DAEHAN HWAHAK HWOEJEE
(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

具廷書 · 金勳燮 · 申桓澈 · 崔柱鉉*

서울大學校 文理科大學 化學科

(1973. 12. 19 접수)

# The Crystal and Molecular Structure of Sulfaguanidine Monohydrate

**Chung Hoe Koo, Hoon Sup Kim, Whanchul Shin** and **Chuhyn Choe***

*Department of Chemnistry, College of Liberal Arts and Sciences,*
*Seoul National University, Seoul, Korea*

(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, $\beta$=91.0±0.2°이다. 결정구조는 패터슨 함수의 해석과 직접법을 병용하여 밝혀냈다. 원자좌표치는 최소자승법으로 정밀화하였으며 1542개의 독립적인 회절반점에 대한 최종 $R$값은 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.

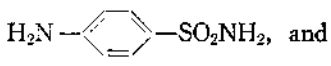
**Abstract.** The crystal and molecular structure of sulfaguanidine monohydrate, $C_7H_{10}N_4O_2S\cdot H_2O$, was determined from visually estimated intensity data from Weissenberg photographs. The crystal data are monoclinic, space group $P2_1/c$ with four molecules in a unit cell of dimensions, $a$=7.57±0.03, $b$=5.44±0.02, $c$=24.76±0.06 Å, $\beta$=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

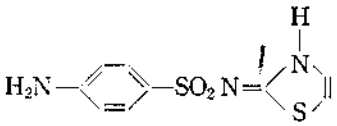
As a part of a series of detailed structure investigation on sulfur-containing compounds which have biologically active groups by X-ray single-crystal diffraction, the crystal and molecular structure of sulfaguanidine monohydrate

*Department of Chemistry, College of Liberal Arts and Sciences. Chonnam National University, Kwangju, Korea

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 $\pi$-electron on the guanidyl residue and the role of hydrated water molecule in the crystal-lattice environment.

## Experimental

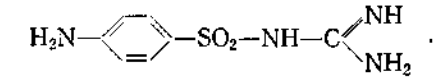
Prismatic crystals of sulfaguanidine monohydrate elongated along the *b* axis were obtained by slow evaporation from an aqueous acetone solution at room temperature.

Unit cell dimensions were determined by a least-squares refinement of $2\theta$ values for 47 indepenpdent reflections measured on the *h0l* and *0kl* Weissenberg photographs calibrated with superimposed Al-lines using Cu-$K_\alpha$ 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,



· $H_2O$,

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-$K_\alpha$ 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 | 208 | 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 | 169 | 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 | 676 | 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 | 176 | 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 | 78 |
| 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 | 89 |
| −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) |

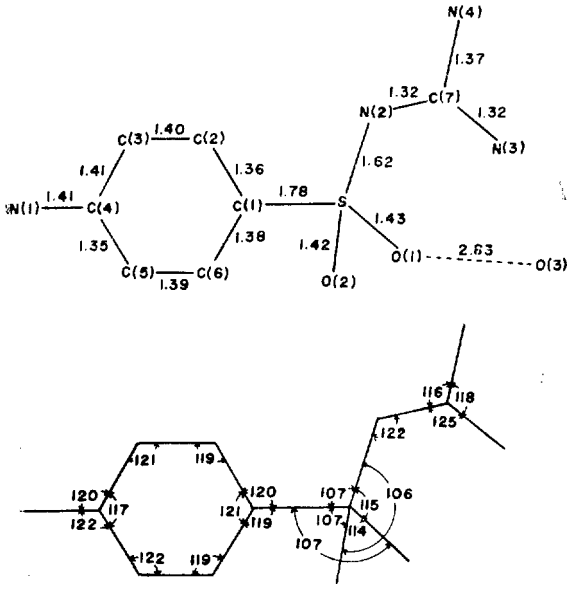



*Fig.* 1. Bond lengths (Å) and angles(°) in sulfaguanidine monohydrate

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.

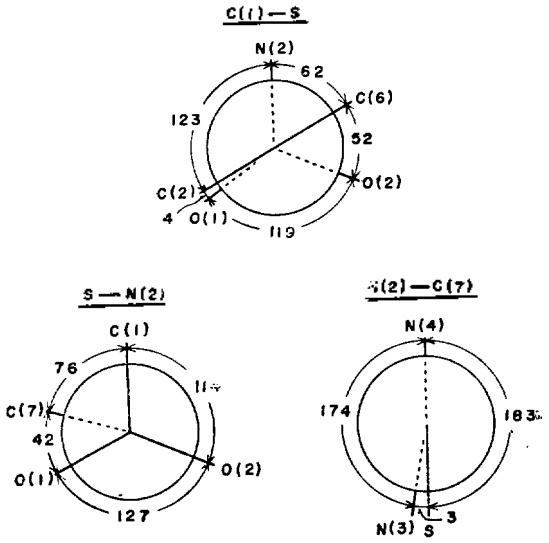



*Fig.* 2. Newman projection down the C(1)—S, S—N(2) and N(2)—C(7) bonds in sulfaguanidine monohydrate showing the conformation angles in degrees.

**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 | |

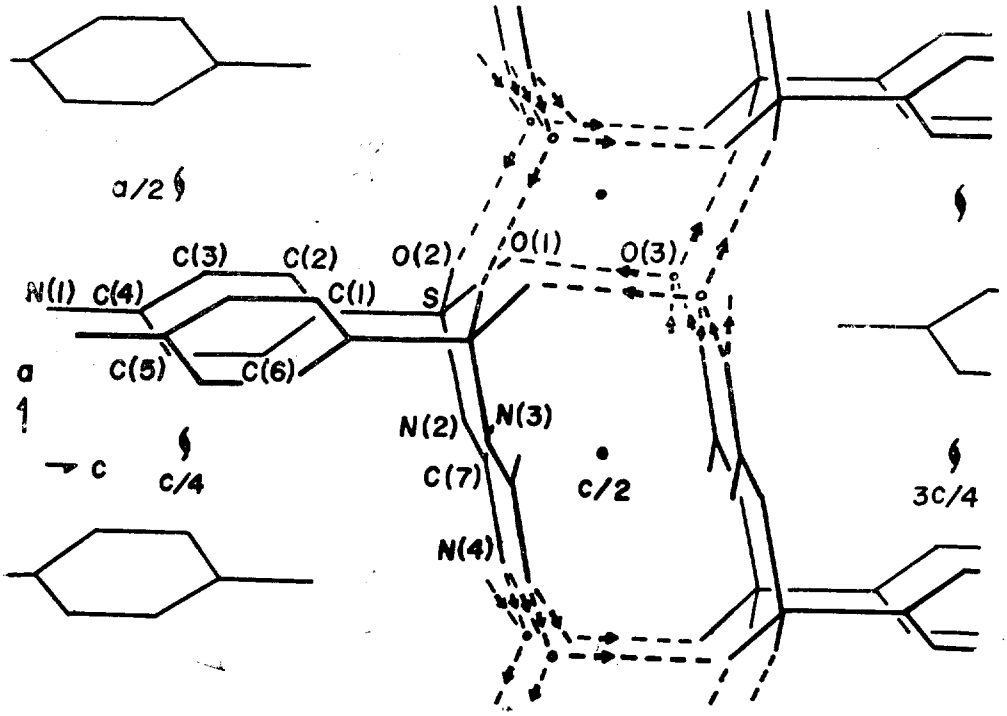



*Fig.* 3. Projection of the crystal structure of sulfaguanidine monohydrate along the *b* axis. Dashed lines are hydrogen bonds; arrows indicate donor direction

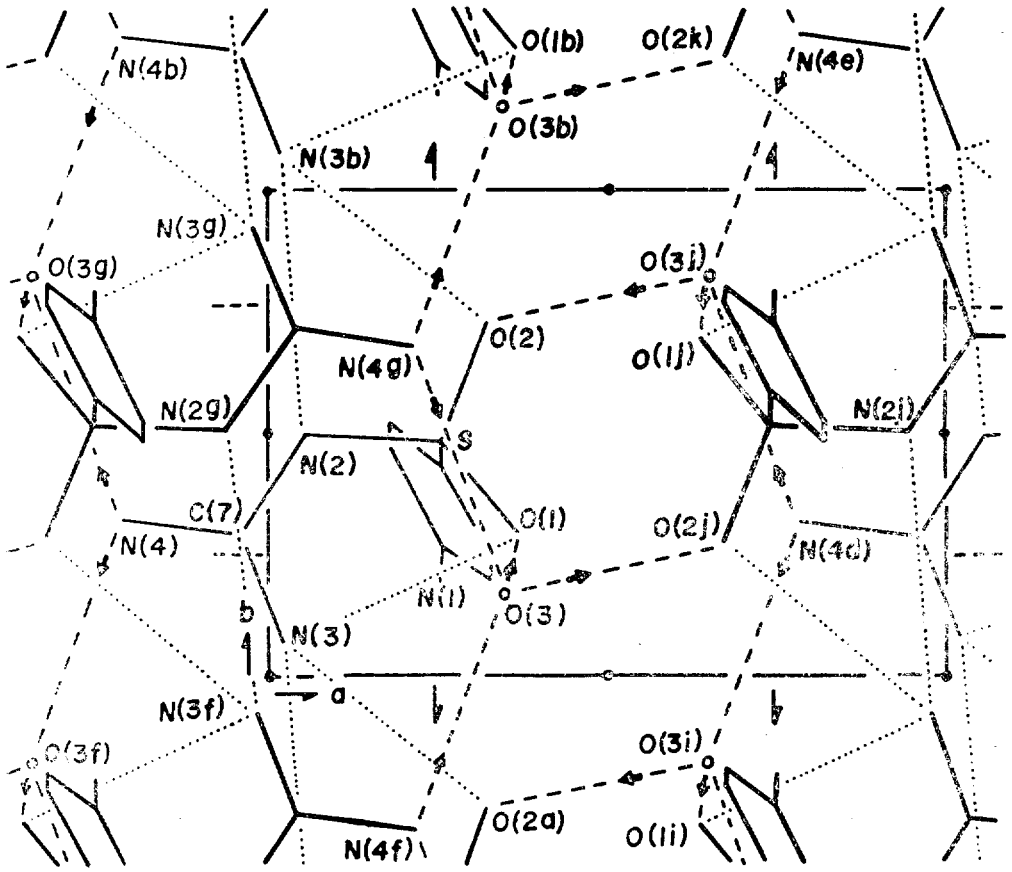



*Fig.* 4. Projection of the crystal structure of sulfaguanidine monohydrate along the *c* axis. Dashed lines are hydrogen bonds;)arrows indicate donor direction. Dotted lines are probable hydrogen bonds.

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(2$j$) | 2.80(2)* | O(1) | O(3) | N(4$f$) | 115(1) |
| O(3) | O(1) | 2.83(2)* | O(1) | O(3) | N(4$g$) | 87(1) |
| O(3) | N(4$f$) | 2.91(2)* | O(1) | O(3) | O(2$j$) | 108(1) |
| O(3) | N(4$g$) | 3.00(2)* | O(2$j$) | O(3) | N(4$f$) | 115(1) |
| N(3) | O(1) | 2.90** | O(2$j$) | O(3) | N(4$g$) | 92(1) |
| N(3) | O(2$a$) | 2.97** | N(4$f$) | O(3) | N(4$g$) | 134(1) |
| N(3) | N(2$a$) | 3.13** | | | | |
| | | | | Symmetry code : | | |
| O(1) | O(2$a$) | 3.18 | | $x$ | $y$ | $z$ |
| N(4) | O(1$c$) | 3.18 | $a$ | $x$ | $-1+y$ | $z$ |
| O(3) | N(3$f$) | 3.20 | $b$ | $x$ | $1+y$ | $z$ |
| O(3) | N(1$h$) | 3.21 | $c$ | $-1+x$ | $y$ | $z$ |
| O(1) | O(3$i$) | 3.40 | $d$ | $1+x$ | $y$ | $z$ |
| O(2) | C(2$b$) | 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

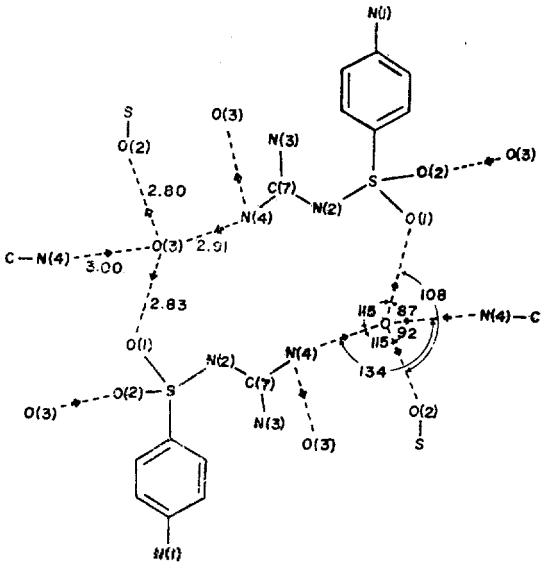



*Fig.* 5. Environment around water oxygen atom O(3) in sulfaguanidine monohydrate

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(2$a$) (intermolecular), two donor N(3)—H···O(1) (intramolecular) and N(3)—H···O(2$a$) (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$\begin{matrix}\cdots O(1)\\ \cdots (2a)\end{matrix}$

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.

## Reference


1. B. H. O'Connor and E. N. Maslen, *Acta Cryst.*, **18**, 363(1965).
2. A. M. O'Connel and E. N. Maslen, *Acta Cryst.*, **22**, 134(1967).
3. M. Alleaume and J. Decap, *Acta Cryst.*, **19**, 934 (1965).
4. G. J. Kruger and G. Gafner, *Acta Cryst.*, **B 27**, 326(1971).
5. G. J. Kruger and G. Gafner, *Acta Cryst.*, **B 28**, 272(1972).
6. A. J. C. Wilson, *Nature*, Lond., **150**, 152(1942).
7. R. Shiono, "IBM 1130 Data Reduction Program, Technical Reports", *Dept. of Crystallography, Univ. of Pittsburgh*, 1968.
8. M. M. Woolfson, "Direct Methods in Crystallography", Clarendon Press, Oxford, 1961.
9. R. Shiono, "IBM 1130 Least Squares Program, Technical Reports", *Dept. of Crystallography, Unit. of Pittsburgh*, 1968.
10. D. W. J. Cruickshank, "Computing Methods and the Phase Problem in X-ray Crystal Structure Analysis", Pergamon Press, New York, 1961.
11. "International Table for X-ray Crystallography", Vol. III Kynoch Press, Birmingham, 1968.
12. E. G. Cox, D. W. J. Cruickshank and J. A. S. Smith, *Proc. Roy. Soc.*, **A247**, 1-21(1958).
13. H. P. Klug, *Acta Cryst.*, **B26**, 1268(1970).
14. S. K. Arona and M. Sundaralingam, *Acta Cryst.*, **B 27**, 1293(1971).
15. L. Pauling, "The Nature of Chemical Bond", 3rd ed., Cornell Univ. Press, NewYork, 1960.
16. M. Alleaume and J. Decap, *Acta Cryst.*, **B 24**, 214(1968).
17. H. P. Klug, *Acta Cryst.*, **B 24**, 792(1968).
18. A. Kalman, B. Deffin and A. Kucsman, *Acta Cryst.*, **B 27**, 586 (1971).
19. M. R. Truter, *J. Chem. Soc.*, 3400(1962).
20. Haas, Harris and Mills, *Acta Cryst.*, **19**, 676 (1965).
21. P. J. Wheatley, *Acta Cryst.*, 8, 224(1955).