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The Crystal and Molecular Structure of Bis-[2-(*N*,*N*-dimethylamino)ethyl]disulfide Dihydrochloride

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The crystal and molecular structure of bis-[2-(*N*,*N*-dimethylamino)ethyl]disulfide dihydrochloride [alternatively named 2,2′-dithiobis-(*N*,*N*-dimethyl)ethylamine dihydrochloride] has been determined crystallographically using the 1972 unique and significant reflections for which $2\theta < 52°$, and refined to an *R* index of 0·043. Counter methods with graphite monochromatized Mo $K\alpha$ radiation were employed. All hydrogen atoms were located on a difference Fourier function. Crystals form in the monoclinic space group $P2_1/c$ with cell dimensions $a = 11{\cdot}646$ (2), $b = 11{\cdot}065$ (2), $c = 12{\cdot}370$ (3) Å and $\beta = 112{\cdot}40$ (2)°. Estimated standard deviations in non-hydrogen bond lengths range from 0·001 to 0·005 Å and, in angles, from 0·11 to 0·26°. The sulfur–sulfur bond length is found to be 2·037 (1) Å with a dihedral angle of 82·4 (2)°. A short intermolecular contact (3·55 Å) between a sulfur atom and a chloride ion is observed.

Introduction

A mean sulfur–sulfur bond length of 2·075 (6) Å and a mean CSSC dihedral angle of 106·3° were found in the X-ray crystallographic structure determination of the complex *cyclo*-di-μ-{bis-[2-(*N*,*N*-dimethylamino)ethyl]-disulfide}dicopper(I) tetrafluoroborate (Ottersen, Warner & Seff, 1973*a*). This bond length differs significantly from those usually found in aliphatic disulfides with dihedral angles near to 90° (Hordvik, 1966; Lee, 1972; Lee & Bryant, 1970; Lee & Bryant, 1969*a*, 1969*b*; Ottersen, Warner & Seff, 1973*b*). The sulfur–sulfur bond lengths reported for dicinnamyl disulfide (Lee & Bryant, 1971) and dibenzyl disulfide (Lee & Bryant, 1969*b*; van Dijk & Visser, 1971) are 2·01 (1) and 2·02 (1) Å, with CSSC dihedral angles of 66·4 and 92·1°, respectively. Hordvik (1966) gives 2·03–2·05 Å as the expected length of an aliphatic S–S bond. Although CSSC dihedral angles of 105 to 110° have been reported (Foss, Johnsen & Tvedten, 1958; Foss & Johnsen, 1965; Oughton & Harrison, 1959), values between 80 and 100° (Hordvik, 1966; Lee & Bryant, 1969) are usually observed. Repulsion between the lone-pair electrons is expected to be at a minimum when the dihedral angle is 90° (Bergson, 1957).

The present structural work was undertaken to determine the differences in molecular geometry between the free and complexed disulfide.

Experimental

Bis-[2-(*N*,*N*-dimethylamino)ethyl]disulfide dihydrochloride, alternatively named 2,2′-dithiobis-(*N*,*N*-dimethyl)ethylamine dihydrochloride, was prepared by the oxidation of 2-(*N*,*N*-dimethylamino)ethanethiol hydrochloride with dimethyl sulfoxide (Yiannos & Karabinos, 1963). The crude product was recrystallized from acidified aqueous ethanol. Hygroscopic colorless needle-shaped crystals were formed. Total elemental analysis yielded results which were in excellent agreement with the theoretical composition.

Oscillation and Weissenberg photographs indicated monoclinic symmetry; all reflections *h*0*l* for *l* odd and 0*k*0 for *k* odd, were systematically absent, uniquely defining the space group $P2_1/c$.

A Syntex four-circle computer-controlled diffractometer with graphite-monochromatized Mo $K\alpha$ radiation ($K\alpha_1$, $\lambda = 0{\cdot}70926$ Å; $K\alpha_2$, $\lambda = 0{\cdot}71354$ Å) and a pulse-height analyzer was used for preliminary experiments and for the measurement of diffraction intensities. The cell constants and their standard deviations were determined by a least-squares treatment of the angular coordinates of fifteen independent reflections with 2θ values up to 25·9°. The program used was written by Robert A. Sparks and is part of the diffractometer program library. The temperature was maintained within 1° of 20°C.

A nearly cylindrical crystal of dimensions 0·5 × 0·2 × 0·2 mm, mounted along its long (*a*) axis in a thin-walled glass capillary tube, was used for data collection. The θ–2θ scanning mode was utilized with scan speed variable from 2 to 24° min^{-1}, depending on the peak intensity of the reflection. Background counting time was equal to the scan time, and the scan range varied from 2·0° at low 2θ to 2·3° at 52°. The variations in the intensities of three check reflections, which were remeasured after every hundred during data collection, were all less than twice their estimated standard deviations, so no decay correction was applied.

Standard deviations were assigned to the individual reflections according to the formula,

$$\sigma(I) = \{\omega^2[CT + B_1 + B_2 + (pI)^2]\}^{1/2},$$

where ω is the scan rate, CT is the total integrated count, B_1 and B_2 are the background counts, and the intensity is $I = \omega(CT - B_1 - B_2)$. A value of 0·02 was assigned to the empirical parameter p to account for

Table 1. *Observed and calculated structure factors*

The running index is l; values of h and k for each group immediately precede that group. The central column is $10|F_o|$; the right-hand column is $10F_c$. All of the X-ray diffraction intensities which were observed to be greater than three times their standard deviations were used in structure refinement and are listed in this table.

H= 0, K= 0		
2	611	-595
4	1098	-1083
6	722	697
8	478	-460
10	345	-335
12	79	76
H= 0, K= 1		
1	496	-446
2	577	-506
3	1460	-1353
4	213	178
5	600	560
6	326	281
7	378	375
10	257	260
12	66	67
H= 0, K= 2		
0	621	550
1	577	612
2	192	174
3	197	195
5	64	58
6	220	-223
7	474	-473
8	120	-113
9	302	-291
13	93	-97
H= 0, K= 3		
1	176	-160
2	908	-841
3	658	840
4	255	249
5	152	-157
6	372	346
7	65	-69
9	174	170
10	334	341
11	115	-113
12	111	99
13	109	-106
H= 0, K= 4		
0	1931	-1828
1	638	625
2	203	203
3	105	100
4	213	216
6	521	-506
8	170	164
9	168	-165
10	204	194
11	135	-124
H= 0, K= 5		
1	358	342
2	608	602
3	485	448
4	842	-821
5	145	-141
6	209	-201
8	252	247
9	82	-75
10	54	-49
11	82	-71
13	102	-88
H= 0, K= 6		
0	562	-518
1	715	700
3	434	418
5	145	-144
6	84	83
7	403	401
8	69	60
9	110	104
11	151	-150
H= 0, K= 7		
1	144	135
2	155	138
3	248	-250
4	449	-432
6	404	-393
8	90	94
-11	96	93
9	185	-189
11	94	93
H= 0, K= 8		
0	403	448
1	381	-364
2	141	-139
3	253	245
5	364	335
6	395	378
7	55	-63
9	57	46
10	92	-94
H= 0, K= 9		
2	446	-437
3	210	-215
4	326	318
5	69	-60
7	102	-97
8	122	-114
10	85	81
H= 0, K= 10		
1	320	-316
3	114	-110
4	101	97
5	66	71
6	71	68
7	192	-201
8	74	73
9	60	-63
H= 0, K= 11		
1	87	-72
3	264	268
4	232	225
5	121	-113
6	219	217
7	172	-176
H= 0, K= 12		
-5	236	232
0	328	-335
1	127	133
3	203	-194
4	117	129
5	241	-232
6	160	-158
H= 0, K= 13		
2	202	198
3	89	74
H= 1, K= 0		
-14	69	68
-10	75	72
-8	207	194
-6	848	-836
-4	729	-743
-2	587	-595
0	96	-86
2	414	426
4	767	804
6	577	619
8	232	233
10	274	-291
H= 1, K= 1		
-11	175	-151
-10	223	231
-9	84	74
-8	87	-96
-7	62	71
-6	458	-446
-5	527	535
-4	211	188
-3	846	813
-2	44	-41
-1	322	322
0	62	-64
1	95	-110
2	365	-357
3	788	-769
4	115	-126
5	565	-543
6	76	71
7	134	-140
8	293	-290
9	69	-78
11	64	78
12	194	197
13	121	130
H= 1, K= 2		
-13	271	-287
-10	72	72
-9	63	64
-8	177	183
-7	91	95
-6	137	-138
-5	214	219
-4	299	-288
-3	308	295
-2	318	-320
-1	226	235
0	73	-101
1	127	119
2	155	154
3	200	-178
4	244	251
5	618	602
6	152	160
7	65	72
8	91	90
10	132	-139
12	76	-69
13	177	-163
H= 1, K= 3		
-14	83	95
-12	163	163
-10	142	145
-9	240	-240
-8	67	-68
-7	206	-227
-6	401	-405
-5	87	-95
-4	170	-188
-3	57	-54
-2	84	-99
-1	203	195
0	94	-98
2	122	-119
3	151	145
5	94	85
6	141	-159
8	255	-259
9	186	183
10	64	64
11	59	55
12	141	148
H= 1, K= 4		
-13	63	-65
-12	111	106
-11	127	-120
-10	158	164
-9	85	-89
-8	85	-80
-7	315	321
-6	594	599
-5	121	132
-4	309	322
-3	145	168
-2	127	-111
-1	57	-39
1	217	-210
2	162	-163
4	47	-73
5	92	85
6	349	-361
7	77	85
8	296	-306
9	200	205
10	137	150
11	79	-83
12	93	-91
13	96	-75
H= 1, K= 5		
-12	85	87
-11	105	110
-10	104	-96
-9	245	-245
-7	180	-185
-6	146	-160
-5	132	-154
-4	239	-242
-3	352	-354
-2	202	209
-1	104	-122
0	339	347
2	415	415
3	206	271
4	56	50
5	337	351
6	183	-183
7	78	95
9	125	136
10	98	-103
12	67	-78
H= 1, K= 6		
-9	54	54
-7	170	177
-6	143	145
-5	85	96
-4	142	156
-3	107	-111
-2	169	179
-1	475	-473
0	67	75
1	396	-399
3	200	-213
4	276	-291
5	202	-200
6	81	-96
7	209	217
8	122	-125
9	118	129
H= 1, K= 7		
-10	82	-80
-9	206	216
-8	127	-132
-7	174	183
-5	166	-160
-2	179	194
-1	155	-150
0	442	455
1	82	88
2	426	424
6	95	-96
7	167	167
9	126	-140
10	111	-113
11	99	-101
H= 1, K= 8		
-10	155	-162
-9	117	111
-6	273	-286
-5	104	103
-4	51	-34
-3	146	-149
-2	204	202
-1	241	-246
1	163	-174
3	72	-85
4	155	-150
6	130	131
7	105	103
8	167	131
11	86	75
H= 1, K= 9		
-10	76	50
-9	208	219
-8	88	-73
-7	142	146
-4	68	71
2	53	-53
7	139	-135
8	109	-130
9	151	-151
H= 1, K= 10		
-8	119	-112
-7	146	-127
-6	56	-53
-3	56	63
-2	75	82
-1	192	187
1	115	121
2	118	-123
3	57	54
4	63	53
5	129	129
7	60	-55
8	104	104
9	115	-103
H= 1, K= 11		
-8	68	57
-3	86	-85
-2	134	-133
0	279	-282
2	217	-226
3	108	113
5	85	83
6	76	78
7	92	-95
H= 1, K= 12		
-6	73	75
-5	77	-76
-4	87	86
-3	68	71
-1	201	203
1	153	156
3	121	111
6	134	-137
H= 1, K= 13		
0	94	-104
H= 2, K= 0		
-14	85	79
-10	90	-96
-8	351	368
-6	731	713
-4	756	-704
-2	942	-905
0	169	-123
2	1872	-1755
4	251	240
6	160	160
10	287	298
12	83	71
H= 2, K= 1		
-11	119	-103
-10	254	254
-8	309	298
-7	198	-180
-6	272	-257
-5	564	-513
-4	622	609
-3	89	50
-2	204	-256
-1	1890	1508
0	444	-406
1	263	255
2	698	650
3	535	511
5	363	342
6	351	-339
7	481	-478
8	114	-109
9	106	-93
11	64	-74
12	113	-104
13	65	-63
H= 2, K= 2		
-13	96	-130
-12	62	-65
-11	126	-120
-9	87	98
-8	70	-77
-7	1050	-1014
-6	265	266
-5	172	-157
-4	684	678
-3	1639	1561
-2	737	-707
-1	1833	-1729
0	395	-376
1	83	-75
3	763	746
4	248	-239
5	222	-221
7	62	48
8	226	226
9	376	373
10	97	86
11	124	135
H= 2, K= 3		
-11	290	289
-10	316	307
-8	547	537
-7	355	-350
-6	298	-308
-5	456	447
-4	550	545
-3	516	-490
-2	140	141
-1	1416	-1322
0	961	-917
1	839	780
2	156	172
3	111	109
4	312	333
5	428	-417
6	215	-211
7	173	180
8	238	-232
12	139	-133
H= 2, K= 4		
-14	124	-131
-12	77	-83
-9	160	-158
-8	283	-272
-7	399	-390
-6	63	50
-4	556	520
-3	665	641
-2	99	100
0	374	360
1	89	79
2	632	779
3	106	-92
4	389	-385
5	51	-42
6	58	71
7	130	-130
8	187	187
10	166	-174
11	146	144
12	94	-90
H= 2, K= 5		
-13	65	62
-12	90	89
-11	160	161
-9	65	-61
-7	132	114
-6	326	329
-5	152	147
-4	323	-315
-3	304	-295
-2	744	-707
-1	616	-583
0	186	184
2	222	-214
3	354	-355
4	137	129
5	290	-291
6	518	517
7	265	268
11	73	65
H= 2, K= 6		
-13	99	74
-10	75	-78
-9	278	-285
-8	122	119
-7	373	373
-6	254	-263
-5	425	418
-4	312	-296
-3	453	-427
-2	593	570
-1	404	757
1	324	323
2	223	223
3	705	-669
4	226	220
5	182	-176
7	133	-134
8	129	-130
9	155	-190
10	65	-45
H= 2, K= 7		
-11	191	-195
-8	158	-150
-7	293	284
-5	228	-235
-4	406	-406
-3	438	439
-2	293	-274
-1	306	290
1	604	-593
2	61	65
4	214	217
5	205	198
6	285	276
7	57	-60
8	164	180
9	76	71
11	88	83
H= 2, K= 8		
-12	90	80
-9	79	71
-8	178	176
-7	161	152
-6	232	-237
-4	470	-460
-3	173	170
-2	202	210
-1	196	192
0	140	-134
1	298	-306
2	264	-287
3	55	67
4	268	271
5	185	-187
6	62	-76
7	121	121
8	235	-239
H= 2, K= 9		
-11	186	-179
-10	96	133
-9	61	60
-7	115	105
-6	252	-252
-4	131	134
-3	219	219
-2	262	254
-1	377	365
0	346	-351
1	218	-209
2	202	198
4	132	135
5	270	271
6	257	-263
8	64	80
H= 2, K= 10		
-10	122	121
-9	69	66
-8	112	-115
-7	205	-212
-5	129	-126
-3	134	146
-2	264	-277
-1	279	-272
0	180	192
1	125	-118
3	341	335
4	142	-143
7	59	53
H= 2, K= 11		
-7	146	-149
-6	121	109
-5	273	277
-4	193	183
-3	54	-54
-2	96	85
-1	228	-226
0	57	58
1	160	164
2	57	-56
3	62	-57
4	185	-175
5	86	-83
6	150	-141
7	96	90
H= 2, K= 12		
-4	199	196
-3	226	-215
-2	194	-179
-1	96	-89
0	128	129
1	141	145
2	227	230
4	63	-75
5	185	174
H= 2, K= 13		
-3	82	-84
-1	142	-150
0	241	245
2	192	-187
H= 3, K= 0		
-10	52	49
-8	45	54
-6	204	202
-4	494	497
-2	471	452
0	503	474
2	441	-429
4	335	-353
6	474	-500
8	250	-251
H= 3, K= 1		
-14	151	-154
-13	112	-114
-11	65	50
-10	85	-61
-9	207	209
-8	48	-65
-7	372	-355
-6	48	48
-5	137	-157
-4	155	150
-3	302	-306
-2	333	349
-1	474	-479
0	194	194
1	273	-279
3	295	310
4	324	311
5	599	608
7	242	250
12	111	-120
H= 3, K= 2		
-14	74	82
-13	174	183
-11	339	355
-10	90	-100
-9	208	-200
-8	47	-52
-6	202	-198
-4	161	160
-3	467	-471
-2	373	407
-1	459	-474
0	386	385
1	361	-345
2	102	81
3	129	117
4	291	-303
6	238	-241
7	456	-467
9	175	185
11	107	109
H= 3, K= 3		
-14	128	-129
-12	106	-102
-11	52	-45
-10	291	-300
-9	168	165
-8	58	65
-7	459	462
-6	170	179
-5	76	63
-4	265	286
-3	98	-96
-2	488	501
-1	286	-298
0	66	98
1	45	-46
2	58	-58
4	172	153
5	232	-233
6	58	-60
7	101	101
8	169	172
10	90	66
11	177	-179
12	177	-176
H= 3, K= 4		
-13	160	163
-12	75	79
-11	127	132
-11	129	132
-10	176	-182
-9	73	75
-8	313	-316
-6	188	-198
-5	399	-389
-4	194	-199
-3	275	-301
-2	61	-67
-1	189	-186
0	81	-93
1	202	279
2	343	340
3	278	284
4	87	116
6	92	113
7	159	-168
8	170	180
10	67	59
11	67	-57
H= 3, K= 5		
-13	71	69
-12	170	-171
-10	101	-111
-9	114	-104
-8	153	151
-7	404	404
-6	75	95
-5	222	227
-4	250	299
-1	156	175
0	253	-259
1	106	106
2	347	-362
3	135	-135
4	355	-355
5	331	-340
6	84	91
7	198	-212
8	116	125
H= 3, K= 6		
-11	87	-73
-10	87	94
-9	196	197
-7	198	-209
-5	267	-273
-4	145	-156
-3	95	97
-2	243	-263
-1	291	302
0	168	-174
1	490	498
3	154	162
4	154	168
5	128	138
6	273	288
7	147	142
9	274	-276
10	73	55
11	108	-114
H= 3, K= 7		
-10	75	83
-9	117	-124
-9	112	-124
-7	267	-270
-6	100	114
-3	143	149
-2	228	-234
-1	221	233
0	262	-273
1	114	122
2	269	-281
3	154	-152
7	65	-72
4	229	-232
5	52	44
6	57	55
9	73	-71
H= 3, K= 8		
-11	59	-67
-10	212	216
-8	229	234
-6	143	152
-3	53	65
-2	72	-79
-1	198	202
0	97	-101
1	70	76
2	150	-147
3	122	123
4	55	47
6	71	63
8	123	-128
H= 3, K= 9		
-9	145	-124
-8	79	-82
-7	315	-315
-6	57	53
-5	125	-131
-3	95	81
-1	73	62
1	63	-40
2	65	61
4	143	141
5	105	107
6	72	-79
H= 3, K= 10		
-8	178	179
-6	127	122
-5	105	111
-3	63	-61
-1	144	-139
0	54	-65
1	227	-231
6	111	-113
7	79	-84
H= 3, K= 11		
-8	89	-78
-2	123	125
0	162	165
2	147	152
4	102	100
5	111	-106
H= 3, K= 12		
-1	152	-157
1	86	-95
3	111	-113
H= 3, K= 13		
0	91	86
2	67	70
H= 4, K= 0		
-14	114	-109
-12	173	-185
-10	374	374
-8	201	-175
-6	756	-731
-4	638	614
-2	337	328
2	663	624
4	602	584
6	193	-195
10	128	-128
H= 4, K= 1		
-15	103	98
-13	84	-77
-12	81	91
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-8	497	-479
-7	307	-320
-5	253	243
-4	153	119
-3	754	711
-2	1065	-1000
-1	673	-613
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1	1118	-1038
2	204	138
3	169	155
4	293	-293
5	202	-203
6	338	326
7	210	-209
8	87	83
9	297	300
11	144	136
H= 4, K= 2		
-15	82	-73
-13	134	-129
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-11	181	181
-10	183	189
-9	259	247
-8	145	-156
-7	46	-39
-6	128	-117
-5	991	936
-3	86	64
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-1	770	-723
0	88	91
1	945	890
2	424	404
3	458	-440
4	267	257
5	395	-369
6	54	-49
7	105	108
8	94	-92
9	106	-105
10	85	-80
11	151	-139
H= 4, K= 3		
-14	70	56
-12	57	62
-11	93	-87
-10	73	-74
-9	361	-362
-8	470	-453
-7	238	228
-6	519	-513
-5	469	448
-4	248	209
-3	131	-127
-2	333	-279
-1	402	379
0	472	420
1	168	184
2	103	104
3	399	-392
4	143	-132
5	98	91
6	208	207
7	151	155
H= 4, K= 4		
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-13	98	-86
-12	257	255
-11	74	-70
-10	180	-188
-9	386	370
-8	225	214
-7	243	220
-6	417	403
-4	970	-902
-3	319	310
-2	212	-199
-1	425	-392
0	189	130
1	443	-443
2	443	-460
4	187	-176
5	244	239
6	165	169
8	119	-116
H= 4, K= 5		
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-11	101	-93
-10	151	-151
-9	236	-224
-8	167	171
-7	240	238
-6	321	-315
-5	216	-207
-4	180	-180
-3	43	-51
-2	1005	966
-1	434	517
0	215	206
1	456	451
2	308	-294
3	69	-81
4	103	103
5	131	124
6	280	-278
7	173	175
8	249	-241
9	180	-181
10	48	54
H= 4, K= 6		
-11	164	-165
-10	49	-84
-8	218	218
-7	293	280
-5	526	-521
-4	59	56
-3	290	-258
-2	223	208
-1	193	136
0	432	-407
1	651	-628
2	136	-93
3	174	166
4	215	-214
5	534	526
7	94	83
8	125	118
10	80	76
H= 4, K= 7		
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-9	234	233
-8	206	198
-7	254	-248
-5	304	-292
-4	70	-59
-3	177	179
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-1	450	-444
1	148	146
2	141	-145
3	409	400
5	181	-179
6	287	-291
8	121	-128
H= 4, K= 8		
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-9	115	-110
-8	68	-66
-7	118	-112
-6	134	-138
-4	563	551
-3	144	-137
-2	248	235
0	213	-214
2	198	202
3	86	81
5	64	50
6	77	-86
7	118	105
8	139	138
H= 4, K= 9		
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-9	157	158
-8	212	-218
-6	117	120
-5	168	-160
-4	173	170
-3	199	-190
-2	330	-315
-1	143	-138
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1	167	-167
2	212	214
3	53	58
4	118	-122
7	124	-126
8	125	115
H= 4, K= 10		
-10	86	-85
-9	72	-77
-8	140	-136
-7	56	-65
-6	159	161
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-1	82	-83
0	342	328
1	294	243
2	156	-158
3	102	-92
5	271	-265
6	93	91
H= 4, K= 11		
-8	130	-128
-7	113	120
-5	57	55
-4	77	-70
-3	304	-309
-2	298	-289
-1	221	221
2	81	81
3	154	-154
5	152	159
H= 4, K= 12		
-6	140	150
-4	123	-118
-3	138	132
-2	125	-125
-1	76	71
0	133	135
2	162	-169
H= 4, K= 13		
-2	95	93
-1	68	74
0	76	-76
H= 5, K= 0		
-14	61	-73
-12	84	93
8	297	304
10	217	217
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-8	137	-144
-6	197	189
-4	117	-108
-2	290	-330
0	729	-698
4	585	578
H= 5, K= 1		
-13	109	106
-12	142	145
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-9	55	49
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-7	111	-103
-6	60	45
-4	196	-183
-3	258	258
-2	323	-343
-1	323	347
0	339	-357
1	96	109
3	181	169
4	52	-54
5	377	-380
6	129	-126
7	299	-315
8	86	87
H= 5, K= 2		
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-11	162	-175
-9	336	-337
-8	163	167
-6	102	114
-5	140	141
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-2	292	-278
-1	363	372
0	279	-279
1	257	263
3	131	132
4	65	68
4	153	152
8	141	138
9	67	67
11	86	-93
H= 5, K= 3		
-14	132	133
-12	230	209
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-9	81	-85
-8	407	401
-7	300	-295
-6	60	-53
-5	159	-165
-4	466	-467
-2	460	-478
-1	97	103
0	277	-269
1	286	268
2	110	107
3	95	-95
6	152	-159
7	101	101
8	81	95
9	62	-68
H= 5, K= 4		
-12	119	-123
-11	172	-175
-10	194	194
-9	179	-187
-8	303	290
-6	112	118
-5	229	228
-4	137	152
-3	355	355
-2	62	67
-1	145	154
0	149	158
1	107	-110
2	108	-106
3	193	-172
4	326	-334
6	89	83
8	108	-117
9	92	108
10	130	-137
H= 5, K= 5		
-14	67	-73
-10	210	218
-9	195	-187
-6	174	-175
-5	135	-146
-4	141	-155
-3	248	-262
-1	149	-164
0	212	210
2	90	97
4	341	354
5	245	256
6	136	137
7	143	134
8	209	-204
H= 5, K= 6		
-13	70	-56
-5	139	139
-2	281	288
0	149	157
1	362	-380
3	352	-353
4	102	-113
6	97	-107
7	84	-83
8	139	-138
H= 5, K= 7		
-13	62	-65
-11	128	117
-10	119	117
-9	62	54
-8	154	-155
-7	185	192
-6	72	-70
-5	87	80
-2	127	130
-1	123	-129
0	151	156
1	189	-194
2	130	131
4	224	218
5	103	101
6	159	153
8	124	-128
H= 5, K= 8		
-12	115	116
-10	105	-111
-9	58	60
-8	295	-300
-7	56	-49
-6	145	-154
-5	71	-75
-4	84	-57
-2	65	48
-1	62	-66
0	54	42
1	60	-63
2	117	122
4	138	135
5	166	-163
6	97	-96
H= 5, K= 9		
-9	134	135
-7	175	177
-5	200	235
-3	113	117
-1	69	-62
0	114	-111
1	125	-126
2	101	96
4	122	-124
5	86	-84
H= 5, K= 10		
-8	80	-91
-6	128	-124
-4	101	-98
-2	75	-78
1	150	151
2	97	105
3	139	141
H= 5, K= 11		
-6	66	67
-2	84	-52
0	79	-76
2	96	-101
3	72	-79
4	111	-122
H= 5, K= 12		
0	100	111
1	76	62
H= 6, K= 0		
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-8	530	-519
-6	330	302
-4	401	360
-2	540	-495
0	346	330
2	306	-292
4	467	-556
6	158	-153
8	97	95
10	91	84
H= 6, K= 1		
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-13	126	-119
-12	55	60
-11	181	178
-10	215	-210
-9	51	39
-8	222	217
-7	163	-156
-6	695	663
-5	204	176
-4	272	-282
-3	434	-398
-2	109	-100
-1	244	232
0	706	742
1	163	166
2	508	-510
3	170	158
4	315	-311
5	349	349
6	190	187
8	51	-98
9	125	-122
H= 6, K= 2		
-13	268	271
-12	59	-57
-11	91	-83
-10	79	-76
-9	362	-355
-8	115	-112
-6	94	78
-5	418	-399
-4	211	187
-3	840	-815
-2	159	142
-1	378	364
1	399	378
2	206	-196
3	176	-169
4	121	-123
5	352	354
6	83	-93
9	62	-39
10	82	70
H= 6, K= 3		
-14	68	-61
-13	102	97
-12	75	-53
-10	121	-114
-9	241	232
-8	362	370
-7	205	198
-6	605	589
-5	554	-516
-4	62	37
-2	186	-183
0	466	445
1	331	-322
2	512	-503
3	154	155
4	307	-305
6	209	208
7	106	-107
10	97	-69
H= 6, K= 4		
-13	115	102
-12	161	-157
-10	274	-268
-8	378	381
-7	386	-368
-6	311	-315
-5	272	-256
-4	229	-217
-3	156	128
-2	979	945
0	155	-142
1	283	279
2	93	-106
3	104	96
4	396	394
5	72	-57
7	74	-71
8	107	-101
H= 6, K= 5		
-13	76	78
-12	184	-194
-11	87	-84
-10	86	99
-9	143	160
-8	119	114
-7	208	170
-6	172	-170
-5	381	-353
-4	262	260
-3	256	240
-2	188	-191
-1	228	-214
0	452	-433
1	332	-319
2	103	104
3	75	-67
4	69	66
5	147	-151
6	74	68
8	169	167
9	75	62
H= 6, K= 6		
-11	105	102
-9	180	187
-7	153	-150
-6	65	-67
-3	332	319
-1	89	85
0	74	68
1	122	128
2	270	264
3	75	74
5	280	-276
6	79	79
7	141	-134
H= 6, K= 7		
-12	68	-63
-11	90	100
-9	242	-251
-8	107	-102
-7	128	-125
-6	135	-136
-5	540	530
-1	231	-232
0	368	-372
1	359	371
2	65	70
3	176	-174
4	206	205
5	195	-192
7	137	145
8	138	135
H= 6, K= 8		
-12	164	170
-11	118	110
-10	191	166
-9	67	-65
-8	173	-181
-7	137	135
-6	230	204
-3	109	-99
-2	701	-692
-1	207	200
2	216	212
4	197	-204
7	66	-59
H= 6, K= 9		
-11	63	-57
-10	84	-98
-9	223	-221
-8	76	-54
-7	76	-77
-6	216	214
-5	200	202
-4	192	-186
-1	208	254
-3	73	71
-2	113	107
0	124	120
1	141	143
2	135	-144
H= 6, K= 10		
-10	81	81
-9	172	-164
-8	146	147
-7	152	145
-4	149	-150
-3	268	-269
-2	115	-108
-1	86	95
0	120	-126
1	63	-51
2	93	-92
3	61	-59
4	52	91
5	145	145
H= 6, K= 11		
-6	84	87
-5	159	-164
-3	86	91
-2	67	44
-1	232	232
0	202	206
1	177	-183
H= 6, K= 12		
-3	97	102
-2	153	144
-1	196	-199
H= 7, K= 0		
-14	127	135
-12	206	207
-8	120	115
-6	61	-60
-2	151	153
0	183	208
2	351	342
4	102	-100
6	332	-338
6	62	-56
H= 7, K= 1		
-11	287	-285
-10	169	-166
-8	93	-99
-7	61	46
-6	88	-82
-5	87	93
-4	137	141
-3	56	46
-2	274	277
-1	368	-372
0	346	343
1	243	-242
3	105	101
5	53	-43
6	69	74
7	223	228
9	165	167
H= 7, K= 2		
-11	130	131
-10	71	76
-9	343	350
-8	84	-89
-7	132	142
-6	123	-125
-5	91	-76
-4	77	-74
-3	444	-462
-2	72	63
-1	311	-338
0	176	178
1	92	-102
2	65	74
3	280	-274
5	103	104
6	61	-63
7	51	79
8	98	-101
H= 7, K= 3		
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-12	112	-116
-11	106	115
-10	294	-303
-9	59	67
-8	66	-99
-7	78	83
-6	94	-105
-5	168	171
-4	182	184
-3	57	52
-2	392	406
-1	172	173
0	398	401
1	162	-151
2	51	-38
3	107	-106
4	56	-47
5	127	126
7	83	-83
H= 7, K= 4		
-11	103	105
-9	195	204
-8	156	-163
-7	95	130
-6	137	-135
-5	109	-116
-4	199	-195
-3	201	-204
-2	76	-87
-1	172	-177
1	65	-63
2	89	-98
3	65	62
4	195	201
5	108	112
6	134	142
H= 7, K= 5		
-12	62	70
-11	138	148
-10	74	-65
-8	98	-98
-7	109	106
-5	90	65
-3	121	119
-2	143	150
-1	198	211
0	66	-63
2	134	-143
3	130	-131
4	61	-62
6	168	-168
7	160	-169
H= 7, K= 6		
-11	56	40
-9	106	-105
-3	53	44
-2	55	-75
0	117	-131
3	319	321
5	150	156
6	106	106
H= 7, K= 7		
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-10	74	80
-9	59	-65
-8	84	-80
-7	70	-79
-5	108	-108
-2	63	-53
0	127	-126
1	206	209
2	66	-76
4	147	-149
5	88	-92
6	85	-99
H= 7, K= 8		
-11	74	-70
-8	138	136
-6	195	193
-4	180	187
0	57	-62
1	95	131
4	176	-171
H= 7, K= 9		
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-7	141	-146
-5	167	-191
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-1	64	-60
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1	147	145
2	71	64
3	149	160
4	94	-97
H= 7, K= 10		
-6	179	196
-4	122	123
-3	64	-57
2	119	-119
3	157	-152
H= 7, K= 11		
-7	79	-81
-5	61	-65
-1	62	70
H= 8, K= 0		
-14	65	-61
-10	161	-153
-8	124	116
-6	452	444
-4	542	-518
0	348	347
2	143	-154
4	350	341
6	244	245
H= 8, K= 1		
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-13	172	170
-11	63	-57
-10	97	-101
-9	238	-239
-8	147	145
-7	222	226
-6	360	-350
-5	64	-61
-4	317	-310
-3	404	-430
-1	642	625
1	117	-112
3	435	-429
4	167	183
5	73	-55
7	85	-77
H= 8, K= 2		
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-10	60	67
-9	359	362
-8	80	65
-7	320	308
-6	131	124
-5	94	-85
-4	93	-87
-3	565	543
-2	225	-217
-1	184	178
1	427	-425
2	52	39
3	134	-125
4	55	59
6	88	89
7	132	-132
H= 8, K= 3		
-14	159	150
-13	87	-90
-7	341	-325
-6	430	-416
-5	54	57
-4	186	-183
-3	366	349
-2	242	-243
-1	172	-170
0	62	60
1	189	161
2	226	222
3	169	179
4	159	155
5	61	-94
H= 8, K= 4		
-13	77	-65
-11	57	-50
-10	235	230
-8	134	129
-6	291	-284
-5	411	410
-4	224	228
-3	124	144
-2	47	-49
-1	175	-179
0	391	-347
3	151	-156
4	149	-147
5	80	-67
6	86	-92
H= 8, K= 5		
-13	93	-102
-12	188	186
-10	145	144
-9	83	87
-8	218	-227
-7	269	-270
-6	157	163
-5	64	-54
-3	510	491
-2	353	-363
-1	269	-268
0	298	293
1	110	102
2	173	176
3	325	328
4	133	-138
H= 8, K= 6		
-12	80	59
-11	66	69
-10	71	-70
-9	367	-353
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-3	368	-300
-2	67	69
-1	170	-166
1	197	203
2	77	-77
3	106	-118
4	65	-83
H= 8, K= 7		
-12	122	116
-10	87	72
-9	103	-135
-7	257	258
-6	148	153
-5	276	-273
-2	113	110
0	83	77
3	61	-89
5	88	99
6	97	-84
H= 8, K= 8		
-10	226	-227
-9	68	-78
-8	144	-132
-6	142	146
-5	86	-93
-2	176	164
0	270	270
1	105	-109
3	82	73
H= 8, K= 9		
-9	120	105
-8	154	157
-7	208	200
-6	119	-120
-4	126	-123
-3	281	-275
-2	250	256
0	177	-172
1	153	-148
3	94	-99
H= 8, K= 10		
-8	145	-144
-7	78	83
-6	136	-139
-5	213	-206
-4	181	182
-3	164	166
-2	113	118
-1	160	168
1	146	-156
2	106	102
H= 8, K= 11		
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-1	153	-146
H= 9, K= 0		
-12	186	-203
-10	118	-122
-8	104	-130
-6	54	69
-2	165	-172
2	143	-145
4	83	-98
6	170	168
H= 9, K= 1		
-12	82	98
-10	83	82
-9	236	242
-8	133	136
-8	69	85
-5	112	-117
-4	196	-203
-2	79	-86
-1	175	176
0	92	-93
1	133	133
2	228	-236
4	89	91
H= 9, K= 2		
-9	175	-184
-8	60	-54
-7	199	-201
-6	82	78
-4	82	89
-3	213	211
-2	70	67
-1	356	375
2	137	-134
-13	72	-74
3	77	65
5	126	128
7	117	-115
H= 9, K= 3		
-14	62	-60
-12	60	65
-11	79	88
-10	94	108
-9	69	-69
-8	249	259
-6	116	118
-5	120	-129
-4	197	-202
-3	124	-122
-2	192	-207
-1	85	-81
0	120	-149
2	238	-250
3	149	148
5	67	74
H= 9, K= 4		
-12	68	79
-9	63	-74
-8	74	90
-7	193	-203
-6	75	63
-4	146	147
-3	114	125
-2	242	250
-1	127	143
0	80	-70
1	160	174
6	155	-159
H= 9, K= 5		
-12	71	-61
-9	156	-153
-8	153	139
-6	93	95
-4	104	99
-3	97	-96
-2	146	-147
-1	175	-181
0	80	-56
2	137	106
3	76	83
6	66	49
H= 9, K= 6		
-3	66	-56
1	69	67
3	50	-76
5	186	-171
H= 9, K= 7		
-5	142	150
-4	103	105
2	152	156
3	174	-174
H= 9, K= 8		
-6	94	-101
-4	164	-170
-3	72	-69
-2	167	-178
0	97	100
2	67	74
3	69	-76
H= 9, K= 9		
-9	73	67
-7	107	99
-5	109	109
-3	153	165
1	71	-74
2	63	-53
H= 9, K= 10		
-6	94	-93
-4	115	-112
H= 10, K= 0		
-14	187	167
-10	81	75
-8	135	122
-6	323	-305
-4	90	-96
-2	294	295
0	163	-163
2	100	-102
6	210	-206
H= 10, K= 1		
-13	98	-91
-12	118	-113
-11	148	-146
-10	166	188
-9	63	84
-7	137	134
-6	81	-87
-4	428	439
0	93	-99
1	124	-120
3	137	136
4	75	-77
5	143	130
H= 10, K= 2		
-14	64	46
-13	74	-79
-12	89	81
-11	64	64
-9	209	212
-8	163	-156
-7	470	-459
-5	114	-110
-2	56	-45
-1	315	-321
0	169	160
1	106	106
3	209	204
4	66	-63
5	61	48
H= 10, K= 3		
-12	128	-119
-11	129	132
-10	157	159
-9	71	-86
-8	126	-125
-7	81	72
-5	264	263
-4	656	643
-3	225	-223
-1	164	-160
0	78	-86
3	175	-167
4	150	-145
5	64	-40
H= 10, K= 4		
-13	63	48
-12	88	107
-11	127	111
-10	87	-80
-9	88	73
-8	263	-254
-7	92	-95
-1	130	-129
-6	133	144
-5	56	-55
-4	94	89
-3	180	-171
-2	166	-174
0	257	251
1	67	67
2	183	185
3	116	114
5	100	90
H= 10, K= 5		
-11	87	84
-10	264	-271
-8	59	-44
-6	160	164
-4	92	-96
-2	108	104
-1	173	-166
0	93	98
2	158	-165
3	91	-91
H= 10, K= 6		
-12	70	-54
-10	82	-77
-8	139	142
-7	286	293
-5	76	-78
-3	124	-123
-2	65	65
-1	170	179
0	76	-74
H= 10, K= 7		
-11	121	-121
-10	183	-184
-5	257	-259
-4	233	-238
-3	196	202
-2	90	94
-1	119	118
2	66	-64
3	77	77
H= 10, K= 8		
-8	284	281
-6	103	-101
-5	60	57
-4	60	-51
-3	73	82
-2	95	107
0	236	-232
H= 10, K= 9		
-7	108	-106
-6	73	-75
-4	102	105
-3	100	90
-1	168	171
0	103	-98
H= 11, K= 0		
-10	195	202
-8	71	65
-2	61	54
-6	61	59
H= 11, K= 1		
-11	127	-127
-10	135	-137
-9	74	-89
-8	66	-55
-2	96	103
-1	105	133
-5	62	-65
1	80	-88
2	118	115
4	101	115
H= 11, K= 2		
-10	97	98
-9	139	146
-7	81	88
-2	92	-83
-1	185	-182
-4	68	-72
1	216	-226
H= 11, K= 3		
-12	80	86
-10	131	-136
-8	131	-135
-6	84	-76
-5	136	139
-3	137	134
-2	76	76
0	119	126
1	61	58
2	161	157
3	93	-67
4	98	99
H= 11, K= 4		
-10	86	-80
-9	92	103
-7	69	86
-6	60	-65
-5	125	129
-4	96	-91
-2	185	-190
-1	115	-126
1	88	-93
2	113	111
H= 11, K= 5		
-11	64	59
-8	71	-79
-6	100	-101
-5	104	120
-4	69	-71
-3	70	78
0	138	149
1	81	71
H= 11, K= 6		
-11	65	-73
-10	94	-93
-7	66	76
-5	134	145
1	62	47
2	62	-59
H= 11, K= 7		
-10	103	111
-6	81	-72
-5	99	-98
-3	104	-106
H= 11, K= 8		
-4	107	112
-2	165	171
H= 12, K= 0		
-12	183	-185
-10	142	154
-8	197	-191
-4	355	356
0	161	-154
2	135	131
H= 12, K= 1		
-11	216	213
-9	89	81
-8	111	-110
-7	255	-259
-2	169	-189
-1	84	-89
H= 12, K= 2		
-11	75	80
-9	107	-109
-6	138	125
-5	288	288
-1	129	121
0	75	-55
2	63	-53
H= 12, K= 3		
-10	104	100
-9	122	-113
-8	63	-75
-7	114	121
-5	135	-136
-4	73	-56
-2	302	-295
-1	168	170
H= 12, K= 4		
-11	96	-91
-10	117	-121
-8	164	168
-7	88	-73
-6	82	82
-5	60	55
-4	151	-154
-3	157	148
-1	108	101
H= 12, K= 5		
-10	81	83
-8	235	236
-7	97	96
-6	125	-127
-5	65	-61
-2	75	71
0	121	-122
H= 12, K= 6		
-7	63	-65
-6	93	-94
-5	236	-241
-3	177	184
H= 12, K= 7		
-5	78	71
-2	141	136
H= 13, K= 0		
-8	118	-121
-6	164	-168
H= 13, K= 1		
-9	71	80
-7	112	104
-4	65	76
-1	93	-69
0	62	-62
H= 13, K= 2		
-11	88	-86
-8	92	-85
-7	72	-79
H= 13, K= 3		
-6	93	90
-4	78	91
-3	119	-124
-2	78	-62
H= 13, K= 4		
-7	65	-72
-6	108	107
-5	81	-93
H= 13, K= 5		
-6	70	75
-3	79	-64
H= 14, K= 0		
-8	104	-117
-4	127	-116
H= 14, K= 1		
-5	159	161
H= 14, K= 2		
-7	131	129
-5	120	-117
-4	80	-78
H= 14, K= 3		
-5	84	-86

instrument instability. The weights, w, used in least-squares were the reciprocal squares of $\sigma(F_o)$. Of the 2932 unique reflections measured, those for which $2\theta < 52°$, 1972 had intensities larger than three times their standard deviations. These were regarded as the observed reflections, and all of the remaining reflections were excluded from further calculations. The intensities were corrected for Lorentz and polarization effects; the contribution of the monochromator crystal was calculated assuming that it was half perfect and half mosaic in character. An absorption correction was not made ($\mu = 6{\cdot}8$ cm^{-1}); the transmission coefficients are estimated to be 0·89 and to show a total range of less than 0·02.

The atomic scattering factors of Doyle & Turner (1968) were used for C^0, N^0, O^0, S^0 and Cl^-. The latter two were modified to account for the real part of the anomalous dispersion correction (*International Tables for X-ray Crystallography*, 1968). Atomic scattering factors used for hydrogen were those of Stewart, Davidson & Simpson (1965).

Crystal data

Bis-[2-(*N*,*N*-dimethylamino)ethyl]disulfide dihydrochloride, or 2,2′-dithiobis-(*N*,*N*-dimethyl)ethylamine dihydrochloride, $C_8H_{22}N_2S_2Cl_2$, monoclinic, $P2_1/c$, $a = 11{\cdot}646$ (2), $b = 11{\cdot}065$ (2), $c = 12{\cdot}370$ (3) Å, $\beta = 112{\cdot}40$ (2)°. Figures in parentheses are estimated standard deviations in the units of the least significant digit given for the corresponding parameter. $V = 1473{\cdot}8$ Å^3; $M = 281{\cdot}3$, $D_{\rm obs}$ (flotation) = 1·26 g cm^{-3}; $D_{\rm calc} = 1{\cdot}268$ g cm^{-3}; $Z = 4$; $F(000) = 600$.

Structure determination

The position of one of the chloride ions was indicated on a three-dimensional Patterson function. Positions

Table 2. *Fractional atomic coordinates and thermal parameters with estimated standard deviations* (*all* × 10^5)

See Fig. 1 for the identities of the atoms. The e.s.d. is in the units of the least significant digit given for the corresponding parameter. The temperature factor is $\exp\,[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)]$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cl(1)	12884 (8)	12952 (7)	15253 (7)	894 (7)	553 (6)	825 (6)	−9 (10)	755 (11)	225 (10)
Cl(2)	64540 (11)	13375 (10)	17620 (11)	772 (10)	543 (8)	938 (10)	−34 (14)	787 (15)	122 (14)
S(3)	70228 (8)	44200 (8)	7268 (7)	713 (7)	832 (7)	609 (6)	−209 (11)	462 (10)	42 (10)
S(4)	74493 (8)	61236 (7)	3769 (7)	808 (7)	588 (6)	629 (6)	48 (11)	248 (10)	−203 (10)
N(5)	97847 (24)	40638 (21)	27562 (25)	587 (21)	494 (19)	862 (23)	38 (30)	660 (35)	−20 (32)
N(6)	50926 (23)	59795 (22)	−30734 (21)	611 (21)	538 (19)	585 (18)	104 (30)	484 (31)	7 (28)
C(7)	83149 (38)	35382 (30)	6892 (31)	1157 (37)	613 (26)	733 (26)	165 (49)	669 (51)	−117 (41)
C(8)	95823 (35)	39673 (30)	14896 (33)	913 (32)	634 (25)	917 (29)	364 (45)	1006 (50)	168 (42)
C(9)	94450 (33)	29580 (31)	32424 (32)	774 (29)	689 (28)	860 (28)	−246 (45)	521 (46)	169 (45)
C(10)	110987 (33)	43853 (39)	34480 (37)	632 (29)	989 (34)	1062 (35)	−309 (53)	474 (50)	34 (56)
C(11)	69189 (29)	61523 (30)	−12050 (27)	658 (26)	715 (25)	635 (22)	−144 (42)	415 (38)	65 (39)
C(12)	55257 (28)	60527 (27)	−17717 (25)	645 (25)	610 (23)	570 (21)	127 (38)	470 (36)	114 (35)
C(13)	53962 (39)	70789 (34)	−35986 (33)	1130 (39)	751 (29)	766 (27)	−233 (53)	560 (54)	384 (46)
C(14)	37467 (33)	57247 (40)	−36046 (31)	651 (29)	1203 (39)	720 (27)	−227 (53)	357 (44)	−80 (51)

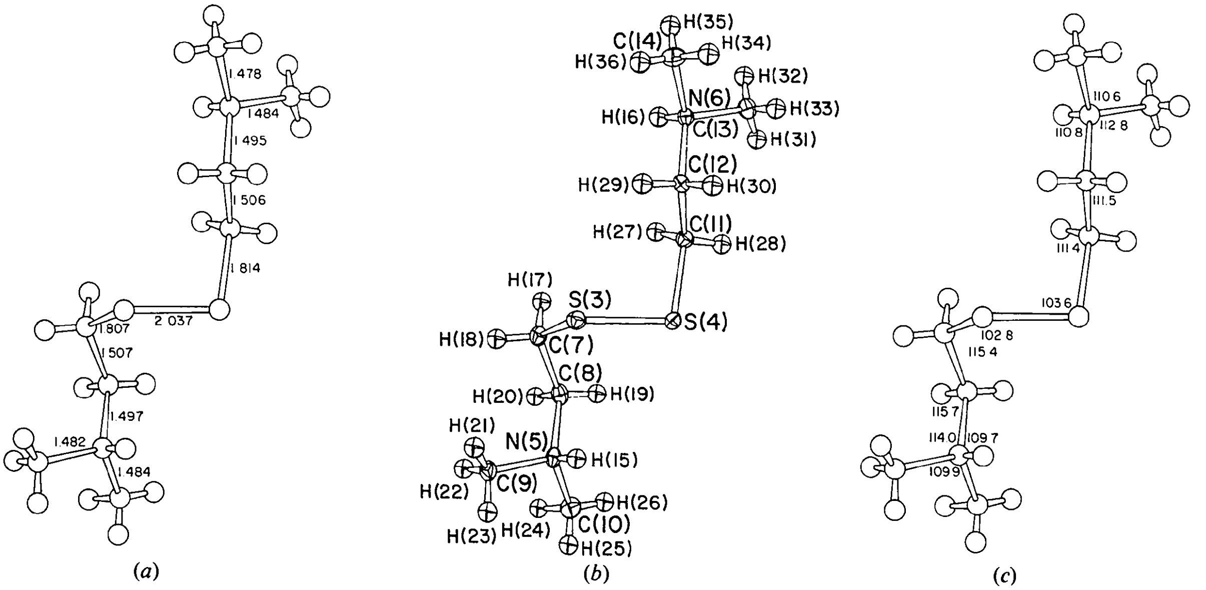

Fig. 1. The bis-[2-(*N*,*N*-dimethylammonium)ethyl]disulfide ion. Ellipsoids of 15% probability are used (Johnson, 1965) in the central figure, and bond lengths and angles are given on the side drawings.

for all other non-hydrogen atoms were found by successive Fourier refinements using the fast-Fourier program *ALFF* (Hubbard, Quicksall & Jacobson, 1971).

The structure model was refined to $R_1=0\cdot104$ $\{R_1=(\sum|F_o-|F_c||)/\sum F_o$ and $R_2=[\sum w(F_o-|F_c|)^2/\sum wF_o]^{1/2}\}$. The introduction of anisotropic thermal parameters for all non-hydrogen atoms followed by least-squares refinement reduced R_1 to 0·065. The program used (*UCLALS*4) was that of Gantzel, Sparks & Trueblood which minimizes $\sum w(\Delta F)^2$.

The positions of all of the hydrogen atoms were carefully estimated from a difference Fourier synthesis prepared with a grid spacing of less than 0·2 Å. The peaks were clear and sharp; subsequent geometry calculations indicated that they were all reasonably placed and that their further refinement would not be likely to generate significant changes in their parameters. These were included in the structure factor calculations with an arbitrarily assigned isotropic temperature factor of 4·0 Å^2.

Table 3. *Fractional atomic coordinates* ($\times 10^3$) *for hydrogen atoms*

See Fig. 1 for the identities of the atoms.

	x	*y*	*z*
H(15)	941	456	281
H(16)	541	544	−322
H(17)	822	278	88
H(18)	836	344	−8
H(19)	977	466	133
H(20)	1013	339	144
H(21)	863	278	300
H(22)	967	308	398
H(23)	992	238	316
H(24)	1133	445	414
H(25)	1164	367	344
H(26)	1133	500	320
H(27)	728	555	−148
H(28)	727	684	−133
H(29)	523	539	−153
H(30)	516	664	−159
H(31)	508	762	−345
H(32)	523	695	−430
H(33)	609	731	−328
H(34)	333	637	−344
H(35)	355	559	−422
H(36)	367	516	−336

Full-matrix least-squares refinement of the positional and anisotropic thermal parameters of all non-hydrogen atoms converged to give the final error indices, $R_1=0\cdot043$ and $R_2=0\cdot046$. The 'goodness-of-fit', $\{[\sum w(F_o-|F_c|)^2]/(m-s)\}^{1/2}$, is 0·97. The number of observations is m, and s, the number of parameters, is 127. In the final cycle of least squares, all shifts were less than 1% of their standard deviations, except for some involving C(14) thermal parameters, which were 2% of their e.s.d.'s. The largest peak on the final difference Fourier function had a density of 0·2 e Å^{-3}; the standard deviation of the electron density is calculated to be 0·09 e Å^{-3}.

The overdetermination ratio, excluding the unrefined hydrogen parameters, is 15·5. Table 1 is a tabulation of the observed and calculated structure factors ($\times$ 10), and the final parameters of the non-hydrogen atoms are listed in Table 2. Positional parameters for the hydrogen atoms are given in Table 3. Standard deviations were calculated from the correlation matrix ignoring standard deviations in cell parameters.

Discussion

Bond lengths and angles involving only non-hydrogen atoms are listed in Table 4 and also in Fig. 1, where the numbering of the atoms is indicated. Average bond lengths and angles involving hydrogen atoms are given in Table 5.

Table 5. *Average bond distances and bond angles involving hydrogen*

	Average value	Number averaged	Range
N—H	0·74 Å	2	0·72–0·76 Å
C—H	0·87	20	0·71–1·01
H···Cl	2·34	2	2·32–2·35
S—C–H	109·6°	4	102–116°
N—C–H	110·2	16	103–120
C—N–H	107·6	6	106–109
C—C–H	110·7	8	106–117
H—C–H	106·9	16	97–117

The S–S bond length of 2·037 (1) Å is significantly shorter than the mean value of 2·075 (6) Å found in

Table 4. *Bond lengths and bond angles for non-hydrogen atoms*

Estimated standard deviations, in parentheses, are in the units of the least significant digit given for the corresponding parameter.

S(3)—S(4)	2·0374 (12) Å	S(3)—S(4)—C(11)	103·59 (11)°
S(3)—C(7)	1·807 (4)	S(4)—S(3)—C(7)	102·77 (12)
S(4)—C(11)	1·814 (3)	S(3)—C(7)—C(8)	115·43 (25)
C(7)—C(8)	1·507 (5)	S(4)—C(11)–C(12)	111·36 (22)
C(11)–C(12)	1·506 (4)	N(5)—C(8)—C(7)	115·75 (26)
N(5)—C(8)	1·497 (5)	N(6)—C(12)–C(11)	111·51 (25)
N(6)—C(12)	1·495 (4)	C(8)—N(5)—C(9)	113·95 (26)
N(5)—C(9)	1·482 (4)	C(8)—N(5)—C(10)	109·70 (26)
N(5)—C(10)	1·484 (4)	C(9)—N(5)—C(10)	109·87 (26)
N(6)—C(13)	1·484 (4)	C(12)–N(6)—C(13)	112·84 (25)
N(6)—C(14)	1·478 (4)	C(12)–N(6)—C(14)	110·77 (24)
		C(13)–N(6)—C(14)	110·56 (26)

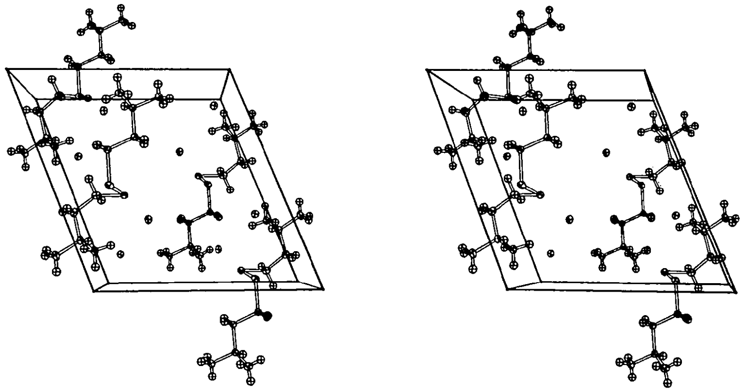

Fig. 2. A stereoview (Johnson, 1965) illustrating the packing of the ions in the crystal. Ellipsoids of 15% probability are used.

the dimeric Cu(I) complex (Ottersen, Warner & Seff, 1973*a*) of the molecule. The uncomplexed value observed in this work, 2·037 (1) Å, lies close to the middle of the range usually found for disulfide bonds (Hordvik, 1966; Lee & Bryant, 1970). Using Pauling's (1960) single-bond value of 2·08 Å and his bond-order relationship (see Lee & Bryant, 1972), this bond has a double-bond character of 13%. It is somewhat longer than the S–S bond length of 2·02 Å found in dibenzyl disulfide (Lee & Bryant, 1969*b*; van Dijk & Visser, 1971).

The central part of the molecule, C(7)–S(3)–S(4)–C(11), adopts a skewed non-planar configuration with a dihedral angle of 82·39 (16)°. According to Hordvik (1966), a relationship exists between bond length and dihedral angle in disulfides. When the dihedral angle is 90°, the repulsions between the lone pairs of electrons on the sulfur atoms are minimized and favorable π overlap is at a maximum. Hordvik (1966) relates the observed dihedral angle of 82° with an S–S bond length of 2·04 Å, in excellent agreement with the current result. Contrary to this, the dihedral angle in dicinnamyl disulfide (Lee & Bryant, 1971), 66·4°, does not lead by Hordvik's (1966) relationship to the observed S–S bond length of 2·01 Å.

The mean S–C bond length of 1·811 Å is in good agreement with the average distance of 1·815 Å found for eleven compounds by Cox & Jeffrey (1951).

The molecule exists as a dication, $[-N(CH_3)_2H^+]_2$, with hydrogen bonds from N(5) to Cl(1) (whose coordinates are related to those given in Table 2 by the symmetry operation: $1-x, 0{\cdot}5+y, 0{\cdot}5-z$) of 3·049 Å, and from N(6) to Cl(2) ($x, 0{\cdot}5-y, -0{\cdot}5+z$) of 3·062 Å. There is a short (3·548 Å) contact between S(4) and Cl(1) ($1-x, 0{\cdot}5+y, 0{\cdot}5-z$), possibly indicating a weak charge-transfer interaction. [The sum of the van der Waals radii for S and Cl is $1{\cdot}85+1{\cdot}80=3{\cdot}65$ Å and that of S and Cl^- is 3·66 Å (Pauling, 1960)]. Otherwise the packing seems to be dominated by van der Waals contacts. A view of the packing is given in Fig. 2.

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