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Crystal Structure of Bis-pyrimidyl-2,2′-disulfide Dihydrate

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The crystal structure of bis-pyrimidyl-2,2′-disulfide dihydrate has been derived from 1690 observed reflections measured by counter methods. The final value of R is 0.030 and the estimated standard deviations in distance between non-hydrogen atoms are 0.001 – 0.003 Å. The C–S–S–C dihedral angle is 82.5°, and both sulfur atoms lie approximately in the plane of both pyrimidine rings. The S–S bond length is 2.016 Å. The bonds C2–N1 and C2–N3 appear to be significantly different.

Continuing our investigations of the structure of pyrimidine derivatives[1] we have carried out an X-ray analysis of bis-pyrimidyl-2,2′-disulfide. The structure of this compound is of interest because it is a derivative of the 2-*thiol* rather than the usual 2-*thio* tautomer of pyrimidine. It would also appear to be of interest to compare the structure of this simple disulfide with those of other aromatic disulfides, such as diphenyl disulfide.[2]

EXPERIMENTAL. STRUCTURE ANALYSIS

The compound was prepared by solving 2-thiopyrimidine in hot ammoniacal water. Oxidation takes place and after some time a crystalline precipitate was formed, which was shown by the structure analysis to be the dihydrate of the disulfide. The crystals disintegrate in air and were sealed off in glass capillaries containing some water during the X-ray exposures.

Unit cell dimensions are $a = 11.835(4)$ Å, $b = 6.944(1)$ Å, $c = 18.655(3)$ Å, and $\beta = 128.94(1)°$. The space group is $P2_1/c$, with four disulfide and eight water molecules in the unit cell. The density was measured by flotation in KI-solutions and found to be 1.44 g/cm.^{3} The calculated value is also 1.44 g/cm.^{3}

The intensity measurements were carried out on a crystal of dimensions $0.24 \times 0.36 \times 0.40$ mm using a Syntex automatic diffractometer and Mo$K\alpha$ radiation ($\lambda = 0.71069$ Å, graphite monochromator). The $\omega/2\theta$ scan technique (rates 1 – 12°/min) was used and 1690 reflections with 2θ less than 55° were recorded with measurable intensities, using a cut-off limit of 2σ. No breakdown of the crystal could be observed. Corrections for absorption or secondary extinction were not made.

The structure was solved by Patterson methods and refined by block diagonal least squares calculations to $R = 0.030$ ($R_w = 0.032$). The weighting scheme was based on standard deviations from counter statistics and an assumed 2 % fluctuation in diffractometer stability. Anisotropic temperature factors were applied to the non-hydrogen atoms, common isotropic ones to the hydrogen atoms. Observed and calculated structure factors are given in Table 1, positional and thermal parameters in Table 2 and corresponding bond lengths and angles in Table 3 and Fig. 1.

Table 1. Observed and calculated structure factors.

h	k	l	F_o	F_c
0	0	2	173	166
0	0	4	1107	-1057
0	0	6	666	670
0	0	8	561	-565
0	0	12	238	235
0	0	14	192	-188
0	0	16	49	51
0	0	18	76	78
0	1	1	433	410
0	1	2	492	-459
0	1	3	635	-615
0	1	4	1540	1650
0	1	5	155	-160
0	1	6	505	-499
0	1	7	131	139
0	1	8	671	-667
0	1	10	365	372
0	1	11	60	-62
0	1	12	96	-83
0	1	16	147	149
0	2	0	1623	-1727
0	2	1	456	-450
0	2	2	330	-313
0	2	3	93	-91
0	2	4	197	-193
0	2	5	136	133
0	2	6	295	-294
0	2	7	256	-266
0	2	8	634	622
0	2	11	41	45
0	2	12	191	-195
0	2	14	159	157
0	2	16	62	-62
0	2	18	93	-91
0	3	1	174	176
0	3	2	199	196
0	3	3	164	156
0	3	4	216	-217
0	3	5	253	-251
0	3	6	119	117
0	3	7	117	113
0	3	8	58	51
0	3	9	211	213
0	3	10	143	-145
0	3	11	76	-79
0	3	12	163	161
0	3	13	58	47
0	3	14	35	29
0	3	15	96	89
0	3	16	124	-122
0	3	17	45	-41
0	4	0	373	361
0	4	1	316	314
0	4	2	169	166
0	4	3	152	156
0	4	4	111	116
0	4	5	45	45
0	4	6	111	109
0	4	7	129	130
0	4	8	211	-210
0	4	9	175	-178
0	4	10	156	-168
0	4	13	144	143
0	4	15	43	-52
0	4	16	55	51
0	5	1	49	-48
0	5	2	196	-195
0	5	3	153	-162
0	5	4	86	85
0	5	5	239	239
0	5	6	74	76
0	5	8	66	-61
0	5	9	136	-138
0	5	13	39	-35
0	6	0	194	-196
0	6	1	242	-241
0	6	2	67	-66
0	6	3	113	-115
0	6	5	56	68
0	6	7	66	-61
0	6	9	126	127
0	6	10	55	52
0	7	1	40	-43
0	7	2	32	-36
0	7	3	54	59
0	7	4	62	-61
0	7	5	42	-47
0	7	6	48	55
0	8	0	78	79
0	8	5	41	56
0	8	7	53	57
0	8	9	97	-84
1	0	-18	84	84
1	0	-14	199	-188
1	0	-12	176	195
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1	0	2	935	887
1	0	4	228	221
1	0	6	259	271
1	0	8	236	-244
1	0	10	233	-205
1	0	14	201	-208
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1	1	-13	254	-260
1	1	-9	312	-316
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1	1	-7	260	261
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1	1	-5	530	491
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1	1	0	370	351
1	1	1	317	301
1	1	2	696	-674
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1	1	7	332	-305
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1	1	14	69	-70
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1	2	-16	70	-69
1	2	-14	183	183
1	2	-13	131	129
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1	2	-9	168	-166
1	2	-8	252	259
1	2	-7	249	245
1	2	-6	154	-161
1	2	-5	181	-187
1	2	-4	29	17
1	2	-3	230	234
1	2	-2	146	-134
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1	2	3	72	75
1	2	4	126	126
1	2	5	249	253
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1	3	-15	76	72
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1	3	-10	135	134
1	3	-9	186	191
1	3	-8	139	-136
1	3	-7	222	-226
1	3	-6	412	-419
1	3	-5	62	-63
1	3	-4	273	273
1	3	-3	469	476
1	3	-2	413	-412
1	3	-1	330	-324
1	3	0	227	-226
1	3	1	79	-81
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1	3	4	251	-256
1	3	5	335	-333
1	3	6	64	66
1	3	7	126	127
1	3	8	215	217
1	3	9	292	299
1	3	10	333	-335
1	3	11	145	-139
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1	3	13	47	-56
1	3	14	117	113
1	3	15	71	68
1	3	16	47	-39
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1	4	-4	179	184
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1	4	1	513	512
1	4	2	227	228
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2	1	-5	92	-88
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2	1	-1	114	-103
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2	1	5	153	150
2	1	6	96	-85
2	1	8	264	-244
2	1	9	125	-127
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2	1	11	82	88
2	1	14	73	-78
2	1	16	119	117
2	2	-17	72	-77
2	2	-12	111	113
2	2	-11	94	-93
2	2	-10	191	177
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2	3	-12	62	-57
2	3	-10	79	-77
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2	4	-6	253	-251
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2	4	14	37	-37
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2	5	-14	95	-91
2	5	-13	54	54
2	5	-12	70	69
2	5	-11	45	46
2	5	-9	66	-65
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2	5	0	191	184
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2	8	1	80	64
2	8	2	111	113
2	8	3	69	75
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3	0	-14	97	99
3	0	-12	165	-162
3	0	-10	439	-402
3	0	-8	283	-268
3	0	-6	133	-138
3	0	-4	69	66
3	0	-2	74	-70
3	0	0	230	224
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3	0	10	261	-260
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3	4	7	153	154
3	4	9	97	-94
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3	4	11	63	-68
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3	5	-14	65	-65
3	5	-10	147	152
3	5	-9	75	77
3	5	-8	154	-155
3	5	-7	132	134
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3	5	-4	93	83
3	5	-2	138	-145
3	5	-1	145	147
3	5	0	133	125
3	5	1	150	153
3	5	2	77	78
3	5	3	84	-81
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3	5	12	70	65
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3	6	-12	111	111
3	6	-11	41	-47
3	6	-9	46	52
3	6	-6	192	194
3	6	-5	53	53
3	6	-4	157	-161
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3	6	1	76	-78
3	6	2	229	-231
3	6	5	67	68
3	6	6	68	69
3	6	7	65	-65
3	6	8	88	-89
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3	7	-14	48	41
3	7	-10	78	-79
3	7	-8	146	145
3	7	-7	52	-68
3	7	-5	46	-48
3	7	-4	79	-74
3	7	-2	158	155
3	7	0	131	-136
3	7	1	67	-72
3	7	2	98	-100
3	7	4	118	118
3	7	6	54	-56
3	7	7	38	-38
3	8	-6	120	-119
3	8	-5	34	-31
3	8	-4	87	84
3	8	-2	47	42
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8	3	-14	154	-160
8	3	-13	212	-210
8	3	-12	93	-91
8	3	-10	958	955
8	3	-9	88	102
8	3	-7	116	-111
8	3	-6	159	-157
8	3	-5	162	-151
8	3	-4	174	143
8	3	1	64	70
8	3	2	53	52
8	3	4	56	-52
8	3	5	76	-81
8	4	-21	72	-63
8	4	-20	44	-49
8	4	-17	133	124
8	4	-16	107	94
8	4	-15	115	-115
8	4	-14	226	-209
8	4	-13	133	-127
8	4	-12	71	-62
8	4	-11	119	121
8	4	-10	161	159
8	4	-6	150	151
8	4	-5	125	126
8	4	-2	156	-152
8	4	1	55	59
8	4	2	39	-41
8	4	3	73	-76
8	5	-20	59	-65
8	5	-19	152	159
8	5	-16	68	55
8	5	-13	36	47
8	5	-12	145	142
8	5	-11	16	-24
8	5	-10	135	-139
8	5	-8	119	-117
8	5	-7	226	226
8	5	-6	33	-35
8	5	-3	65	-70
8	5	-1	113	116
8	5	2	82	-77
8	6	-16	71	-65
8	6	-15	33	33
8	6	-14	95	95
8	6	-11	65	-52
8	6	-10	95	-91
8	6	-9	67	64
8	6	-8	90	94
8	6	-6	33	-34
8	6	-5	116	-119
8	6	-4	36	-41
8	6	-3	42	37
8	6	-2	98	88
8	6	1	46	-47
8	7	-13	45	-52
8	7	-12	114	-109
8	7	-10	49	33
8	7	-8	57	69
8	7	-7	59	-67
8	7	-6	52	-42
8	7	-4	56	73
9	0	-24	132	-94
9	0	-20	136	125
9	0	-18	142	-149
9	0	-16	135	-139
9	0	-12	73	97
9	0	-8	56	-91
9	0	-6	79	77
9	0	-4	99	-87
9	0	0	122	115
9	0	2	113	-117
9	0	4	63	-63
9	1	-24	67	-64
9	1	-20	57	-53
9	1	-19	32	44
9	1	-18	59	56
9	1	-17	73	-79
9	1	-16	159	164
9	1	-15	83	-82
9	1	-13	166	166
9	1	-9	61	-52
9	1	-7	223	224
9	1	-6	269	-242
9	1	-5	86	-92
9	1	-4	165	-155
9	1	-3	137	-132
9	1	-1	96	95
9	1	4	53	55
9	1	6	49	-57
9	2	-21	67	64
9	2	-20	66	63
9	2	-18	77	76
9	2	-15	214	215
9	2	-14	77	76
9	2	-12	67	68
9	2	-11	292	-292
9	2	-10	111	-123
9	2	-9	214	212
9	2	-8	55	69
9	2	-7	139	139
9	2	-5	165	-165
9	2	-3	101	106
9	2	-2	75	-76
9	2	-1	49	-47
9	2	2	131	134
9	2	3	111	105
9	3	-21	59	61
9	3	-20	46	46
9	3	-19	75	-77
9	3	-18	63	-69
9	3	-17	91	-36
9	3	-16	66	-66
9	3	-15	41	49
9	3	-13	71	-74
9	3	-12	244	-249
9	3	-9	292	277
9	3	-7	201	-197
9	3	-6	151	166
9	3	-5	111	120
9	3	-4	101	96
9	3	-3	67	66
9	3	-1	131	-131
9	3	1	38	47
9	3	4	51	-61
9	3	5	59	-77
9	4	-18	46	-42
9	4	-16	41	41
9	4	-15	49	-56
9	4	-13	88	-131
9	4	-12	46	-42
9	4	-9	46	-52
9	4	-5	114	127
9	4	-4	111	112
9	4	-3	136	-162
9	4	-2	99	-96
9	4	1	93	92
9	4	3	38	-36
9	5	-16	121	132
9	5	-15	69	52
9	5	-14	56	51
9	5	-13	89	97
9	5	-12	115	113
9	5	-10	63	-59
9	5	-9	97	-94
9	5	-8	132	-121
9	5	-7	116	119
9	5	-5	45	49
9	5	-4	139	-131
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9	5	0	42	47
9	6	-16	45	-79
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9	6	-14	56	61
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9	7	-12	117	-112
9	7	-11	57	-54
9	7	-9	33	34
10	0	-22	112	-111
10	0	-20	111	107
10	0	-18	76	-73
10	0	-16	161	-166
10	0	-14	136	137
10	0	-8	246	235
10	0	-2	59	65
10	0	0	132	-127
10	0	4	97	91
10	1	-24	121	-119
10	1	-23	69	-64
10	1	-22	51	63
10	1	-20	53	54
10	1	-19	71	77
10	1	-18	125	-126
10	1	-16	131	128
10	1	-12	113	-109
10	1	-10	123	124
10	1	-8	165	-136
10	1	-6	237	-262
10	1	-2	102	-104
10	1	-1	69	-71
10	1	0	113	-112
10	1	2	39	45
10	1	4	49	51
10	2	-21	56	55
10	2	-20	57	-64
10	2	-19	175	174
10	2	-16	59	72
10	2	-15	133	132
10	2	-14	227	-236
10	2	-8	206	-206
10	2	-5	139	116
10	2	-4	39	46
10	2	-3	39	35
10	2	-2	45	-55
10	2	-1	97	-88
10	3	-17	53	51
10	3	-15	137	-141
10	3	-14	37	36
10	3	-13	127	126
10	3	-10	125	-127
10	3	-9	90	-85
10	3	-7	114	-137
10	3	-6	113	115

Table 1. Continued.

h	k	l	F_o	F_c
10	3	-5	125	128
10	3	-4	131	120
10	3	-3	83	90
10	3	-2	56	67
10	4	-19	97	-100
10	4	-17	36	-29
10	4	-15	159	-157
10	4	-11	65	66
10	4	-10	173	179
10	4	-9	129	-132
10	4	-6	35	-34
10	4	-5	71	71
10	4	-4	59	61
10	4	-3	60	-66
10	5	-16	69	69
10	5	-15	65	-66
10	5	-13	69	76
10	5	-12	89	89
10	5	-8	72	-73
10	5	-7	65	66
10	5	-6	94	87
10	5	-4	67	-70
10	5	-2	101	-101
10	6	-15	60	59
10	6	-13	69	69
10	6	-10	106	-110
10	6	-5	39	-36
11	0	-24	50	55
11	0	-22	99	-102
11	0	-20	136	137
11	0	-18	44	-63
11	0	-16	186	197
11	0	-12	144	-144
11	0	-8	273	278
11	0	-6	48	46
11	0	-2	73	77
11	0	0	72	-76
11	0	2	73	-81
11	1	-22	45	47
11	1	-20	77	81
11	1	-19	126	-122
11	1	-18	225	228
11	1	-16	107	108
11	1	-12	107	-108
11	1	-10	117	109
11	1	-8	116	-129
11	1	-6	157	-162
11	1	-5	96	-99
11	1	-4	73	75
11	1	-3	36	31
11	1	0	51	-54
11	1	1	52	-54
11	2	-22	72	77
11	2	-20	158	-156
11	2	-18	31	26
11	2	-16	172	173
11	2	-15	97	96
11	2	-14	133	-149
11	2	-13	82	-93
11	2	-10	71	71
11	2	-9	95	95
11	2	-8	135	-134
11	2	-7	96	-91
11	2	-6	73	-83
11	2	-5	67	-59
11	2	0	49	52
12	0	-8	261	261
12	0	-6	127	-127
12	0	-2	98	89
12	1	-20	193	179
12	1	-19	147	-150
12	1	-17	38	-46
12	1	-16	106	104
12	1	-15	56	61
12	1	-14	224	217
12	1	-13	64	66
12	1	-12	141	-139
12	1	-11	67	-63
12	1	-10	105	103
12	1	-8	65	61
12	1	-7	54	47
12	1	-6	178	-191
12	1	-4	69	69
12	2	-22	89	94
12	2	-21	96	57
12	2	-20	77	-74
12	2	-19	69	-64
12	2	-18	74	-76
12	2	-17	59	-56
12	2	-16	155	156
12	2	-15	48	32
12	2	-14	76	-74
12	2	-10	72	79
12	2	-9	35	31
12	2	-8	131	-127
12	2	-7	49	-44
12	2	-5	59	-66
12	2	-4	64	64
12	2	-2	67	-65
11	3	-21	48	33
11	3	-20	107	-107
11	3	-19	67	-67
11	3	-18	99	95
11	3	-17	111	111
11	3	-16	123	-126
11	3	-14	117	-122
11	3	-13	113	-104
11	3	-12	116	117
11	3	-11	59	56
11	3	-10	51	-56
11	3	-8	107	109
11	3	-5	92	83
11	4	-21	73	-75
11	4	-20	59	56
11	4	-16	110	-124
11	4	-15	97	-93
11	4	-14	55	53
11	4	-13	63	39
11	4	-9	87	-80
11	4	-8	134	135
11	4	-5	66	61
11	4	-2	41	46
11	5	-16	63	66
11	5	-10	56	53
11	5	-6	87	-84
11	5	-5	73	-72
11	6	-12	41	35
12	0	-22	95	-106
12	0	-20	95	79
12	0	-16	175	-190
12	0	-14	217	246
12	0	-10	242	-247
12	3	-20	95	-90
12	3	-18	46	-65
12	3	-16	115	-105
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12	4	-7	77	75
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12	5	-9	60	51
13	0	-19	48	47
13	0	-14	146	-147
13	0	-12	53	61
13	0	-10	90	-87
13	0	-8	57	51
13	1	-20	141	147
13	1	-19	43	45
13	1	-17	54	-63
13	1	-16	116	111
13	1	-15	72	75
13	1	-12	101	-109
13	1	-10	69	59
13	1	-8	66	56
13	1	-6	114	-104
13	2	-22	65	66
13	2	-18	92	-86
13	2	-16	128	123
13	2	-15	68	67
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13	2	-10	122	119
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13	2	-6	76	71
13	3	-20	79	-85
13	3	-19	48	-76
13	3	-18	43	50
13	3	-17	61	61
13	3	-16	147	-165
13	3	-13	106	-106
13	3	-12	56	62
13	3	-11	65	70
13	3	-9	63	-55
13	3	-6	71	73
13	3	-5	58	59
13	4	-18	49	65
13	4	-15	75	-80
13	4	-13	47	41
13	4	-10	99	-95
14	0	-16	76	-79
14	0	-10	61	66
14	0	-4	56	51
14	1	-12	98	-89
14	2	-18	53	-67
14	2	-16	51	57
14	2	-15	90	53
14	2	-10	59	62
14	3	-16	70	77
15	0	-10	57	49

Table 2. Positional (× 10^5 for non-hydrogens, × 10^4 for hydrogens) and thermal (× 10^5 for non-hydrogens) parameters with estimated standard deviations. The anisotropic temperature factor is $\exp[-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{23}hl+B_{23}kl)]$.

Atom	x	y	z	B_{11}(B)	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ring A									
S	−2459	69050	32958	1022	3059	400	864	751	−2
	5	8	3	6	15	2	16	6	10
N1	−4284	71924	45977	1105	1811	600	311	1199	155
	15	20	10	19	37	9	43	23	29
C2	5323	74542	44556	981	1475	439	617	868	222
	17	23	11	21	37	9	48	24	33
N3	18956	80218	50536	1004	2450	490	−96	913	−110
	15	23	10	19	42	8	45	22	30
C4	23345	83735	59019	1208	2529	507	−25	851	−213
	20	30	13	25	53	11	63	29	40
C5	14526	81567	61339	1696	2413	499	634	1284	114
	22	30	13	31	51	11	68	32	40
C6	630	75539	54557	1663	1911	668	729	1705	444
	21	27	13	29	45	12	61	34	39
H4	3325	8725	6333	5.3					
	19	26	12	0.2					
H5	1827	8386	6732	5.3					
	18	28	12	0.2					
H6	−608	7355	5551	5.3					
	20	26	13	0.2					
Ring B									
S	13482	74597	32197	1836	2232	615	1390	1700	790
	6	7	3	8	12	3	17	9	11
N1	34725	54857	35648	1310	2411	543	152	1291	65
	15	23	10	21	42	9	49	25	31
C2	24534	53549	36630	982	1914	346	71	777	−86
	17	25	10	21	42	9	50	24	31
N3	22281	39382	40284	1128	1999	451	139	948	210
	14	21	9	19	37	8	44	22	28

Table 2. Continued.

C4	31308	24325	43125	1545	2126	553	368	1176	336
	22	28	13	29	49	11	63	32	40
C5	42027	23844	42280	1578	2700	655	1587	1318	423
	23	31	14	31	58	13	71	35	44
C6	43474	39617	38571	1300	3282	654	657	1365	86
	21	33	14	27	63	13	68	33	47
H4	3005	1378	4581	5.3					
	19	28	12	0.2					
H5	4806	1332	4409	5.3					
	18	28	12	0.2					
H6	5081	4116	3808	5.3					
	18	28	12	0.2					
Water molecules									
W1	46851	87353	32207	1524	2638	893	−107	1651	14
	14	21	10	20	40	10	47	26	33
W2	35345	14793	18496	1186	5313	1168	124	1266	2195
	15	29	13	20	65	14	60	29	49
H1W1	4255	7982	3278	6.9					
	23	33	15	0.3					
H2W1	5180	8077	3185	6.9					
	22	33	14	0.3					
H1W2	2695	1469	1418	6.9					
	22	32	14	0.3					
H2W2	3720	758	2186	6.9					
	21	32	14	0.3					

Table 3. Bond lengths (Å) and angles (°). Estimated standard deviations in parentheses. Corrected bond lengths in brackets {}.

	Ring A		Ring B			Ring A	Ring B
S–S	2.016(1)						
C2–S	1.781(2)	{1.788}	1.781(2)	{1.789}	C2–S–S	104.6(1)	104.9(1)
N1–C2	1.332(2)	{1.342}	1.332(2)	{1.342}	N1–C2–S	111.0(1)	110.1(1)
C2–N3	1.319(2)	{1.329}	1.316(2)	{1.325}	N3–C2–S	121.0(1)	121.7(1)
N3–C4	1.342(2)	{1.347}	1.343(2)	{1.350}	N1–C2–N3	128.0(2)	128.2(2)
C4–C5	1.366(3)	{1.374}	1.373(3)	{1.381}	C2–N3–C4	114.4(1)	114.6(1)
C5–C6	1.366(3)	{1.374}	1.363(3)	{1.370}	N3–C4–C5	123.1(2)	122.5(2)
C6–N1	1.339(2)	{1.345}	1.334(3)	{1.341}	C4–C5–C6	117.1(2)	117.3(2)
C4–H	0.95(2)		0.95(2)		C5–C6–N1	122.0(2)	122.2(2)
C5–H	0.92(2)		0.92(2)		C6–N1–C2	115.4(2)	115.3(2)
C6–H	0.93(2)		0.93(2)		N3–C4–H4	114(1)	117(1)
					C5–C4–H4	123(1)	120(1)
					C4–C5–H5	119(1)	122(1)
W1–H1	0.78(2)		N1$_A$···W2	2.928(2)	C6–C5–H5	124(1)	120(1)
W1–H2	0.78(2)		N1$_B$···W1	2.953(2)	C5–C6–H6	123(1)	124(1)
W2–H1	0.79(2)		W1···W2	2.766(2)	N1–C6–H6	115(1)	114(1)
W2–H2	0.72(2)		W1···W2′	2.694(2)			

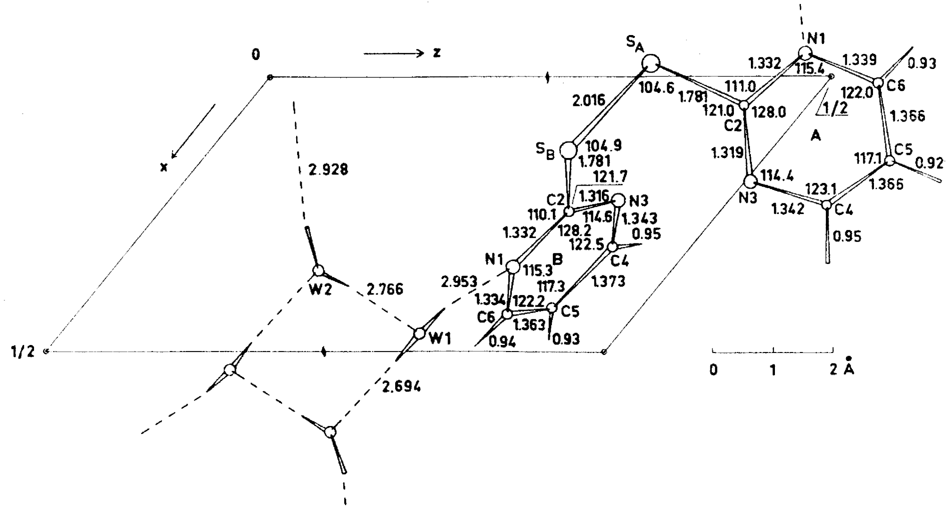

Fig. 1. The *b* projection of one molecule.

The effect of libration was examined by assuming each pyrimidine ring and its sulfur atom to be a rigid body. The root mean square differences between atomic vibration tensor components calculated from Table 1 and those derived from the rigid body model were 0.0019 Å^2 and 0.0015 Å^2, respectively. Corrected bond lengths are included in Table 2.

The atomic scattering factors were those of Hanson *et al.*[3] for non-hydrogen atoms and of Stewart *et al.*[4] for hydrogen atoms. All programs used are described in Ref. 5.

DISCUSSION

Because of the inherent uncertainties in the correction procedure, the discussion is based on uncorrected values of bond lengths and angles.

In Table 4 deviations from least square planes are given for the two pyrimidine residues A and B. It is seen that one ring (A) is almost exactly planar, whereas some of the atoms of the other are significantly displaced from planarity. The angle between the two planes is 80.7°. The hydrogen atoms lie in the ring planes to within 0.06 Å. Those at C4 and C6 appear to be bent away from that at C5.

Table 4. Deviation of atoms from least squares planes defined by the ring atoms and one sulfur atom.

	Ring A	Ring B	Ring A	Ring B
N1	0.004 Å	0.017 Å	C5 −0.003 Å	−0.024 Å
C2	0.005	0.009	C6 −0.003	0.001
N3	0.001	0.016	S_A −0.002	−0.220
C4	0.000	−0.002	S_B 0.052	−0.009

The dihedral angle C–S–S–C is 82.5°, which is within the range of 90 ± 10° usually found for organic disulfides. It has been pointed out,[6] that the values of the angle between the ring plane and the C–S–S plane fall into two general classes, being either *ca.* 0° or *ca.* 90°. The present compound clearly belongs to the former class, as the angles are 1.7° and 6.1° for rings A and B, respectively. This means that both sulfur atoms lie approximately in the plane of both pyrimidine rings. There would seem to be considerable strain in the molecule caused by steric repulsion between N3 and the sulfur atom of the other ring. This is shown by the great distortion of the external angles at C2, which differ by about 10°. The distances $N3_A \cdots S_B$ and $N3_B \cdots S_A$ are 3.08 Å and 3.11 Å, respectively, still somewhat shorter than the sum of van der Waals radii (about 3.3 Å). The length of the S–S bond is 2.016 Å, of the C–S bonds 1.781 Å. The S–S–C angles are 104.6° and 104.9°, respectively. These structural parameters are very similar to those found in other aromatic disulfides of this class, such as diphenyl disulfide[2] (2.03 Å, 1.80 Å, 106°), and 4,4′-di(thiouridine)[7] (2.022 Å, 1.79 Å, 104°) but different from those of the other class (2.074 Å, 1.75 Å, 101°).[6] The corresponding parameters in $(CH_3)_2S_2$ are 2.022 Å, 1.806 Å, and 104.4°, with a dihedral angle of 84° about S–S.[9]

Corresponding bond lengths and bond angles in the two pyrimidine rings agree very well. The angle N1–C2–N3 is large (128.2° and 128.0°), as is found in pyrimidine itself (128.2°),[8] but widely different from that in 2-*one* and 2-*thio* derivatives (near 120°). Each pyrimidine ring should be expected to have a plane of symmetry through C2···C5. Within the limits of error this is also found except that C2 – N3 is shorter than C2 – N1 by 0.013 Å in ring A and by 0.016 Å in ring B. A full matrix least squares refinement of the parameters of C2, N1, N3, and S did not give significant changes and the relevant correlation coefficients were small. The observed differences are therefore probably significant. They may be related either to the angular deformation at C2 or to a N3···S interaction. The effect has apparently not been observed in other structures of this class,[2,7] which, however, all are less accurately determined than the present one.

The water molecules are linked together in infinite spirals around the two-fold screw axes by nearly linear O–H···O hydrogen bonds of lengths 2.694 Å and 2.766 Å (Fig. 1). Each water molecule is also linked to N1 by an O–H···N bond of length 2.953 Å or 2.928 Å. The angles at the hydrogen atoms involved in hydrogen bonding are 167°, 178°, 164°, and 168° for H1W1, H2W1, H1W2, and H2W2, respectively.

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