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(Journal of the Korean Chemical Society)
Vol. 18, No. 2, 1974
Printed in Republic of Korea

Sulfaguanidine Monohydrate의 結晶 및 分子構造

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(1973. 12. 19 접수)

The Crystal and Molecular Structure of Sulfaguanidine Monohydrate

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(Received Dec. 19, 1973)

요 약. sulfaguanidine 1수화물의 결정 및 분자구조를 X-선 회절법으로 규명하였다. 결정은 일사측계에 속하며 단위세포내에는 4분자가 있으며 공간군은 $P2_1/c$이다. 단위세포상수는 a=7.57±0.03, b=5.44±0.02, c=24.76±0.06Å, β=91.0±0.2°이다. 결정구조는 패터슨 함수의 해석과 직접법을 병용하여 밝혀냈다. 원자좌표치는 최소자승법으로 정밀화하였으며 1542개의 독립적인 회절반점에 대한 최종 R값은 0.14이었다. sulfaguanidine분자의 guanidyl기의 질소원자는 2개의 다른 물분자를 매개로 하여 수소결합을 하므로써 다른 분자들의 sulfonyl기의 산소원자에 연결되어 있다. 수소결합 형성에 있어서 물분자는 donor와 acceptor의 역할을 겸하고 있으며 수소결합은 사면체의 방향을 하고 있다. 2차원적인 수소결합에 의하여 연결된 분자들은 (001)면에 평행한 분자층을 형성하고 있다.

Abstract. The crystal and molecular structure of sulfaguanidine monohydrate, $C_7H_{10}N_4O_2S \cdot H_2O$, was determined from visually estimated intensity data from Weissenberg photographs. The crystal data are monoclinic, space group $P2_1/c$ with four molecules in a unit cell of dimensions, a=7.57±0.03, b=5.44±0.02, c=24.76±0.06 Å, β=91.0±0.2°. The structure has been solved by an interpretation of a Patterson map and with a help of a direct procedure on a procejection. The parameters were refined isotropically by block-diagonal least-squares methods using 1542 observed independent reflections to give R=0.14. By hydrogen bonding a guanidyl nitrogen of a sulfaguanidine molecule is linked to the sulfonyl oxygens of the other molecules indirectly through two different water molecules. The role of water molecule is both a donor and an acceptor in hydrogen-bonding formation and these hydrogen bonds are tetrahedrally oriented. The hydrogen-bonding networks form infinite molecular layers parallel to (001) plane.

Introduction

As a part of a series of detailed structure investigation on sulfur-containing compounds which have biologically active groups by X-ray single-crystal diffraction, the crystal and molecular structure of sulfaguanidine monohydrate

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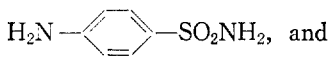
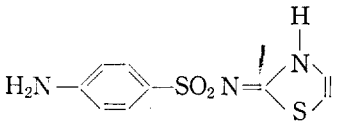
has been determined in this work. Among the sulfa drugs containing sulfanilamido group, the crsytal structures of sulfanilamide[1,2,3],



and sulfathiazole,[4,5]



, have been studied previously. In this experiment, we have investigated the conformation of the sulfaguanidine molecule, delocalization of π-electron on the guanidyl residue and the role of hydrated water molecule in the crystal-lattice environment.

Experimental

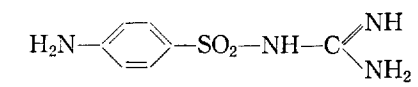
Prismatic crystals of sulfaguanidine monohydrate elongated along the *b* axis were obtained by slow evaporation from an aqueous acetone solution at room temperature.

Unit cell dimensions were determined by a least-squares refinement of 2θ values for 47 indepenpdent reflections measured on the *h0l* and *0kl* Weissenberg photographs calibrated with superimposed Al-lines using Cu-K_α radiation.

The density of single crystals, measured by flotation method in a mixture of benzene and carbon tetrachloride, was found to be consistent with 4 formular units in a unit cell.

The existence of water molecule in the single crystal was confirmed by the strong O—H stretching peak at 3400 cm^{-1} in IR-spectrum.

The crystal data are as follows; sulfaguanidine monohydrate,



monoclinic, $a=7.57\pm0.03$, $b=5.44\pm0.02$, $c=24.76\pm0.06$ Å, $\beta=91.0\pm0.2°$, $V=1019.1$ Å^3, M. W. $=232.26$, $Z=4$, $D_m=1.50$, $D_c=1.51$ g cm^{-3}, space group : $P2_1/c$ from systematic absence *h0l* for *l* odd, *0k0* for *k* odd.

Intensity data were collected from equi-inclination Weissenberg photographs taken with Cu-K_α radiation by use of the multiple-film method. The layers from *h0l* to *h4l* for the *b* axis and from *0kl* to *1kl* for the *a* axis were recorded. The approximate dimensions of the crystals used for gathering the intensity data for the *a* and *b* axes were $0.2\times0.2\times1.0$ and $0.2\times0.3\times0.7$ mm respectively. The relative intensities were measured by visual comparison with a calibrated intensity scale prepared from the same crystals with the X-ray beam under carefully controlled conditions.

The intensities were corrected for spot-shape, Lorentz and polarization effects, but not for extinction or absorption, and converted into the observed structure factors. Interlayer scaling constants were calculated from common equivalent reflections and the structure factors were placed approximately on an absolute scale following the Wilson method[6]. The final number of observed independent reflections was 1542. All calculations were performed on an IBM 1130 computer with a series of programs by Shiono (1968)[7].

Structure Determination and Refinement

After the structure factors were converted into the normalized structure factors, *E*, a three-dimensional sharpened Patterson function was computed using 985 normalized structure factors greater than $|E|=0.58$. The sulfur-sulfur Harker peak was easily identified, and trial positional parameters for the six atoms of S, O(1), O(2), C(1), N(1) and N(2) whose numberings are shown in *Fig.* 1 were obtained

from the inspection of the Patterson map.

Independently a Fourier projection of electron density on($0kl$) plane with 78 reflections whose phases were determined manually by an application of the direct method[8] showed the positions of some of fifteen atoms and these were consistent with those obtained from the Patterson map.

The initial R factor, $R=\sum||F_o|-|F_c||/\sum|F_o|$, based on these six atoms with all assumed $B=3.0$ Å^2 was 0.56 for 1005 reflections. The R factor based on sulfur coordinates alone was 0.70. Two cycles of isotropic block-diagonal least-squares refinement[9] reduced the R factor to 0.45. A three-dimensional Fourier synthesis, which was computed using the 1005 observed structure factors phased on the contribution of the six atoms, gave the clear picture of the molecule consistent with a chemically reasonable model. The refinement with isotropic temperature parameters was carried out successively and stopped after several cycles attaining a minimum R value of 0.14 for all the 1542 observed reflections. The function minimized was $w(|F_o|-|F_c|)^2$ and the weighting scheme w proposed by Cruickshank(1965)[10] was used throughout the refinement. The form of the function, w, was $(A+B|F_o|+C|F_o|^2)^{-1}$, where $A=2|F_{min}|=3.14$, $B=1.00$, $C=2/|F_{max}|=0.0164$. No allowance was made for the hydrogen atoms. Atomic scattering factor values were taken from the International Table for X-ray Crystallography[11]. The final positional and thermal parameters for the atoms are given in *Table* 1 with their estimated standard deviations as calculated from the least-squres refinement. Observed and calculated structure factors based on the parameters in *Table* 1 are listed in *Table* 2. The atoms are numbered according to *Fig.* 1.

Table 1. Final atomic coordinates and isotropic thermal parameters in sulfaguanidine monohydrate. The estimated standard deviations given in parentheses refer to the last decimal positions

	x	y	z	B
S	0.2588(5)	0.4835(7)	0.4041(1)	1.68(6)
O(1)	0.366(2)	0.301(2)	0.4306(5)	2.6(2)
O(2)	0.323(2)	0.727(2)	0.4098(5)	2.9(2)
O(3)	0.349(2)	0.168(2)	0.5410(5)	2.7(2)
N(1)	0.266(3)	0.273(4)	0.1671(9)	5.1(4)
N(2)	0.055(2)	0.493(2)	0.4226(5)	2.7(2)
N(3)	0.022(2)	0.065(3)	0.4261(7)	3.2(3)
N(4)	−0.215(2)	0.324(3)	0.4386(7)	3.2(3)
C(1)	0.257(2)	0.412(3)	0.3339(6)	2.4(3)
C(2)	0.338(3)	0.205(4)	0.3160(8)	2.8(3)
C(3)	0.338(3)	0.157(4)	0.2603(9)	3.5(4)
C(4)	0.261(3)	0.322(4)	0.2230(8)	3.3(3)
C(5)	0.175(3)	0.519(4)	0.2433(9)	3.8(4)
C(6)	0.175(3)	0.573(4)	0.2983(8)	3.0(3)
C(7)	−0.038(2)	0.291(3)	0.4302(6)	2.2(3)

Table 2. Observed and calculated structure factors. Columns are Index, $10|F_{obs}|$ and $10|F_{cal}|$

h=0 k=0			16	366	268	9	65	72	15	166	152	8	195	202	5	62	55	8	556	505
4	324	319	18	285	219	10	451	490	16	271	268	9	268	225	6	53	54	9	56	12
6	1058	1548	20	72	56	11	597	612	17	336	303	10	330	285	8	58	43	10	623	599
8	1157	1098	22	191	130	12	357	326	19	292	215	11	62	52	9	85	79	11	198	211
10	114	36	26	163	148	13	47	65	21	107	112	12	199	170	h=0 k=2			12	119	100
12	835	815	28	111	99	14	234	168	22	190	170	13	220	231	0	95	124	13	86	93
16	158	154	h=5 k=0			15	79	67	23	87	81	15	249	206	2	57	61	14	177	192
18	77	100	2	200	188	16	191	121	25	136	141	16	238	224	3	444	499	15	160	172
22	64	62	4	444	406	17	347	267	27	216	193	17	189	132	4	212	192	16	403	365
26	107	107	6	332	273	18	415	417	28	108	123	21	79	68	5	197	167	17	153	130
30	180	173	8	158	169	19	209	186	30	53	57	22	80	58	6	354	295	18	177	172
h=1 k=0			10	132	135	20	128	135	h=3 k=1			23	133	113	7	310	279	20	225	230
0	159	148	12	181	148	21	63	89	0	40	62	24	52	48	8	360	342	21	77	98
2	676	1037	14	417	350	22	107	110	1	428	412	25	87	97	9	65	43	22	81	91
4	94	115	16	107	107	23	59	42	2	310	287	26	45	34	10	356	377	23	79	88
6	747	800	18	381	282	24	139	127	3	120	134	h=6 k=1			11	143	146	25	56	59
8	1047	1076	20	196	159	25	133	138	4	272	236	1	341	307	12	163	152	26	82	123
10	558	496	22	119	131	26	115	127	5	91	109	2	90	90	13	106	98	28	43	58
12	746	699	24	98	96	27	154	175	6	328	291	4	162	143	14	104	119	29	49	57
14	774	710	26	123	119	28	70	78	7	367	328	5	307	259	16	275	228	h=3 k=2		
16	419	324	h=6 k=0			29	204	31	9	258	254	7	210	162	17	245	212	0	439	423
18	325	255	2	77	86	31	121	150	10	130	120	11	181	179	18	198	140	1	53	41
20	286	289	4	184	152	h=1 k=1			13	266	221	12	67	69	19	163	120	2	236	212
24	130	143	6	74	50	0	567	659	14	158	117	13	151	118	20	303	227	4	71	87
26	98	107	8	75	75	1	645	876	15	192	167	15	261	197	26	122	130	6	219	166
28	182	131	10	239	208	2	520	624	17	288	241	17	127	108	28	74	69	7	289	253
30	108	77	12	258	188	3	369	380	18	360	322	18	244	190	29	31	34	8	282	242
h=2 k=0			16	405	324	4	298	239	19	143	142	21	170	142	30	51	67	10	58	87
0	960	1364	22	154	144	5	297	268	22	76	53	23	95	94	h=1 k=2			11	241	198
2	191	186	h=7 k=0			6	336	278	23	171	150	h=7 k=1			0	499	565	12	303	219
6	210	168	0	103	83	7	339	357	24	88	86	0	149	139	1	183	171	13	109	120
8	352	299	2	275	190	8	76	93	25	166	157	1	85	96	2	506	534	14	134	119
10	104	80	4	297	247	9	367	340	26	107	92	3	270	236	3	122	129	15	110	103
12	98	87	6	199	156	10	297	204	29	162	148	4	59	54	4	124	103	16	113	102
14	325	271	8	180	151	11	93	59	h=4 k=1			6	131	121	5	117	133	17	88	114
16	438	400	10	209	163	12	191	104	0	287	243	7	194	160	6	348	263	18	302	268
18	433	359	14	205	135	13	383	314	1	264	208	9	237	189	7	418	379	21	41	40
20	298	275	18	156	130	14	242	211	2	147	156	10	132	114	8	389	281	22	79	124
22	419	357	20	73	46	15	236	167	3	298	255	12	184	131	9	52	36	23	35	26
26	373	313	h=8 k=0			16	59	54	4	92	90	13	276	231	10	51	45	24	196	199
28	267	231	0	177	140	17	374	281	5	237	224	14	60	72	11	252	222	25	33	31
30	111	72	2	138	120	18	267	179	6	139	113	15	217	175	12	315	297	27	35	30
h=3 k=0			6	69	66	19	292	260	8	191	154	16	53	74	14	262	196	28	101	128
0	854	959	10	114	71	20	132	103	10	166	138	17	123	80	16	87	80	h=4 k=2		
2	456	345	12	149	114	22	88	89	11	207	196	18	85	70	18	526	406	0	394	391
4	382	339	14	80	63	23	99	109	12	191	137	19	90	85	19	31	16	1	249	251
6	56	87	16	171	134	25	184	187	15	260	226	20	83	54	21	73	66	2	282	294
8	270	254	h=9 k=0			29	149	147	17	212	195	h=8 k=1			24	231	203	3	81	115
10	131	63	0	97	71	h=2 k=1			18	181	161	1	173	176	25	36	29	4	159	114
12	181	155	2	226	134	0	589	735	19	119	106	4	117	102	26	68	65	5	132	105
14	195	182	4	153	130	1	251	260	20	49	26	5	147	125	27	38	30	6	313	316
18	389	279	8	121	101	2	265	254	21	129	141	7	81	71	28	76	101	7	85	83
24	196	165	10	184	147	3	302	249	23	96	100	8	71	69	29	32	32	8	68	75
28	215	214	h=0 k=1			4	309	309	25	54	71	10	127	87	30	86	102	9	38	39
h=4 k=0			1	478	683	5	435	425	27	138	141	11	140	129	h=2 k=2			10	250	208
0	391	388	2	154	217	6	61	36	h=5 k=1			13	83	76	0	100	64	11	37	41
2	414	345	3	328	298	8	540	481	0	94	93	15	190	146	1	180	165	12	76	93
4	107	102	4	427	421	10	359	320	1	106	100	16	93	82	2	463	378	13	125	133
6	307	248	5	572	662	11	476	410	2	379	340	h=9 k=1			3	336	266	14	33	38
8	272	338	6	141	136	12	106	111	3	265	189	0	159	113	4	458	356	15	39	70
12	286	268	7	307	324	13	92	73	4	373	328	3	202	149	5	193	182	16	197	172
14	232	215		127	105	14	138	110	7	255	213	4	82	72	6	259	261	17	47	45
															7	218	202			

18	43	62
19	58	52
20	133	130
21	43	55
22	76	81
23	57	75
24	70	67
25	57	67
26	83	104
27	30	18
h=5 *k*=2		
0	42	39
1	79	75
2	391	380
3	111	128
4	373	324
7	61	62
8	338	318
10	323	288
11	91	99
12	57	60
13	69	98
14	259	243
15	117	119
17	79	79
18	29	51
20	55	69
21	101	95
22	48	55
24	107	120
25	71	54
h=6 *k*=2		
0	336	270
1	80	108
2	195	158
4	371	312
6	283	260
8	74	59
9	61	34
10	259	245
12	229	217
13	83	98
14	53	64
15	43	24
16	260	233
18	86	103
20	83	83
22	187	180
h=7 *k*=2		
0	76	156
1	161	136
2	97	94
3	163	149
4	39	35
5	142	120
6	39	18
7	140	123
8	109	91
9	41	31
11	105	101
12	151	140
13	85	85
14	269	218
16	116	112
17	44	36
18	135	135
h=8 *k*=2		
0	115	99
2	253	208
4	101	94
6	95	95
8	105	90
10	36	43
12	124	114
13	61	71
h=9 *k*=2		
0	192	135
1	53	45
2	106	120
4	145	125
6	36	37
h=0 *k*=3		
1	172	212
2	240	248
3	154	197
4	83	77
5	17	19
6	177	194
7	41	44
8	93	69
9	297	279
10	88	95
11	145	153
12	57	65
13	128	139
14	338	333
15	394	402
16	125	113
17	188	195
18	100	115
19	53	49
20	36	55
21	254	251
22	29	42
23	168	156
24	93	86
25	43	55
26	85	82
27	42	57
28	38	40
h=1 *k*=3		
0	107	54
1	170	183
2	164	178
3	107	125
4	274	248
5	89	63
6	219	215
7	387	357
8	44	52
9	199	173
10	131	135
11	92	88
12	188	195
13	376	350
14	22	39
15	51	49
16	193	173
17	46	49
18	54	68
19	221	177
20	47	44
21	17	41
22	88	92
23	203	174
24	27	38
25	61	81
27	68	87
28	77	74
h=2 *k*=3		
0	14	5
1	101	105
2	291	276
3	462	457
4	20	42
5	429	394
6	102	80
7	202	188
8	161	119
9	276	252
10	163	166
11	314	287
12	84	80
13	85	85
14	283	258
15	288	285
17	156	144
18	34	40
19	59	55
20	100	94
21	138	162
22	35	32
23	144	143
25	96	92
26	68	69
27	43	62
h=3 *k*=3		
0	54	65
2	189	159
3	309	330
4	63	51
5	42	37
6	23	41
7	171	136
8	100	102
9	142	130
10	63	39
11	179	162
12	102	100
13	275	237
15	138	137
16	56	26
17	157	164
18	88	80
19	144	154
20	35	36
21	61	46
22	30	28
23	121	123
24	26	16
25	42	63
26	25	8
h=4 *k*=3		
0	187	177
1	179	145
2	180	183
3	80	93
4	45	10
5	358	310
6	203	184
7	220	181
9	85	116
10	206	204
11	202	210
12	46	30
13	153	166
14	34	32
15	99	107
17	56	77
18	50	47
19	86	72
21	68	64
23	44	52
25	137	134
h=5 *k*=3		
0	192	201
1	73	77
2	133	118
3	495	505
4	51	93
5	197	207
8	193	170
9	210	196
12	96	84
13	54	36
14	54	49
15	116	104
16	40	40
17	119	119
18	58	51
19	101	107
21	26	11
22	57	32
23	87	86
h=6 *k*=3		
1	141	123
2	118	108
4	85	91
5	316	254
7	166	140
9	168	149
10	94	80
11	265	237
13	174	150
14	42	45
15	121	107
17	156	164
19	27	21
h=7 *k*=3		
1	63	54
3	219	198
4	37	21
5	42	45
6	123	89
7	50	57
9	122	112
10	35	14
11	70	56
12	27	28
13	111	82
14	50	57
15	32	38
16	31	32
17	117	117
h=8 *k*=3		
0	56	43
1	125	95
3	76	82
5	57	51
6	30	29
7	90	77
8	52	39
9	76	62
10	97	19
h=0 *k*=4		
0	337	388
2	133	134
3	88	104
4	236	285
5	283	314
6	333	346
7	157	174
8	56	52
9	19	4
10	279	271
11	120	128
12	274	284
13	306	321
14	40	47
16	119	143
17	95	109
18	103	103
19	161	137
20	60	82
21	35	39
22	121	98
23	141	120
24	26	38
25	81	105
26	72	103
h=1 *k*=4		
0	23	67
1	301	342
2	291	289
4	350	394
6	141	140
7	114	128
8	283	296
9	113	111
10	178	158
12	217	210
13	55	54
14	150	143
15	196	211
16	74	55
17	90	102
18	22	210
20	162	147
21	113	111
22	25	38
23	49	61
24	193	176
25	38	67
h=2 *k*=4		
0	151	169
1	33	14
2	104	124
3	189	264
4	99	116
5	149	162
6	162	116
7	105	116
7	105	116
8	59	46
9	200	187
10	223	208
11	89	70
12	186	169
13	274	283
14	151	156
15	151	159
16	179	186
17	59	67
18	70	82
19	31	42
20	102	123
21	101	110
22	131	147
23	34	42
24	21	15
25	10	12
h=3 *k*=4		
0	108	101
1	237	239
2	42	28
3	222	249
4	280	309
5	161	168
6	46	34
7	135	152
8	99	108
9	28	20
10	127	116
11	131	144
12	72	69
13	166	179
14	36	33
15	129	124
18	123	126
19	28	26
20	44	53
21	40	39
22	100	97
23	56	76
24	113	141
h=4 *k*=4		
0	156	178
1	70	72
2	212	196
3	158	184
5	63	60
6	161	156
7	49	45
8	63	75
9	37	36
10	87	101
11	30	6
12	121	128
13	114	109
14	39	33
15	32	46
16	108	113
17	28	34
18	72	81
19	56	70
20	36	43
22	86	81
h=5 *k*=4		
0	92	66
1	198	190
2	52	48
3	50	40
4	192	183
5	25	11
6	25	43
7	86	93
8	187	182
9	94	83
10	82	68
11	25	1
12	57	54
13	99	84
14	141	127
16	51	55
17	41	35
18	67	77
19	58	57
20	84	97
h=6 *k*=4		
0	121	118
1	28	24
2	79	87
3	151	154
4	71	68
5	53	49
6	174	151
7	28	11
8	43	52
9	76	78
10	144	120
11	45	46
12	83	75
14	23	17
15	120	38
h=7 *k*=4		
0	34	38
2	76	59
4	67	83
5	46	43
8	158	153
9	54	41

10	21	0	−2	602	584	−8	147	98	−16	223	245	h=7 k=1			−9	103	93	−4	190	204
11	77	14	−4	206	160	h=1 k=1			−17	242	237	−1	109	121	−10	100	88	−5	121	125
h=0 k=5			−6	755	807	−1	590	702	−18	136	166	−3	189	167	−11	33	26	−6	68	70
1	237	270	−8	579	547	−2	226	213	−19	140	165	−4	64	81	−12	51	33	−7	73	66
2	95	97	−10	73	50	−3	431	430	−20	97	104	−7	66	78	−13	101	106	−8	394	394
3	82	110	−12	790	778	−4	524	641	−23	117	170	−8	103	97	−14	188	206	−10	289	304
4	109	120	−14	214	205	−6	418	390	−24	98	139	−13	80	89	−15	263	275	−12	36	29
5	361	438	−16	189	196	−7	255	235	−25	76	85	−14	60	54	−16	41	21	−13	132	142
7	214	240	−18	500	451	−8	89	93	−27	72	107	−15	83	53	−17	136	143	−14	304	313
11	159	199	−20	372	288	−9	276	291	−29	133	156	−17	165	166	−18	87	110	−15	186	194
13	121	138	−22	319	233	−10	316	315	h=4 k=1			−18	123	116	−19	124	128	−18	45	38
17	120	110	−24	230	185	−11	118	121	−1	358	355	−21	63	80	−20	247	268	−19	43	51
h=1 k=5			−26	198	139	−13	418	454	−2	73	94	h=8 k=1			−21	128	141	−20	44	49
1	143	173	−28	87	92	−14	132	132	−3	184	432	−3	71	50	−23	40	53	−21	105	116
3	218	237	h=4 k=0			−15	148	147	−4	430	439	−5	90	119	−24	91	102	−23	60	74
4	188	200	−2	310	296	−17	195	219	−5	254	235	−7	80	70	−26	161	211	−25	28	26
5	101	118	−4	745	689	−18	67	39	−6	80	98	−9	117	116	h=3 k=2			h=6 k=2		
7	273	287	−6	132	98	−19	226	244	−7	41	44	−11	99	97	−1	79	36	−1	305	276
9	153	169	−8	327	270	−20	120	125	−8	42	40	−12	62	47	−2	489	478	−2	200	176
10	109	122	−10	516	501	−23	55	72	−9	190	197	−13	90	78	−3	88	92	−4	122	124
12	107	125	−12	381	314	−25	83	113	−10	160	153	−15	169	164	−4	189	149	−6	145	129
13	145	176	−14	254	213	−26	56	60	−11	250	225	h=9 k=1			−5	102	105	−7	127	103
15	142	136	−16	365	292	−27	81	74	−13	67	74	−1	215	165	−6	34	12	−8	47	33
17	67	81	−20	202	171	−28	55	69	−14	125	118	−3	113	90	−7	38	53	−9	139	147
19	214	170	−24	76	84	−29	140	134	−15	365	404	−4	93	82	−8	97	90	−10	123	159
21	70	61	−26	262	182	h=2 k=1			−16	106	95	−5	93	64	−9	89	67	−11	47	46
h=1 k=6			−28	47	41	−1	619	712	−18	67	76	−7	164	131	−12	139	133	−12	64	75
5	96	101	h=5 k=0			−2	251	226	−19	76	100	h=1 k=2			−13	39	54	−13	59	66
6	93	125	−2	72	42	−3	560	643	−21	228	273	−1	227	244	−14	260	284	−14	148	181
8	102	113	−4	164	161	−4	318	240	−25	59	111	−2	316	420	−15	111	117	−15	37	40
9	73	86	−6	188	132	−5	391	436	−27	93	148	−3	242	193	−16	226	212	−16	53	93
12	138	128	−8	109	90	−6	103	149	h=5 k=1			−4	174	120	−18	124	122	−20	98	109
14	99	102	−10	224	174	−7	326	319	−1	269	242	−5	403	368	−19	41	48	−22	65	96
15	62	50	−12	385	310	−8	243	212	−3	204	196	−6	352	276	−22	86	121	h=7 k=2		
h=1 k=0			−16	349	295	−9	66	87	−4	104	96	−7	165	163	−23	75	84	−1	109	106
−2	537	722	−18	379	288	−10	278	279	−5	254	247	−8	141	99	−24	151	168	−2	238	224
−4	309	312	−20	101	80	−11	342	398	−6	144	134	−9	29	21	−28	132	165	−3	90	67
−6	117	80	−22	227	167	−13	41	45	−7	237	249	−10	74	67	h=4 k=2			−4	42	31
−8	727	672	−24	176	134	−14	139	134	−8	59	68	−11	200	209	−1	165	130	−5	67	56
−10	668	553	−26	56	33	−15	172	204	−9	153	152	−12	34	16	−2	205	191	−6	222	201
−12	509	390	h=6 k=0			−16	128	129	−10	50	42	−13	139	156	−3	178	168	−8	210	201
−16	126	59	−4	425	351	−17	45	54	−11	121	127	−14	129	119	−4	385	390	−9	48	53
−18	132	126	−10	174	124	−18	56	70	−12	48	29	−15	42	59	−5	113	99	−12	200	197
−20	78	81	−16	235	150	−19	141	144	−13	332	358	−16	131	123	−6	303	313	−13	74	60
−22	416	300	−18	339	239	−20	48	41	−16	150	157	−18	173	180	−7	48	40	−14	136	153
−24	131	118	−24	130	117	−21	156	180	−17	179	212	−19	53	51	−8	69	73	−16	104	122
−28	253	213	h=7 k=0			−25	122	133	−18	91	73	−20	81	90	−9	47	40	−18	126	153
−30	110	97	−4	85	88	−26	78	60	−19	95	113	−21	70	45	−10	242	261	h=8 k=2		
h=2 k=0			−6	80	77	−27	120	152	−23	122	169	−23	46	55	−12	255	270	−1	145	121
−2	683	812	−8	133	81	h=3 k=1			−25	58	82	−24	97	98	−13	82	78	−2	67	79
−4	310	304	−10	118	97	−1	281	279	−26	73	81	−25	42	20	−14	196	203	−4	201	181
−6	89	19	−12	156	124	−2	382	337	h=6 k=1			−27	41	52	−15	92	104	−6	61	83
−8	429	401	−16	324	209	−3	109	161	−1	242	231	−28	103	139	−16	124	169	−8	136	139
−10	509	515	−18	115	94	−4	172	142	−3	104	109	−30	42	55	−17	90	99	−10	240	239
−12	420	313	−20	55	53	−5	358	342	−4	140	144	h=2 k=2			−18	106	112	−12	24	40
−14	732	616	h=8 k=0			−6	149	132	−5	208	224	−1	199	177	−19	96	103	−14	159	164
−16	465	368	−4	317	231	−7	214	190	−7	78	55	−2	412	361	−20	118	114	−15	40	51
−18	91	100	−10	118	74	−8	212	207	−9	87	97	−3	286	218	−22	144	175	h=9 k=2		
−20	368	319	−14	58	57	−9	56	100	−11	181	178	−4	129	91	−26	66	82	−2	184	146
−22	110	97	−16	195	136	−10	248	213	−12	93	81	−5	101	60	−27	35	59	−3	29	31
−26	267	210	h=9 k=0			−11	199	212	−15	204	238	−6	22	19	h=5 k=2			−4	26	41
−30	204	155	−2	176	118	−12	190	188	−18	86	84	−7	24	10	−1	42	31	−7	128	120
h=3 k=0			−6	182	130	−13	440	466	−21	75	115	−8	44	18	−2	616	646	−8	34	46

h=1 k=3		
−1	254	256
−2	196	208
−3	122	149
−4	154	126
−5	140	179
−6	137	167
−7	312	331
−8	73	87
−9	256	222
−11	104	122
−12	112	98
−13	212	196
−14	54	60
−15	90	107
−16	143	145
−17	72	70
−18	69	79
−19	166	168
−20	32	43
−21	143	163
−22	39	52
−23	110	150
−25	64	71
−26	69	94
−27	73	96
h=2 k=3		
−1	300	303
−2	77	94
−3	207	157
−4	14	7
−5	207	188
−6	131	130
−7	34	34
−8	123	119
−9	159	172
−10	39	32
−11	24	31
−12	35	34
−13	146	132
−14	137	144
−15	201	219
−16	230	222
−17	83	92
−18	96	126
−19	84	8_9
−20	91	108
−21	128	140
−23	27	24
−24	99	121
−25	96	130
−27	38	57
h=3 k=3		
−1	191	155
−2	109	87
−3	334	313
−4	167	113
−5	189	203
−7	180	168
−8	154	132
−9	135	123
−10	71	79
−13	88	83
−14	136	140
−15	147	172
−16	95	125
−17	53	73
−18	99	115
−19	179	200
−20	68	81
−21	174	195
−22	33	54
−23	134	173
−25	96	99
−26	98	128
h=4 k=3		
−1	141	124
−2	241	245
−3	115	135
−4	44	53
−5	206	182
−6	131	137
−7	32	46
−8	62	58
−9	286	323
−10	52	64
−11	213	228
−12	90	89
−14	118	132
−15	182	200
−16	63	58
−17	177	178
−18	56	73
−19	68	65
−20	49	51
−21	93	119
−22	45	43
−23	24	25
−24	37	42
−25	56	98
h=5 k=3		
−1	221	195
−2	73	62
−3	489	498
−4	68	62
−5	71	89
−6	71	75
−7	216	223
−8	53	76
−9	244	249
−10	39	62
−12	39	43
−13	141	151
−15	93	96
−16	31	28
−17	25	31
−18	32	35
−19	98	134
−22	40	48
h=6 k=3		
−1	182	169
−2	50	71
−3	78	102
−4	120	113
−5	214	214
−6	35	30
−7	95	105
−8	43	38
−9	136	143
−11	88	104
−12	55	53
−13	38	50
−14	37	29
−15	89	104
−16	52	65
−17	29	42
−18	27	41
−19	90	111
−20	69	66
−21	23	67
h=7 k=3		
−1	53	28
−2	186	145
−3	188	165
−5	131	117
−6	61	58
−7	162	194
−8	42	31
−9	51	54
−10	91	102
−11	135	151
−13	149	162
−14	91	92
−15	40	51
−16	21	28
−17	80	131
h=8 k=3		
−1	51	66
−2	70	89
−3	146	144
−4	82	69
−5	123	130
−8	41	44
−9	132	140
−11	22	50
h=1 k=4		
−1	123	137
−2	238	252
−3	93	89
−4	249	286
−5	78	59
−6	20	2
−7	27	37
−8	224	285
−9	99	90
−10	32	7
−11	100	91
−12	171	161
−14	82	93
−15	228	245
−17	56	49
−18	138	167
−20	59	81
−21	69	79
−22	76	100
−23	66	73
−24	114	131
−25	23	38
−26	55	67
h=2 k=4		
−1	93	79
−2	22	56
−3	107	107
−4	213	238
−5	72	80
−6	178	186
−7	91	89
−8	106	118
−9	64	80
−10	218	241
−11	29	37
−12	37	69
−13	52	47
−14	144	165
−15	43	59
−16	147	169
−17	39	53
−18	22	5
−19	26	42
−20	165	200
−21	71	76
−22	105	119
−23	63	76
−24	57	67
−25	34	49
h=3 k=4		
−1	44	65
−2	191	211
−3	76	85
−6	195	157
−8	285	358
−9	33	35
−10	144	157
−12	191	212
−14	137	163
−15	31	42
−16	39	57
−18	132	170
−19	104	124
−20	56	89
−21	24	26
−22	96	134
−23	25	44
−24	70	102
h=4 k=4		
−1	81	82
−2	39	27
−3	26	41
−4	191	199
−5	21	33
−6	145	157
−7	35	62
−8	129	132
−9	31	13
−10	183	184
−11	85	89
−13	23	41
−14	131	153
−15	37	41
−16	141	167
−17	119	122
−19	73	99
−20	126	184
−21	46	57
−22	70	90
k=5 k=4		
−1	38	11
−2	157	141
−3	162	163
−4	38	27
−5	157	161
−6	134	114
−7	55	78
−8	219	250
−9	104	107
−10	65	99
−11	110	125
−12	86	108
−13	85	93
−14	97	100
−16	44	59
−17	56	67
−18	77	102
−19	37	61
−20	21	39
h=6 k=4		
−1	52	42
−2	38	51
−3	72	69
−4	84	96
−6	79	78
−8	78	80
−10	77	89
−11	30	51
−13	26	54
−14	66	88
−16	64	91
h=7 k=4		
−2	72	81
−3	157	147
−4	74	63
−5	49	50
−6	58	63
−8	74	94
−9	47	52
−10	28	32
−11	43	53
−12	19	48
h=1 k=5		
−2	101	106
−3	184	210
−4	73	112
−5	116	139
−7	149	193
−8	89	97
−12	60	42
−13	71	121
−15	60	75
−17	79	119
−18	74	95
−19	53	83
−20	46	20
h=1 k=6		
−4	79	104
−5	82	124
−6	68	115
−8	109	134
−12	51	64
−14	46	76
−15	43	52

Description and Discussion of the Structure

Bond lengths and angles. The intramolecular bond lengths and angles are given in *Table* 3 and *Fig.* 1.

The benzene ring is slightly distorted from regular hexagon. Ring C—C bond lengths vary from 1.35 to 1.41 Å with the average 1.39 Å which is in agreement with the C—C bond length in crystalline benzene of 1.392±0.010 Å (Cox, Cruickshank & Smith, 1958)[12]. It has been pointed out by many authors, namely, Klug (1970)[13], Arona & Sundaralingam (1917)[14], Kruger & Gafner (1971)[4] etc., that there is a tendency of stabilization of p-substituted benzene ring with the distortion from regular hexagonal form. It was found that the internal valence angles (range 117∼122°) at the substituted benzene ring show significant deviations from the ideal hexagonal value of 120°.

The C(4)—N(1) bond length is 1.41 Å which

Table 3. Bond lengths and angles in sulfaguanidine monohydrate.
The estimated standard deviations given in parentheses refer to the last decimal positions of respective values

i	j	D_{ij}(Å)	i	j	k	$\angle_{ijk}$(°)
S	C(1)	1.78(2)	O(1)	S	O(2)	114.3(7)
S	O(1)	1.43(1)	O(1)	S	C(1)	106.8(7)
S	O(2)	1.42(1)	O(1)	S	N(2)	115.2(7)
S	N(2)	1.62(1)	O(2)	S	C(1)	107.4(8)
C(1)	C(2)	1.36(3)	O(2)	S	N(2)	105.8(7)
C(2)	C(3)	1.40(3)	C(1)	S	N(2)	107.0(7)
C(3)	C(4)	1.41(3)	C(2)	C(1)	S	120(1)
C(4)	C(5)	1.35(3)	C(6)	C(1)	S	119(1)
C(5)	C(6)	1.39(3)	C(1)	C(2)	C(3)	119(2)
C(6)	C(1)	1.38(3)	C(2)	C(3)	C(4)	121(2)
C(4)	N(1)	1.41(3)	C(3)	C(4)	C(5)	117(2)
N(2)	C(7)	1.32(2)	C(4)	C(5)	C(6)	122(2)
C(7)	N(3)	1.32(2)	C(5)	C(6)	C(1)	119(2)
C(7)	N(4)	1.37(2)	C(6)	C(1)	C(2)	121(2)
			N(1)	C(4)	C(3)	120(2)
			N(1)	C(4)	C(5)	122(2)
			S	N(2)	C(7)	122(1)
			N(2)	C(7)	N(3)	125(2)
			N(2)	C(7)	N(4)	116(1)
			N(3)	C(7)	N(4)	118(2)

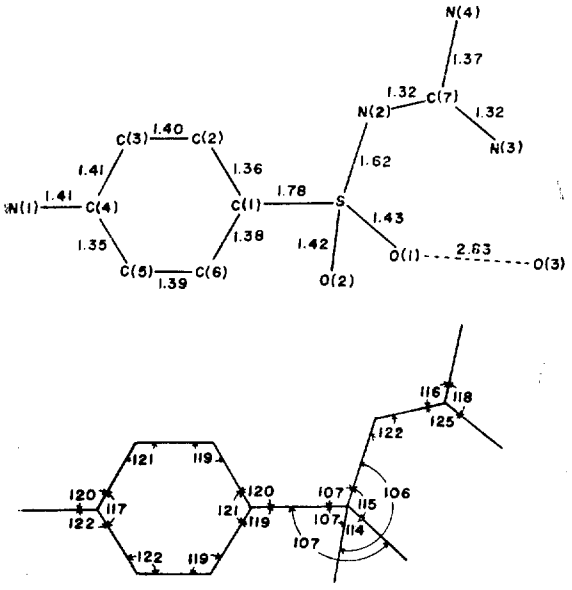


Fig. 1. Bond lengths (Å) and angles(°) in sulfaguanidine monohydrate

is shorter than the C—N single bond of 1.47 Å (Pauling, 1950)[15].

Bonding around the sulfur atom is distorted from the ideal tetrahedral. The maximum and minimum values for O—S—N are 115° and 106° respectively as in sulfathiazole II[4]. Distortion from the tetrahedral symmetry is the general property of the derivatives of sulfanilamide. Reference to *Table* 4 shows a comparison of bond lengths and angles around the tetrahedral sulfur atom obtained from the compounds containing sulfonyl group with their average values. The S—C(1) bond length 1.78 Å is longer than the theoretical S—C(sp^2) value 1.75 Å calculated from the atomic radii and electronegativities given by Truter (1962)[19]. The S—O(1) and S—N(2) bond lengths are in good agreement with the results of the other related compounds. The S—O(2) bond length 1.42 Å is insignificantly shorter than the S—O

Table 4. A comparison of bond lengths and angles around sulfur atom in sulfaguanidine monohydrate and related compounds.

Compound	Bond length (Å)				Range of angle(°)
	S—O		S—N(2)	S—C(1)	
Sulfaguanidine monohydrate	1.42(1)	1.43(1)	1.62(1)	1.78(2)	105.8~115.2
α-Sulfanilamide[1]	1.41	1.47	1.61	1.74	105.7~119.0
β-Sulfanilamide[2]	1.448(2)	1.454(2)	1.620(2)	1.750(2)	105.5~118.2
γ-Sulfanilamide[3]	1.44(2)	1.45(1)	1.67(2)	1.74(1)	106.4~117.3
Sulfanilamide monohydrate[16]	1.448(9)	1.463(9)	1.620(11)	1.748(12)	106.0~118.3
Sulfathiazole II[4]	1.435(2)	1.444(2)	1.589(3)	1.759(3)	104.7~116.6
Sulfathiazole I & III[5]	1.434(10)	1.440(6)	1.606(9)	1.754(9)	104.8~117.1
Methanesulfonanilide[17]	1.425(2)	1.443(2)	1.633(2)	1.746(2)	105.3~118.6
2′-Hydroxymethanesulfonanilide[13]	1.435(6)	1.447(6)	1.617(6)	1.736(6)	106.5~119.2
S, S-Diphenyl-N-p-tolylsulfonyl sulfilimine[18]	1.430(8)	1.435(8)	1.598(8)	1.756(8)	104.8~118.1
Average	1.44		1.62	1.75	109.5

average value 1.44 Å (see *Table* 4).

In the guanidyl residue the C—N bond lengths range from 1.32 Å in C(7)—N(2) to 1.37 Å in C(7)—N(4), angles from 116° in N(2)—C(7)—N(4) to 125° in N(2)—C(7)—N(3). There is a considerable deviation from the dimensions of free guanidium ion, $[C(NH_2)_3]^+$ (Haas, Harris & Mills, 1965)[20] of the average values 1.323 Å and 120°. This fact indicates that there is a flexibility in the geometry of guanidyl residue, subjecting to the crystal lattice environment.

By Wheatley's order/length curve for C—N bonds[21], the C—N bond length of 1.319 Å corresponds to 50 % double bond character. Thus the bond lengths and the good planarity of guanidyl residue (see *Table* 5) suggest that the guanidyl residue has a delocalized π-electron system.

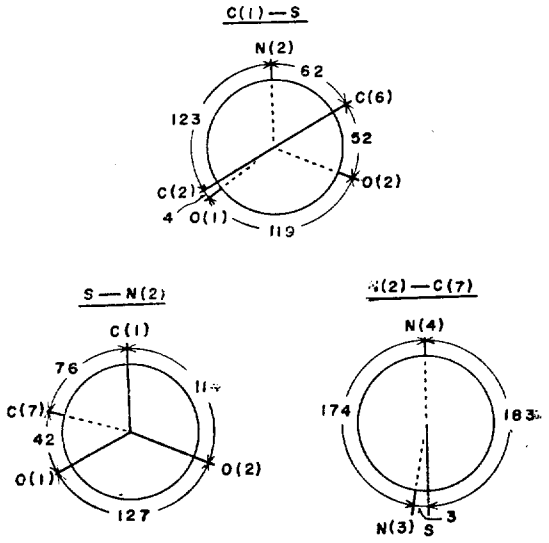


Fig. 2. Newman projection down the C(1)—S, S—N(2) and N(2)—C(7) bonds in sulfaguanidine monohydrate showing the conformation angles in degrees.

Planarity and molecular conformation The least-squares planes are listed in *Table* 5. The benzene ring is planar within the errors of the structure determination. The sulfur atom and the nitrogen atom N(1) are displaced 0.07 and 0.05 Å respectively from the ring plane. The small distortion may result from the crystal packing forces. The guanidyl residue is planar within the experimental error and the sulfur atom is displaced −0.18 Å from the guanidyl plane which makes an angle of 96° with the benzene ring plane.

Table 5. Least-squares planes in sulfaguanidine monohydrate.
Equation for plane : $Ax+By+Cz=D$, where x, y, z are in Å

Atoms in plane	Atoms out of plane	Distance in Å from best plane	Constant
A. Benzene ring			
C (1)		0.01	A=0.857
C (2)		0.00	B=0.511
C (3)		−0.01	C=−0.073
C (4)		0.03	D=2.072
C (5)		−0.02	
C (6)		0.00	
	S	0.07	
	N (1)	0.05	
	O (1)	0.19	
B. Guanidyl residue			
N (2)		−0.01	A=0.182
N (3)		−0.01	B=0.018
N (4)		−0.01	C=0.983
C (7)		0.03	D=10.383
	S	−0.18	
	O (1)	0.60	
	O (2)	0.07	

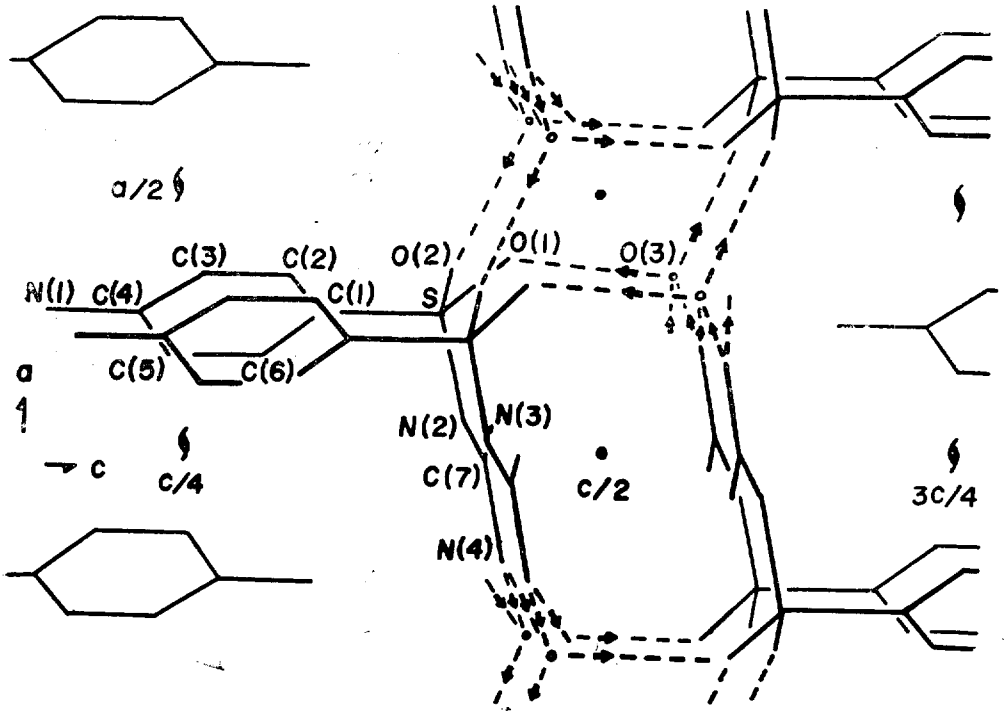


Fig. 3. Projection of the crystal structure of sulfaguanidine monohydrate along the *b* axis. Dashed lines are hydrogen bonds; arrows indicate donor direction

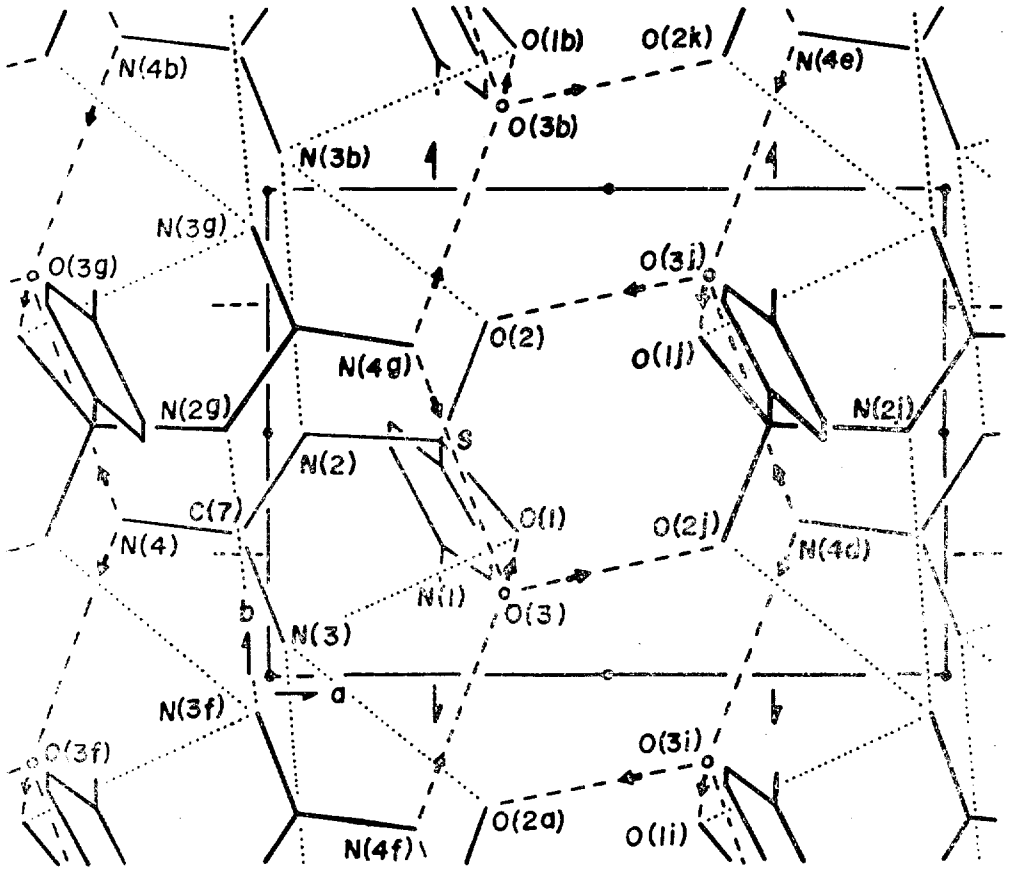


Fig. 4. Projection of the crystal structure of sulfaguanidine monohydrate along the *c* axis. Dashed lines are hydrogen bonds;)arrows indicate donor direction. Dotted lines are probable hydrogen bonds.

The conformation angles about the C(1)—S, S—N(2) and N(2)—C(7) bonds are given in *Fig.* 2. The molecule does not take a symmetrical form. The benzene ring makes a torsional angle of 62° rather than 90° to the S—N(2) bond while in the case of sulfanilamide monohydrate[16] it is 88°.

Hydrogen bonding and crystal packing. The structure of sulfaguanidine monohydrate projected along the *b* and *c* axis is shown in *Fig.* 3 and *Fig.* 4. The molecules related by the symmetry centers are joined indirectly by two N—H(guanidyl)···O—H(water)···O(sulfonyl) hydrogen bonds. As shown in *Fig.* 5, the water molecule plays an important role in the hydrogen-bonding scheme by forming a distorted tetrahedral configuration and water oxygen O(3) is involved in two donor O(3)—H ···O hydrogen bonds to sulfonyl oxygen O(1) and O(2*j*) and two acceptor N—H···O(3) hydrogen bonds from N(4*f*) and N(4*g*). The close inter- and intramolecular approaches less than 3.5 Å are listed in *Table* 6. Though the positions of the hydrogen atoms can not be located under the present accuracy it may be possible to suppose the hydrogen location and then consider the possi-

Table 6. Intermolecular distances and angles in sulfaguanidine monohydrate

i	j	D_{ij}(Å)	i	j	k	$\angle_{ijk}$(°)
O(3)	O(2j)	2.80(2)*	O(1)	O(3)	N(4f)	115(1)
O(3)	O(1)	2.83(2)*	O(1)	O(3)	N(4g)	87(1)
O(3)	N(4f)	2.91(2)*	O(1)	O(3)	O(2j)	108(1)
O(3)	N(4g)	3.00(2)*	O(2j)	O(3)	N(4f)	115(1)
N(3)	O(1)	2.90**	O(2j)	O(3)	N(4g)	92(1)
N(3)	O(2a)	2.97**	N(4f)	O(3)	N(4g)	134(1)
N(3)	N(2a)	3.13**				
				Symmetry code :		
O(1)	O(2a)	3.18		x	y	z
N(4)	O(1c)	3.18	a	x	$-1+y$	z
O(3)	N(3f)	3.20	b	x	$1+y$	z
O(3)	N(1h)	3.21	c	$-1+x$	y	z
O(1)	O(3i)	3.40	d	$1+x$	y	z
O(2)	C(2b)	3.49	e	$1+x$	$1+y$	z
			f	$-x$	$-y$	$1-z$
			g	$-x$	$1-y$	$1-z$
			h	x	$1/2-y$	$1/2+z$
			i	$1-x$	$-y$	$1-z$
			j	$1-x$	$1-y$	$1-z$
			k	$1-x$	$2-y$	$1-z$

*hydrogen bonds, **probable hydrogen bonds

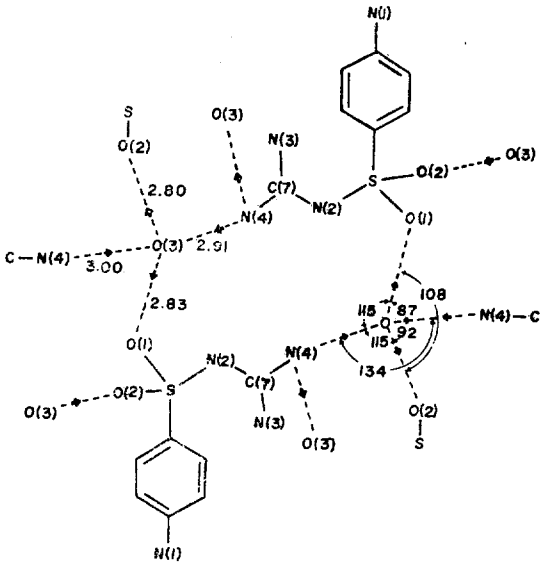


Fig. 5. Environment around water oxygen atom O(3) in sulfaguanidine monohydrate

bilities of forming the hydrogen bonds. Analogy in this way may lead to the conclusion that N(3) can be involved in the three hydrogen bonds, one acceptor N(3)···H—N(2a) (intermolecular), two donor N(3)—H···O(1) (intramolecular) and N(3)—H···O(2a) (intermolecular). These are shown as the dotted lines in *Fig.* 4. But as there is only one hydrogen atom available to N(3), the latter two donor hydrogen bonds may exist as bifurcated hydrogen bond in the form of N(3)—H$\begin{matrix}\diagup\,\mathrm{O(1)}\\ \diagdown\,(2a)\end{matrix}$

These hydrogen-bonding networks form infinite molecular layers parallel to (001) plane and the interlayer force appears to be van der Waals in character.

Acknowledgement

It is a pleasure to thank Prof. H. S. Shin, Dongguk Univ., for helpful discussions. This.

work has been supported by the research grant from the Ministry of Science and Technology.

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