ACTA CHEMICA SCANDINAVICA 26 (1972) 2670–2680

The Crystal and Molecular Structure of 5-Hydroxydopamine Hydrochloride

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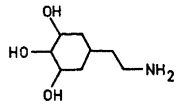
The crystal structure of 5-hydroxydopamine hydrochloride has been determined by X-ray methods using 1310 reflections collected by counter methods. The crystals are orthorhombic, space group *Pbca*, with unit cell dimensions $a = 14.58_8$ Å, $b = 16.34_5$ Å and $c = 7.83_5$ Å. The refinements yielded an *R*-factor of 0.039; standard deviations in bond lengths are 0.002 – 0.003 Å and in angles 0.2°. The molecular structure is close to that found for other hydroxylated phenylethylamine derivatives. The crystal structure is stabilized through an extensive hydrogen bond system.

Interference with the chemistry of synaptic transmission is one of the mechanisms through which drug molecules may influence a biological system. Drugs with a biological activity which may be explained by the structural analogy to normal neuro transmitters represent a source of information about the transmission mechanism.[1] Whereas noradrenaline is considered to be a normal transmitter in adrenergic synapses, it seems probable that structurally related compounds can be stored in nerve terminals and released by stimulation.[2] It appears that the biological activity of 3,4,5-trihydroxyphenylethylamine (5-hydroxydopamine), at least partly, is based on this mechanism and thus may act as a "false" transmitter,[3] and it is also closely related to the hallucinogen mescaline (3,4,5-trimethoxyphenylethylamine). The structure determinations of 5-hydroxydopamine hydrochloride seems to be of value for the study of the influence of the environment on the side chain conformation in analogous compounds.[4,5]

EXPERIMENTAL

Crystals of 5-hydroxydopamine hydrochloride were formed by slow diffusion of ethyl ether into a concentrated solution of the compound in alcohol. The thin, needle-formed crystal plates could be directly used for the X-ray experiments.

Oscillation and Weissenberg photographs indicated orthorhombic symmetry. Absent reflections were $0kl$ for k odd, $h0l$ for l odd and $hk0$ for h odd which uniquely show the space group to be *Pbca*.



Unit cell parameters were determined from diffractometer measurements of the eight equivalents of six unique reflections on a manual Picker diffractometer using Cu$K\beta$ radiation ($\lambda = 1.3922$ Å). The take-off angle was 0.5°. The computer program employed in the least squares treatment of the measurements as well as the other programs applied during the structure investigation are described in Ref. 6.

Three-dimensional intensity data were recorded on an automatic Picker diffractometer using graphite crystal monochromated MoK-radiation. The take-off angle was 4°. A crystal of dimensions $0.04 \times 0.17 \times 0.39$ mm^3 was mounted with the needle axis (c) along the diffractometer ϕ axis. The $\omega - 2\theta$ scanning mode was employed, the 2θ scan speed being 1° min^{-1} through the scan range from 0.5° below $2\theta(\alpha_1)$ to 0.6° above $2\theta(\alpha_2)$. Background counts were taken for 20 sec at each of the scan range limits. Intensities of three standard reflections were measured for every 100 reflections of the data set. They showed no decrease during the run. Fluctuations up to 4 % occurred, however, and the intensities were accordingly adjusted. Estimated standard deviations in the intensities were taken as the square root of the total counts; 2 % was added to allow for the uncertainty in the adjustments.

The measurements included 2614 unique reflections with sin θ/λ less than 0.7 Å^{-1}. Of these 1310 had net intensities larger than $3\sigma(I)$; the rest were regarded as unobserved and excluded from the further calculations.

The intensity data were corrected for Lorentz, polarization and absorption effects.

Atomic form factors used were those of Hanson *et al.*[7] for oxygen, nitrogen, and carbon, of Stewart *et al.*[8] for hydrogen. For the chlorine ion the values listed in *International Tables* (1962) were employed.

CRYSTAL DATA

5-Hydroxydopamine hydrochloride, $C_8H_{12}O_3NCl$, orthorhombic. $a = 14.588(0.005)$ Å; $b = 16.345(0.003)$ Å; $c = 7.835(0.003)$ Å. Figures in parentheses are estimated standard deviations. $V = 1868$ Å^3; $M = 205.64$; $F(000) = 864$; $\mu = 0.38$ mm^{-1}; $Z = 8$.

D_{obs} (flotation) $= 1.46$ g cm^{-3}, $D_{calc} = 1.462$ g cm^{-3}. Space group *Pbca*.

STRUCTURE DETERMINATION

The parameters of the chlorine ion were determined from a sharpened Patterson function. A weighted Fourier map [9] gave information about the position of all non-hydrogen atoms; one Fourier refinement yielded an R-factor of 0.15. Two cycles of least squares refinement of the parameters lowered R to 0.10. Two additional cycles, introducing anisotropic thermal parameters for the heavy atoms and including the six hydrogen atoms bonded to carbon atoms in calculated positions yielded an R-factor of 0.073. A difference Fourier map calculated at this point did not give sufficient information about the remaining six hydrogen atoms, and the hydrogen bond system in the crystal was therefore considered. Probable hydrogen bonds with the heteroatoms acting as hydrogen donors were found. Tentative positions of the six hydrogen atoms were chosen on the connecting line between the hydrogen bond partners. Least squares refinements led to hydrogen atomic positions giving bond angles

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

0 2 0	614	- 563	10 19 0	127	119
0 4 0	667	- 659	12 0 0	181	176
0 6 0	1622	-1654	12 1 0	98	- 98
0 8 0	726	- 713	12 2 0	388	383
0 10 0	444	- 449	12 3 0	393	393
0 12 0	278	275	12 5 0	516	- 528
0 16 0	484	500	12 7 0	136	133
0 18 0	207	216	12 8 0	321	- 321
0 22 0	156	- 121	12 9 0	132	- 136
2 0 0	185	193	12 10 0	434	- 433
2 1 0	513	481	12 11 0	118	102
2 2 0	286	291	12 12 0	105	- 86
2 3 0	1521	-1564	12 14 0	170	161
2 4 0	786	786	12 18 0	396	342
2 5 0	1352	-1382	14 1 0	264	- 272
2 6 0	731	- 744	14 2 0	183	183
2 7 0	96	79	14 3 0	342	- 352
2 8 0	76	66	14 5 0	440	- 449
2 9 0	290	- 282	14 8 0	237	- 229
2 10 0	502	- 496	14 11 0	278	280
2 11 0	234	230	14 13 0	181	177
2 12 0	235	241	14 14 0	166	156
2 13 0	760	774	14 15 0	174	165
2 14 0	335	- 341	14 16 0	165	153
2 15 0	214	221	16 0 0	361	- 356
2 16 0	242	247	16 2 0	178	- 181
2 17 0	80	- 57	16 4 0	125	109
2 18 0	145	144	16 5 0	219	- 222
2 19 0	223	- 224	16 6 0	168	156
2 20 0	201	171	16 7 0	103	- 103
2 21 0	156	- 170	16 8 0	136	120
4 0 0	227	222	16 11 0	222	208
4 1 0	377	- 379	16 13 0	133	113
4 2 0	865	- 873	18 0 0	264	- 272
4 3 0	696	- 668	18 2 0	157	- 166
4 4 0	166	154	18 6 0	194	200
4 5 0	1437	-1464	18 8 0	202	222
4 6 0	816	802	18 9 0	167	142
4 7 0	797	791	20 0 0	172	- 138
4 8 0	947	941	19 2 1	188	160
4 9 0	524	526	19 3 1	166	- 129
4 10 0	425	429	18 9 1	104	70
4 11 0	764	775	18 4 1	125	139
4 13 0	460	461	18 3 1	106	- 67
4 15 0	162	- 169	18 2 1	200	201
4 16 0	248	- 255	17 2 1	132	- 118
4 17 0	96	- 88	17 3 1	95	- 62
4 18 0	169	- 171	17 7 1	115	114
4 21 0	152	- 152	17 11 1	213	192
6 0 0	1206	-1214	16 11 1	151	133
6 1 0	157	154	16 7 1	102	100
6 2 0	503	- 520	16 4 1	110	103
6 3 0	602	606	16 1 1	168	- 172
6 4 0	557	- 542	15 1 1	83	96
6 5 0	382	358	15 2 1	125	- 132
6 6 0	532	521	15 3 1	132	- 162
6 7 0	87	- 96	15 4 1	209	- 203
6 8 0	943	941	15 6 1	151	- 144
6 9 0	399	- 416	15 10 1	210	202
6 10 0	513	509	15 13 1	101	- 66
6 14 0	284	- 277	14 14 1	110	82
6 15 0	171	182	14 10 1	107	108
6 16 0	342	- 353	14 9 1	161	164
6 18 0	198	- 182	14 7 1	149	173
6 19 0	112	- 77	14 5 1	118	140
6 21 0	119	103	14 4 1	292	- 290
8 0 0	735	- 717	14 3 1	173	- 179
8 1 0	308	- 293	14 2 1	101	- 90
8 2 0	229	- 230	14 1 1	138	- 147
8 3 0	1253	1246	13 1 1	205	217
8 4 0	194	195	13 2 1	330	- 339
8 5 0	326	324	13 3 1	229	231
8 6 0	352	345	13 4 1	189	201
8 7 0	125	125	13 5 1	313	- 319
8 8 0	152	154	13 6 1	97	- 102
8 10 0	262	261	13 7 1	128	- 123
8 11 0	171	- 176	13 9 1	136	- 148
8 12 0	192	- 213	12 12 1	170	178
8 13 0	416	- 426	12 10 1	108	105
8 15 0	185	- 167	12 9 1	103	- 100
8 16 0	103	- 88	12 8 1	107	- 119
8 19 0	131	135	12 6 1	274	- 279
8 20 0	106	- 72	12 5 1	103	- 107
10 0 0	594	586	12 2 1	135	- 117
10 1 0	134	134	12 1 1	144	153
10 2 0	507	513	11 1 1	207	212
10 3 0	801	797	11 2 1	66	67
10 4 0	321	- 324	11 3 1	270	278
10 5 0	375	377	11 4 1	243	- 247
10 7 0	155	150	11 5 1	352	- 349
10 8 0	673	- 691	11 6 1	277	- 288
10 9 0	522	- 536	11 7 1	203	- 208
10 10 0	80	- 80	11 8 1	164	151
10 11 0	107	130	11 9 1	89	91
10 13 0	293	- 308	11 10 1	77	- 80
10 14 0	170	163	11 11 1	156	- 186
10 15 0	122	- 109	11 14 1	194	- 186
10 16 0	155	163	11 15 1	154	155
10 15 1	135	134	3 18 1	86	- 77
10 13 1	109	111	2 22 1	103	- 67
10 11 1	98	- 116	2 16 1	84	79
10 10 1	157	191	2 15 1	187	- 185
10 9 1	107	- 104	2 14 1	296	303
10 7 1	312	- 314	2 12 1	140	- 141
10 5 1	168	- 169	2 11 1	106	105
10 4 1	215	- 209	2 10 1	114	- 116
10 3 1	258	258	2 9 1	186	197
10 2 1	179	- 176	2 8 1	87	- 78
10 1 1	237	233	2 7 1	358	358
9 2 1	227	219	2 6 1	271	260
9 3 1	149	- 160	2 5 1	278	- 277
9 4 1	276	272	2 4 1	72	- 76
9 5 1	233	236	2 3 1	486	- 481
9 6 1	245	236	2 2 1	325	- 328
9 8 1	354	355	2 1 1	440	447
9 9 1	132	- 132	1 1 1	89	- 71
9 10 1	527	- 530	1 2 1	290	- 289
9 11 1	161	183	1 3 1	441	439
9 12 1	160	- 175	1 4 1	612	- 611
9 13 1	117	- 115	1 5 1	607	608
8 17 1	93	85	1 6 1	320	- 318
8 15 1	132	106	1 7 1	612	- 611
8 13 1	97	- 107	1 8 1	130	146
8 12 1	150	- 148	1 9 1	302	- 306
8 10 1	329	- 341	1 10 1	288	280
8 9 1	110	- 94	1 11 1	314	- 311
8 8 1	129	132	1 12 1	374	370
8 6 1	165	160	1 13 1	71	64
8 4 1	245	250	1 14 1	365	- 381
8 2 1	238	233	1 15 1	269	276
8 1 1	146	141	1 16 1	111	129
7 1 1	93	- 98	1 17 1	149	138
7 2 1	424	- 406	1 19 1	114	- 84
7 3 1	327	- 325	1 20 1	161	- 139
7 4 1	106	107	0 14 1	277	- 278
7 6 1	285	281	0 12 1	403	401
7 7 1	255	261	0 10 1	488	489
7 8 1	154	163	0 8 1	230	223
7 9 1	131	121	0 6 1	532	- 533
7 11 1	108	97	0 4 1	514	- 497
7 12 1	210	-.217	0 2 1	552	- 540
7 13 1	78	90	0 0 2	1338	1295
7 14 1	125	- 108	0 2 2	1892	-1919
7 15 1	157	- 168	0 4 2	289	289
7 19 1	170	- 169	0 6 2	182	177
6 18 1	126	134	0 8 2	531	533
6 14 1	142	- 150	0 10 2	116	116
6 13 1	92	98	0 12 2	256	253
6 12 1	173	- 168	0 14 2	380	- 379
6 11 1	97	106	0 18 2	171	- 174
6 9 1	114	111	1 21 2	101	- 117
6 8 1	119	- 127	1 20 2	111	70
6 7 1	60	- 66	1 19 2	108	- 124
6 6 1	195	201	1 17 2	129	- 126
6 5 1	458	- 450	1 16 2	107	- 99
6 4 1	235	240	1 15 2	135	144
6 3 1	145	- 152	1 14 2	230	- 228
6 2 1	305	318	1 13 2	483	491
6 1 1	358	355	1 12 2	96	- 93
5 1 1	1013	-1013	1 11 2	350	347
5 2 1	561	- 559	1 10 2	111	122
5 4 1	159	- 163	1 8 2	531	521
5 5 1	809	791	1 7 2	175	- 186
5 6 1	358	- 348	1 6 2	425	429
5 7 1	157	163	1 5 2	1012	-1015
5 8 1	399	396	1 4 2	274	270
5 9 1	135	144	1 3 2	513	- 521
5 15 1	134	- 143	1 2 2	148	132
5 16 1	174	173	1 1 2	172	161
5 17 1	106	- 93	1 0 2	2669	-2727
5 18 1	91	- 94	2 0 2	733	727
4 17 1	110	- 110	2 1 2	1129	1123
4 15 1	96	- 116	2 2 2	548	- 552
4 14 1	106	- 114	2 3 2	526	527
4 13 1	182	- 190	2 4 2	127	126
4 11 1	92	85	2 5 2	92	84
4 10 1	129	- 129	2 6 2	612	- 602
4 9 1	156	151	2 7 2	340	338
4 8 1	264	260	2 8 2	561	551
4 7 1	214	220	2 9 2	208	- 207
4 5 1	601	593	2 10 2	84	64
4 3 1	116	112	2 11 2	86	- 93
4 2 1	169	- 172	2 12 2	293	295
4 1 1	888	- 891	2 14 2	329	- 328
3 2 1	870	- 860	2 15 2	276	- 285
3 4 1	385	- 382	2 21 2	97	87
3 6 1	52	- 48	3 20 2	114	- 91
3 7 1	164	157	3 18 2	236	- 223
3 8 1	90	- 91	3 16 2	327	- 324
3 9 1	176	- 168	3 15 2	156	- 151
3 10 1	94	83	3 13 2	103	- 95
3 11 1	290	- 297	3 12 2	104	- 112
3 12 1	102	103	3 11 2	302	302
3 13 1	392	398	3 10 2	482	476
3 14 1	235	241	3 9 2	203	200
3 16 1	106	96	3 8 2	592	587
3 7 2	294	- 288			
3 6 2	247	250			
3 4 2	53	- 49			
3 3 2	184	192			
3 2 2	806	- 803			
3 1 2	537	- 521			
3 0 2	604	- 610			
4 0 2	858	860			
4 1 2	249	237			
4 2 2	154	151			
4 3 2	625	622			
4 4 2	71	68			
4 5 2	278	273			
4 6 2	653	- 653			
4 8 2	302	300			
4 9 2	78	- 68			
4 10 2	144	- 142			
4 11 2	256	- 260			
4 12 2	155	- 154			
4 16 2	99	96			
5 21 2	175	163			
5 16 2	132	- 138			
5 15 2	80	75			
5 14 2	93	- 93			
5 13 2	291	- 292			
5 11 2	373	- 374			
5 10 2	63	62			
5 9 2	461	- 468			
5 8 2	122	119			
5 7 2	188	184			
5 6 2	649	643			
5 5 2	780	782			
5 4 2	136	- 133			
5 3 2	1055	1051			
5 1 2	528	515			
5 0 2	1102	-1091			
6 1 2	189	- 182			
6 2 2	447	446			
6 3 2	114	118			
6 4 2	153	155			
6 5 2	60	- 63			
6 6 2	268	- 272			
6 7 2	223	- 227			
6 8 2	270	- 269			
6 9 2	90	- 93			
6 10 2	171	- 176			
6 11 2	202	208			
6 14 2	126	140			
6 17 2	113	- 113			
6 21 2	126	111			
7 19 2	146	149			
7 18 2	148	136			
7 16 2	144	162			
7 15 2	197	- 194			
7 14 2	147	135			
7 13 2	347	- 349			
7 12 2	173	- 177			
7 11 2	161	- 166			
7 10 2	166	- 159			
7 9 2	119	124			
7 8 2	342	- 337			
7 7 2	149	143			
7 5 2	204	195			
7 4 2	253	256			
7 3 2	598	592			
7 2 2	187	186			
7 1 2	134	- 117			
7 0 2	144	147			
8 0 2	118	120			
8 1 2	295	- 286			
8 2 2	166	167			
8 3 2	371	367			
8 4 2	257	- 256			
8 5 2	464	- 469			
8 7 2	405	- 401			
8 8 2	121	- 123			
8 10 2	158	176			
8 11 2	325	328			
8 12 2	128	- 112			
8 13 2	82	72			
8 20 2	101	- 68			
9 16 2	210	205			
9 14 2	217	212			
9 12 2	87	107			
9 10 2	304	- 301			
9 9 2	275	- 267			
9 8 2	667	- 683			
9 6 2	116	- 121			
9 5 2	125	128			
9 4 2	182	- 190			
9 3 2	83	- 80			
9 2 2	492	488			
9 1 2	78	78			
9 0 2	684	679			
10 0 2	296	- 290			
10 1 2	237	- 237			
10 2 2	128	- 128			
10 4 2	196	- 198			

Table 1. Continued.

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10	6	2	442	464	12	2	3	352	- 365	3	11	3	239	- 249	5	6	4	175	179	14	5	5	124	- 101
10	7	2	159	163	11	1	3	207	- 209	3	12	3	168	- 173	5	5	4	273	- 272	14	4	5	134	114
10	9	2	268	- 284	11	3	3	73	- 71	3	14	3	324	- 334	5	3	4	293	- 295	14	2	5	139	146
10	10	2	161	162	11	4	3	172	- 157	3	18	3	91	86	5	2	4	258	261	14	1	5	348	345
10	11	2	228	237	11	5	3	87	- 89	2	17	3	166	- 151	5	1	4	269	- 270	13	2	5	155	148
10	15	2	98	77	11	7	3	293	294	2	15	3	268	- 272	5	0	4	426	- 419	13	9	5	105	110
11	18	2	173	151	11	9	3	199	204	2	14	3	265	271	6	0	4	459	460	12	14	5	112	- 98
11	15	2	101	83	11	10	3	98	- 93	2	13	3	162	166	6	1	4	260	- 264	12	12	5	211	- 203
11	13	2	242	255	11	11	3	104	88	2	12	3	103	- 112	6	2	4	246	240	12	4	5	395	408
11	11	2	116	100	11	12	3	155	155	2	11	3	151	163	6	6	4	372	- 368	12	2	5	124	115
11	10	2	221	- 214	11	16	3	124	107	2	9	3	116	123	6	7	4	69	- 73	11	1	5	109	- 103
11	9	2	180	183	11	17	3	135	- 135	2	7	3	481	472	6	8	4	148	- 138	11	2	5	89	- 105
11	5	2	502	- 497	10	17	3	109	115	2	4	3	173	- 177	6	9	4	132	132	11	7	5	151	153
11	4	2	133	139	10	15	3	95	109	2	3	3	335	- 338	6	12	4	186	- 186	11	8	5	90	76
11	3	2	266	- 259	10	12	3	123	126	2	2	3	472	- 471	6	14	4	135	123	11	11	5	114	102
11	2	2	74	63	10	11	3	175	- 195	2	1	3	405	- 409	6	16	4	155	148	11	13	5	91	23
11	1	2	109	- 109	10	10	3	229	222	1	1	3	500	- 498	6	18	4	156	147	10	15	5	133	- 112
11	0	2	66	- 104	10	9	3	188	181	1	2	3	836	840	6	20	4	110	- 60	10	14	5	134	- 121
12	0	2	447	- 453	10	8	3	160	- 156	1	3	3	264	- 269	7	11	4	171	168	10	10	5	133	- 140
12	3	2	404	407	10	7	3	122	- 136	1	4	3	257	260	7	10	4	123	127	10	9	5	266	261
12	5	2	201	- 209	10	6	3	71	- 65	1	5	3	437	439	7	9	4	102	108	10	8	5	104	- 97
12	6	2	180	189	10	5	3	446	- 452	1	6	3	349	345	7	7	4	224	- 233	10	7	5	316	323
12	7	2	107	117	10	4	3	286	- 288	1	7	3	194	194	7	6	4	291	298	10	6	5	163	172
12	9	2	125	- 115	10	3	3	156	157	1	8	3	172	- 171	7	5	4	276	- 280	10	5	5	110	- 98
12	11	2	140	123	10	2	3	99	- 96	1	9	3	267	265	7	4	4	72	- 67	10	4	5	303	307
12	12	2	103	100	10	1	3	307	317	1	10	3	278	- 282	7	3	4	175	182	10	2	5	276	276
12	16	2	194	- 170	9	1	3	109	102	1	11	3	84	72	7	2	4	132	- 138	10	1	5	274	- 273
12	17	2	96	- 80	9	2	3	467	- 470	1	12	3	201	- 205	7	1	4	208	- 211	9	2	5	146	- 152
13	13	2	134	129	9	4	3	556	- 561	1	15	3	120	- 121	7	0	4	286	- 283	9	4	5	170	- 173
13	11	2	168	177	9	6	3	149	- 144	1	16	3	112	105	8	0	4	213	211	9	8	5	98	- 91
13	10	2	241	227	9	7	3	174	- 185	1	17	3	101	119	8	1	4	155	- 157	9	9	5	104	101
13	9	2	131	133	9	8	3	164	158	1	19	3	151	- 146	8	2	4	281	281	9	10	5	110	99
13	7	2	94	- 81	9	10	3	153	152	0	20	3	259	- 247	8	3	4	81	- 79	9	12	5	113	101
13	5	2	157	- 163	9	11	3	107	111	0	16	3	292	293	8	4	4	195	- 196	8	17	5	206	- 198
13	3	2	492	- 493	9	12	3	233	237	0	14	3	190	- 186	8	5	4	104	- 104	8	15	5	99	- 127
13	1	2	126	- 132	8	19	3	178	161	0	12	3	331	338	8	6	4	103	104	8	13	5	126	- 118
13	0	2	239	- 243	8	15	3	105	110	0	10	3	232	230	8	7	4	109	- 108	8	12	5	110	114
14	3	2	313	305	8	10	3	202	- 206	0	8	3	91	91	8	8	4	429	- 440	8	11	5	251	234
14	6	2	84	72	8	9	3	211	- 217	0	6	3	173	- 169	8	9	4	102	- 117	8	9	5	437	437
14	7	2	89	81	8	8	3	126	132	0	4	3	711	- 694	8	11	4	171	174	8	7	5	104	117
14	12	2	144	- 116	8	7	3	350	- 353	0	2	3	1190	-1187	8	13	4	126	127	8	4	5	269	- 266
14	13	2	128	- 105	8	6	3	91	- 94	0	0	4	240	- 245	8	14	4	104	109	8	3	5	156	- 163
15	8	2	248	250	8	4	3	116	117	0	2	4	580	- 576	8	15	4	105	135	8	2	5	171	- 177
15	6	2	208	202	8	3	3	227	222	0	4	4	90	77	9	9	4	170	- 179	8	1	5	213	- 231
15	5	2	108	95	8	2	3	327	324	0	6	4	130	131	9	7	4	288	280	7	1	5	87	76
15	3	2	82	89	8	1	3	256	253	0	8	4	670	665	9	6	4	457	465	7	5	5	82	76
15	2	2	236	- 237	7	1	3	269	262	0	10	4	174	172	9	5	4	75	60	7	6	5	257	- 254
15	0	2	347	- 346	7	2	3	306	- 317	0	14	4	459	- 452	9	3	4	115	- 118	7	7	5	160	- 160
16	4	2	108	100	7	3	3	57	61	0	18	4	108	- 124	9	2	4	225	- 223	7	8	5	143	152
16	8	2	147	- 132	7	4	3	131	- 133	1	19	4	101	98	9	0	4	371	- 377	7	9	5	144	- 144
16	10	2	120	- 111	7	6	3	164	- 158	1	15	4	152	- 153	10	0	4	285	- 288	7	18	5	105	- 66
17	11	2	180	- 172	7	7	3	121	- 114	1	14	4	109	119	10	1	4	88	- 97	6	14	5	220	213
17	9	2	129	94	7	8	3	228	234	1	11	4	81	- 85	10	5	4	556	- 558	6	13	5	148	- 144
17	7	2	96	77	7	9	3	303	- 309	1	10	4	94	- 110	10	6	4	205	195	6	12	5	331	322
17	5	2	199	205	7	10	3	86	- 89	1	9	4	217	- 219	10	10	4	91	103	6	10	5	104	108
17	1	2	119	112	7	11	3	139	- 139	1	8	4	112	- 115	10	11	4	264	261	6	9	5	147	149
17	0	2	103	- 81	7	14	3	98	107	1	7	4	200	192	10	12	4	126	- 121	6	8	5	80	80
18	3	2	91	- 71	7	15	3	117	129	1	6	4	397	- 383	10	13	4	161	164	6	7	5	218	220
18	8	2	109	- 68	7	16	3	95	106	1	4	4	89	96	10	16	4	107	- 79	6	6	5	356	- 361
19	6	2	128	- 105	7	17	3	195	196	1	3	4	516	509	11	16	4	102	- 89	6	5	5	133	- 133
19	5	2	193	185	7	18	3	105	- 89	1	2	4	627	624	11	11	4	163	- 174	6	4	5	518	- 522
19	3	2	133	140	6	12	3	280	- 286	1	1	4	500	504	11	10	4	93	- 72	6	3	5	100	- 100
19	0	2	157	124	6	11	3	69	- 36	1	0	4	403	- 405	11	7	4	146	150	6	2	5	266	- 269
19	1	3	173	153	6	10	3	260	- 258	2	0	4	604	608	11	4	4	94	85	5	1	5	193	189
19	4	3	134	- 135	6	6	3	625	626	2	2	4	602	- 603	11	3	4	227	225	5	2	5	120	- 119
18	2	3	185	161	6	5	3	347	- 348	2	3	4	66	69	11	2	4	86	74	5	3	5	117	122
17	1	3	157	170	6	4	3	292	291	2	4	4	240	248	11	1	4	132	139	5	4	5	127	140
17	3	3	95	- 37	6	3	3	91	- 86	2	5	4	367	363	11	0	4	215	- 228	5	5	5	107	- 115
16	10	3	124	- 116	6	1	3	386	396	2	6	4	226	- 227	12	0	4	339	- 336	5	6	5	142	149
16	9	3	93	44	5	1	3	512	521	2	7	4	180	179	12	1	4	100	- 114	5	7	5	76	- 70
16	7	3	100	109	5	3	3	180	181	2	8	4	245	239	12	4	4	162	- 147	5	8	5	82	- 71
16	4	3	133	130	5	4	3	390	399	2	9	4	122	- 128	12	5	4	83	- 74	5	9	5	128	- 130
15	2	3	92	108	5	5	3	174	- 182	2	11	4	132	- 136	12	6	4	128	114	5	11	5	176	- 180
15	4	3	329	326	5	6	3	185	188	2	12	4	184	189	12	8	4	152	176	5	12	5	118	- 102
15	5	3	115	- 109	5	7	3	507	- 507	2	13	4	109	- 113	12	10	4	196	195	5	13	5	174	173
15	6	3	109	114	5	9	3	349	- 354	2	15	4	163	- 164	12	11	4	139	154	4	17	5	98	84
15	10	3	91	- 81	5	11	3	97	- 78	2	16	4	124	- 135	12	16	4	113	- 68	4	16	5	107	106
15	11	3	139	127	5	12	3	238	- 233	3	16	4	135	143	13	12	4	145	- 146	4	15	5	97	104
15	12	3	142	- 160	5	13	3	267	287	3	14	4	285	290	13	11	4	163	- 143	4	14	5	149	134
14	15	3	106	- 110	5	14	3	126	- 134	3	12	4	317	- 318	13	5	4	156	167	4	13	5	133	138
14	11	3	110	86	5	15	3	98	91	3	11	4	84	77	13	2	4	90	73	4	11	5	88	85
14	10	3	154	135	5	16	3	93	85	3	10	4	150	- 150	14	0	4	226	- 225	4	10	5	108	100
14	9	3	125	123	4	15	3	141	- 142	3	8	4	199	- 190	14	2	4	163	- 162	4	9	5	421	- 417
14	7	3	117	115	4	14	3	239	- 236	3	7	4	307	- 312	14	3	4	317	329	4	7	5	506	- 519
14	6	3	103	- 90	4	13	3	300	- 307	3	6	4	116	- 116	14	6	4	197	199	4	6	5	135	- 124
14	5	3	151	150	4	12	3	139	- 129	3	3	4	416	429	14	8	4	111	108	4	5	5	125	- 129
14	4	3	142	- 145	4	11	3	293	293	3	2	4	369	375	14	9	4	122	- 112	4	4	5	331	- 330
14	3	3	227	- 217	4	10	3	149	- 141	3	1	4	174	- 180	14	13	4	107	- 90	4	3	5	79	85
14	2	3	211	204	4	9	3	418	423	3	0	4	132	124	15	10	4	118	- 97	4	2	5	205	- 199
14	1	3	95	- 95	4	8	3	344	339	4	0	4	137	- 132	15	8	4	134	- 115	4	1	5	595	603
13	1	3	287	- 286	4	7	3	192	195	4	1	4	613	620	15	6	4	125	- 108	3	1	5	65	- 76
13	6	3	109	102	4	6	3	54	42	4	2	4	201	196	15	3	4	189	- 191	3	2	5	237	241
13	7	3	203	206	4	5	3	108	102	4	3	4	624	628	15	2	4	106	113	3	3	5	147	148
13	8	3	87	93	4	4	3	136	141	4	5	4	217	214	16	1	4	103	125	3	4	5	146	145
13	9	3	260	259	4	3	3	328	- 330	4	6	4	134	- 136	16	3	4	97	114	3	6	5	101	90
13	12	3	137	- 137	4	2	3	306	300	4	9	4	147	- 141	17	9	4	104	93	3	7	5	100	- 107
13	13	3	95	- 61	4	1	3	303	- 304	4	10	4	185	- 185	17	3	4	135	- 144	3	8	5	132	126
13	15	3	106	- 110	3	1	3	447	- 445	4	11	4	264	- 268	18	0	4	174	178	3	9	5	129	127
12	17	3	102	- 70	3	2	3	83	- 84	4	19	4	96	88	16	9	5	160	- 135	3	10	5	185	- 184
12	14	3	162	153	3	3	3	183	178	5	19	4	143	- 114	16	6	5	117	- 93	3	14	5	222	- 226
12	12	3	123	115	3	4	3	348	344	5	15	4	112	108	16	4	5	170	- 146	2	19	5	137	129
12	11	3	130	125	3	5	3	233	230	5	14	4	116	129	16	1	5	105	89	2	17	5	236	240
12	8	3	95	- 98	3	6	3	301	300	5	11	4	354	356	15	2	5	99	- 62	2	15	5	127	113
12	7	3	108	95	3	7	3	91	97	5	10	4	90	- 105	15	4	5	167	157	2	14	5	100	- 101
12	6	3	200	- 203	3	8	3	307	314	5	8	4	326	- 332	14	9	5	205	- 219	2	12	5	223	- 227

Table 1. Continued.

2	11	5	212	- 217	2	12	6	92	- 105	10	3	6	188	189	5	4	7	97	- 95	5	10	8	105	89
2	10	5	152	- 158	2	16	6	114	102	10	4	6	84	- 46	5	6	7	163	- 155	5	7	8	91	- 79
2	9	5	277	- 276	3	16	6	113	110	10	6	6	101	- 98	5	7	7	365	367	5	3	8	235	238
2	8	5	261	271	3	14	6	175	172	10	7	6	163	156	5	9	7	353	337	5	0	8	172	- 173
2	7	5	193	- 194	3	12	6	180	- 165	10	11	6	137	- 128	5	12	7	142	129	6	2	8	96	- 100
2	6	5	254	252	3	8	6	164	- 156	11	13	6	109	- 106	5	15	7	134	- 131	6	4	8	146	- 137
2	5	5	324	- 323	3	7	6	149	- 145	11	8	6	100	86	4	10	7	108	59	6	6	8	192	209
2	4	5	236	238	3	3	6	292	287	11	5	6	115	119	4	3	7	151	- 152	6	8	8	99	90
2	3	5	130	128	3	2	6	348	355	11	3	6	87	57	4	1	7	132	127	6	9	8	121	102
2	2	5	440	- 451	3	1	6	104	105	11	0	6	273	- 267	3	4	7	459	- 471	6	11	8	108	- 88
2	1	5	546	556	3	0	6	181	- 187	12	2	6	117	130	3	6	7	232	- 247	7	8	8	105	- 124
1	1	5	150	- 145	4	0	6	156	- 142	12	8	6	104	- 109	3	7	7	116	- 110	7	6	8	145	128
1	2	5	240	246	4	3	6	378	- 378	12	9	6	100	- 89	3	10	7	106	127	7	3	8	89	95
1	3	5	237	- 242	4	5	6	87	- 96	12	12	6	112	- 90	3	12	7	287	290	8	7	8	200	203
1	5	5	177	183	4	8	6	83	76	13	11	6	173	- 170	3	14	7	107	120	8	11	8	126	- 113
1	6	5	124	124	4	12	6	110	109	13	5	6	145	129	2	4	7	84	72	9	8	8	112	- 96
1	7	5	139	139	4	13	6	92	78	13	3	6	110	114	2	3	7	97	106	9	7	8	107	87
1	10	5	73	- 83	5	14	6	157	136	14	5	6	124	- 121	2	2	7	137	- 142	10	2	8	160	150
1	12	5	156	- 155	5	11	6	259	253	15	8	6	120	- 116	2	1	7	83	- 73	10	3	8	133	127
1	14	5	85	95	5	8	6	219	- 211	15	6	6	104	- 128	1	1	7	383	395	12	0	8	172	165
1	15	5	139	- 117	5	7	6	123	- 117	15	2	6	134	132	1	2	7	304	- 306	13	3	8	103	- 88
1	16	5	92	- 89	5	5	6	98	- 92	15	2	7	146	- 145	1	4	7	223	- 218	10	4	9	116	- 64
0	18	5	194	171	5	4	6	69	61	15	4	7	163	- 146	1	5	7	92	- 105	9	7	9	107	111
0	16	5	138	125	5	3	6	121	- 116	14	4	7	122	112	1	6	7	123	- 138	8	5	9	119	- 98
0	14	5	369	- 368	5	2	6	153	150	13	1	7	154	164	1	7	7	224	- 219	8	1	9	143	143
0	12	5	369	- 368	5	1	6	302	- 312	13	4	7	158	- 170	1	8	7	118	104	7	7	9	115	- 91
0	10	5	232	- 224	6	0	6	192	- 204	13	6	7	101	- 73	1	9	7	243	- 248	7	8	9	128	- 115
0	8	5	86	- 73	6	1	6	104	- 72	13	9	7	142	- 143	1	11	7	91	- 97	7	10	9	131	106
0	6	5	477	475	6	2	6	147	- 141	11	1	7	213	216	1	12	7	116	129	6	4	9	151	161
0	4	5	525	523	6	3	6	136	150	11	4	7	101	103	1	14	7	155	159	6	2	9	137	133
0	2	5	688	693	6	6	6	124	133	11	7	7	213	- 193	1	17	7	190	174	5	3	9	130	124
0	2	6	212	208	6	7	6	151	- 154	11	9	7	222	- 210	0	8	7	107	97	5	5	9	126	- 118
0	4	6	259	253	6	9	6	106	98	10	7	7	125	103	0	6	7	144	138	4	9	9	112	102
0	6	6	370	- 372	6	10	6	95	122	10	6	7	109	- 72	0	4	7	208	- 208	4	7	9	145	168
0	8	6	159	- 156	7	11	6	155	152	10	5	7	152	- 140	0	0	8	154	161	4	4	9	130	137
0	10	6	157	- 158	7	8	6	165	- 168	9	2	7	242	250	0	2	8	124	109	4	1	9	168	- 165
0	12	6	168	153	7	7	6	105	- 99	9	4	7	319	321	0	4	8	101	97	3	1	9	93	74
1	16	6	116	- 104	7	6	6	334	333	9	6	7	179	191	0	6	8	219	- 215	3	2	9	190	172
1	15	6	132	- 119	7	5	6	205	- 199	9	10	7	139	- 136	0	10	8	158	- 174	3	6	9	93	76
1	14	6	164	164	7	4	6	102	- 93	9	12	7	137	- 131	0	14	8	112	86	2	9	9	172	167
1	12	6	85	74	7	1	6	106	- 99	9	14	7	137	- 156	1	13	8	121	108	2	7	9	99	99
1	10	6	125	- 121	7	0	6	148	- 150	8	8	7	110	- 101	1	11	8	97	104	2	4	9	125	- 124
1	9	6	156	- 160	8	0	6	73	- 55	8	7	7	86	- 98	1	10	8	91	14	2	1	9	124	- 141
1	8	6	137	- 119	8	1	6	95	81	8	1	7	82	31	1	8	8	100	99	1	4	9	149	133
1	7	6	105	80	8	4	6	139	- 143	7	1	7	223	- 230	1	7	8	92	- 96	0	12	9	132	123
1	6	6	160	- 150	8	5	6	80	88	7	4	7	235	244	1	6	8	95	102	0	6	9	123	- 129
1	5	6	81	98	8	6	6	226	227	7	5	7	124	135	1	5	8	97	- 93	0	4	9	239	- 219
1	3	6	243	236	8	7	6	144	152	7	6	7	110	100	2	3	8	169	- 159	0	2	9	149	- 154
1	2	6	129	118	8	9	6	115	- 107	7	9	7	209	210	3	10	8	93	62	0	0	10	215	- 187
1	1	6	206	221	8	10	6	134	113	7	11	7	136	135	3	6	8	93	92	0	2	10	123	- 125
1	0	6	65	43	9	8	6	108	102	7	12	7	130	- 127	3	4	8	84	- 48	0	6	10	113	89
2	0	6	376	380	9	7	6	150	152	7	14	7	116	- 91	3	0	8	309	- 325	1	0	10	97	- 68
2	2	6	173	- 175	9	6	6	213	215	6	10	7	172	- 147	4	0	8	114	- 122	2	1	10	99	48
2	4	6	71	53	9	5	6	192	- 195	6	8	7	177	167	4	2	8	99	- 83	3	0	10	181	- 163
2	7	6	221	- 221	9	4	6	89	82	6	5	7	83	- 66	4	3	8	199	- 206	4	5	10	170	166
2	8	6	80	70	9	2	6	192	- 194	6	1	7	88	88	4	4	8	104	81	5	3	10	118	119
2	10	6	89	- 79	9	0	6	287	- 284	5	1	7	359	- 366	4	7	8	108	- 91	6	0	10	118	120
2	11	6	154	153	10	2	6	172	163	5	2	7	125	- 126	4	11	8	151	156					

closer to tetrahedral values on the hetero atoms; the magnitudes of the thermal parameters were also quite reasonable. Finally, six least squares refinement cycles with variation of all parameters yielded a conventional R-factor of 0.039 and a weighted R of 0.032.

A comparison of observed and calculated structure factors is given in Table 1. Final parameters are listed in Table 2 for non-hydrogen atoms and in Table 3 for hydrogen atoms.

Magnitudes and directions of the principal axes of the vibrational ellipsoids are given in Table 4. The r.m.s. discrepancy between the atomic vibration tensor components obtained in the structure determination and those calculated from a rigid-body analysis of the cation was 0.0036 Å^2. The translational r.m.s. amplitudes of vibration are 0.18 Å, 0.17 Å, and 0.16 Å along the principal axes and the r.m.s. librational amplitudes 5.3°, 1.9°, and 1.2°. The coordinates were adjusted according to this libration.

DISCUSSION

Bond lengths and angles are presented in Table 5. The bond lengths corrected for the librational effects and the bond angles may be found in

Table 2. Fractional atomic coordinates and thermal parameters with estimated standard deviations for non-hydrogen atoms ($\times 10^5$). 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}
Cl	8383	6128	14243	271	284	1253	−40	145	0
	4	4	8	3	2	11	5	11	10
O1	29518	8075	14617	252	204	2304	9	122	155
	10	9	23	8	6	40	11	31	28
O2	46948	10445	4424	295	282	1495	188	−168	−48
	10	9	21	8	7	35	13	29	27
O3	53036	25844	−2628	268	358	2840	26	531	455
	10	11	26	8	8	50	14	35	34
N	9505	44143	22376	305	229	1322	34	59	13
	12	11	24	10	7	38	15	34	30
C1	28909	30246	7148	272	213	1026	45	40	9
	14	13	30	11	9	44	17	40	35
C2	26062	22417	11239	203	229	1342	10	102	−26
	14	14	30	10	9	50	16	39	36
C3	32116	15948	10627	265	188	1079	6	−14	16
	14	13	29	11	9	49	18	37	33
C4	41118	17155	5953	247	212	1111	121	−87	−44
	15	13	29	10	9	44	17	43	35
C5	44049	24978	1996	215	310	1179	13	92	66
	14	16	32	10	10	51	19	39	36
C6	37993	31501	2743	301	204	1344	−41	175	129
	15	13	30	11	10	50	18	42	37
C7	22018	37164	6740	318	213	1369	103	111	145
	15	13	32	12	9	51	17	44	37
C8	17323	38312	23697	399	261	1408	257	99	96
	15	14	35	13	9	54	22	46	43

Fig. 1 where the numbering of the atoms is indicated. The estimated standard deviations were calculated from the correlation matrix. Standard deviations in carbon-carbon and carbon-nitrogen bond lengths are 0.003 Å and in carbon-oxygen bond lengths 0.002 Å. The standard deviation in angles involving carbon, nitrogen, or oxygen atoms is 0.2°.

The pyrogallol part is nearly planar. The C1 to C6 carbon atoms deviate less than 0.009 Å from a least squares benzene ring plane whereas the deviation for C7 is 0.104 Å and for the oxygen atoms −0.015 Å (O1), 0.085 Å (O2) and 0.015 Å (O3). The geometry of the carbon frame is quite normal, the average C−C bond length is 1.386 Å. The C3−O1 and C5−O3 bond lengths, 1.380 Å and 1.370 Å, respectively, agree well with the value commonly found in phenols. The C4−O2 bond of 1.394 Å is significantly longer, but is the expected single bond length between an oxygen and an sp^2 hybridized carbon atom.

The distortions of the external C−C−O angles from 120° at the C3 and C5 atoms are in accordance with those usually observed in hydroxylated benzenes where the phenolic hydrogen atom is involved in a hydrogen bond roughly coplaner with the benzene ring. The larger angle seems in such cir-

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

Atom	x	y	z	B
HO1	2406	770	1553	3.4
	19	15	35	7
HO2	5047	1003	1266	3.8
	19	17	35	7
HO3	5400	3022	−545	4.8
	21	18	40	9
HN1	733	4504	3278	4.9
	19	17	41	8
HN2	494	4231	1544	4.5
	19	16	37	8
HN3	1163	4914	1809	3.7
	17	17	32	7
H2	2000	2139	1498	1.7
	13	11	26	4
H6	4018	3681	−4	2.4
	17	16	34	7
H71	1710	3581	−197	3.2
	14	14	30	5
H72	2489	4237	312	3.6
	17	13	29	5
H81	2192	4074	3175	4.6
	16	15	33	7
H82	1491	3331	2773	4.2
	16	15	35	7

Table 4. R.m.s. amplitudes of vibration $(\bar{u}^2)^{\frac{1}{2}}$ (Å) and B values (Å^2) along the principal axes of vibration given by the components of a unit vector e in fractional coordinates.

Atom	$(\bar{u}^2)^{\frac{1}{2}}$	B	e_x	e_k	e_z
Cl^-	.202	3.23	.0262	.0227	.1080
	.196	3.04	−.0032	.0555	.0535
	.165	2.15	.0632	.0122	−.0421
O1	.269	5.73	.0054	.0069	.1264
	.165	2.14	.0367	.0510	−.0175
	.164	2.11	−.0577	.0331	.0007
O2	.226	4.02	.0333	.0316	−.0900
	.207	3.37	.0237	.0384	.0890
	.151	1.80	.0551	−.0356	.0161
O3	.311	7.62	.0150	.0179	.1188
	.210	3.48	.0114	−.0582	.0331
	.158	1.97	.0659	.0060	−.0327
N	.204	3.28	.0146	.0050	.1243
	.184	2.67	.0549	.0341	−.0282
	.172	2.34	−.0384	.0506	.0068
C1	.182	2.60	.0376	.0253	.0928
	.176	2.44	.0331	.0338	−.0867
	.162	2.08	−.0468	.0443	.0131
C2	.205	3.33	.0097	−.0040	.1261
	.176	2.44	.0056	.0609	.0070
	.146	1.69	−.0676	.0045	.0186

Table 4. Continued.

C3	.183	2.65	−.0051	.0036	.1271
	.169	2.25	.0679	.0073	.0086
	.159	2.00	.0079	−.0606	.0086
C4	.194	2.96	.0348	.0322	−.0870
	.179	2.53	.0276	.0340	.0929
	.142	1.59	.0522	−.0394	.0089
C5	.207	3.39	.0058	.0569	.0457
	.190	2.86	.0112	−.0225	.1168
	.150	1.79	−.0674	.0012	.0235
C6	.211	3.52	.0240	.0108	.1174
	.181	2.59	−.0555	.0337	.0258
	.157	1.94	.0323	.0499	−.0428
C7	.215	3.65	.0283	.0228	.1061
	.187	2.76	.0522	.0221	−.0688
	.157	1.94	.0343	−.0523	.0171
C8	.238	4.46	.0510	.0361	.0398
	.206	3.35	.0183	.0101	−.1212
	.152	1.83	.0420	−.0483	.0044

Table 5. Bond lengths (Å) and bond angles (°).

Bond lengths		Corrected	Bond angles	
C1 – C2	1.383	1.388	C1 – C2 – C3	120.6
C2 – C3	1.379	1.379	C2 – C3 – C4	120.7
C3 – C4	1.377	1.382	C3 – C4 – C5	119.1
C4 – C5	1.384	1.388	C4 – C5 – C6	120.3
C5 – C6	1.386	1.387	C5 – C6 – C1	120.4
C6 – C1	1.386	1.389	C6 – C1 – C2	118.8
C1 – C7	1.513	1.514	C2 – C1 – C7	119.8
C7 – C8	1.507	1.512	C6 – C1 – C7	121.3
C8 – N	1.490	1.491	C1 – C7 – C8	112.2
C3 – O1	1.377	1.380	C7 – C8 – N	111.5
C4 – O2	1.393	1.394	C2 – C3 – O1	122.2
C5 – O3	1.368	1.370	C4 – C3 – O1	117.1
			C3 – C4 – O2	119.5
			C5 – C4 – O2	121.3
			C4 – C5 – O3	116.9
			C6 – C5 – O3	122.8
Hydrogen bond lengths				
O1 – Cl^-	3.100			
O2 – Cl^-(a)	3.051		(a) $x+\frac{1}{2}$, y , $-z+\frac{1}{2}$	
O3 – Cl^-(b)	3.181		(b) $x+\frac{1}{2}$, $-y+\frac{1}{2}$, $-z$	
N – Cl^- (c)	3.285		(c) x , $-y+\frac{1}{2}$, $z+\frac{1}{2}$	
O1 – N (d)	2.850		(d) $-x+\frac{1}{2}$, $y-\frac{1}{2}$, z	
O2 – N (b)	2.886			
Other short contacts				
O3 – H2(b)	2.70			
C3 – C6 (c)	3.44			

cumstances always to be *cis* to the hydrogen atom relative to the C – O bond. A slight shortening of these bonds also appears to be associated with this coplanarity.

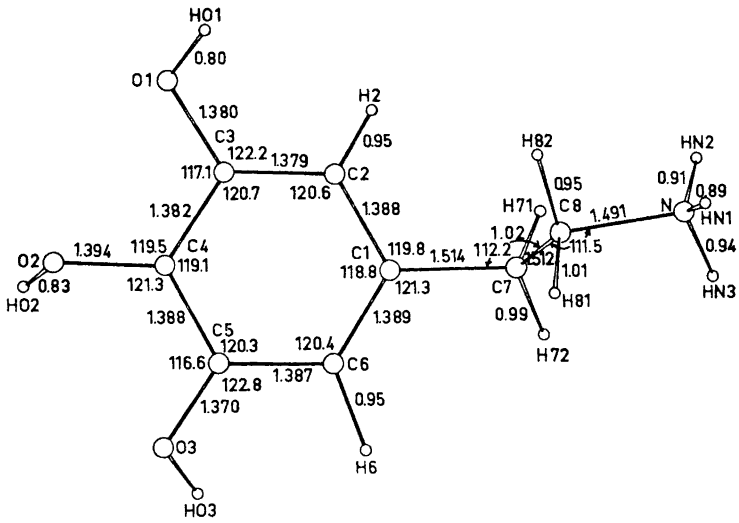

Fig. 1. Bond lengths (corrected) (Å) and angles (°).

The bond lengths in the ethylammonium part are not significantly different from those found in 6-hydroxydopamine hydrochloride.[5]

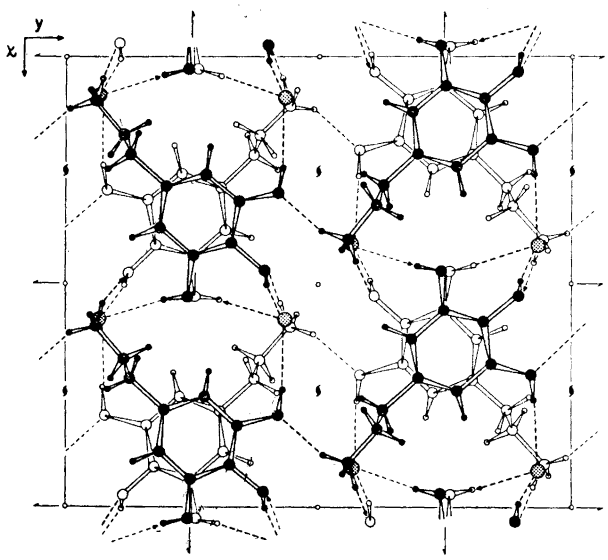

Fig. 2. The structure as viewed down the *c*-axis.

The angle between the benzene ring plane and the plane defined by C1, C7, and C8 is 57.7°, the C7–H72 bond is thus situated in the plane of the benzene ring. The dihedral angle C1–C7–C8–N is 189.6° and the conformation about the C8–N bond is staggered. This extended structure is always found in hydrochloric salts of phenylethylamines.[5,10] Of the two torsional angles determining the molecular conformation, the one about C1–C7 seems to be less rigidly fixed than does the one about C7–C8 in these structures. However, MO calculations on the free *N*-protonated dopamine showed a preferred *gauche* conformation of the nitrogen atom relative to the phenyl ring; this was supported by an NMR analysis of the molecule.[11] Thus, the structures of the hydrochlorides alone are not conclusive as to the preferred conformation of this type of molecules.

The crystal structure (Fig. 2) is mainly stabilized by hydrogen bonds of the types N–H···O, N–H···Cl^- and O–H···Cl^-.

The phenol oxygen atoms act as hydrogen donors in hydrogen bonds to chlorine ions, the lengths being 3.100 Å (O1), 3.051 Å (O2), and 3.181 Å (O3). The H–Cl^- distances are 2.30 Å, 2.24 Å, and 2.42 Å, respectively. The nitrogen atom is hydrogen donor in a corresponding bond, the N–Cl^- distance being 3.285 Å and the HN1–Cl^- distance 2.48 Å. O1 and O3 are hydrogen acceptors in hydrogen bonds to nitrogen atoms of neighbouring molecules; the hydrogen bond lengths are 2.850 Å and 2.886 Å, respectively.

Each 5-hydroxydopamine ion is thus hydrogen bonded to four chlorine ions and to four other organic moieties. There are two additional neighbouring organic ions along the *z* axis with van der Waals contacts as listed in Table 5. The chlorine ions are hydrogen bonded to four organic ions as shown in Fig. 3.

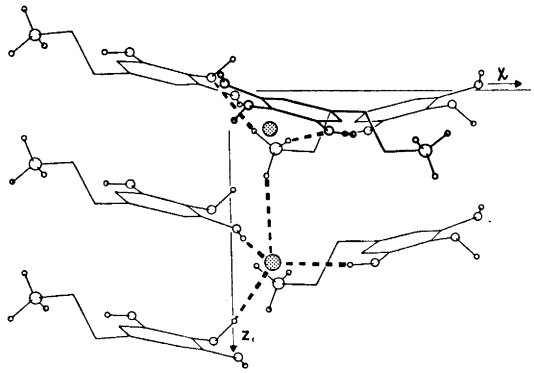

Fig. 3. The coordination about the chlorine ion (dotted).

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Received November 27, 1971.