	98
-1	359	-324
-2	312	-317
-3	50	59
-4	129	139
-5	72	100
K= 11, L= 5		
0	179	177
1	90	87
2	188	182
3	210	136
4	43	-65
5	190	-198
-1	75	97
-3	22	-108
-4	256	-249
-5	69	-83
K= 11, L= 6		
0	271	316
1	52	50
2	32	-31
3	142	137
4	59	74
-2	48	-49
-3	215	-206
-4	254	-255
-5	99	93
K= 11, L= 7		
0	51*	-20
1	50	70
2	135	138
3	159*	-71
4	97	-100
5	124	-124
-1	74	88
-2	120	110
-4	248	-235
-5	117	-131
K= 11, L= 8		
0	121	-155
2	74	93
-1	136	-123
-4	129	143
-5	102	96
K= 11, L= 9		
0	81	77
1	178	168
2	223	259
3	94	98
4	66	-67
-1	139	-146
-2	177	-175
-3	196	-194
-4	225	-229
K= 11, L= 10		
1	82	-83
3	123	126
-1	78	-122
-2	196	-177
-3	149	-149
K= 11, L= 11		
1	99	121
2	140	142
-1	39	-51
-2	112	-87
-3	100	-118
K= 11, L= 12		
1	134	136
-1	81	-109
-2	31*	57
-4	43*	76
-5	34*	61
K= 11, L= 13		
-2	122	-130
-3	114	-128
K= 12, L= 0		
0	72	58
2	168	-145
5	103	-102
-2	172	-145
K= 12, L= 1		
0	72	-72
1	182	205
2	338	369
3	279	332
4	66	66
5	162	-166
-1	118	-121
-2	172	-146
-3	50*	-94
K= 12, L= 2		
0	155	-135
2	46	59
-2	86	72
-5	68	88
K= 12, L= 3		
0	127	115
2	80	-90
4	45	54
5	57	62
-2	292	-240
-3	152	-129
-5	169	168
K= 12, L= 4		
0	51	-47
1	240	216
2	161	145
3	152	-156
-1	107	-96
-2	110	-113
-3	50	57
-5	83	-114
K= 12, L= 5		
0	254	-288
1	30	73
2	212	246
3	43*	276
4	163	159
-1	278	-294
-2	169	-169
-3	111	-125
-5	94	101
K= 12, L= 6		
0	51	62
1	70	-77
4	38*	61
-1	91	-92
-2	223	-215
-3	154	144
-4	119	133
K= 12, L= 7		
1	48	43
2	243	206
3	119	148
-1	176	-154
-2	216	-216
-3	115	116
-4	202	225
-5	154	153
K= 12, L= 8		
1	31*	-51
-2	100	101
-4	63	-73
-5	108	107
K= 12, L= 9		
0	165	-162
3	30*	58
-1	250	-271
-2	42*	-78
-5	161	170
K= 12, L= 10		
-1	56	-72
-2	172	-160
-3	53	-76
K= 12, L= 11		
0	134	-129
-1	193	-186
-2	155	-156
-4	130	149
-5	124	149
K= 13, L= 0		
1	105	103
3	56	87
-2	224	-219
K= 13, L= 1		
0	179	-160
1	221	-209
2	65	-85
4	99	111
5	167	171
-4	224	198
-5	78	99
K= 13, L= 2		
0	51	45
1	89	-87
2	102	-116
3	151	166
4	43	47
-5	62	66
K= 13, L= 3		
0	102	-116
1	89	-121
2	45	-60
3	62	75
4	168	195
5	83*	134
-1	92	-79
-3	206	232
-4	161	175
K= 13, L= 4		
0	86	-116
1	202	174
2	64	90
3	43	-57
-1	182	-167
-2	187	-166
-4	125	129
-5	133	123
K= 13, L= 5		
0	210	-246
1	224	-224
2	43*	-73
4	53*	87
-1	50	-72
-4	124	131
-5	122	134
K= 13, L= 6		
4	67	98
5	95	105
-2	136	-127
K= 13, L= 7		
0	156	-150
1	154	-123
-2	61	89
-3	138	163
-4	42*	105
K= 13, L= 8		
0	59	-71
-1	62	-67
-3	146	167
-4	131	143
K= 13, L= 9		
0	163	-143
1	188	-173
-2	175	161
-3	225	239
-4	35*	67
K= 13, L= 10		
0	147	-123
1	79	-72
-1	33*	-68
-5	78	98
K= 13, L= 11		
-3	98	115
K= 14, L= 0		
0	230	-194
4	109	120
-1	114	-118
K= 14, L= 1		
1	85	-95
2	109	-135
3	73	-101
-1	153	144
-2	180	148
-4	61	-77
K= 14, L= 2		
0	147	-137
1	156	-136
2	61	80
3	95	101
4	38*	69
5	124	112
-3	79	91
-4	134	150
K= 14, L= 3		
0	69	69
2	129	-129
3	141	-144
5	124	115
-1	50*	80
-3	128	141
-4	86	102
K= 14, L= 4		
0	67	-75
2	40	-54
3	37*	-59
5	54	66
-1	121	-121
-3	124	126
-4	42*	68
K= 14, L= 5		
0	110	88
1	63	-73
2	123	-144
-1	142	138
-2	129	125
-5	128	-119
K= 14, L= 6		
1	163	-169
2	199	-191
4	136	150
-3	57	78
-4	88	94
-5	91	-86
K= 14, L= 7		
1	38*	61
3	131	-144
-2	38	58
K= 14, L= 8		
0	61	-82
1	58*	-91
-2	59	75
-3	152	142
-4	129	149
-5	37*	68
K= 14, L= 9		
-1	79	113
-2	212	212
-3	153	142
-5	155	-195
K= 14, L= 10		
-3	88	109
K= 15, L= 0		
5	41	-53
-2	92	112
-3	114	132
-4	26*	21
K= 15, L= 1		
5	156	-159
-1	81	101
-3	57	-80
-4	76	-101
K= 15, L= 2		
1	191	-161
2	204	-197
3	110	-122
5	43	47
-1	213	192
-2	125	118
-4	38*	1
K= 15, L= 3		
3	49	-76
5	71	87
-1	78	94
-2	91	88
K= 15, L= 4		
0	127	130
2	35*	-84
3	138	-153
4	45	-58
-1	185	200
-2	153	163
-3	37*	89
-4	202*	-31
-5	91	-82
K= 15, L= 5		
0	56*	95
2	32*	64
-2	92	-103
-4	157*	-19
-5	28*	-54
K= 15, L= 6		
1	34	48
2	58*	-102
3	123	-131
-2	59	91
-5	43*	-71
K= 15, L= 7		
0	103	106
-1	78	87
-3	59	-81
-4	87	-109
K= 15, L= 8		
0	101	101
-1	139	168
-2	25*	65
K= 16, L= 0		
0	169	194
4	151	-192
-1	140	164
-2	35*	66
-3	57	-61
K= 16, L= 1		
0	111	-103
3	54	64
-5	37*	-65
K= 16, L= 2		
0	66	64
-1	38	57
-3	57	-81
K= 16, L= 3		
2	54	-52
-5	71	-99
K= 16, L= 4		
0	69	91
1	108	125
2	98	107
3	39	-56
-1	63	87
-3	147	-145
K= 16, L= 5		
0	32	34
1	110	132
2	22*	37
-2	104	-105
-3	120	-109
K= 16, L= 6		
0	195	233
-1	125	134
-2	46	-64
-3	108	-112
K= 16, L= 7		
-1	63	71
K= 17, L= 0		
-2	79	-99
-3	67	-84
K= 17, L= 1		
0	69	96
2	65	68
-1	94	88
-3	55	-52
K= 17, L= 2		
1	150	162
2	40*	68
-1	159	-145
-2	166	-162
-3	122	-146
K= 17, L= 3		
0	117	-94
-1	130	-114
K= 17, L= 4		
0	117	-147
K= 17, L= 5		
0	137	192

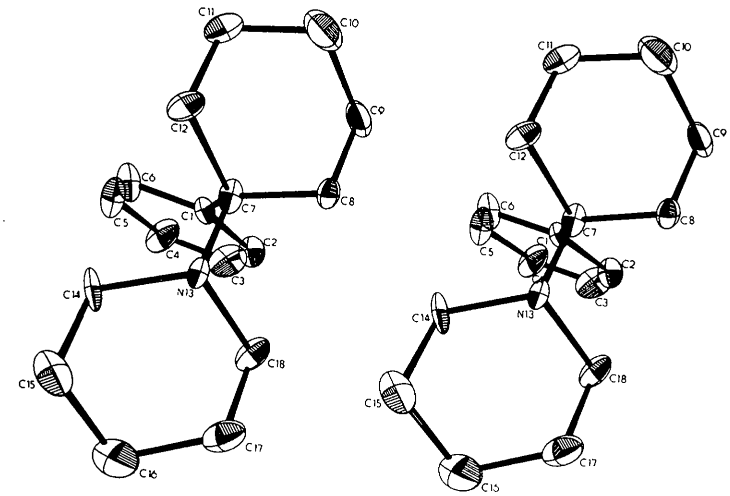

Fig. 2. Stereoscopic view of the sernyl rings. C(1) to C(6) form the phenyl ring while C(7) to C(12) and N(13) to C(18) form respectively the cyclohexane and piperidine rings with C(9) below, C(12) above and C(18) below, C(15) above the mean atomic planes formed by the remaining four atoms of the respective rings, demonstrating their chair conformations. Atoms with the largest number of covalent bonds [C(1), C(7), and N(13)] have the smallest ellipsoids of vibration. The ellipsoids increase in size on moving from C(7) to C(8) to C(9) to C(10), from C(7) to C(12) to C(11) to C(10), and similarly from N(13) to C(16) and from C(1) to C(4). The orientations of the ellipsoids are scaled to include 50% probability (Johnson, 1965).

Table 2 (*cont.*)

(*b*) Calculated structure factors (× 10) for unobserved reflections.
The ** or * indicate respectively reflections not estimated as they were barely visible or in high background regions on the film; the remaining reflections were simply not visible.

H	K	L	10FC
0	0	10	27
-1	0	6	8
-5	0	0	6
2	0	2	-43
6	0	8	27
0	1	12	-16
-2	1	7	7
-3	1	8	-23
-3	1	14	38
-3	1	14	38
-6	1	10	60**
-8	1	4	28
-8	1	9	-2
-9	1	7	-12
1	1	4	16
1	1	12	27
2	1	12	16
3	1	3	-26
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5	7	3	15
0	8	10	-11
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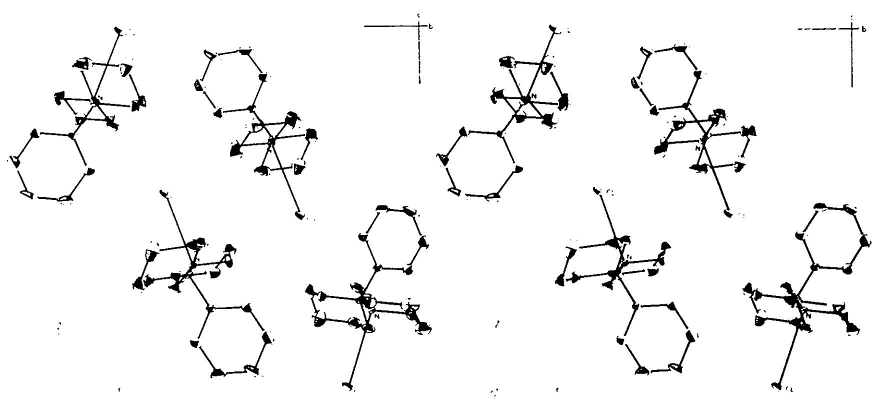

Fig. 3. Stereoscopic projected view of the unit cell along the reciprocal lattice a^* axis, illustrating the molecular packing. The nitrogen atoms are shown bonded to the chlorine atoms. The thermal ellipsoids are scaled to include 50% probability (Johnson, 1965).

block-diagonal least-squares refinement (Cruickshank, 1964; Ahmed, Hall, Pippy & Saunderson, 1967) with weighting scheme

$$w^{1/2}=F_o/F^*,\ F_o\le F^*,\ F^*=18\ , \tag{1}$$

$$w^{1/2}=F^*/F_o,\ F_o\ge F^*, \tag{2}$$

correlated to the most inaccurately estimated (very weak and very strong) reflections and with the minimization of

$$R^*=\sum_F w(|F_o|-|F_c|)^2\ , \tag{3}$$

where the sum is over non-equivalent reflections with weight w, reduced R to 0·11. Schomaker's correction was applied to the thermal parameter shifts (Ahmed *et al.*, 1967); convergence-acceleration factors were employed in the block-diagonal refinement (Hodgson & Rollett, 1963). One-hundred and fifty-two reflections (indicated by an asterisk in Table 1) for which $||F_o|-|F_c||/|F_o|\ge 0{\cdot}56$ were deleted from the refinement and the R index calculation as 2 suffered from extinction, 15 were mis-estimations, and 135 with estimated intensity about 1 or 2 were close to the background intensity and thus incorrectly estimated. Three-dimensional Fourier difference syntheses indicated the hydrogen positions as resolved maxima. The resulting observed

Table 3. *Final positional parameters* (x,y,z) *expressed as fractions* $(\times 10^5)$ *of the a, b, and c unit-cell dimensions respectively and estimated standard deviations in parentheses (fractional,* $\times 10^5$*) and deviations* Δ' *from the mean molecular planes* (Å)

An asterisk indicates an atom not used to determine the molecular plane. Hydrogen positional parameters are fractional and multiplied by 10^3.

	x	y	z	Δ'
C(1)	17659 (66)	43338 (37)	23249 (39)	0·0100
C(2)	16325 (77)	39425 (46)	13273 (43)	−0·0074
C(3)	19871 (94)	44954 (58)	05350 (47)	0·0014
C(4)	24493 (99)	54460 (58)	07089 (53)	0·0020
C(5)	25582 (97)	58297 (50)	16804 (60)	0·0008
C(6)	22154 (80)	52984 (43)	24791 (47)	−0·0068
C(7)	13130 (75)	37471 (39)	32040 (39)	−0·0018
C(8)	16210 (72)	26621 (41)	31328 (45)	0·0018
C(9)	33757 (86)	24267 (48)	33957 (54)	−0·6679*
C(10)	42264 (94)	27905 (61)	44920 (61)	−0·0019
C(11)	39433 (79)	38627 (50)	45797 (45)	0·0019
C(12)	21947 (76)	40875 (48)	43239 (42)	0·6787*
N(13)	−04478 (55)	38830 (31)	31137 (31)	0·0050
C(14)	−09534 (81)	49192 (41)	31170 (47)	−0·0050
C(15)	−26413 (85)	49914 (50)	31320 (48)	0·6635*
C(16)	−37164 (85)	45047 (54)	21989 (51)	0·0050
C(17)	−31950 (81)	34567 (51)	21828 (50)	−0·0050
C(18)	−15014 (79)	33623 (46)	21941 (44)	−0·6379*
Cl(19)	−11704 (21)	31246 (12)	51560 (11)	
				Bonded to
H(20)	155	321	109	C(2)
H(21)	200	422	−009	C(3)
H(22)	283	579	018	C(4)
H(23)	303	650	161	C(5)
H(24)	257	568	332	C(6)
H(25)	113	228	370	C(8)
H(26)	124	237	233	C(8)
H(27)	406	266	286	C(9)
H(28)	382	174	344	C(9)
H(29)	342	236	512	C(10)
H(30)	535	262	456	C(10)
H(31)	483	450	399	C(11)
H(32)	419	407	543	C(11)
H(33)	166	360	480	C(12)
H(34)	217	489	460	C(12)
H(35)	−065	364	375	N(13)
H(36)	−092	511	243	C(14)
H(37)	−035	545	399	C(14)
H(38)	−278	458	393	C(15)
H(39)	−296	571	325	C(15)
H(40)	−375	472	155	C(16)
H(41)	−473	451	214	C(16)
H(42)	−347	310	280	C(17)
H(43)	−395	315	147	C(17)
H(44)	−121	370	169	C(18)
H(45)	−113	265	234	C(18)

Table 4. *Carbon, nitrogen and chlorine anisotropic temperature factors* ($Å^2 \times 10^5$) *as they appear in* $\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{23}kl+\beta_{13}hl+\beta_{12}hk)]$ *with estimated standard deviations in parentheses* ($Å^2 \times 10^5$)

	β_{11}	β_{22}	β_{33}	β_{23}	β_{13}	β_{12}
C(1)	537 (82)	136 (27)	152 (28)	44 (45)	359 (79)	130 (84)
C(2)	965 (103)	252 (33)	231 (33)	−12 (53)	511 (96)	87 (101)
C(3)	1452 (131)	558 (48)	154 (32)	2 (65)	410 (103)	−270 (134)
C(4)	1415 (130)	544 (48)	330 (42)	−528 (75)	612 (122)	−201 (138)
C(5)	1338 (121)	231 (34)	576 (49)	−279 (69)	927 (126)	−173 (116)
C(6)	1083 (109)	168 (31)	358 (38)	6 (55)	717 (107)	145 (101)
C(7)	1003 (98)	124 (26)	119 (28)	64 (44)	406 (87)	−65 (91)
C(8)	651 (91)	129 (29)	326 (36)	3 (52)	276 (93)	−166 (89)
C(9)	1051 (112)	223 (34)	459 (45)	25 (62)	252 (114)	426 (107)
C(10)	1015 (120)	375 (41)	593 (52)	234 (73)	130 (126)	326 (122)
C(11)	838 (104)	372 (39)	212 (34)	−168 (58)	100 (91)	−259 (109)
C(12)	822 (95)	331 (35)	131 (29)	−136 (54)	151 (84)	−162 (104)
N(13)	682 (71)	87 (21)	111 (23)	3 (35)	308 (65)	−103 (67)
C(14)	1109 (114)	90 (27)	367 (38)	−18 (51)	583 (108)	327 (96)
C(15)	1106 (110)	310 (36)	301 (38)	94 (58)	455 (106)	523 (107)
C(16)	839 (106)	453 (43)	340 (39)	51 (67)	300 (105)	240 (117)
C(17)	703 (98)	436 (41)	308 (38)	−177 (62)	119 (98)	−199 (110)
C(18)	952 (103)	327 (37)	170 (31)	−277 (52)	226 (91)	−233 (102)
Cl(19)	1202 (24)	245 (7)	189 (7)	−214 (14)	432 (20)	124 (28)

Table 5. *Atomic least-squares planes determined by the method of Schomaker, Waser, Marsh & Bergman* (1959)

The equations of the planes are in the form $lX+mY+nZ=p$, where X, Y, Z are in Å units referred to the crystallographic axes.

Plane	Atoms	l	m	n	p
A	C(1), C(2), C(3), C(4), C(5), C(6)	0·91120	−0·29231	0·05205	−0·17920
B	C(7), C(8), C(10), C(11)	−0·65966	−0·17647	0·87283	1·97872
C	N(13), C(14), C(16), C(17)	−0·44326	−0·14842	0·96702	3·31590

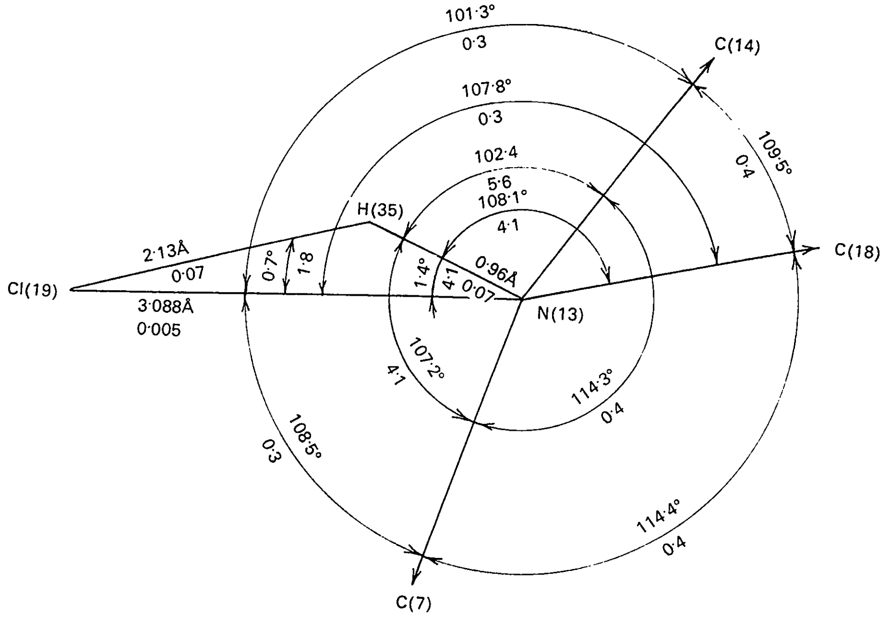

Fig. 4. Coordination angles (degrees) at the nitrogen atom. Estimated standard deviations are given below the corresponding interatomic bond distance (Å) and bond angles (degrees).

and unobserved calculated structure factors are given in Table 2(*a*) and (*b*) respectively, the fractional atomic coordinates in Table 3, and the temperature factors in Table 4. The estimated standard deviations were obtained in the usual way from the variance–covariance matrix. The mean shift was 0·4 of the estimated standard deviations in the final refinement cycle.

Discussion

The bond lengths uncorrected for thermal motions and bond angles are shown in Fig. 1. Their standard deviations were calculated by the method developed by Cruickshank & Robertson (1953). Stereoscopic views of the sernyl rings are illustrated in Fig. 2 and the molecular packing arrangement in Fig. 3.

The equations of the least-squares mean atomic planes are given in Table 5 while the distances of the atoms from the planes are listed in Table 3. The phenyl ring is equatorial while the piperidine and cyclohexane rings are in chair conformations. The average C–C bond distance and C–C–C bond angle for the phenyl, cyclohexane, and piperidine rings are respectively 1·382 Å and 120·0°, 1·536 Å and 110·9°, and 1·512 Å and 111·1°. The average piperidine bond distance and angle involving nitrogen and carbon atoms are 1·508 Å and 111·1°. The angles between normals to planes *A* and *B*, *B* and *C*, and *A* and *C* (Table 5) are 110, 15, and 96° respectively.

The smallest intermolecular contacts (excluding hydrogen atoms) are 3·443 Å between Cl(19) and C(18′) and 3·497 Å between C(12) and C(15′) where the fractional positions of C(18′) and C(15′) are (−0·15014, 0·16377, 0·71941) and (0·26413, 0·50086, 0·68680) respectively. These contacts are acceptable in consideration of the van der Waals radii, 1·6 and 1·8 Å, for carbon and chlorine respectively (Pauling, 1960). Intermolecular contacts less than 4 Å are listed in Table 6 (excluding hydrogen atoms).

Table 6. *Intermolecular distances less than* 4 Å (*excluding hydrogen atoms*)

The (x,y,z) coordinates of the unprimed atoms are listed in Table 3 while those of the primed atoms are listed here.

C(2) —Cl(19′)	$x, \frac{1}{2}-y, z-\frac{1}{2}$	3·853 Å
C(3) —C(3′)	$-x, 1-y, -z$	3·727
C(3) —C(4′)	$-x, 1-y, -z$	3·867
C(3) —C(16′)	$1+x, y, z$	3·890
C(4) —C(16′)	$1+x, y, z$	3·710
C(5) —C(16′)	$1+x, y, z$	3·695
C(5) —C(17′)	$-x, \frac{1}{2}+y, \frac{1}{2}-z$	3·922
C(6) —C(16′)	$1+x, y, z$	3·881
C(9) —C(15′)	$-x, y-\frac{1}{2}, \frac{1}{2}-z$	3·896
C(10) -Cl(19′)	$1+x, y, z$	3·987
C(11) -C(11′)	$1-x, 1-y, 1-z$	3·694
C(11) -C(15′)	$-x, 1-y, 1-z$	3·819
C(12) -C(15′)	$-x, 1-y, 1-z$	3·497
C(14) -Cl(19′)	$-x, 1-y, 1-z$	3·727
C(15) -C(11′)	$-x, 1-y, 1-z$	3·819
C(18) -Cl(19′)	$x, \frac{1}{2}-y, z-\frac{1}{2}$	3·443
Cl(19)–C(4′)	$-x, y-\frac{1}{2}, \frac{1}{2}-z$	3·965
Cl(19)–C(5′)	$-x, y-\frac{1}{2}, \frac{1}{2}-z$	3·993

The C–H bond distances, expected to be 1·09 Å, the accepted distance for an sp^3 hybridized carbon atom bonded to hydrogen (Pauling, 1960), have average length 1·09 Å for the cyclohexane and piperidine rings with mean standard deviation 0·10 Å. This length agrees well with 1·08 Å given by Sutton (1965). The average C–H distance for the phenyl ring is 1·03 Å (mean standard deviation, 0·09 Å) close to the expected 1·07 Å. The fifty-two N–C–H, C–C–H, and H–C–H piperidine and cyclohexane angles have mean value 108·7° (mean standard deviation, 5·7°), close to the tetrahedral value 109·5°; the ten C–C–H angles of the phenyl ring have mean value 119·4° (mean standard deviation, 5·0°), close to the expected 120°.

The shortest chlorine to nitrogen or carbon inter- or intramolecular distance is 3·088 Å (standard deviation, 0·005 Å) between Cl(19) and N(13) with coordinates given in Table 3. A N–Cl distance of at least 3·29 Å would be expected in consideration of the 1·48 Å radius of the NH_4^+ ion and 1·81 Å radius of the Cl^- ion (Pauling, 1960). Thus, due to the shorter observed distance and the tetrahedral nature of the coordination angles at the nitrogen atom, illustrated in Fig. 4, each chlorine atom is hydrogen-bonded to a nitrogen atom. For SHCl, the N–H and H–Cl distances of 0·96 and 2·13 ± 0·07 Å respectively agree well with 1·02 Å and 2·17 Å in methyglyoxal bisguanylhydrazone dihydrochloride monohydrate (Hamilton & LaPlaca, 1968).

Thermal motion

The thermal motion ellipsoids of the individual atoms are illustrated in Figs. 2 and 3. The molecular motion has been analyzed in terms of the rigid-body modes of translation, libration, and screw motion, using the approach described by Schomaker & Trueblood (1968). The root mean square differences between calculated and observed U_{ij} values were respectively 84, 54, 52, and 87×10^{-4} Å^2 for the phenyl, cyclohexane, piperidine rings taken individually and all together. The origin in each case was the unweighted centroid. The r.m.s. values, though unsatisfactory because of the visually estimated intensities, seem to indicate independent thermal motion for each of the rings.

Computer programs

The computer programs employed in the analysis were (1) Data reduction, SFLS, Fourier, and SF table by F. R. Ahmed, S. R. Hall, M. E. Pippy & C. P. Saunderson (1967) for the IBM 360; (2) intensity corrections, independent reflections, Wilson's method, interatomic and intermolecular distances, standard deviations, and least-squares plane by R. Shiono, D. Hall & S. Chu (1963) for the IBM 1620; (3) absorption corrections by W. T. Kane (1966) for the IBM 1710; (4) *ORTEP* by C. K. Johnson (1965) for the IBM 360; and (5) ACA Library Program No. 1 by V. Schomaker & K. N. Trueblood (1968).

The authors wish to acknowledge the continued guidance and assistance of L.K. Walford of Southern Illinois University (Edwardsville, Illinois); F.S. Mathews of the Washington University School of Medicine (St. Louis, Missouri); H.B. Donahoe and H.U. Rhoads of Saint Louis University; and the Saint Louis University computer staff. Special mention is made of the support of the Washington University computing facilities through National Science Foundation Grant G-22296. The research was supported in part by the United States Army Chemical Center under Grant DA-18-108-CML-6601 and the American Cancer Society; the authors are particularly indebted to H.B. Wilson of the Center for numerous helpful discussions. PA held a National Aeronautics and Space Administration Traineeship during part of the research. The Saint Louis University chemistry department made available the Weissenberg camera.

References

AHMED, F. R., HALL, S. R., PIPPY, M. E. & SAUNDERSON, C. P. (1967). *NRC Crystallographic Programs for the IBM* 360 *System*, Divisions of Pure Physics and Chemistry, National Research Council, Ottawa, Canada.

BUSING, W. R. & LEVY, H. A. (1957). *Acta Cryst.* **10**, 180.

CRUICKSHANK, D. W. J. (1964). *The Equations of Structure Refinement*, Glasgow (Private communication to F. R. Ahmed).

CRUICKSHANK, D. W. J. & ROBERTSON, A. P. (1953). *Acta Cryst.* **6**, 698.

HAMILTON, W. C. & LAPLACA, S. J. (1968). *Acta Cryst.* B**24**, 1147.

HODGSON, L. T. & ROLLETT, J. S. (1963). *Acta Cryst.* **16**, 329.

International Tables for X-ray Crystallography (1952). Vol. I. Birmingham: Kynoch Press.

International Tables for X-ray Crystallography (1959). Vol. II, p. 202. Birmingham: Kynoch Press.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

JOHNSON, C. K. (1965). *A Fortran Thermal-Ellipsoid Plot Program for Crystal Structure Illustration*, Oak Ridge National Laboratory, Chemistry Division, Oak Ridge, Tennessee.

KANE, W. T. (1966). Ph. D. Dissertation, Univ. of Missouri, Columbia, Missouri.

PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed. Ithaca: Cornell Univ. Press.

PHILLIPS, D. C. (1954). *Acta Cryst.* **7**, 746.

PHILLIPS, D. C. (1956). *Acta Cryst.* **9**, 819.

SCHOMAKER, V. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* B**24**, 63.

SCHOMAKER, V., WASER, J., MARSH, R. E. & BERGMAN, G. (1959). *Acta Cryst.* **12**, 600.

SHIONO, R., HALL, D. & CHU, S. (1963). *Crystallographic Computing Programs for IBM* 1620, Crystallography Laboratory, Univ. of Pittsburgh, Pittsburgh, Pennsylvania.

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

SWANSON, H. & TATGE, E. (1953). *Nat. Bur. Stand. Circ.* 539. Washington, D. C.: U. S. Government Printing Office.

TAYLOR, S. & SINCLAIR, H. (1945). *Proc. Phys. Soc.* (*London*), **57**, 126.

WELLS, M. (1960). *Acta Cryst.* **10**, 180.

WILSON, A. J. C. (1942). *Nature, Lond.* **150**, 152.

Acta Cryst. (1970). B**26**, 61

The Crystal Structure of Acid Potassium Hydrogen Fumarate: A Re-determination

BY M. P. GUPTA AND R. G. SAHU
Department of Physics, University of Ranchi, Ranchi-8, India

(*Received* 18 *February* 1969)

The crystal structure of acid potassium hydrogen fumarate (empirical formula $2KC_4H_3O_4+C_4H_4O_4$ or $2C_4H_4O_4+K_2C_4H_2O_4$) has been redetermined with final R values: $R(hk0)=0{\cdot}084$, $R(0kl)=0{\cdot}083$, $R(h0l)=0{\cdot}081$. It has been shown that the X-ray analysis favours the formula $2KC_4H_3O_4+C_4H_4O_4$ rather than $2C_4H_4O_4+K_2C_4H_2O_4$. Values of inter- and intramolecular distances are given. The fumarate groups are non-planar with one of the COOH groups twisted by about 8° from the plane of the rest of the atoms in the molecule. Hydrogen bonds of 2·58 Å link the fumarate groups to form extensive chains along the [010] axis, these chains being cross-linked by hydrogen bonds of 2·57 Å from the molecule of fumaric acid occupying a special position ($\frac{1}{2}\ \frac{1}{2}\ \frac{1}{2}$). K^+–O distances range from 2·71 to 3·01 Å giving a sevenfold coordination.

Several potassium salts of fumaric acid ($KC_4H_3O_4$, $K_2C_4H_2O_4.2H_2O$, $2KC_4H_3O_4+C_4H_4O_4$) have been investigated in the past and their structures reported (Gupta, 1956; Roy, 1967) the earliest one being what Gupta & Barnes (1956) called acid potassium hydrogen fumarate and whose empirical chemical formula could be given either as $2KC_4H_3O_4+C_4H_4O_4$ or $K_2C_4H_2O_4+2C_4H_4O_4$, both giving the same value of 423·8 atomic mass units for the total unit-cell content in a triclinic unit cell. Gupta (1956) published a description of the gross features of this structure but neither the positions of the atoms nor the inter- or intramolecular distances were given by him. The unusual point of interest remained unsettled, however,