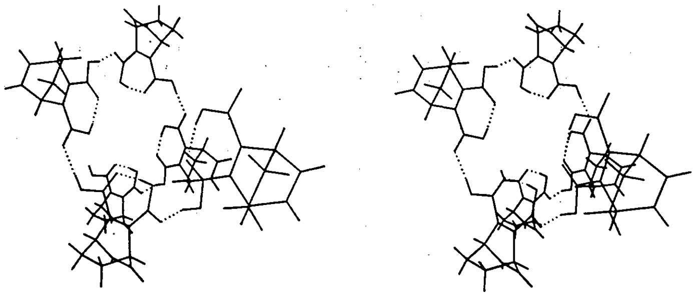

Fig. 6. Stereobild eines Ausschnitts der Molekülschraube entlang der zweizähligen Schraubenachse.

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The Crystal Structure of DL-Histidine Hydrochloride Dihydrate

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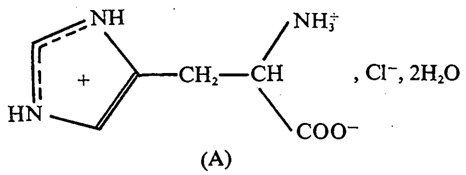
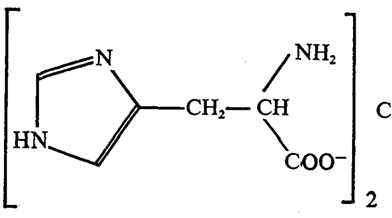
The structure of DL-histidine hydrochloride dihydrate has been determined from three-dimensional X-ray diffraction data and refined until $R = 0{\cdot}108$ and the standard deviations in bond lengths are $\sim 0{\cdot}01$ Å. The crystals are monoclinic, $P2_1/a$, with $a = 8{\cdot}87$, $b = 15{\cdot}30$, $c = 8{\cdot}48$ Å, $\beta = 114{\cdot}5°$. The histidine cation, $C_3N_2H_4^+.CH_2.CH(NH_3^+).COO^-$, is fully extended, with the imidazole group *trans* to the carboxyl group across the C_α–C_β bond; in this and a number of smaller ways the conformation is different from that of the chemically identical cation in L-histidine hydrochloride monohydrate [Donohue & Caron, *Acta Cryst.* (1964) **17**, 1178]. Despite the difference in conformation the bond lengths in the DL- and L-crystals agree very closely.

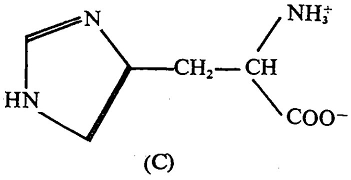
We have determined the crystal structure of DL-histidine hydrochloride dihydrate (A) so that we can compare the histidine cation here with the histidine group in various other situations. The structure of L-histidine hydrochloride monohydrate is already known (Donohue, Lavine & Rollett, 1956; Donohue & Caron, 1964, subsequently referred to as DC), and also the structure of various metal complexes containing the histidine anion such as (B), bis(histidino)cobalt(II) (Candlin & Harding, 1969; Harding & Long, 1968*a*; see also the review by Freeman, 1967).



(A)



(B)



(C)

Table 1. *Observed and calculated structure factors*

(10 × absolute values)

K 0		
2	-173	-227
4	364	409
6	139	133
8	-107	-103
10	-330	-369
12	290	339
14	-142	-146
16	37	47
18	-61	-65
0 K 1		
0	-659	-373
1	226	217
2	349	321
3	224	251
4	44	95
5	-1105	-1035
6	-699	-669
7	707	664
8	277	297
9	-197	-238
10	37	53
11	114	116
12	95	93
13	-95	-108
15	185	208
16	72	79
17	-118	-119
19	55	60
0 K 2		
0	-1456	-1367
1	-1120	-952
2	-419	-314
3	447	416
4	-697	-620
5	134	145
6	205	219
7	169	193
8	-79	-96
9	-37	-25
10	393	420
11	186	198
12	-117	-122
13	-44	-21
15	43	46
16	-95	-91
18	73	77
19	62	82
0 K 3		
0	-26	-26
1	503	407
2	411	342
3	-431	-369
4	-360	-352
5	155	155
6	233	243
7	-64	-62
8	-115	-129
10	127	124
11	-136	-142
12	-268	-295
14	62	64
15	-92	-83
16	-206	-192
17	46	38
0 K 4		
0	315	277
1	192	181
2	273	246
3	-372	-376
4	-48	-26
5	174	150
6	-235	-248
7	-251	-266
8	-57	-65
9	172	188
10	-113	-136
11	-151	-177
12	81	93
14	-68	-64
15	-85	-89
16	52	55
17	87	91
0 K 5		
0	428	366
1	-144	-138
2	-228	-214
3	-39	-38
4	189	179
5	87	86
6	-230	-236
7	55	53
8	112	120
9	114	130
10	-44	-40
12	191	191
13	62	65
16	56	90
0 K 6		
0	133	136
1	-94	-79
2	-103	-106
3	60	54
4	61	48
5	-149	-136
6	76	86
7	218	188
8	103	90
9	-143	-144
11	75	67
12	86	81
13	-119	-94
14	-61	-53
15	52	47
0 K 7		
0	-251	-241
1	-96	-92
2	106	113
3	63	69
4	-68	-61
5	-114	-117
6	43	30
7	93	72
9	-65	-53
11	-34	-30
12	-111	-108
13	-46	-43
K 8		
0	-34	-33
2	34	26
3	-54	-50
4	-42	-50
5	68	69
6	49	44
7	-30	-25
8	-118	-117
9	81	76
10	51	47
0 K 9		
1	58	51
3	-90	-84
5	54	45
6	-31	-37
7	-24	-23
1 K 0		
1	356	408
2	-123	-204
3	462	432
4	52	64
5	107	103
6	307	285
7	-298	-275
8	101	113
9	-245	-244
10	82	81
11	-60	-67
12	167	166
15	65	61
16	44	40
17	33	27
19	29	34
1 K -1		
1	127	155
2	-123	-59
3	65	116
4	-80	-129
5	250	204
6	-470	-387
7	-164	-161
8	64	76
10	-199	-196
11	35	31
12	-194	-178
13	164	175
14	-111	-125
15	86	77
16	51	48
17	147	130
18	-29	-37
19	-53	-35
1 K -2		
1	-148	-122
2	-90	-99
3	-219	-302
4	-155	-148
5	431	370
6	210	201
7	492	470
8	31	9
9	33	40
10	48	18
11	-35	-25
12	-55	-45
13	103	113
15	-55	-39
16	37	26
19	-44	-40
1 K -3		
1	-462	-551
2	-699	-572
3	57	62
4	347	333
5	138	122
6	251	216
7	-84	-52
8	-56	-54
9	53	93
10	300	303
11	-75	-76
13	-152	-158
18	42	37
1 K -4		
1	-192	-153
2	267	190
3	379	323
4	-579	-457
5	-231	-231
6	-113	-103
7	-93	-91
8	202	199
9	-82	-74
10	-196	-215
11	-109	-99
12	-96	-88
13	-109	-114
15	60	50
16	-44	-43
17	-51	-47
18	26	15
1 K -5		
1	367	306
2	-126	-116
3	-287	-250
5	76	79
6	50	50
7	64	64
9	-192	-200
11	57	95
12	-37	-40
13	90	77
14	-48	-43
16	45	42
1 K -6		
1	-57	-87
2	-144	-125
3	37	45
4	53	59
5	-53	-56
6	55	61
7	-55	-48
8	87	91
9	136	132
10	39	38
12	70	74
13	113	92
15	42	32
1 K -7		
4	-77	-52
5	77	70
7	67	60
13	-37	-34
14	-20	-13
1 K -8		
2	-37	-23
3	53	32
9	44	38
11	-42	-55
12	-47	-51
1 K -9		
1	-33	-29
3	-45	-33
5	-66	-46
7	-26	-22
8	39	27
1 K 1		
1	-166	-177
3	-125	-151
4	-87	-117
5	319	261
6	89	69
7	351	352
8	203	200
9	-273	-255
12	93	97
13	-156	-156
14	87	93
17	-96	-90
19	-29	-27
1 K 2		
1	-1192	-1262
2	292	231
3	-50	-88
4	-266	-245
5	-63	-62
6	-73	-69
7	42	53
8	-83	-96
9	236	257
10	-87	-50
11	-129	-132
12	-102	-111
15	74	65
16	-60	-47
17	-74	-74
18	50	46
19	26	43
1 K 3		
1	-60	-71
2	-112	-115
4	238	258
5	100	119
6	31	30
7	-45	-38
8	-177	-172
9	-101	-118
10	159	168
12	-104	-109
13	39	36
17	107	96
18	-29	-29
1 K 4		
1	540	469
3	264	268
4	-33	-40
5	163	166
7	-63	-42
8	37	39
9	-66	-79
10	-57	-94
11	-77	-80
12	109	118
13	37	47
14	-50	-20
15	31	25
1 K 5		
1	-35	-31
3	-100	-94
6	-65	-65
7	67	71
8	-39	-34
10	39	33
13	95	92
15	26	20
16	48	66
1 K 6		
7	67	76
8	94	88
10	-51	-47
11	58	56
13	-67	-59
14	64	64
1 K 7		
8	-96	-73
11	-47	-58
2 K 0		
0	-292	-410
1	333	444
2	738	846
3	-333	-326
4	-751	-693
5	65	65
6	29	25
7	-458	-426
8	-322	-281
9	-14	0
11	-65	-54
12	-215	-202
14	88	72
16	-147	-143
17	48	48
18	78	71
19	-61	-65
2 K -1		
1	79	55
2	468	469
3	351	419
4	-360	-364
5	-463	-459
6	282	296
7	341	338
8	-432	-433
9	-402	-375
10	249	242
11	71	68
12	-119	-116
14	170	194
16	-75	-71
17	-116	-104
18	14	26
19	114	111
2 K -2		
0	-25	-9
1	-942	-999
2	-305	-309
3	835	919
4	-74	-94
5	108	116
6	-118	-119
7	43	29
8	191	163
9	-92	-87
10	41	35
11	313	336
12	-386	330
13	-124	-141
14	-38	-27
15	153	148
16	110	108
17	-71	-61
18	48	46
19	20	29
2 K -3		
0	46	19
1	664	519
2	-370	-322
3	-20	-15
4	630	541
5	-175	-197
6	-382	-350
7	-66	-76
8	528	472
9	-114	-113
10	-118	-119
12	46	37
14	-141	-157
16	95	90
18	-86	-81
19	-32	-58
2 K -4		
0	673	487
1	320	294
2	-65	-81
3	-175	-139
4	205	188
5	238	244
6	155	162
7	-372	-370
8	25	16
9	157	149
10	-133	-139
11	-208	-206
12	-110	-117
13	169	156
14	-105	-103
15	-118	-111
16	-75	-71
17	41	39
18	-60	-64
2 K -5		
0	-710	-570
1	176	180
2	166	195
3	-43	-31
4	-330	-296
5	205	201
6	-48	-44
7	-113	-109
8	-201	-205
9	38	37
10	87	82
11	-48	-58
13	105	101
14	107	94
15	-74	-77
16	-29	-17
2 K -6		
0	-131	-133
1	35	35
2	74	57
3	128	125
4	-171	-179
5	-203	-178
6	60	62
7	71	65
8	-169	-164
9	-96	-94
10	116	121
11	84	79
13	-63	-65
14	133	141
15	96	87
2 K -7		
0	240	217
1	-187	-161
3	182	171
4	99	105
5	-96	-108
6	110	120
7	72	86
8	38	41
9	-60	-68
10	-72	-71
11	93	96
14	-35	-41
15	20	38
2 K -8		
0	-38	-34
1	-38	-35
2	-66	-65
4	145	154
5	79	80
6	-58	-55
7	-52	-53
8	114	125
10	-60	-71
11	38	41
12	20	24
13	-25	-45
2 K -9		
0	-43	-48
1	123	122
2	-65	-78
3	-52	-56
4	32	30
6	-38	-45
7	-29	-26
8	14	19
10	14	21
2 K-10		
2	29	32
4	-46	-61
5	20	29
2 K 1		
0	-646	-404
1	693	624
2	-542	-445
3	-75	-78
4	-63	-35
5	236	260
6	-141	-190
7	-81	-92
8	29	88
9	441	435
10	89	88
11	-245	-252
12	-29	-43
13	344	356
14	-148	-152
15	-83	-77
16	-35	-35
17	38	51
19	-25	-25
2 K 2		
0	317	218
2	-464	-437
3	-121	-84
4	433	426
5	352	345
6	-318	-334
7	-159	-174
8	427	417
9	-130	-120
10	98	67
11	93	103
12	61	61
13	-83	-85
14	-107	-113
15	-35	-36
16	83	95
17	32	31
18	-124	-130
2 K 3		
0	750	563
1	-371	-286
2	102	90
3	229	199
4	-150	-142
5	-294	-308
6	-66	-76
7	60	53
8	88	98
9	-231	-259
10	-197	-201
11	63	65
12	260	254
13	-157	-170
16	35	40
17	-38	-47
18	14	34
2 K 4		
0	276	249
1	-202	-178
2	309	275
3	198	207
4	-68	-83
5	-168	-173
7	145	161
8	-218	-241
9	56	56
10	124	144
11	54	64
12	-65	-68
14	65	71
15	121	129
16	-29	-25
17	-43	-68
2 K 5		
1	161	164
2	143	139
3	-145	-137
4	-144	-145
5	82	91
6	129	121
7	38	68
8	-63	-65
10	128	134
12	-83	-88
13	61	81
15	-32	-34
2 K 6		
4	-87	-90
5	102	103
6	-29	-32
7	-133	-142
8	150	101
9	79	94
11	-126	-127
3 K 0		
1	75	53
2	-151	-193
3	381	388
4	-184	-189
5	197	219
6	161	154
7	-77	-77
8	-41	-30
10	-103	-98
11	-142	-141
12	50	45
13	-239	-213
15	-118	-115
16	-79	-69
18	92	87
3 K -1		
1	-212	-216
2	106	109
3	-121	-112
4	-178	-215
5	-174	-189
6	94	104
7	-265	-286
8	192	206
9	-94	-89
10	112	108
12	-201	-211
13	66	44
14	131	129
15	72	67
16	-48	-43
3 K -2		
1	99	110
2	-191	-236
3	-477	-444
4	-120	-139
5	-179	-191
6	-29	-21
7	98	99
8	66	72
9	-146	-171
10	-12	-45
11	177	149
13	198	195
14	-94	-49
15	76	56
18	46	51
19	35	55
3 K -3		
1	321	274
2	-317	-325
3	464	419
4	67	93
5	121	109
6	114	120
7	171	160
9	57	79
10	70	74
12	100	92
13	-132	-128
14	-64	-60
16	123	99
17	-50	-44
18	-29	-29
3 K -4		
1	-48	-34
5	206	192
8	133	138
9	110	95
10	-35	-24
11	-64	-59
13	-76	-72
15	-94	-100
17	35	30
3 K -5		
1	-221	-193
2	-41	-72
4	-105	-97
5	-200	-225
6	-81	-77
7	-50	-50
9	110	115
10	-76	-75
11	-54	-46
12	-118	-116
13	63	68
14	-59	-63
3 K -6		
1	171	169
2	66	57
3	-172	-163
4	-162	-167
5	-90	-55
6	103	122
7	-64	-62
8	38	35
9	-85	-89
11	64	74
13	48	29
14	72	80
16	-41	-48
3 K -7		
1	82	91
2	-110	-116
4	118	118
5	-54	-48
7	-66	-48
9	-35	-37
10	94	102
13	-38	-14
3 K -8		
3	66	51
4	-64	-59
7	80	73
10	-61	-55
3 K -9		
2	-35	-23
3	-32	-37
4	32	32
5	48	38
9	56	66
3 K-10		
5	-38	-42
3 K 1		
1	-257	-221
2	-278	-272
3	-131	-145
4	107	78
5	126	118
6	-94	-89
7	173	202
9	148	139
11	-203	-202
13	100	99
14	-82	-83
15	-50	-49
16	69	71
17	-54	-55
3 K 2		
1	-750	-195
2	153	145
3	-59	-69
4	-38	-46
5	59	67
6	45	54
7	48	39
8	194	216
11	193	202
15	72	81
16	50	52
17	72	53
18	-45	-70
3 K 3		
1	227	182
2	-269	-235
3	272	259
4	64	73
7	-52	-47
8	-82	-80
9	-64	-78
12	35	46
14	-80	-82
15	77	98
17	-20	-20
3 K 4		
1	120	108
2	50	49
3	-141	-137
4	-148	-161
9	-165	-174
11	-63	-62
12	-59	-40
15	-45	-44
16	-20	-30
3 K 5		
2	55	76
3	38	38
8	75	82
9	80	79
10	50	39
11	-32	-42
13	-50	-51
14	56	69
3 K 6		
6	70	9
7	32	7
8	-32	-25
9	41	50
10	-38	-28
4 K 0		
0	-277	-212
1	-131	-119
2	-54	-50
3	595	599
4	478	446
5	-438	-397
6	-281	-258
7	219	204
8	255	236
9	-35	-31
12	-153	-156
13	-35	-41
14	-112	-113
15	148	129
16	66	53
17	-112	-99
18	-66	-72
4 K -1		
0	374	383
1	218	246
2	-557	-617
3	-103	-88
4	-116	-108
5	-62	-65
6	-64	-76
7	-171	-165
8	212	197
9	-135	-133
10	-404	-405
11	-137	-132
12	287	300
14	-111	-113
15	-75	-70
16	52	45
17	-27	-24
4 K -2		
1	199	298
2	-38	-48
3	-307	-356
4	152	156
5	445	467
6	-303	-300
7	-292	-255
8	-108	-101
9	114	114
10	108	111
12	-35	-31
13	198	204
14	79	85
15	-162	-148
16	31	35
17	124	100
18	-75	-60
4 K -3		
0	-207	-190
1	211	219
2	317	334
3	130	153
4	-247	-260
5	35	26
6	297	320
7	-49	-60
8	-343	-332
9	339	321
10	120	99
11	-87	-79
12	-62	-60
13	181	177
14	49	49
15	79	59
16	-44	-35
18	89	97
4 K -4		
0	-349	-318
1	-167	-160
2	314	290
3	135	153
4	-77	-87
5	-111	-115
6	361	336
8	79	69
9	-319	-307
11	166	150
12	-145	-146
13	-210	-207
14	102	99
15	70	50
16	-119	-104
4 K -5		
1	-283	-229
2	-234	-220
3	223	248
4	284	284
5	-139	-150
6	31	27
7	141	139
8	97	97
9	-207	-200
10	62	73
11	62	86
13	-68	-78
14	-78	-66
16	78	89
17	-35	-48
4 K -6		
0	-98	-94
1	76	72
2	-217	-186
4	87	97
6	-241	-262
7	35	26
8	125	122
9	125	116
10	-157	-158
12	139	146
13	70	65
14	-31	-33
16	58	75
4 K -7		
0	191	177
1	185	202
2	-35	-90
3	-209	-196
5	71	70
7	-89	-101
8	-102	-101
9	49	46
10	-108	-103
11	-54	-52
13	58	64
15	-31	-48
4 K -8		
0	-92	-92
2	109	112
3	-35	-31
4	-35	-26
5	71	69
6	126	128
7	-78	-68
8	-75	-75
9	47	38
10	31	35
11	-47	-43
12	-92	-87
4 K -9		
0	-173	-172
1	-49	-48
3	47	51
4	-47	-42
6	87	94
7	49	43
8	27	14
9	-35	-23
10	35	58
4 K-10		
1	-38	-35
2	-27	-25
3	35	37
4	49	47
5	-47	-55
6	-31	-28
7	31	46
4 K 1		
0	125	95
1	-277	-277
2	246	245
3	170	187
4	58	90
5	100	92
6	332	317
7	70	48
8	-87	-106
9	-272	-258
10	244	230
11	334	345
12	-103	-104
13	-111	-120
15	76	71
16	-47	-43
4 K 2		
0	-354	-241
1	103	97
2	248	239
3	-38	-53
4	-416	-445
5	31	12
6	141	136
7	-96	-104
8	-70	-63
9	35	28
10	52	54
11	-52	-60
12	60	63
13	68	64
14	179	205
15	-38	-43
16	-52	-57
4 K 3		
0	214	196
1	235	203
2	-116	-110
3	-98	-89
4	-47	-49
5	137	130
6	-60	-66
7	-152	-196
8	-149	-169
9	167	193
11	-115	-131
12	82	96
15	-31	-41
4 K 4		
1	177	162
2	-35	-24
3	-79	-73
4	177	167
6	-155	-162
8	113	120
9	-60	-59
10	-125	-127
13	35	41
14	-100	-129
4 K 5		
3	148	130

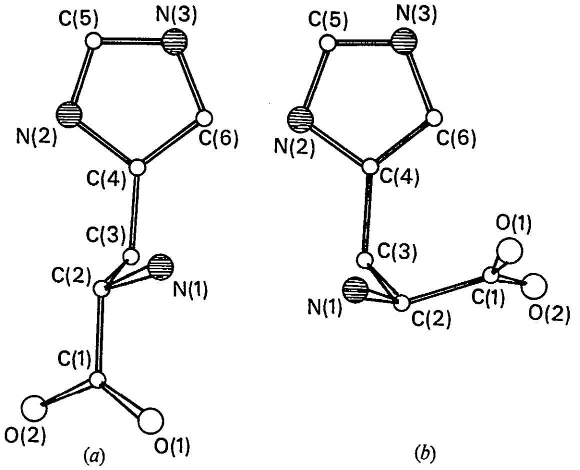
Table 1 (*cont.*)

	5	-79	-63
	6	-35	-35
	8	113	118
	9	-52	-47
5	K	0	
	1	128	93
	2	-174	-149
	3	35	32
	4	47	30
	5	94	92
	6	-64	-68
	7	180	161
	8	-94	-92
	9	25	22
	10	-50	-44
	11	-59	-57
	12	-100	-106
	13	-61	-55
	14	-25	-25
	17	-56	-57
5	K	-1	
	1	-112	-98
	2	-69	-59
	3	201	189
	6	61	67
	7	77	66
	9	89	81
	10	-178	-101
	11	81	66
	12	56	54
	13	-61	-96
	14	-136	-129
5	K	-2	
	1	-92	-123
	2	59	61
	3	-144	-226
	4	-25	-24
	5	-153	-148
	6	-18	-12
	7	-232	-241
	8	231	230
	9	66	58
	10	112	110
	11	87	79
	12	-66	-45
	13	66	50
	14	59	55
	15	31	28
	16	64	55
	17	35	37
5	K	-3	
	1	308	293
	2	-53	-66
	3	-188	-196
	4	-144	-156
	5	-170	-150
	6	-43	-43
	7	-43	-42
	8	56	50
	9	-124	-113
	10	-59	-61
	11	-121	-132
	12	66	50
	13	181	173
	14	61	54
	15	-92	-75
	16	-69	-61
	17	-25	-11
5	K	-4	
	1	215	216
	2	-228	-217
	3	194	175
	4	137	127
	5	90	90
	6	95	112
	8	-238	-235
	9	-35	-43
	10	115	105
	11	66	55
	13	-150	-134
	14	-64	-58
	15	-79	-62
	16	35	27
	17	31	37
5	K	-5	
	1	-181	-196
	2	195	174
	3	47	48
	5	-43	-49
	7	25	36
	8	105	111
	9	210	212
	11	61	50
	12	-92	-100
	13	-47	-54
	14	53	48
	15	71	81
5	K	-6	
	2	-40	-40
	3	-120	-114
	4	35	16
	5	-73	-84
	6	-59	-58
	7	106	129
	10	-56	-57
	11	73	67
	12	47	47
	13	64	72
	14	-35	-35
5	K	-7	
	1	135	134
	3	92	96
	4	-66	-68
	5	-35	-30
	6	25	32
	7	-128	-137
	8	-35	-21
	12	61	71
	13	-40	-40
5	K	-8	
	1	56	96
	3	-81	-53
	4	35	38
	6	-59	-49
	7	-73	-50
	8	56	29
	12	-35	-42
	13	18	20
5	K	-9	
	1	-64	-69
	5	-40	-43
	7	61	67
	8	-43	-86
	9	31	32
5	K	-10	
	4	18	18
	5	40	50
	6	18	16
	7	18	18
5	K	1	
	1	73	47
	3	47	41
	4	-59	-49
	5	-53	-32
	6	125	118
	8	69	52
	10	179	169
	11	56	45
	14	64	68
	15	43	48
	16	25	22
5	K	2	
	1	81	64
	2	-100	-91
	3	-112	-99
	6	53	25
	7	-75	-82
	8	47	48
	9	-61	-63
	10	43	44
	11	59	60
	13	87	95
	15	-35	-42
5	K	3	
	1	-73	-71
	3	-35	-23
	4	-103	-110
	5	-56	-57
	7	-90	-82
	9	25	21
	10	-102	-115
	11	-75	-82
	12	-31	-27
	13	25	26
	14	-18	-17
5	K	4	
	2	-35	-28
	4	71	70
	5	25	28
	7	59	62
	9	-31	-29
5	K	5	
	2	25	27
	3	56	63
	4	-40	-35
	5	40	44
	7	31	37
	8	43	44
	9	40	49
	10	-18	-21
5	K	6	
	2	-59	-70
	4	18	22
	5	-25	-17
6	K	0	
	0	198	170
	1	409	359
	2	116	107
	3	-238	-222
	4	189	164
	5	123	115
	6	155	134
	7	-137	-105
	8	-49	-38
	9	42	35
	11	-99	-94
	12	-71	-60
	13	96	91
	14	-60	-58
	15	-60	-65
	16	-24	-33
6	K	-1	
	0	-532	-376
	1	-93	-79
	2	75	69
	3	145	129
	4	-119	-125
	5	190	184
	6	286	266
	7	-172	-160
	8	-54	-50
	9	122	112
	10	199	211
	11	-69	-63
	12	-168	-159
	13	-39	-47
	14	116	118
	16	-30	-36
6	K	-2	
	1	-42	-33
	2	94	92
	3	151	129
	4	-234	-202
	5	-291	-274
	6	117	87
	7	232	227
	8	30	27
	9	-197	-186
	10	60	65
	11	92	46
	13	-39	-31
	14	117	101
	15	46	38
	16	-30	-15
6	K	-3	
	0	278	408
	1	-199	-215
	2	-86	-94
	3	83	91
	4	209	220
	5	-151	-148
	6	-109	-105
	7	118	118
	8	-116	-106
	9	-130	-126
	10	-144	-130
	11	207	207
	12	49	47
	13	-64	-55
	14	-79	-75
	15	73	67
	16	17	13
6	K	-4	
	0	196	185
	1	24	32
	2	-168	-161
	3	17	27
	4	180	177
	5	-24	-11
	6	-210	-212
	7	-46	-47
	8	49	28
	9	156	138
	10	-242	-214
	11	-113	-102
	12	67	59
	13	54	58
	14	-112	-106
	16	91	100
6	K	-5	
	0	-181	-168
	1	159	171
	2	-97	-112
	3	-211	-214
	4	-60	-65
	5	249	242
	6	114	105
	7	-191	-195
	8	142	136
	9	69	51
	10	-60	-52
	11	-102	-98
	12	39	44
	14	46	41
	15	-57	-63
	16	-17	-32
6	K	-6	
	0	-214	-221
	1	39	34
	2	93	102
	3	-60	-52
	4	39	25
	5	86	95
	6	189	189
	8	-60	-55
	10	214	222
	12	-128	-117
	13	52	61
	14	49	52
	15	-39	-51
6	K	-7	
	0	-100	-94
	1	-100	-94
	2	174	189
	3	102	105
	4	-42	-43
	5	-134	-135
	6	30	29
	7	69	85
	8	-67	-73
	9	39	30
	11	24	27
	12	-39	-41
	13	-42	-41
	14	17	31
6	K	-8	
	0	150	148
	1	-30	-30
	2	-60	-54
	5	-131	-126
	6	-57	-57
	7	93	94
	9	-110	-129
	10	-91	-103
	11	17	25
	12	39	42
6	K	-9	
	0	30	38
	1	-64	-56
	2	-112	-129
	4	52	54
	5	46	52
	6	-42	-39
	7	-39	-35
	9	24	24
	10	-17	-19
6	K	-10	
	0	-57	-74
	1	49	62
	3	-34	-43
	5	42	53
6	K	1	
	0	64	68
	1	-116	-100
	2	-83	-58
	3	-81	-75
	4	110	104
	5	102	99
	6	-242	-249
	7	-154	-168
	8	94	102
	9	69	72
	10	-153	-164
	11	30	30
	12	93	102
	13	57	59
	14	-54	-48
	15	-17	-17
6	K	2	
	0	211	203
	1	-77	-59
	2	-304	-289
	3	157	140
	4	-52	-49
	5	-113	-114
	6	-42	-46
	7	134	148
	8	-30	-33
	9	-39	-30
	10	-54	-59
	11	69	76
	12	94	110
6	K	3	
	0	-176	-169
	2	121	112
	3	109	107
	5	-123	-120
	6	30	25
	7	77	93
	9	-103	-114
	10	34	38
	12	-54	-61
	13	-46	-90
6	K	4	
	0	-116	-120
	1	-39	-35
	2	114	127
	4	-24	-20
	5	39	25
	6	83	89
	7	-71	-64
	8	-24	-13
	10	49	63
	11	-24	-41
6	K	5	
	0	52	53
	1	49	39
	2	-24	-17
	3	-54	-61
	5	71	93
	6	24	32
	7	-42	-67
6	K	6	
	0	24	44
	1	24	46
7	K	0	
	1	158	134
	2	-83	-51
	3	-102	-93
	4	38	51
	8	-42	-37
	9	-100	-106
	10	72	62
	11	-70	-63
	12	24	25
7	K	-1	
	1	-109	-104
	2	-96	-88
	4	103	92
	5	24	38
	6	24	21
	7	-48	-41
	8	-120	-112
	9	157	168
	10	-24	-30
	12	-38	-53
7	K	-2	
	1	-83	-80
	2	149	148
	3	131	136
	4	-54	-51
	6	38	37
	7	93	103
	8	83	75
	9	143	140
	11	74	55
	12	-45	-36
7	K	-3	
	1	116	171
	2	38	34
	3	-113	-113
	4	-38	-54
	6	42	36
	9	-125	-116
	10	91	73
	11	45	69
	12	38	22
	14	54	45
	15	24	31
7	K	-4	
	1	70	68
	2	-103	-117
	3	85	77
	5	-152	-157
	6	-134	-133
	7	-122	-107
	8	-48	-24
	9	-59	-52
	10	-94	-87
	11	-74	-65
	14	-38	-33
	15	17	20
7	K	-5	
	2	68	68
	3	-109	-105
	4	-24	-15
	9	-59	-48
	10	-70	-54
	11	72	58
	12	38	46
	14	-48	-57
7	K	-6	
	1	-110	-109
	3	-24	-51
	4	129	140
	5	24	43
	6	24	20
	9	143	153
	10	105	116
	11	-45	-35
	12	-24	-29
	14	24	28
7	K	-7	
	1	70	58
	2	48	36
	3	76	90
	4	-42	-51
	7	56	56
	10	-61	-45
	12	17	8
7	K	-8	
	1	81	85
	3	70	57
	4	-42	-41
	7	-45	-36
	8	-63	-66
	9	-38	-43
7	K	-9	
	1	-24	-27
	2	24	22
	3	-61	-60
	7	-38	-28
	8	24	50
	9	-34	-6
7	K	1	
	1	110	94
	2	68	60
	4	-116	-111
	5	-48	-25
	6	-70	-55
	7	-56	-60
	8	138	149
	9	-76	-70
	10	-45	-48
	11	24	37
	12	34	64
7	K	2	
	1	-107	-101
	2	-59	-50
	4	24	22
	5	-81	-77
	6	24	28
	7	-24	-28
	8	-45	-44
	9	59	69
7	K	3	
	2	48	45
	3	-61	-52
	5	38	38
	6	38	37
	7	74	85
7	K	4	
	3	81	100
7	K	5	
	2	-24	-48
8	K	0	
	1	-254	-247
	2	87	88
	3	49	56
	4	-79	-84
	5	-77	-76
	8	-150	-146
	9	-75	-85
	10	-52	-48
	11	79	79
9	K	0	
	1	82	87
	2	52	60
	4	-82	-86
	5	-28	-27
	6	-37	-36
	10	-16	-23
10	K	0	
	0	104	111
	3	-52	-54
	4	88	94

The structure of histidine itself (C) has also just been determined in this laboratory (Edington, 1969). From these studies we hope to discover how the conformation, and perhaps bond lengths or angles can vary from one situation to another. In the structure of DL-bis(histidino)cobalt there are two chemically identical histidine groups which differ in conformation by small rotations about several bonds, a difference which must be due to their different hydrogen bonding environments in the crystal lattice. In crystals of L-cysteine (Harding & Long, 1968*b*) two molecules in the asymmetric unit were found to have major differences in their conformations; N–C$_{\alpha}$–C$_{\beta}$–S is *trans* in one and *gauche* in the other, showing that the energy of the two forms is sufficiently close for van der Waals forces or hydrogen bonds to be able to tip the balance in favour of either. We have now found that the conformation of the histidine cation in our DL crystals is quite different from that in L-histidine hydrochloride (DC).

Crystal data

DL-Histidine hydrochloride dihydrate, $C_6H_9N_3O_2.HCl.2H_2O$, $M=227\cdot5$, monoclinic needles elongated along **c**. $a=8\cdot87\pm0\cdot01$, $b=15\cdot30\pm0\cdot02$, $c=8\cdot48\pm0\cdot01$ Å, $\beta=114\cdot5\pm0\cdot20°$, $V=1047$ Å^3, $D_m=1\cdot44$ g.cm^{-3}, $Z=4$, $D_{\rm calc}=1\cdot44$ g.cm^{-3}.
Space group $P2_1/a$, Cu $K\alpha$ radiation, $\mu=24\cdot3$ cm^{-1}.



(*a*) (*b*)

Fig. 1. The conformation of the L-histidine cation in (*a*) DL-histidine hydrochloride dihydrate, and (*b*) L-histidine hydrochloride monohydrate. In both cases the atomic positions are projected onto the plane of the imidazole ring.

Experimental

Commercial DL-histidine hydrochloride was recrystallized from aqueous alcohol (1:1). The intensities of 1469 reflexions were estimated visually from multiple film Weissenberg photographs of the layers *0kl* to *7kl* and *hk*0. Lorentz and polarization corrections were applied and all the reflexions placed on a common scale.

Solution and refinement of the structure

The chlorine atom positions were found from a three-dimensional sharpened Patterson series. They are very nearly on the glide planes at $y=0{\cdot}25$ and $y=0{\cdot}75$ and so there was an ambiguity in the relation of the set of chlorine atom positions to the symmetry elements of the cell; they may be placed at 0·04, 0·25, 0·20 and symmetry related positions, or at $0{\cdot}04+\frac{1}{4}$, 0·25, 0·20 *etc.* A chlorine-phased electron density series was calculated, which is the same for either set of chlorine positions and has a false mirror plane at $y=0{\cdot}25$. In its interpretation the first set of chlorine positions was initially assumed and a plausible trial structure found; however electron density difference maps failed to show how it could be improved and the agreement of observed and calculated structure amplitudes was very poor for reflexions with h odd ($R\sim0{\cdot}7$) although quite good for those with h even ($R<0{\cdot}4$). When the alternative set of chlorine positions was assumed, the histidine cation already found could be displaced by $a/4$, and the structure was then easily refined, first by difference electron density series (to $R=0{\cdot}33$) and then by 'least squares'.

A full-matrix least-squares refinement program based on that of Busing, Martin & Levy (1962) was used. Form factors were taken from *International Tables for X-ray Crystallography* (1962), the carboxyl oxygen atoms were treated as $O^{1/2-}$. For weighting purposes $\sigma(F)$ was taken as $0{\cdot}05|F|$ when $|F|>10{\cdot}0$ and $5/|F|$ otherwise. Refinement of positional parameters, isotropic thermal parameters and layer scale factors reduced R to 0·15. A difference electron density series indicated seven hydrogen atoms and some large anisotropic thermal vibrations. Layer scale factors were not adjusted after this, but the contributions of these seven hydrogen atoms to F_{calc} were included, and anisotropic thermal vibration parameters were refined. The refinement converged, with $R=0{\cdot}108$.

A difference electron density series then showed no peaks greater than 0·5 e.Å^{-3} except near the sites of the two water molecules (peaks of 0·7 and 0·9 e.Å^{-3}) and in possible positions for the amino hydrogen atoms which had not been included in the structure factor calculation.

Observed and calculated structure factors are given in Table 1, positional and thermal parameters in Table 2, and bond lengths and angles in Table 3.

Table 3. *Bond lengths, bond angles and angles between planes, in the histidine cation*

Those in the DL-crystals (this work) are compared with those in the L-crystals (DC).

	DL-crystals	L-crystals
(*a*) Bond lengths ($\sigma=0{\cdot}01$ Å)		
C(1)–C(2)	1·515 Å	1·530 Å
C(2)–C(3)	1·545	1·527
C(3)–C(4)	1·487	1·508
C(4)–C(6)	1·355	1·358
C(4)–N(2)	1·403	1·386
C(5)–N(2)	1·306	1·319
C(5)–N(3)	1·331	1·314
C(6)–N(3)	1·381	1·359
C(2)–N(1)	1·504	1·495
C(1)–O(1)	1·235	1·240
C(1)–O(2)	1·262	1·265

Table 2. *Positional and vibrational parameters*

The numerals in parentheses are 10⁴ times the estimated standard deviations of the positional parameters. The vibrational parameters, U, are 10³ times the mean square vibration amplitudes (Å²), with estimated standard deviations ~3. The hydrogen parameters are those used in the structure factor calculations and have not been refined.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{31}	U_{12}
Cl	0·2908 (2)	0·2460 (1)	0·1975 (2)	49	38	61	1	23	−7
C(1)	0·7909 (7)	0·3082 (3)	0·6412 (8)	36	31	50	0	25	2
C(2)	0·8038 (7)	0·2109 (3)	0·6161 (8)	30	32	48	1	17	2
C(3)	0·8771 (7)	0·1670 (3)	0·7964 (8)	34	33	46	2	11	−1
C(4)	0·8957 (7)	0·0710 (3)	0·7836 (8)	36	30	39	1	10	1
C(5)	0·9978 (8)	−0·0515 (4)	0·7326 (9)	49	32	52	−2	19	−1
C(6)	0·8070 (8)	0·0050 (4)	0·8102 (9)	43	38	48	6	14	0
N(1)	0·6334 (6)	0·1748 (3)	0·5119 (7)	30	32	48	−6	10	0
N(2)	1·0140 (6)	0·0334 (3)	0·7345 (7)	38	31	51	−1	19	−3
N(3)	0·8739 (7)	−0·0705 (3)	0·7766 (7)	47	33	55	0	18	−8
O(1)	0·6537 (5)	0·3389 (3)	0·6146 (7)	36	33	85	−7	29	4
O(2)	0·9246 (5)	0·3504 (2)	0·6864 (7)	42	34	75	−9	27	−6
O(*W*1)	0·6661 (6)	0·0270 (3)	0·3354 (7)	62	37	65	−6	22	3
O(*W*2)	0·4380 (8)	0·0988 (4)	0·0340 (8)	106	65	69	6	34	8

	x	y	z	$\bar{U}$		x	y	z	$\bar{U}$
H(C2)	0·8828	0·1970	0·5480	25	H(C6)	0·7058	0·0112	0·8486	25
H(C3)	0·7780	0·1778	0·8470	25	H(N2)	1·0985	0·0688	0·7054	25
H(C3)	0·9907	0·1992	0·8818	25	H(N3)	0·8298	−0·1319	0·7875	25
H(C5)	1·0720	−0·0958	0·6992	25					

Table 3 (*cont.*)

(*b*) Bond angles (σ=0·4–0·6°)

O(1)–C(1)–O(2)	126·3°	125·8°
O(1)–C(1)–C(2)	118·3	120·0
O(2)–C(1)–C(2)	115·4	114·2
C(1)–C(2)–N(1)	109·1	109·4
C(1)–C(2)–C(3)	108·3	113·3
N(1)–C(2)–C(3)	109·2	111·1
C(2)–C(3)–C(4)	111·8	114·9
C(3)–C(4)–C(6)	129·4	131·6
C(3)–C(4)–N(2)	123·1	122·1
C(6)–C(4)–N(2)	107·5	106·2
C(4)–N(2)–C(5)	108·6	108·5
N(2)–C(5)–N(3)	108·3	108·7
C(5)–N(3)–C(6)	110·5	109·6
N(3)–C(6)–C(4)	105·1	106·9

(*c*) Dihedral angles (σ=0·5–1·0°)

O(1)–C(1)–C(2)–N(1)	−16·7°	0·4°	ψ_2*
O(1)–C(1)–C(2)–C(3)	102·1	125·0	
O(2)–C(1)–C(2)–N(1)	162·6	179·5	ψ_1
O(2)–C(1)–C(2)–C(3)	−78·6	−55·9	
C(1)–C(2)–C(3)–C(4)	179·4	−52·1	
N(1)–C(2)–C(3)–C(4)	−61·9	71·5	χ_1
C(2)–C(3)–C(4)–N(2)	−70·6	−120·5	χ_{21}
C(2)–C(3)–C(4)–C(6)	107·7	61·1	χ_{22}

* The signs, and symbols ψ_1, χ_{12} *etc.* correspond to those used by Lakshminarayanan, Sasisekharan & Ramachandran (1967).

Discussion

The crystals are racemates, containing equal numbers of D and L molecules, so throughout the discussion we shall compare the L-histidine cation found here with the same cation in L-histidine hydrochloride monohydrate (DC).

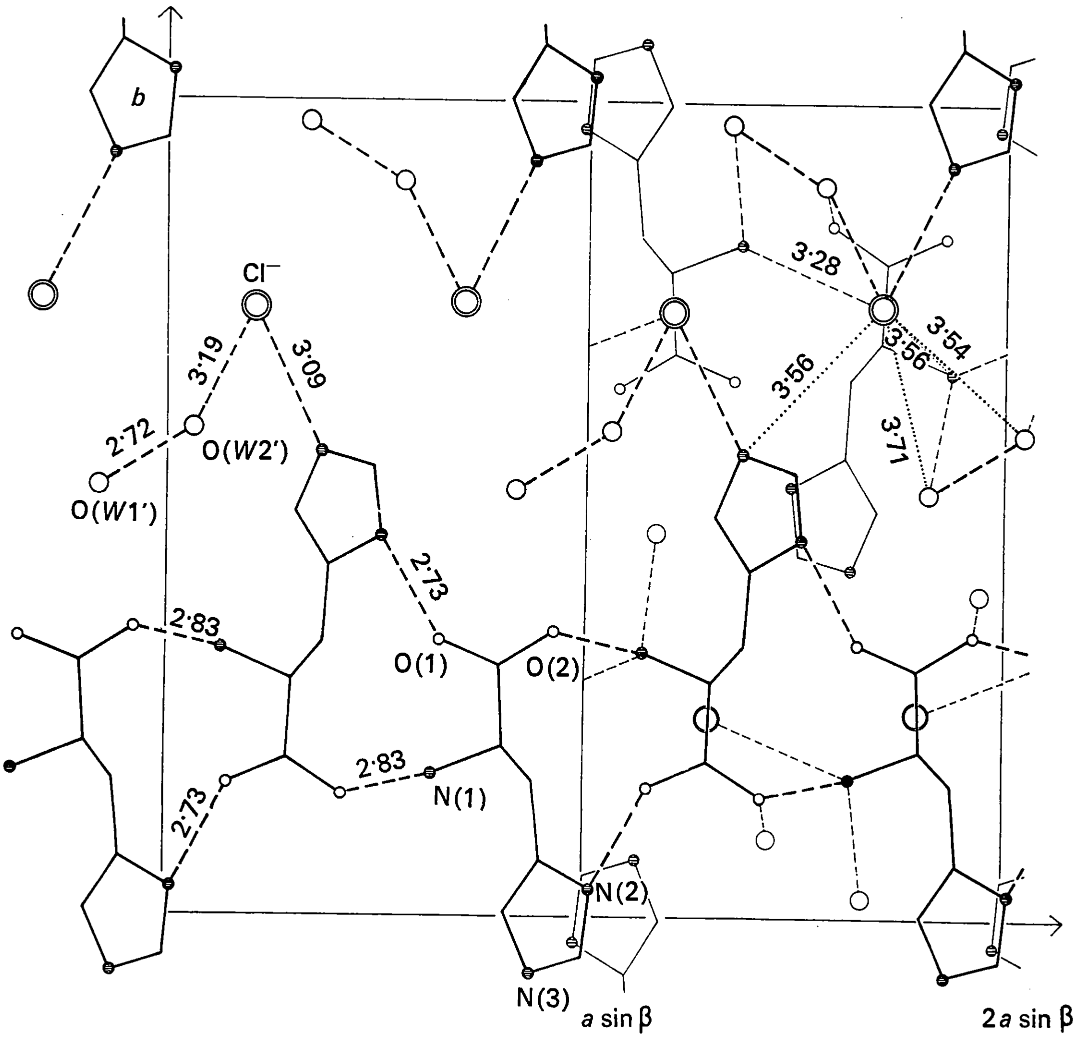


Fig. 2. *c*-Axis projection of the structure. The heavier lines represent molecules whose atoms have $\frac{1}{2}<z<1$ and hydrogen bonds between them (broken lines); only these molecules are shown in the unit cell on the left. On the right the remainder of the cell contents is included (thinner lines) and thin broken lines show hydrogen bonds joining the two regions. Additional close contacts are shown around one chloride ion as dotted lines.

Conformation

As shown in Table 3(*c*) and Fig. 1, the conformations are different, chiefly around the bonds C(2)–C(3) and C(3)–C(4). The imidazole ring is rotated about C(3)–C(4) by nearly 180°, thus interchanging the carbon and nitrogen atoms in relation to amino group; C(2) is nearer C(6) than N(2) in the L-crystals, but nearer N(2) than C(6) in the DL-ones. The C(3)–C(4) conformation is such that C(1) is *trans* in the DL crystals with respect to C(4), and N(1) is *gauche*, but in the L-crystals both C(1) and N(1) are in *gauche* positions. There are smaller differences in other torsion angles.

In the extended form found in the DL crystals interatomic repulsions within the cation should be less than in the compact form in the L crystals, but the latter would have a more favourable electrostatic energy since the negative carboxylate group is closer to the positively charged imidazole ring. In each crystalline form the molecules participate in seven hydrogen bonds which may appreciably affect the relative energies of the different conformations. Crystallization from a solution containing D- and L-histidine yields the racemic crystals rather than separate D and L crystals so the former must be more favourable. These results emphasize that predictions about conformation made on the basis of minimum energy or any other calculations for an isolated molecule, may not always be correct for the molecule in a crystal – or in any other environment.

Bond lengths and angles

Despite the difference in conformation the bond lengths [Table 3(*a*)] are in close agreement in the L- and

Table 4. *Intermolecular contacts*

Hydrogen bonds			
N(1)·····O(2)	at	$-\frac{1}{2}+x, \frac{1}{2}-y, z$	2·84 Å
N(1)·····O(*W*1)			2·79
N(2)·····O(1)	at	$\frac{1}{2}+x, \frac{1}{2}-y, z$	2·73
O(2)·····O(*W*2)	at	$\frac{1}{2}+x, \frac{1}{2}-y, 1+z$	3·00
O(*W*1)···O(*W*2)			2·75
Cl·······N(1)			3·29
Cl·······N(3)	at	$1-x, -y, 1-z$	3·11
Cl·······O(*W*2)			3·19
Other contacts less than 3·5 Å			
C(1)···O(*W*2)	at	$\frac{1}{2}+x, \frac{1}{2}-y, 1+z$	3·35
C(2)···O(1)	at	$\frac{1}{2}+x, \frac{1}{2}-y, z$	3·20
C(3)···O(1)	at	$\frac{1}{2}+x, \frac{1}{2}-y, z$	3·40
C(4)···O(1)	at	$\frac{1}{2}+x, \frac{1}{2}-y, z$	3·45
C(6)···O(*W*2)	at	$1-x, -y, 1-z$	3·37

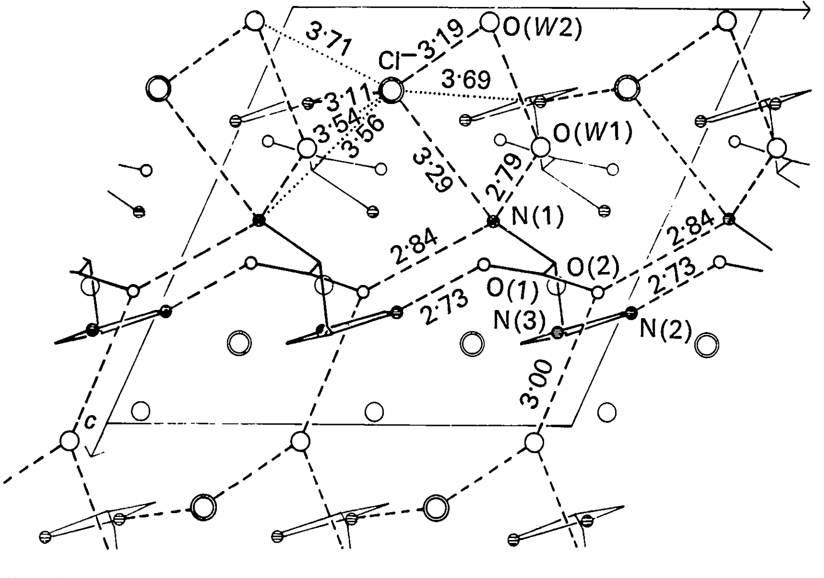


Fig. 3. *b*-Axis projection of the structure. Double circles represent chloride ions, open circles oxygen and shaded circles nitrogen atoms. Heavy lines represent molecules straddling the glide plane at $y=\frac{1}{4}$, and the water molecules near it, with hydrogen bonds shown as broken lines. Thinner lines represent the molecules straddling the glide planes at $y=-\frac{1}{4}$ or $\frac{3}{4}$ (hydrogen bonds are not shown for these). Additional close contacts are shown around one chloride ion.

DL-crystals; there are no differences greater than 3σ (0·03 Å); the root mean square difference is 0·015 Å ($1·5\sigma$), and the variations in bond lengths around the imidazole ring follow the same pattern.

Only four of the bond angles [Table 3(*b*)] in the DL crystals differ by more than 3σ from those in the L crystals, and only two of these differences are much greater than 3σ. C(1)–C(2)–C(3) and C(2)–C(3)–C(4) are both 5° smaller in the DL than in the L crystals, and in the DL crystals their values (108·3 and 111·8°) are closer to the tetrahedral angle; this suggests that these angles have increased (to 113·3 and 114·9°), in the compact conformation in the L-crystals, to relieve the repulsions between non-bonded atoms within the molecule.

Like the bond lengths, the bond angles in and adjacent to the imidazole ring follow the same pattern in both L and DL crystals. It is particularly noteworthy that C(3)–C(4)–C(6) is large (131·6, 129·4°) compared with C(3)–C(4)–N(2) (123·1, 122·1°); this cannot be attributed to repulsion by C(2) since this atom is nearer to C(6) in the L-crystals, but to N(2) in the DL-crystals; it must presumably be a result of the electronic structure around C(4), and connected with the fact that C(4)–C(6) is shorter, and so of higher bond order, than C(4)–N(2).

The variation in angles at C(1) is the same in the two crystal forms. As in other amino-acids O(1)–C(1)–C(2) is slightly greater than O(2)–C(1)–C(2). The average values for amino-acids given by Marsh & Donohue (1967) are 118·0 and 116·4°, but both histidine cations have smaller values still for the second angle.

C(3) and all the atoms of the imidazole ring are coplanar, within 0·01 Å or two standard deviations. C(2) and the atoms of the carboxyl group are coplanar, within 0·004 Å, but N(1) is 0·42 Å out of this plane.

Hydrogen bonds and intermolecular contacts

These are listed in Table 4, and Figs. 2 and 3 illustrate the packing in the crystal lattice.

Ribbons of histidine cations, each straddling a glide plane, extend in the **a** direction. Each histidine cation forms four strong N–H···O bonds to its two glide plane related neighbours. In the **b** direction the edges of neighbouring ribbons are held together by the interaction of chloride ions with positively charged imidazole groups, and by one N(3)–H···Cl^- bond from each molecule (see Fig. 2). In the **c** direction, neighbouring ribbons are displaced by $b/2$ relative to each other, so that the chloride ions can interact with the $-NH_3^+$ groups (see Fig. 3). In addition, hydrogen bonds involving the water molecules hold the ribbons together. The imidazole groups of molecules in neighbouring ribbons lie in parallel planes 3·40 Å apart.

The chloride ion makes three short hydrogen bonded contacts with an N(1), an N(3) (both positively charged), and an O(*W*1) (see Table 4); there are also a number of more distant neighbours the closest of which are an O(*W*1) at 3·54 Å, an N(1) at 3·56 Å an N(3) at 3·69 Å and an O(*W*2) at 3·71 Å. This may be contrasted with the situation in the L crystals where the chloride ion makes hydrogen bonds to two $-NH_3^+$ groups and a water molecule, but has no imidazole ring atoms as neighbours.

Donohue & Caron (1964) have pointed out that in the L-crystals and in both bis(histidino)zinc complexes the conformation around C(2)–N(1) is the expected staggered one and that even the atoms involved in the hydrogen bonds N–H···X are appropriately placed for this. Fig. 4 shows a projection along C(2)–N(1) in the DL-crystals. The three hydrogen bonded atoms are slightly rotated from the ideal staggered positions. The hydrogen atom positions have not been accurately determined, but in the final difference electron density series there are two peaks of 0·6 e.Å^{-3} at the positions indicated in Fig. 4, corresponding closely to a staggered conformation (and at the appropriate position for the third hydrogen of the $-NH_3^+$ group there is a region 0·4 e.Å^{-3} high.)

Thermal vibrations

For the whole histidine cation and for the chloride ions the vibration amplitudes found in the least-squares refinement are greater for vibrations perpendicular to the *ab* plane than for vibrations in it. The large vibration amplitudes are across the planes of the ribbons, which is consistent with the looseness of the molecular packing in this direction; the vibrations are particularly large for the carboxyl oxygen atoms.

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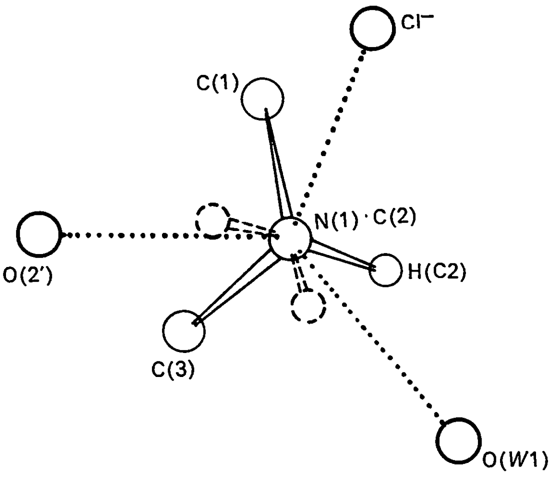

Fig. 4. Projection along the bond N(1)–C(2) showing the positions of atoms involved in hydrogen bonds with N(1) and the tentative positions (broken circles) of two of the three amino hydrogen atoms.

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An Accurate Crystal Structure Determination of 2,2′-Diaminodiphenyl Disulphide

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The crystal structure of 2,2′-diaminodiphenyl disulphide has been determined accurately from three-dimensional X-ray diffraction data. Crystals are orthorhombic, and belong to the space group *Pbca*. The unit cell has dimensions $a=8{\cdot}21$, $b=13{\cdot}14$, $c=22{\cdot}77$ Å, and contains eight molecules. The positional and anisotropic thermal parameters for the sulphur, carbon and nitrogen atoms have been refined by Fourier and full-matrix least-squares methods on 1313 independent observed reflexions. In the later stages hydrogen atoms were included in fixed positions, and the final agreement factor $R=8{\cdot}6\%$. The central part of the molecule C–S–S–C has a skewed non-planar conformation similar to H_2O_2, with a dihedral angle of 90·5°. The S–S bond length is 2·06 Å, which is longer than in diphenyl and dibenzyl disulphides. The C–S bond lengths of 1·77 and 1·75 Å are significantly short. This indicates some double bond character, which may be explained by $p\pi$–$d\pi$ bonding. The benzene rings are inclined at an angle of 32·5° to each other.

Experimental

A sample of 2,2′-diaminodiphenyl disulphide, $H_2N–C_6H_4–S–S–C_6H_4–NH_2$, kindly provided by the Clayton Aniline Co. Ltd, was recrystallized from ethanol and gave pale yellow-green platelets elongated along **a**. These showed straight extinction parallel and perpendicular to **a** when viewed under the polarizing microscope. The density of the crystals was measured by flotation in aqueous potassium iodide solution, and was found to be 1·33 (5) g.cm^{-3}.

Considerable difficulty was experienced in selecting a single crystal for X-ray examination. Many crystals consisted of several parallel platelets, indistinguishable from a single crystal under the polarizing microscope but detectable on higher layer line Weissenberg photographs. Eventually a suitable single crystal of dimensions 0·4 × 0·3 × 0·15 mm was used to obtain oscillation photographs about the *a* and *b* axes, and equi-inclination Weissenberg photographs for the 0*kl* – 6*kl* and *h*0*l* zones. The relative intensities of 1313 independent reflexions were measured visually and corrected for Lorentz and polarization factors. Absorption corrections were considered unnecessary, in view of the small crystal size compared with the optimum size of 0·50 mm (Buerger, 1958). Reflexions too weak to be observed were omitted.

Crystal data

$C_{12}H_{12}N_2S_2$, M.W. 248·36,

Orthorhombic; $a=8{\cdot}21\pm0{\cdot}02$, $b=13{\cdot}14\pm0{\cdot}02$, $c=22{\cdot}77\pm0{\cdot}03$ Å

$U=2456{\cdot}4$ Å^3, $Z=8$, $D_m=1{\cdot}33$ (5) g.cm^{-3}, $D_c=1{\cdot}343$ g.cm^{-3}

$F(000)=1040$, Cu $K\alpha$, $\lambda=1{\cdot}5418$ Å, $\mu=39{\cdot}7$ cm^{-1}.

Absent reflexions *hk*0 when *h* odd, 0*kl* when *k* odd, *h*0*l* when *l* odd. The space group is thus uniquely determined as *Pbca* (D_{2h}^{15}), no. 61.

Structure analysis

The determination of the crystal structure of 2,2′-diaminodiphenyl disulphide was undertaken as part of a series of accurate structure determinations on disul-