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THE CRYSTAL STRUCTURE AND HYDROGEN BONDING OF MAGNESIUM SULFATE HEXAHYDRATE

Berkeley, California

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THE CRYSTAL STRUCTURE AND HYDROGEN BONDING OF
MAGNESIUM SULFATE HEXAHYDRATE

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The Crystal Structure and Hydrogen Bonding of

Magnesium Sulfate Hexahydrate[*]

by Allan Zalkin, Helena Ruben and David H. Templeton

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Crystals of $MgSO_4 \cdot 6H_2O$ are monoclinic, space group C2/c,

with 8 formula units in the unit cell with dimensions

a = 10.110, b = 7.212, c = 24.41 Å; β = 98.30°.

The atomic parameters were refined by least squares using

x-ray counter data for 2576 independent reflections. The

positions of the hydrogen atoms found by least squares

and by Fourier methods confirm the assignment of hydrogen

bonds made previously for the isomorphous $CoSO_4 \cdot 6H_2O$.

Average interatomic distances are: water coordinated to

Mg, Mg-O = 2.06 Å; hydrogen bonds, O-O = 2.82 Å; sulfate

ion, S-O = 1.473 Å (uncorrected), 1.486 Å (corrected for

thermal motion).

Introduction

The crystal structure of $CoSO_4 \cdot 6H_2O$ has been described in a previous paper (Zalkin, Ruben, and Templeton, 1962). Hydrogen bonds were assigned on the basis of the positions of the heavier atoms, but the x-ray diffraction data did not give direct evidence of the hydrogen atom positions. The present paper describes a study of the isomorphous substance $MgSO_4 \cdot 6H_2O$ for which we have obtained more accurate diffraction data. These better data permit a more accurate description of the structure and indicate hydrogen atom positions in agreement with those deduced in the earlier work.

Crystals of $MgSO_4 \cdot 6H_2O$ are known in nature as the mineral hexahydrite (Dana and Ford, 1948). Other substances which have the same structure are $MgSeO_4 \cdot 6H_2O$, $NiSO_4 \cdot 6H_2O$, $CoSeO_4 \cdot 6H_2O$, and $ZnSO_4 \cdot 6H_2O$ (Groth, 1908).

Experimental

Crystals of $MgSO_4 \cdot 6H_2O$ were grown from saturated aqueous solution at $50 - 55^{\circ}C$. Once grown and separated from the mother liquor, the exposed crystals could be kept at room temperatures for several months with no visible deterioration. A small single crystal with diameter of the order of 0.02–0.08 mm was mounted on a glass fiber and oriented for rotation about its b axis with the use of a Weissenberg camera. It was then transferred to a goniostat mounted on a General Electric XRD-5 apparatus, which was equipped with a molybdenum x-ray tube, a scintillation counter, and a pulse-height discriminator, for further alignment and data taking. Cell dimensions and crystal settings were calculated with $\lambda(K\alpha_1) = 0.70929$ Å. Intensities were measured for 2,576 reflections of which 289 were recorded

as zero. All of the reciprocal lattice points with positive h and k, out to a limit of $\sin\theta/\lambda$ = 0.705 (2θ = 60°) were measured. Step scanning of the angle θ was done for selected reflections to verify the settings for peak intensity. For the typical reflection, a single count was made for a fixed time of 20 seconds.

The absorption coefficient μ for this crystal with Mo Kα x-rays is estimated as 5.0 cm^{-1}. The resulting μR is less than 0.02. No correction was made for absorption or extinction.

An accurate trial structure was provided by the structure of $CoSO_4 \cdot 6H_2O$ (Zalkin, Ruben and Templeton, 1961); corresponding coordinates for atoms other than hydrogen differ by 0.01 or less in the two structures. Least-squares refinements were started using the programs of Busing and Levy (1959) on the IBM 704 computer. We then used the IBM 709 computer with the programs of Gantzel, Sparks, and Trueblood (1961). Each program utilizes the full matrix, and each minimizes the function $\Sigma w(|F_o|-|F_c|)^2/\Sigma w|F_o|^2$, where w is the weighting factor and F_o and F_c are the observed and calculated structure factors. In this calculation, each independent reflection was assigned unit weight. The atomic scattering factors used in the refinements were Mg^{++} and neutral S (Tomiie and Stam, 1958), neutral O (Hoerni and Ibers, 1954), and neutral H (Ibers, 1961).

Space Group and Cell Dimensions

The space group is C2/c (C_{2h}^6). Reflections are absent with h+k odd, and h0ℓ reflections are absent with ℓ odd. The cell dimensions are:

$a = 10.110 \pm 0.005,$ $b = 7.212 \pm 0.004,$ $c = 24.41 \pm 0.01$ Å

(10.06)   (7.16)   (24.39)

$\beta = 98.30 \pm 0.05°$ $z = 8$ $V = 1761.2$ Å^3.

(98.57)

The values in parentheses are those reported by Ide (1938), changed from kX.

The calculated x-ray density is 1.723 g/cm^3. The a:b:c ratios found in this

and in previous work are:

1.402 : 1 : 3.384, $\beta = 98.30°$ (this work),

1.404 : 1 : 3.404, $\beta = 98.57°$ (Ide, 1938),

1.404 : 1 : 3.337, $\beta = 98.57°$ (Marignac, 1855).

The eight Mg atoms occupy two sets of positions 4(a) and 4(e) (notation

of International Tables, 1952):

4(a): (0, 0, 0; 0, 0, 1/2) + C centering,

4(e): (0, y, 1/4; 0, -y, 3/4) + C centering.

The sulfur atoms, ten sets of oxygen atoms, and twelve sets of hydrogen

atoms occupy general positions:

8(f): $\pm$(x, y, z; -x, y, $\frac{1}{2}$ - z) + C centering.

Refinement Procedure

At first hydrogen was omitted from the calculations. With isotropic

temperature factors of the form $\exp(-B\sin^2\theta/\lambda^2)$ for each atom,

$R = \Sigma||F_o|-|F_c||/\Sigma|F_o|$ was reduced to 0.120 by three cycles of least

squares. After correction of a few blunders in the original measurements,

two more cycles reduced R to 0.118.

A three-dimensional Fourier synthesis with (F_o-F_c) as coefficients

was calculated to seek out the hydrogen atoms. The resulting maps showed

a peak wherever a hydrogen atom was expected, as well as many others of

about the same size which resulted from the anisotropic motion of oxygen

atoms. A final isotropic refinement which included the hydrogen atoms

but did not refine them resulted in R = 0.115.

Anisotropic temperature factors of the form $\exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}\ell^2$

$-2\beta_{12}hk - 2\beta_{13}h\ell - 2\beta_{23}k\ell)$ were introduced for Mg, S and O. Three cycles with

hydrogen atoms included but not refined gave R = 0.056.

A set of $(F_o - F_c)$ values were calculated with the resulting parameters

for Mg, S, and O, but omitting hydrogen. Another "difference Fourier" was

calculated using these numbers as coefficients. The twelve largest peaks

in this function (Fig. 1) were near the predicted locations of the twelve

hydrogen atoms.

As our least-squares programs at this time could not handle at one time

a refinement of all the atoms including hydrogen with all the anisotropic

thermal parameters, we ran a series of calculations refining parts of the

structure separately. Temperature factors were isotropic for H and anisotropic

for the other atoms. First the H atoms were included but not refined, while

all other parameters were refined. Next H atoms were refined with the other

atoms fixed. Then Mg atoms were held fixed while all other atoms were refined;

in this series only the 923 reflections with $\sin\theta/\lambda$ less than 0.5 were included.

Finally the H atoms were fixed and all other atoms were refined. All this

calculation reduced R to 0.053 for the 2576 reflections, including those with

zero intensity. In the final cycle no coordinate or thermal parameter shifted

more than 0.000001.

The resulting observed and calculated structure factors are shown in

Table 1. The corresponding coordinates are listed in Table 2 and the thermal

parameters in Tables 3 and 4.

By a special patch, the program was modified to allow simultaneous

refinement of all 159 variable parameters. Two cycles yielded no significant

improvement in R nor any substantial change in coordinates. The results are

not reported because parameters were oscillating; further cycles to attain

convergence (at 0.9 hours per cycle with IBM 7090) seemed unjustified.

Standard deviations of coordinates estimated by least squares, with

neglect of the possible error in the hydrogen parameters, are reported in

Table 2. The similar estimates of standard deviations of the anisotropic

thermal parameters range from 2 to 4 percent of the diagonal elements of

the β matrices. They are not reported in detail because the effects of

systematic errors in the data and of coupling with hydrogen parameters are

of unknown and possibly considerably larger magnitude.

The standard deviations of hydrogen coordinates correspond to about 0.05 Å

when estimated by least squares with neglect of errors in heavy atom parameters.

The least-squares results and the Fourier results (Fig. 1) disagree in several

cases by about 0.1 Å. The best evidence of the accuracy of these coordinates

comes from the O-H bond distances which are expected to be somewhere in the

range 0.9 to 1.0 Å, depending on the magnitude of the thermal motion effect.

We find values 0.7 to 1.1 Å for eleven such bonds and 0.5 Å for O(7)-H(6).

This scatter suggests 0.2 Å as the standard deviation of position of hydrogen,

including the effects of systematic errors in the data.

The standard deviations of isotropic thermal parameters of hydrogen are

estimated by least squares to range from 0.7 $Å^2$ for H(11) to 1.5 $Å^2$ for H(6).

The scatter from the average value of 3.4 $Å^2$ corresponds to $\sigma = 1.8$ $Å^2$. This

latter value may be a better estimate of the true accuracy, since there is no

obvious reason for the thermal motions of the various hydrogen atoms to be

substantially different.

Discussion

The structure of $MgSO_4 \cdot 6H_2O$ differs very slightly from that already described for $CoSO_4 \cdot 6H_2O$ (Zalkin, Ruben and Templeton, 1962). The hydrogen bonds which were assigned in the previous study on the basis of oxygen positions are fully confirmed by the present results.

Table 5 lists some interatomic distances and their standard deviations, with no correction for thermal motion. The S-O bond distances in the sulfate ion average 1.473 Å with an average deviation of 0.007 Å. When a librational correction is made assuming the oxygen atoms to ride on the sulfur atom, the average becomes 1.486 Å with an average deviation of 0.004 Å. In the Tutton salt $Mg(NH_4)_2(SO_4)_2 \cdot 6H_2O$ Margulis and Templeton (1962) also found 1.486 Å for the average S-O distance when it was corrected in the same way.

The average Mg-O distance is 2.06 Å without correction for thermal motion. Magnesium is also in water octahedra in $Mg(NH_4)_2(SO_4)_2 \cdot 6H_2O$ with average Mg-O = 2.07 Å and in $Ce_2Mg_3(NO_3)_{12} \cdot 24H_2O$ (Templeton, Zalkin and Forrester, 1962) with average Mg-O = 2.06 Å. In all three crystals the average Mg-O distance is 2.07 Å with thermal correction assuming in-phase motion.

The hydrogen bond O-O distances average 2.82 Å, but scatter over a considerable range as is common in complicated hydrate structures. The accuracy of the hydrogen positions, about 0.2 Å, does not justify tabulation of the many bond distances and angles involving hydrogen atoms.

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Table 1. Observed and calculated structure factors, each multiplied

by 10.

(table to be reproduced photographically in two parts)

```
   H,K= 0, 0
  L FOBS FCAL
  2   50   42
  4  453 -459
  6   73   62
  8  525  513
 10  597 -582
 12  710 -724
 14  480  423
 16 1161 1159
 18  468  495
 20  657 -671
 22    0  -27
 24  522  540
 26  146 -174
 28    0   42
 30  121 -107
 32  340  338

   H,K= 2, 0
  L FOBS FCAL
 32    0   46
 30  102 -117
 28  119  131
 26  229  231
 24    0    7
 22  479 -464
 20  401  397
 18 1492 1519
 16  368  370
 14  326 -339
 12 1930 1964
 10  436 -435
  8   46   40
  6 2511-2634
  4 1447 1436
  2 1574 1560
  0  903  822
 -2 1620-1637
 -4 1255 1218
 -6 1513 1552
 -8 2195-2256
-10  383 -362
-12  323  292
-14  674  693
-16  140  151
-18  672 -657
-20  645  655
-22  190  205
-24  505  512
-26  206 -208
-28  304  333
-30  193  176
-32  184  158
-34  187 -191

   H,K= 4, 0
  L FOBS FCAL
 30  256  278
 28  180  186
 26  105  -92
 24  119 -135
 22    0  -29
 20  683  699
 18  416 -408
 16  140  144
 14  409  396
 12 1197 1182
 10 1312-1289
  8  199  216
  6  221  208
  4   73   28
  2  683 -668
  0  736 -741
 -2 1841 1853
 -4  662  687
 -6  832  813
 -8 1000  979
-10  616  598
-12 1284 1303
-14  571 -541
-16  188  186
-18  156 -200
-20  863  854
-22  203 -194
-24  157  164
-26    0    9
-28  257  253
-30   81   72
-32  204 -190
-34    0    1

   H,K= 6, 0
  L FOBS FCAL
 28    0   16
 26  111 -128
 24  339  331
 22   77  -65
 20    0    9
 18  479 -466
 16  473  471
 14  204  176
 12  537  543
 10  549  569
  8  638  635
  6  953  944
  4  294 -245
  2  227 -234
  0  434  447
 -2   36   49
 -4  650  641
 -6  230  209
 -8  883  885
-10  351 -340
-12  729  749
-14 1054-1057

-16  275  268
-18  394  346
-20   34   49
-22  208 -185
-24   82   95
-26  224  218
-28    0   -5
-30  157 -123
-32  123  122

   H,K= 8, 0
  L FOBS FCAL
 24  422  458
 22   84  -87
 20  121 -112
 18   55   77
 16  124   92
 14  108  103
 12  206  196
 10  259  249
  8  632  608
  6  240  269
 .4  498 -490
  2  466 -484
  0  872  879
 -2  635 -634
 -4  377  370
 -6  218 -210
 -8  667  630
-10  146 -144
-12  120 -136
-14  123  116
-16  682  706
-18  293  296
-20  131 -140
-22  274  267
-24  244  247
-26  135 -133
-28   42  -37
-30    0   29

   H,K=10, 0
  L FOBS FCAL
 20   88  105
 18  225  244
 16  226 -239
 14  237 -251
 12  244  236
 10  201  163
  8  344  342
  6  511 -522
  4  405  403
  2  436  444
  0  446  420
 -2  419 -409
 -4  610  608
 -6  451  454
 -8   70  -86
-10  238 -190
-12  176  177
-14  510  511
-16    0   50
-18   86 -121
-20  256  259
-22   41   43
-24    0    8
-26  241 -243

   H,K=12, 0
  L FOBS FCAL
 14  147  159
 12  407  423
 10  244 -281
  8    0   13
  6  200  174
  4  497  516
  2   98  -97
  0  158 -120
 -2  286  304
 -4  245  252
 -6  142 -138
 -8  295 -268
-10  143  151
-12  284  273
-14  223 -209
-16    0  -14
-18   42   -1
-20  212  259
-22  140 -150

   H,K=14, 0
  L FOBS FCAL
  0  193  182
 -2   88  107
 -4   44  -78
 -6   62  -66
 -8  285  298
-10   99  125

   H,K= 1, 1
  L FOBS FCAL
 33   88   96
 32   61  -41
 31   59   64
 30   41   25
 29   98  -71
 28  368  343
 27  239 -230
 26  296 -296
 25   63   35
 24   35  -44
 23    0    2
 22  455  463
 21  203 -187
 20  524  519
 19  118 -100

 18  325 -324
 17  432  434
 16   68  -57
 15  243  233
 14  659  641
 13   56  -54
 12 1348 1396
 11  502 -511
 10  333 -299
  9  313  326
  8  707  667
  7   54  -71
  6 1285 1292
  5   94 -106
  4 1712 1734
  3  586 -563
  2 1280-1254
  1  358  342
  0  169  128
 -1  426  391
 -2 1246 1249
 -3  380  385
 -4 2056 2252
 -5  815 -793
 -6 1142-1162
 -7  404  384
 -8  416 -431
 -9 1088 1089
-10  896  898
-11  385  370
-12  269 -225
-13  296 -274
-14  907 -909
-15   83  -78
-16  225  227
-17    0    6
-18  209  189
-19   30  -41
-20  640  649
-21  443 -448
-22  157 -116
-23  112 -125
-24   35  -43
-25  228  229
-26  132  130
-27  155  143
-28  351  341
-29  163 -132
-30  210 -208
-31   59   50
-32  104  104
-33   61   55
-34  108  111

   H,K= 3, 1
  L FOBS FCAL
 32  213  224
 31   43  -12
 30  159  155
 29   42   37
 28   41  -51
 27   40   29
 26   39   -7
 25    0  -20
 24  570  556
 23  233 -225
 22  182  200
 21   34   -6
 20   66 -128
 19    0   24
 18  300 -305
 17  233  235
 16  961  946
 15   50  -38
 14  549  562
 13  101  114
 12   98  -85
 11  206  179
 10  193 -211
  9  384  374
  8 1672 1665
  7  427 -410
  6 1352-1308
  5  234  219
  4 1073-1062
  3  375  373
  2  366 -376
  1   92   87
  0  352  326
 -1  433 -432
 -2 1042 1030
 -3 1074-1091
 -4  465  429
 -5  609 -616
 -6  501 -532
 -7  542  532
 -8 1186 1230
 -9  194 -166
-10    0    4
-11  159 -144
-12  502 -471
-13  557  571
-14  283  277
-15  515  520
-16 1066 1068
-17  349 -335
-18  223  241
-19  322 -317
-20  510  542
-21  191 -186
-22  227 -241
-23  152  148
-24  312  328
-25   95 -107
-26  176  178
-27   84  -80

-28    0   15
-29   68   86
-30   99  -92
-31  199  188
-32  232  235
-33    0  -40
-34  117  102

   H,K= 5, 1
  L FOBS FCAL
 30  148 -169
 29   97   95
 28  121  119
 27    0   30
 26  319  326
 25    0   -6
 24   55   59
 23   38  -54
 22  413 -392
 21    0    5
 20  101   99
 19  115 -125
 18    0  -19
 17  100  113
 16  587  588
 15  167 -160
 14  321 -304
 13   43  -35
 12  199  210
 11    0   17
 10  804  799
  9  215 -214
  8  231  256
  7  206 -208
  6  965 -933
  5  545  531
  4  754  759
  3  171  173
  2  671  687
  1    0  -21
  0  730  746
 -1   74  -77
 -2  937  918
 -3   91  -85
 -4  500  508
 -5  341 -350
 -6  242  235
 -7  399 -412
 -8 1025 1040
 -9  432 -438
-10  593 -602
-11  656  667
-12  321  325
-13  400  414
-14  438  415
-15   71   81
-16  667  680
-17   30   -8
-18  265 -263
-19  244  251
-20   73  -73
-21   34  -61
-22   34  -32
-23  136 -144
-24  270  272
-25  152 -163
-26  189 -180
-27  102   98
-28    0  -28
-29    0   18
-30  180  190
-31    0  -18
-32  198  200
-33    0  -30

   H,K= 7, 1
  L FOBS FCAL
 27    0  -18
 26   44  -22
 25    0  -29
 24  119 -120
 23    0   -2
 22   81   58
 21   56   56
 20  359  351
 19    0  -18
 18    0   15
 17    0   19
 16  253  254
 15   86 -103
 14    0  -21
 13   75   80
 12  602  605
 11  126 -107
 10  448  461
  9  196 -214
  8  141 -136
  7   30   24
  6    0  -30
  5  275  256
  4  865  851
  3   76   62
  2  183  193
  1  415  426
  0  157  163
 -1  257  263
 -2  453 -453
 -3   97  104
 -4  443  424
 -5  366 -369
 -6   40  -71
 -7  111 -116
 -8   64  -70
 -9  175 -182
-10  411 -436
-11   73  -65

-12  727  738
-13  157 -158
-14  261  274
-15  186  198
-16   33  -45
-17  133  127
-18  285 -292
-19   84   73
-20  339  341
-21   72  -93
-22  237  235
-23    0   -5
-24   66  -62
-25    0  -45
-26    0  -30
-27   40  -15
-28  380  381
-29   84  -76
-30   86   96
-31    0    5

   H,K= 9, 1
  L FOBS FCAL
 23   63   71
 22  251  247
 21    0    3
 20  133  144
 19   41  -43
 18  153 -167
 17  114  111
 16   39   36
 15  123  120
 14  143  162
 13   38   38
 12  185  195
 11  168 -158
 10  316 -313
  9   71.  -74
  8   50   43
  7  130 -129
  6  281  286
  5   34  -32
  4  498  515
  3  115 -118
  2  446 -449
  1  164  165
  0   33  -49
 -1  103  104
 -2  280  265
 -3  203  203
 -4  536  548
 -5  103  -87
 -6  451 -442
 -7    0  -41
 -8  293  290
 -9  194 -206
-10  646  645
-11   96 -102
-12  397  404
-13   69  -88
-14  185 -190
-15    0  -12
-16   62   58
-17    0    6
-18  274  265
-19  145  151
-20  288  302
-21    0   13
-22  171 -174
-23   40   25
-24  100   98
-25    0   15
-26  178  187
-27    0   23
-28  115  122
-29   44  -44

   H,K=11, 1
  L FOBS FCAL
 17  125  133
 16  295  308
 15    0   -6
 14   42   13
 13    0    0
 12  149 -173
 11    0   31
 10   70   47
  9   69   58
  8  596  604
  7  203 -190
  6   67   83
  5  128 -135
  4    0   36
  3  137 -158
  2   53  -69
  1  135  133
  0  487  493
 -1  112  119
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24   68  -49    -29  154 -165    16    0   -1    -16    0    7    -14  261  264     7  281 -285    24   63   72     6    0  -15     19    0   18      -8  178  168    14   43  -40    -10   60  -68
23  226 -237    -30   43  -49    15    0   13    -17  277  270    -15  222  169     6  143  156    23  130  129     5  130 -145     18   72   91      -9    0   12    13   42   51    -11   74  -81
22   53   35    -31  108 -105    14    0  -15    -18  255  275    -16   36    3     5    0  -23    22   60   72     4   68   60     17  338  347     -10  121  136    12  237 -221    -12  121 -110
21  312  310                    13  175  203    -19  172  168    -17  407 -419     4   85  103    21  188  185     3  236  235     16  210 -200     -11  226  226    11  161 -161    -13  106 -101
20  343  344     H,K= 6, 4      12   94   89    -20  323  328    -18   52   45     3  104 -110    20   41  -47     2  122  106     15   80   54     -12   41  -28    10  136  136    -14   75   87
19  195  205     L FOBS FCAL    11  223 -223    -21  522 -523    -19  345  324     2   60   77    19  307 -320     1  374  365     14   68  -54     -13   41  -23     9   82  -85    -15    0   29
18  374  367    25  240  244    10   41   58    -22  215  195    -20   54   53     1  279  278    18  233  230     0   86  -89     13  295 -302     -14   59   81     8  295  292
17  385 -384    24   43   33     9    0  -29    -23   87  -73    -21  145  134     0    0  -31    17   56  -49    -1  294 -292     12  435  431     -15  178 -186     7   90   92     H,K= 3, 9
16  236  233    23    0   -2     8   57   53    -24   40   21    -22   68   80    -1   72  -77    16  267  269    -2   93   85     11  126  123     -16  104  108     6  271  274     L FOBS FCAL
15  383 -386    22   59   74     7   98 -100    -25   99  101    -23   40   19    -2   93   85    15  267 -269    -3   54   75     10  209  203     -17  113 -102     5   79   56    12  153  142
14  177  188    21  158 -151     6    0   14    -26   82   75    -24  115  -65    -3  204 -198    14    0  -18    -4  223  217     9  257  260     -18  188  190     4  177 -177    11   98   98
13  631  643    20   69   78     5  360  366    -27  133  134    -25  212 -214    -4  132  128    13  153  168    -5   38   41     8  127 -128     -19  223  231     3  222 -213    10  363  369
12  341  330    19  341 -323     4  160  139    -28   74   41    -26   84   50    -5  172  158    12    0  -26    -6  243  237     7  203 -197     -20  132 -131     2  271  264     9   74   30
11  283 -277    18    0  -17     3   55  -67    -29  368 -371    -27    0   18    -6   42   57    11  461 -468    -7    0   23     6  284  292                     1   55  -44     8  234 -234
10    0  -38    17  360  375     2  196  187                    -28  124  142    -7  310  298    10  409  421    -8    0   24     5  160 -164     H,K= 9, 7        0  135  133     7   85  -89
 9  585  585    16   37   42     1  245 -239     H,K= 3, 5                       -8   42   19     9  231  228    -9  365 -356     4  333  335     L FOBS FCAL    -1   39   38     6   42   30
 8    0  -29    15    0   -8     0  209  221     L FOBS FCAL     H,K= 7, 5       -9   59  -27     8   70   67    -10  209 -214     3  100  106    19    0   -1    -2  110  172     5   73  -92
 7  360 -363    14  161  144    -1  312 -313    27   98  103    22    0  -34    -10   84   95     7  236  250    -11  209  203    2  247  249    18   88   72    -3  170  169     4   42   52
 6   60   26    13  253 -268    -2  114  120    26  105  101    21  185  198    -11  170 -182    -12   56   55     1  304  369     17   98  110    -4  434 -435     3   72  102
 5  440  447    12  184  181    -3  284  282    25  158 -162    20   43  -39    -12   60   46     5  148  140    -13   57   48     0  136 -137    16    0  -40    -5   78  -66     2  256  257
 4  368  398    11  291 -301    -4   76   91    24    0  -13    19  190 -196    -13   96   98     4  106   95    -14   99   98    -1   86  -96     5  106 -102    -4  167  167     1   72   82
 3  533  490    10  200  189    -5  194 -193    23  129 -124    18   72   70    -14   61  -71     3  532 -545    -15  191  204    -2  244  258     4    0  -44     6   39  -37     0    0   -5
 2  619  612     9  581  570    -6   54   51    22    0  -19    17   41  -55    -15  163  160     2  408  415    -16   41  -50    -3  122 -124     3  227 -218    -8  254  235    -1   41  -43
 1  321  330     8  373  394    -7  148 -149    21  175 -177    16    0   20    -16   44  -16     1  128 -113    -17  172 -170    -4   35   48     2  165  173    -9    0    0    -2  138 -133
 0  399  392     7  282  283    -8   66   72    20   38   60    15  120 -128    -17  117 -111     0   93   96    -18  179  173    -5  375 -383     1    0   25    -10  251  246    -3   72  -48
-1  948 -936     6  429  434    -9    0   24    19    0    5    14  142  131                    -1  104   96    -19   43  -38    -6  458  477     0  180  187    -11   81   80    -4  239  221
-2  171 -166     5  658 -656   -10   39   20    18  212  214    13  142  131     H,K= 0, 6      -2   47   39    -20  129  131    -7  329  321    -1  243  252    -12  168 -167    -5   42   41
-3  831  864     4   89   93   -11  344  349    17  506  511    12  179  191     L FOBS FCAL    -3  160  172    -21  131  123    -8  486 -505    -2   85  -72    -13   71  -77    -6  490  488
-4  135  122     3  513 -503   -12   68   44    16  123  131    11    0   -8     0  372 -353    -4   47   54    -22   94  -77    -9  277 -273    -3  104 -101    -14   42   45    -7    0   -1
-5  559  545     2  111 -100   -13  153  139    15  244 -251    10    0  -29    -1  894 -886    -5  491 -496                      -10  137  145    -4    0   -5    -15   78  -83    -8  119 -116
-6  456  479     1  724  727   -14  120  131    14  327  340     9  144  166    -2  514  508    -6  349  355     H,K=10, 6       -11  105  -97    -5  296 -274    -16  220  229    -9   73  -83
-7  298 -299     0  159  170   -15   90  -92    13   34  -13     8  340 -339    -3  224 -211    -7    0  -24     L FOBS FCAL     -12  257  275    -6  275  264    -17   43   51    -10  135 -143
-8  227  217    -1    0   -4   -16  168  158    12  408  414    11   33   53     7  355  364    -8  179  178     9  146 -149    -13   53   64    -7  120  114    -18   43  -32    -11   61  -70
-9  544 -537    -2   86   95   -17   82  -98    11   33   53     7  355  364    -9   76   92    -8   76   71    14  236  225    -14  236  225    -8   85   90    -19   98  102    -12  184  186
-10   62  -58    -3  175 -163   -18   59   51    10  375  375     6  179  177    -10  105 -106    -10  134  129    -9  128  114    -10    0   23    13    0   25
-11    0  -47    -4    0   17   -19  173  184     9 1087 1091     5  448  439    -7  449  457    -11  342  333     0  178  178    -15    0   -5    -10    0   23     H,K= 6, 8
-12  184  200    -5    0   41   -20    0   -9     8  132 -160     4  122  125    -8  368 -380    -12   61  -71     5  148  158    -16   75  -87     0  135  133     L FOBS FCAL    -14  152  163
-13    0   34    -6  247  244   -21   43  -11     7  781 -799     3  178 -185    -9  212 -242    -13  335 -338     4   43    5    -17  107  119    -11   75  -87    13   44  -65    -15   44   40
-14  428  447    -7  881  879   -22    0   22     6    0  -29     2  104  102    -10  188  117    -14  173  194     3   94  -77    -18  107  119    -12   61  -74     12    0   21
-15  279 -285    -8  496  507   -23    0  -60     5   30  -14     1    0  -25    -11   58  -33    -15   52   71     2   94  -77    -19  116 -125    -13  255 -265     11    0  -50     H,K= 5, 9
-16   71   93    -9  138  136                     4  202 -207     0    0   -5    -12  346  336    -16   74   63     1  138 -169    -20  117  125    -14  197  195     10  353  344     L FOBS FCAL
-17  549 -561   -10  204  198     H,K=12, 4       3   42   34    -1   34  -39    -13  186  186    -17  242  242    0    0  -15    -21   74   73                    9  142  126     8  146 -152
-18  220 -222   -11    0  -11     L FOBS FCAL      2   88  -93    -2  146  154    -14  375  378    -18   77   75    -1  162 -163    -23   61   59     H,K= 0, 8       8   95  -81     7   44   -5
-19  447  449   -12   46  -40     8   44  -18     1  248  236    -3  527  512    -15  419  422    -19  233  223    -2  283  276    -24  108  -92     L FOBS FCAL     7   64  -28     6  347  354
-20  120  111   -13  618 -616     7  139  124     0  307  309    -4    0    3    -16   73  -73    -20   39  -21    -3  364  354                     0  328 -321     6   73  -67     5   43   31
-21  146  144   -14   75  -78     6    0  -10    -5  360 -387    -17  369 -375    -21  238 -243    -4   72    5                     H,K= 5, 7        5  156 -153     4  115  106
-22  346  345   -15  514  511     5  179  191    -2  593  608    -6    0  -13    -18  164  163    -22  141  155     H,K= 5, 7       19  161  177     4  254 -216     3    0   13
-23    0   -5   -16  159  182     4  161  170     3  783 -800    -7  130  146    -19  288 -301    -23    0  -31    -6    0   13    18   43   39     3  189 -180     2  178 -167
-24  227  203   -17   35  -41     3   96 -105    -4  546  554    -8   93   76    -20  151  158    -24   60   78    -7  162 -168    17    0    3    -5  113   86     1   43  -40
-25  346 -347   -18   72   95     2  128  131     5    0  -10    -9  178 -166    -21  119  125    -25    0   18    -6  100  84?    16   49   46     2  504  513     0  142 -133
-26   40   17   -19  258 -266     1   60   75    -5  375  374    -22   81   5    -11  225  209                     -9   59   44    15   59   46    -1    0  -25
-27  162  160   -20    0   22     0   42   52    -7  543  542    -11  370 -371    -22  151 -133     H,K= 6, 6      -11  225  209    14  170  170     6   86  -96     -2  219  201
-28  124  115   -21  131 -144    -1  127  124    -6  177  183    -12  125  127    -24  151 -133     L FOBS FCAL    -12    0   38    13   41   43    -9  129 -122     -3   43   18
-29    0   -8   -22   77   44    -2   60   47    -9  370 -371    -13  361 -366    -25  185 -200                    -13    0   38    12   98  102   -10  260  253     -4   82   82    -5    0   17
-30  149  147   -23  263  261    -3   42   38   -10   70   79    -14  104  105    -26  106  132     H,K= 6, 6     -14  117 -106    11  164 -175   -11  112   88     -5   43  -32     -7    0   -1
-31   44  -54   -24  169  172    -4  151  94   -11  125  130    -15  205 -205    -27    0   -1    20    0   43   -15  131 -125    10  118 -137   -12   40   37     -6  263  266     -8  242 -237
                -25   41   -7    -5  206 -227   -11  121 -120   -16  125  125                    19  149 -154   -14  216   76   9   116 -106    14  310  330     -9  177  164    -11   0    0
   H,K= 4, 4    -26   41   27    -6   42   34   -13  149  151   -17   38  -51     H,K= 2, 6      26    0    9    -15  122 -131     8  450 -457   -15  143  143    -10   94  -82    -11  409  410
   L FOBS FCAL  -27   84  -83    -7   60  -27   -14    0   -5   -18  160  146     L FOBS FCAL    25  137 -138    16   71  -76     7  450 -457   -16  270 -270   -10  103 -124
28    0   14    -28    0  -11    -8   85  -68   -15  554  558    -19  163  155    26    0    9    24   60  -64    15  122 -131     6  309  297   -17  144 -150   -11   94  -82     H,K= 0,10
27  106 -108    -29  116 -118    -9  175  174   -16  249  244    -20    0  -23    25  137 -138    23  272 -278    14  120  130     5  135 -161   -14  113  112    13  165 -160    L FOBS FCAL
26    0    0                   -10    0   12   -17  394 -394    -21    0   -4    24   60  -64    22  189  184    13  142 -139     4    1   37   -13  125  107     3    0   13
25   42   24     H,K= 8, 4    -11  121  130   -18  312  315   -22    0   18    23  272 -278    21  286  281    12   78   81     3   42   59   -19  152 -141    14  317  320    1   76  -60
24   71   68     L FOBS FCAL  -12  167  158   -19    0   17   -23  112  110    22  189  184    20  200 -204    11  176 -180    20  200 -204   -14  152 -141    13   92   93    0  334 -331
23  126  113    21    0   16  -13  216 -201   -20    0    5   -24  112  110    21  286  281    19  340 -333    10  147  105     2  276 -273   -15   75   79     1  376 -368    -1   76  -66
22   78   83    20   61  -30  -14   62   80   -21  281 -281   -25   75   61    20   56  -47    18    0   -6   -23  442   44    11   40   40     6  276 -273     -2  271  260
21  491  500    19  268  274  -15    0   14   -22  127  128   -26  124  121    19  203  199    17    0   -6     H,K= 9, 5      10  146 -143    -7   44   32     -3   76  -81
20  225  213    18   59   36  -16   44  -28   -23  442  44    H,K= 9, 5   18  238  252     9   94  154   -24   80  -86    11   40   26    -8  124 -117     0  169  154
19  370 -361    17    0   -6                 -24   80  -86     L FOBS FCAL   17  397  389     8   53   71     9 -1  168 -163     -4  254  293    -5  125   66
18  157  168    16  114  112    H,K= 1, 5   -25  152 -141   17   89   98     9  155 -163     6  172  141   -12  123  137    10   40   26    -1  168 -163
17  132 -137    15  311 -314    L FOBS FCAL  -27  152 -144   16    0   -3     8  166 -187     5  166 -187   -13  337  330    9  118 -136     H,K= 2,10
16  151  160    14   56   58   29    0   49   -29    0  -23   15  156  158    13  100  108     4  100 -124   -14   89   71   -3  133 -107     L FOBS FCAL
15  201  195    13  164  158   28   29  -21               14  167 -168    12  118 -124    3   95  -95   -13  100  108   8   55  -70    2   44  -46
14  137  132    12   93   98   27  316 -318   H,K= 5, 5   13   73  -97    11    0  -34   11  303 -370    -6  258 -256   -10  143  138   1  147 -141
13  492  504    11  299  301   26   42    1   13   73  -97   12    0   11     1  507  507   -8  258 -256   -7  154 -147   -9  293  285   0   44  -40
12  178  135    10  193  191   25   62  -59   10  122  111    9   67  -73     0   71  -58   -9  204 -208   -7  154 -147  -10   92   93   -1   44  -10
11  621 -630     9  322  317   24  120  122    8  277  263    8  133  186    -1  433  431  -11   45 -370   -12  146 -143   -11   92   93
10    0   25     8  277  263   23  209  293    7  281 -296    7  167 -157    -2  271  267   -5  133  186   -11   40   26   -2  274  293
 9    0   10     7  281 -296   22  149  144    6   61  -38    6  391  408   -3  123  107  -12  391  408    1  168 -168   -4  264  252
 8  137  119     6  101  103   21  278 -279    5   62   52    5  131  128   -6  162  170    9  162  170    9  110 -136   -5  106  112
 7   51   48     5   61  -38   20   75  -16    4  367 -378    4  180  191    -9  133 -121   -6   90  -37   -2  118 -136   -6   61  -61
 6  294  291     4    0   37   19  367 -378    3  138  137    3  515 -504   -10    0   34  -15   89   71   -9  110 -136   -7   44   32
 5  372  357     3  374  357   18  138  137    2  235 -231    2  201 -200  -16  151  148    6  306  306   -4  306  306   -8    0   61
 4  400  382     2   77   96   17    0   27   1  105  -99    1  109  112   -2  215  227   -7  212  247   -7  215  227   -9  158 -157
 3  823 -846     1  178  174   16  249  253   17  207  190   -17  114 -107    8  124 -117   -9  119  -93   -8  124 -117
 2    0   -3     0  137  118   15  342  345    2  199  186   -18  203  206   -3  255  250  -16  203  206   -9  158 -157
 1  807 -761    -1  914 -914   14  195  192  -10  146  124    1  190  198  -14  425 -420  -21  190  198   -10    0  -15   11   74  -76
 0  214 -205    -2   48  -28   13   32   32   15  596 -605    0    0   25  -20  609  633  -11  609  633  -22    0   -4    0   93  -92
-1  242  240    -3  201  205   12    0  -23   14  361  385   -1   54  -53  -21   61  -65  -15  140 -140    10  240  240
-2  153  175    -4  167  182   11  639 -637   13  283  277  -18   -2   -3  -23  271 -258  -14  278 -286   -3  119  112
-3  622  590    -5  108  119   10  219  211   12  285  260  -14  157  165  -25  429 -435  -14  278 -286   -2  480  487
-4  465  452    -6  149  162    9  537  553   11  378  374   -6  669  674  -18  215  221    5    0   -0   -3  178  165
-5  314  249    -7  154  147    8  194  108   10  209  209  -16  197  191  -19  114 -125   -9  165 -110   -4   13    0
-6  314  309    -8   60   64    7  102  102    9  225 -234   -7  232  243  -17  197  191  -10  167  136   -5   28   13
-7  233 -258    -9  357 -369    6  104  102    8  111   99  -10  367  373  -19  114 -125  -11    0    0   -6    0    0
-8  341  334   -10   35   14    5  322  315    7  467 -465  -11  486  491  -20  164  147  -11    0    0  11  74  -76
-9  148  148   -11   61   67    4  113 -114    6   33  -10   -9  149  133  -11  486  491  -20  164  147  -20  164  147  10  240  240
```

Table 2. Atomic coordinates in $MgSO_4 \cdot 6H_2O$.

	Atom	x	y	z
	Mg(1)	0	0	0
	Mg(2)	0	0.9425	$\frac{1}{4}$
	S	0.8659	0.4490	0.1241
Sulfate oxygens	O(1)	0.7747	0.5986	0.1361
	O(2)	0.9834	0.4442	0.1665
	O(3)	0.9069	0.4854	0.0692
	O(4)	0.7961	0.2688	0.1211
Water about Mg(1)	O(5)	0.5852	0.7125	0.0483
	O(6)	0.5393	0.3162	0.0640
	O(7)	0.3126	0.5464	0.0231
Water about Mg(2)	O(8)	0.8887	0.1486	0.2820
	O(9)	0.8884	0.7405	0.2811
	O(10)	0.8598	0.9435	0.1799
Water (5)	H(1)	0.643	0.698	0.076
	H(2)	0.539	0.806	0.045
Water (6)	H(3)	0.495	0.232	0.060
	H(4)	0.614	0.340	0.093
Water (7)	H(5)	0.304	0.650	0.055
	H(6)	0.271	0.544	0.014
Water (8)	H(7)	0.840	0.092	0.312
	H(8)	0.937	0.235	0.296
Water (9)	H(9)	0.940	0.651	0.291
	H(10)	0.820	0.733	0.275
Water (10)	H(11)	0.849	0.831	0.164
	H(12)	0.847	0.033	0.164

Estimated standard deviations are: for Mg(2), $\sigma(y) = 0.0002$;

for S, $\sigma(x) = 0.00006$, $\sigma(y) = 0.00008$, $\sigma(z) = 0.00002$; for

oxygen, $\sigma(x) = 0.0002$, $\sigma(y) = 0.0003$, $\sigma(z) = 0.0001$.

Table 3. Anisotropic thermal parameters (each multiplied by 10^4) and root mean square amplitudes of vibration in $MgSO_4 \cdot 6H_2O$.

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	u, Å
Mg(1)	45	86	8.7	2	-1	-3	0.16
Mg(2)	46	72	7.8	(0)[a]	2	(0)[a]	0.15
S	46	72	9.8	0	-1	-1	0.16
O(1)	64	103	12.4	23	4	-1	0.18
O(2)	79	107	17.6	10	-17	-5	0.21
O(3)	81	120	13.2	0	10	-1	0.19
O(4)	68	85	15.4	-19	-4	6	0.19
O(5)	67	129	13.6	17	-5	-12	0.19
O(6)	87	175	19.0	-51	-15	26	0.23
O(7)	54	216	16.7	7	3	-21	0.21
O(8)	67	94	12.6	-2	6	-7	0.18
O(9)	61	113	16.0	-5	5	12	0.19
O(10)	103	103	10.7	-8	-8	2	0.20

[a]Zero because of 2-fold axis.

Table 4. Isotropic thermal parameters for H in $MgSO_4 \cdot 6H_2O$.

Atom	B, Å^2	Atom	B, Å^2	Atom	B, Å^2
H(1)	1.9	H(5)	5.6	H(9)	5.9
H(2)	5.2	H(6)	1.9	H(10)	3.1
H(3)	1.9	H(7)	4.3	H(11)	2.6
H(4)	5.7	H(8)	1.3	H(12)	2.0

Table 5. Interatomic distances and standard deviations in $MgSO_4 \cdot 6H_2O$.

Atoms	Distance	σ
Water octahedron about Mg(1)		
Mg(1) - 2 O(5)	2.046 Å	0.002 Å
- 2 O(6)	2.044	0.003
- 2 O(7)	2.080	0.002
O(5) - O(6)	2.930	0.004
- O(6)	2.854	0.004
- O(7)	2.986	0.003
- O(7)	2.847	0.003
O(6) - O(7)	2.889	0.004
- O(7)	2.943	0.003
Water octahedron about Mg(2)		
Mg(2) - 2 O(8)	2.083	0.002
- 2 O(9)	2.054	0.002
- 2 O(10)	2.059	0.003
O(8) - O(8)	2.917	0.004
- O(9)	2.943	0.003
- O(10)	2.876	0.003
- O(10)	2.973	0.003
O(9) - O(9)	2.896	0.003
- O(10)	2.850	0.003
- O(10)	2.972	0.003

Table 5 (cont.)

Sulfate ion

S	- O(1)	1.476	0.002
	- O(2)	1.460	0.003
	- O(3)	1.482	0.002
	- O(4)	1.475	0.002

Hydrogen bonds

O(5) - H(1) -- O(1)	2.785	0.003
O(5) - H(2) -- O(3)	2.766	0.003
O(6) - H(3) -- O(3)	2.747	0.003
O(6) - H(4) -- O(4)	2.785	0.004
O(7) - H(5) -- O(4)	2.906	0.003
O(7) - H(6) -- O(3)	2.934	0.004
O(8) - H(7) -- O(1)	2.796	0.003
O(8) - H(8) -- O(2)	2.707	0.003
O(9) - H(9) -- O(2)	2.721	0.003
O(9) - H(10) -- O(8)	3.068	0.003
O(10) - H(11) -- O(1)	2.794	0.003
O(10) - H(12) -- O(4)	2.777	0.003

(Figure caption)

Fig. 1. Electron density sections through hydrogen atoms, with other atoms subtracted out. Contour interval approx. 0.1 e/Å^3; zero and negative contours omitted. Grid lines and contours were reproduced photographically from cathode ray tube of computer.

Hydrogen in $MgSO_4 \cdot 6H_2O$

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