dihedral angle of 5·3 (3)°, such that the two halves of the chelate system are each more planar than the ligand as a whole. The carboxylate and peptide groups have retained their expected planarity, and the steric requirements associated with this retention of planarity in these groups and the formation of the Cu–N(8) bond probably precludes a more coplanar arrangement of the complete ligand framework. The copper atom, as in the trihydrate, is significantly displaced from the equatorial plane about 0·13 Å toward the loosely bound water molecule. In this respect, it is interesting that the copper lies nearly in the plane of the carboxylate half of the ligand (deviation 0·006 Å), while it lies substantially out of the plane of the peptide half of the ligand (deviation 0·091 Å).

The crystal packing in $[(\mathrm{glygly})\mathrm{Cu^{II}}].2\mathrm{H_2O}$ is dominated by a series of intermolecular hydrogen bonds involving all of the acidic protons in the complex (Table 4). As noted in the structure of glycylglycinatocopper(II) trihydrate (Strandberg, Lindqvist & Rosenstein, 1961), the strongly coordinated water molecule $[\mathrm{O(1)H_2}]$ forms two strong hydrogen bonds while the loosely bound water molecule $[\mathrm{O(2)H_2}]$ is involved in a weaker set of hydrogen bonds.

There also seems to be some contribution to packing stability owing to the presence of weak dimerization of the complexes about centers of symmetry $(\frac{1}{2}-x,\frac{1}{2}-y,-z)$ involving Cu and O(1) [Cu–O(1) distance 3·083 (1) Å]. This weak dimerization extends, in a very qualitative sense, the coordination of the copper from five to six.

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Refinement of the Crystal Structure of $\mathrm{Fe_3BO_6}$

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$\mathrm{Fe_3BO_6}$ is orthorhombic with $a=10·048$ (2), $b=8·531$ (2), $c=4·466$ (1) Å, space group *Pnma* (No. 62), $Z=4$. It is isostructural with the mineral norbergite, $\mathrm{Mg_3SiO_4(OH)_2}$ [White, Miller & Nielsen, *Acta Cryst.* (1965). **19**, 1060–1061]. X-ray intensities were collected on a four-circle automatic single-crystal diffractometer with Zr-filtered Mo $K\alpha$ radiation. The structure has been refined from 1241 reflexions to $R=0·031$. Both structurally independent iron atoms are surrounded by six oxygen atoms forming distorted octahedra. Boron is exclusively tetrahedrally coordinated by oxygen.

Introduction

The crystal structure of $\mathrm{Fe_3BO_6}$ (White, Miller & Nielsen, 1965) has been found to be isotypic with that of the mineral norbergite, $\mathrm{Mg_3SiO_4(OH)_2}$ (Taylor & West, 1929). In the paper of White *et al.* (1965) only mean bond distances Fe–O (2·04 Å) and B–O (1·47 Å) are reported because of the limited accuracy of the oxygen coordinates which resulted from an overlap of the oxygen positions in an *h*0*l* electron density projection. Isotropic temperature factors are given: 0·52 Å^2 for the iron atoms, 0·90 Å^2 for the lighter atoms. $\mathrm{Fe_3BO_6}$ is a so-called easy-axis ferrimagnet with a Néel temperature of 235 °C. In order to study the optical and magnetic properties of this substance accurate structural information is required. We therefore decided to perform a full-matrix least-squares refinement of the Fe_3BO_6 crystal structure including anisotropic thermal coefficients for all atoms.

Polycrystalline Fe_3BO_6 was synthesized by reacting a mixture of Fe_2O_3 and B_2O_3 in a molar ratio of 1:1·5 at 850°C and subsequently leaching off the excess B_2O_3. Prismatic single crystals were obtained by chemical transport with chlorine in evacuated quartz ampoules (Diehl, 1975).

Crystal data

Lattice constants of Fe_3BO_6 were obtained by least-squares refinement from Guinier powder photographs

Table 1. *Crystal data for* Fe_3BO_6

	Present study	White *et al.* (1965)			
a	10·048 (2) Å	10·05 (1) Å	$\varrho_{exp}=4{\cdot}78\ (2)\ \mathrm{g\ cm^{-3}}$	$\varrho_x=4{\cdot}76\ \mathrm{g\ cm^{-3}}$	$Z=4$
b	8·531 (2)	8·55 (1)	F.W. 274·35	$F(000)=524$	
c	4·466 (1)	4·470 (5)			
V	382·82 Å³	384·1 Å³			

Table 2. *Observed and calculated structure factors*

```
    H,0,0
 2  129   121
 4 1223 -1346
 6  808  -746
 8  847   783
10  259   245
12  680  -648
14  407   392
16   18*  -32
18  392  -405

    H,1,0
 2   73   -30
 4  731   647
 6  603   558
 8  415  -368
10  618  -562
12  307   275
14  517   497
16   48    55
18  365  -360

    H,2,0
 0   44    37
 2   72   -57
 4  606  -523
 6 1030  1022
 8  990   954
10  629  -581
12  936  -940
14  103  -101
16  624   657
18   64    60

    H,3,0
 2 1240 -1505
 4  524  -459
 6 1576  1949
 8  136   111
10  225  -204
12   16*  -15
14  357   362
16  285  -284

    H,4,0
 0  640   578
 2  384   356
 4  567  -526
 6  788  -754
 8  243  -210
10  371   340
12  548   534
14  129   115
16  234  -225

    H,5,0
 2  827  -812
 4  827   806
 6  756   751
 8  242  -210
10 1000 -1025
12  433   394
14  752   753
16  101   -93

    H,6,0
 0 1887 -2283
 2  263  -247
 4  893   896
 6  555   526
 8  452  -432
10  207  -190
12  369   341
14  254  -244
16   67    71

    H,7,0
 2  776  -775
 4  348  -323
 6  260   239
 8  243   231
10   73   -68
12  115   -93
14   55   -52
16  190  -187

    H,8,0
 0  231  -197
 2   75   -60
 4  721   729
 6  706  -687
 8  906  -914
10  559   553
12  815   835
14  144   141

    H,9,0
 2  642   626
 4  221   201
 6  761  -660
 8  244  -247
10   77   -45
12   18*   35
14   56   -73

    H,10,0
 0  891  -905
 2  129  -119
 4  738   732
 6  388   385
 8  282  -282
10  175  -168
12   45   -28

    H,11,0
 2  522   505
 4  513  -504
 6  435  -428
 8   87    75
10  786   815
12  337  -333

    H,12,0
 0  653   625
 2  244   253
 4  215  -211
 6  345  -334
 8   18*  -33
10  128   135

    H,13,0
 2  715   725
 4  234   219
 6  528  -540
 8   41   -38

    H,14,0
 0   18*   25
 2   66    51
 4  364  -355
 6  420   422

    H,15,0
 2   59   -54
 4   95   -84

    H,0,1
 1  549   478
 2   92   -60
 3 1089 -1094
 4 1092 -1071
 5  294  -242
 6   56   -42
 7 1407  1478
 8  344  -301
 9  426   397
10  302   285
11 1071 -1087
12  213  -200
13  619  -618
14  171  -176
15  623   651
16   18*   25
17  491   498
18   19*   52

    H,1,1
 0   87    86
 1  837  -771
 2  330   276
 3 1231 -1259
 4   47    30
 5  293   242
 6  118    95
 7  717   652
 8  124   101
 9  136   131
10  100   -91
11  104   -35
12   69   -54
13   61   -68
14  138  -128
15  118  -114
16   18*   -1
17  249  -255

    H,2,1
 1 1477  1573
 2  374   312
 3  956  -892
 4  191   160
 5 1152 -1157
 6  338  -308
 7  147   112
 8  243  -221
 9  582   540
10  162  -143
11  132  -124
12   40*  -18
13   17*   22
14   17*   33
15  251   250
16  118   119
17  118  -134

    H,3,1
 0  196   164
 1  799  -707
 2  970   898
 3  156  -145
 4  255  -232
 5 1162  1172
 6  167   137
 7  326   303
 8   96    88
 9 1129 -1137
10  264  -224
11  505  -493
12   62   -52
13  820   838
14  197  -194
15  480   488
16   88    97
17  412  -410

    H,4,1
 1  796  -726
 2  330  -283
 3  169   149
 4   48    37
 5  573   522
 6   47   -18
 7  490   452
 8  380   351
 9  223  -194
10  115    93
11  308  -290
12   61    48
13  226  -226
14   73   -75
15   87    90
16  134  -135
17  265   271

    H,5,1
 0  534   495
 1 1116 -1111
 2  103   -97
 3 1313 -1371
 4  106    79
 5  671   617
 6   37*   19
 7  867   829
 8  114    94
 9  261  -237
10   16*    3
11  238  -235
12  215  -219
13  231   232
14   18*    8
15   18*   12
16   67   -70
17  450  -468

    H,6,1
 1  134  -118
 2  107    94
 3  591   548
 4  518   459
 5   49    47
 6  142   122
 7  981  -959
 8  150   125
 9  200  -204
10  168  -154
11  755   767
12  137   141
13  466   473
14  130   140
15  465  -470
16   19*    8

    H,7,1
 0  116    97
 1   54   -49
 2  205  -189
 3  380   343
 4   15*  -13
 5  368   343
 6   57   -48
 7  131  -115
 8   32*  -18
 9  548  -527
10   92    79
11  213  -216
12   61   -46
13  418   419
14  161   158
15  256   265
16   19*   10

    H,8,1
 1 1073 -1081
 2  153  -132
 3  756   736
 4  205  -193
 5  887   895
 6  361   349
 7  292  -280
 8   17*   19
 9  516  -504
10  179   177
11  257   256
12   31*  -29
13   63    73
14   18*   -9
15  353  -358

    H,9,1
 0  186  -184
 1  149   136
 2  204  -177
 3  193  -180
 4   16*  -16
 5  446  -419
 6   16*  -47
 7   17*   -7
 8   17*    6
 9  556   546
10  110    85
11  235   241
12   95   100
13  469  -465
14   67    76

    H,10,1
 1   32*   25
 2   61    60
 3  300   293
 4   17*   20
 5   17*   -7
 6   54    46
 7  647  -639
 8  215  -207
 9  134   -99
10  114  -129
11  530   538
12   55   -18
13  328   345

    H,11,1
 0  333  -322
 1  630   635
 2  117   103
 3  792   791
 4   64   -66
 5  406  -397
 6   25*    9
 7  552  -551
 8   47   -53
 9  208   198
10   55   -39
11  207   205
12  232   232

    H,12,1
 1  207  -207
 2  138  -145
 3   39*  -12
 4  161  -142
 5  189   188
 6  148  -148
 7  340   335
 8   18*   22
 9   58   -51
10   19*  -10
11  298  -291

    H,13,1
 0   18*  -17
 1  259   260
 2   18*   29
 3   44    27
 4   85    92
 5  441  -455
 6   18*   11
 7  103  -107
 8   19*  -41
 9  578   604

    H,14,1
 1  554   562
 2   52    63
 3  368  -369
 4  134   125
 5  496  -504
 6  231  -232
 7  122   122

    H,15,1
 0  160   155
 1  158   169
 2   49    31
 3  322   345

    H,0,2
 0 1038  1000
 1  591   514
 2  371   315
 3  437   394
 4 1801 -2109
 5  170  -153
 6  288  -265
 7  358  -334
 8  975   934
 9   15*  -32
10   70    52
11   87    87
12  180  -163
13  113  -114
14  172   154
15   74    88
16  520   543
17   91   104

    H,1,2
 1   57   -14
 2  124    95
 3   62   -47
 4  724   650
 5   97    83
 6  220   182
 7  209   175
 8  677  -628
 9   42    20
10  789  -767
11  198  -194
12  152   141
13   81   -81
14  579   585
15   70    80
16  107   126
17   76    85

    H,2,2
 0  414   372
 1   11*    7
 2  581  -506
 3  126  -112
 4  686  -619
 5  334  -290
 6  520   467
 7   14*    4
 8  797   766
 9  302   284
10  347  -313
11   92    79
12  677  -677
13  183  -167
14  189   174
15  163  -160
16  557   571
17   79    91

    H,3,2
 1  453   409
 2 2168 -2231
 3  167  -155
 4  508  -455
 5  397  -357
 6  644   609
 7  177   169
 8  430   395
 9   90    82
10  671  -641
11   16*  -28
12   81    80
13   58   -67
14  184   173
15   54    58
16  362  -370
17   49   -19

    H,4,2
 0  801   714
 1  123    99
 2  213   185
 3  270   244
 4   72   -61
 5  135   118
 6  418  -385
 7  100   -88
 8  198  -182
 9  152  -145
10  291   266
11   17*  -32
12  159   152
13   86    94
14   51    37
15  103   108
16  303  -310

    H,5,2
 1   95    85
 2  448  -403
 3  195  -186
 4  427   383
 5   47    36
 6  841   810
 7  213   198
 8  809  -764
 9   93   101
10  947  -929
11  261  -261
12  170   156
13   96  -104
14  781   820
15  141   142
16   67    81

    H,6,2
 0  859  -829
 1  329  -298
 2  138  -117
 3  286  -257
 4 1233  1184
 5   95    76
 6  279   262
 7  239   231
 8  575  -538
 9   63    53
10  134  -124
11   46   -45
12   63    58
13   76    60
14  152  -154
15   78   -80
16  328  -341

    H,7,2
 1  135   132
 2  707  -670
 3  100   -78
 4  458  -430
 5  159  -155
 6  489   463
 7   16*  -26
 8  364   327
 9   41    55
10  211   196
11   46    63
12   18*  -32
13   31*   28
14  166  -152
15   19*   10

    H,8,2
 0  656  -623
 1   46   -13
 2  452   431
 3   41   -21
 4  592   548
 5  254   249
 6  340  -326
 7   40    28
 8  743  -738
 9  271  -266
10  221   204
11   44   -73
12  794   813
13  156   159
14  303  -298

    H,9,2
 1  185  -181
 2  878   840
 3   53    43
 4  421   403
 5  175   163
 6  236  -236
 7   17*  -35
 8  230  -214
 9   39*  -42
10  164   142
11   18*  -16
12   18*   39
13   90    24
14   64    71

    H,10,2
 0  851  -833
 1  170  -160
 2   91  -109
 3  209  -199
 4  523   519
 5   17*    8
 6  206   190
 7  113   117
 8  286  -282
 9   18*   23
10  127  -103
11   18*  -25
12  203   193
13   19*   -3

    H,11,2
 1   62   -59
 2  270   262
 3  136   135
 4  186  -176
 5   18*  -26
 6  613  -627
 7  127  -128
 8  575   595
 9   88   -90
10  659   665
11  210   211

    H,12,2
 0  259   253
 1  102    87
 2   31*  -27
 3   98   110
 4  303  -279
 5   18*    8
 6  234  -235
 7  111  -105
 8   18*   24
 9   48   -91
10  223   218

    H,13,2
 1  136  -127
 2  726   749
 3   98    97
 4  177   181
 5  157   157
 6  512  -516
 7   52   -53
 8  240  -242

    H,14,2
 0  303   303
 1   18*   -7
 2  278  -273
 3   19*    5
 4  254  -254
 5  146  -146
 6  188   194

    H,0,3
 1  346   302
 2  227   219
 3  885  -831
 4  386   322
 5  304  -281
 6  508  -478
 7 1039  1017
 8  681   637
 9  371   355
10  233   230
11  838  -828
12   54    68
13  407  -418
14  309  -327
15  569   582
16   19*  -36

    H,1,3
 0  471   438
 1  814  -758
 2  110   101
 3  881  -815
 4  518  -475
 5   19*   27
 6  327  -295
 7  521   478
 8   54*   21
 9  147   139
10  178   177
11   87   -87
12  175   168
13   18*   15
14  118   104
15  194  -202
16   56    18

    H,2,3
 1  960   906
 2  598   555
 3  639  -591
 4   82    79
 5  916  -870
 6  112   -82
 7  262   236
 8  131  -128
 9  369   349
10  279   267
11   77   -66
12   17*  -20
13   59   -38
14   18*  -13
15  246   243
16   19*   52

    H,3,3
 0  198   175
 1  507  -464
 2  810  -729
 3  154  -130
 4   76   -76
 5  894   836
 6  224   209
 7  163   144
 8  397   376
 9  983  -977
10   17*  -22
11  454  -451
12  482  -486
13  696   691
14  297   291
15  463   472
16  215   219

    H,4,3
 1  376  -354
 2  196  -186
 3   60    41
 4  212  -201
 5  515   475
 6   48    10
 7  169   161
 8   39*   48
 9   65   -46
10   17*  -16
11  313  -301
12   18*  -58
13  133  -134
14  108  -111
15   49*   57
16   19*   22

    H,5,3
 0  342   325
 1  958  -895
 2  274   253
 3 1067 -1017
 4  712  -675
 5  413   385
 6  213  -192
 7  678   646
 8  105   191
 9  178  -177
10   59    56
11  229  -211
12  146   158
13  240   226
14   18*   28
15   19*  -47

    H,6,3
 1  100   -91
 2  127  -121
 3  526   491
 4  187  -152
 5   82    74
 6  244   239
 7  691  -662
 8  479  -443
 9  239  -230
10  201  -193
11  616   612
12   54   -49
13  305   311
14  239   256
15  435  -433

    H,7,3
 0  136  -138
 1  139   119
 2   49   -55
 3  227   205
 4   99   103
 5  404   385
 6  259   251
 7   88   -83
 8  163   161
 9  483  -471
10  195  -199
11  123  -122
12  238  -242
13  294   292
14   79   -84

    H,8,3
 1  789  -776
 2  540  -523
 3  606   586
 4   59    54
 5  729   706
 6   17*   28
 7  318  -297
 8  201   192
 9  412  -403
10  379  -368
11  220   219
12   55    75
13  111   105

    H,9,3
 0  112   114
 1   17*   34
 2  353   319
 3   92   -85
 4   29    16
 5  368  -355
 6  152  -154
 7   52    56
 8  214  -207
 9  545   537
10   65    56
11  207   218
12  238   233
13  352  -352

    H,10,3
 1   87   -72
 2   17*  -30
 3  288   277
 4  116   114
 5   70   -57
 6  160   159
 7  423  -423
 8   72   -82
 9  145  -159
10  109  -108
11  509   514
12   81    74

    H,11,3
 0  145  -151
 1  551   561
 2  227  -217
 3  681   684
 4  507   501
 5  304  -298
 6  118   111
 7  476  -475
 8  163  -171
 9  163   168
10   26*   -8

    H,12,3
 1  157  -166
 2   18*    8
 3   92   -85
 4   18*   24
 5  171   178
 6   18*   57
 7  187   181
 8  199   192
 9   37*   33

    H,13,3
 0   84   -91
 1  152   154
 2  106   106
 3   37*   39
 4   18*   10
 5  454  -464
 6  176  -172
 7   93  -104

    H,14,3
 1  436   446
 2  310   320
 3  329  -325

    H,0,4
 0 1745  1806
 1  528   517
 2  141   143
 3  588   566
 4  842  -840
 5  198  -189
 6  351  -328
 7  335  -332
 8  491   485
 9  150   166
10  103   111
11  173   176
12  447  -438
13  124  -114
14  238   234
15   19*   11

    H,1,4
 1  197  -180
 2   50    13
 3  228  -221
 4  297   277
 5  287   274
 6  376   370
 7  244   232
 8  288  -278
 9  141   133
10  315  -302
11  380  -378
12  157   136
13  148  -140
14  368   363
15  161   166

    H,2,4
 0  201   192
 1   14*   12
 2  155  -145
 3   78    72
 4  289  -265
 5  329  -320
 6  518   490
 7   39*   25
 8  564   568
 9  414   404
10  347  -332
11   89   100
12  704  -686
13  241  -230
14   18*  -24
15  238  -248

    H,3,4
 1  539   514
 2  930  -899
 3  382  -367
 4  195  -185
 5  585  -566
 6 1017   994
 7   88    93
 8  130   120
 9  222   215
10  200  -196
11   30*   27
12   25*   42
13   18*   30
14  268   270
15  192   197

    H,4,4
 0  229   219
 1  248   232
 2  245   235
 3   78    65
 4  309  -302
 5  120   126
 6  395  -377
 7  169  -158
 8  131  -118
 9  243  -237
10  192   179
11   25*   22
12  422   411
13   18*   47
14   18*   30
15  185   203

    H,5,4
 1   15*  -20
 2  454  -429
 3  299  -295
 4  400   387
 5   95    76
 6  542   537
 7  399   395
 8  176  -176
 9  168   163
10  630  -636
11  433  -444
12  248   238
13  203  -206
14  558   567

    H,6,4
 0 1186 -1159
 1  404  -383
 2  200  -191
 3  385  -376
 4  591   572
 5   75    82
 6  294   278
 7  241   237
 8  313  -291
 9   77   -79
10   84   -90
11  144  -142
12  241   234
13   61    73
14  135  -134

    H,7,4
 1  311   300
 2  502  -487
 3   17*    8
 4  128  -136
 5  386  -366
 6   90    89
 7   30*   -7
 8  214   203
 9   18*  -20
10  133  -120
11  176   178
12   18*  -19
13   19*   40

    H,8,4
 0  235  -223
 1  126  -129
 2   17*   14
 3   90   -81
 4  453   449
 5  300   301
 6  428  -421
 7   18*   41
 8  650  -636
 9  380  -379
10  366   371
11  141  -155
12  632   649

    H,9,4
 1  265  -255
 2  423   415
 3  136   134
 4   74    75
 5  367   370
 6  422  -403
 7   18*  -12
 8  210  -221
 9   84   -79
10   18*   15
11  131  -131

    H,10,4
 0  534  -544
 1  293  -287
 2  142  -130
 3  167  -174
 4  548   548
 5   70    60
 6  241   235
 7  229   225
 8  218  -220
 9   52*   43
10   85   -89

    H,11,4
 1   18*   -6
 2  360   357
 3  201   204
 4  326  -326
 5   18*  -14
 6  374  -367
 7  333  -340
 8   74    72
 9  113  -114

    H,12,4
 0  406   385
 1  185   188
 2  230   232
 3  134   131
 4  171  -170
 5   69    71
 6  219  -216
 7  103   -91

    H,13,4
 1  290  -309
 2  546   554
 3  113   116

    H,0,5
 1  258   255
 2  179   182
 3  487  -466
 4  555  -534
 5   16*   12
 6  335  -335
 7  616   610
 8   82    89
 9  298   299
10  553   573
11  690  -712
12  257  -259
13  393  -406
14  467  -498

    H,1,5
 0  468   454
 1  308  -278
 2  182   188
 3  407  -394
 4  295  -300
 5  150   143
 6  169  -171
 7  302   298
 8  118   113
 9   63    28
10   64    53
11   51*  -36
12   41*   23
13   18*   14
14   32*  -34

    H,2,5
 1  643   627
 2  585   582
 3  332  -316
 4  209   206
 5  534  -533
 6  402  -400
 7  101    91
 8  256  -257
 9  232   224
10   18*  -17
11   60   -60
12   18*    2
13   45*   -7
14   37*    7

    H,3,5
 0  165   172
 1  243  -223
 2  164   134
 3  193  -184
 4  300  -290
 5  588   585
 6  329   317
 7  234   229
 8  397   395
 9  543  -540
10  336  -323
11  191  -187
12  398  -424
13  427   434

    H,4,5
 1  352  -330
 2  334  -326
 3   57    34
 4  151  -150
 5  232   228
 6   54    44
 7  245   235
 8  360   362
 9   84   -74
10  156   153
11  156  -166
12   18*  -23
13  132  -132

    H,5,5
 0  737   742
 1  529  -528
 2   60    56
 3  518  -499
 4  380  -390
 5  324   318
 6  145  -153
 7  421   425
 8  252   252
 9  160  -160
10   66    71
11  191  -194
12  202  -217
13  221   240

    H,6,5
 1  123  -113
 2   42*  -39
 3  292   276
 4  404   403
 5   83   -55
 6  262   264
 7  482  -467
 8  106  -115
 9  181  -179
10  403  -410
11  537   541
12  197   201

    H,7,5
 0   67   -62
 1   67   -68
 2  193  -191
 3   97    88
 4   43*   25
 5  144   145
 6  144   166
 7   72   -55
 8  108   115
 9  273  -283
10   81   -79
11  145  -141

    H,8,5
 1  584  -581
 2  511  -521
 3  345   336
 4  239  -226
 5  567   567
 6  513   530
 7  231  -235
 8  113   113
 9  227  -228
10   19*  -19

    H,9,5
 0   69   -77
 1   67    56
 2   65   -30
 3   70   -40
 4   48    30
 5  284  -272
 6  209  -214
 7   69   -66
 8  169  -175
 9  271   266

    H,10,5
 1   52*   34
 2   18*   24
 3  209   211
 4  147   153
 5   18*    7
 6  142   152
 7  405  -408
 8  290  -299

    H,11,5
 0  620  -641
 1  433   437
 2   18*   13
 3  357   372
 4  287   282
 5  258  -251

    H,12,5
 1   93  -105

    H,0,6
 0  338   341
 1  486   507
 2  141   151
 3  551   570
 4  763  -764
 5  254  -265
 6   71   -55
 7  465  -478
 8  406   420
 9   18*  -25
10   37*  -39
11   87   102
12   64   -54

    H,1,6
 1   51   -56
 2   17*    6
 3   57   -30
 4  137   145
 5   18*   38
 6  145   139
 7  343   362
 8  281  -292
 9   58   -63
10  299  -302
11  264  -266
12   45    25

    H,2,6
 0  216   220
 1  118   106
 2  195  -213
 3  194  -186
 4  215  -210
 5  338  -346
 6  159   136
 7   65   -20
 8  318   339
 9  428   450
10  112   -73
11  191   208

    H,3,6
 1  529   539
 2  762  -766
 3  275  -273
 4  123   -85
 5  423  -429
 6  177   179
 7  149   171
 8  193   202
 9  240   235
10  406  -415
11  131  -143

    H,4,6
 0  171   170
 1   43*   43
 2  126   112
 3  386   394
 4   64   -49
 5  130   116
 6  114  -113
 7  123  -119
 8   86   -77
 9  212  -223
10   89    95
11  117  -121

    H,5,6
 1  114   104
 2  190  -188
 3  214  -224
 4   71    71
 5   31*  -37
 6  417   420
 7  417   430
 8  307  -402
 9   76    93
10  411  -415

    H,6,6
 0  243  -243
 1  346  -351
 2   88   -92
 3  463  -471
 4  550   544
 5  169   183
 6   71    72
 7  375   385
 8  268  -276
 9   76    74

    H,7,6
 1  224   220
 2  288  -293
 3  184  -186
 4  141  -131
 5  169  -174
 6  186   184
 7  138  -142
 8  172   176

    H,8,6
 0  394  -387
 1  117  -120
 2  260   264
 3   18*   32
 4  245   253
 5  393   402
 6  127  -112
 7   72    78

    H,9,6
 1  322  -342
 2  402   405
 3  147   140
 4  126   148
 5  220   220

    H,10,6
 0  357  -370
 1  191  -210
 2   81   -84
 3  440  -456

    H,0,7
 1  134   125
 2  202   222
 3  301  -316
 4   87   -89
 5  176  -180
 6  353  -383
 7  314   320
 8  421   429
 9  177   190

    H,1,7
 0  458   475
 1  299  -316
 2   18*   48
 3  197  -194
 4  326  -332
 5   37*  -56
 6  252  -269
 7  147   152
 8   74    67

    H,2,7
 1  192   198
 2  499   522
 3  172  -171
 4  173   194
 5  291  -307
 6  270  -283
 7  290   208
 8  111  -115

    H,3,7
 0  116   120
 1  143  -155
 2  292  -292
 3  103  -103
 4  223  -232
 5  281   285
 6  274   276
 7   32*   31
 8  414   441

    H,4,7
 1   18*  -16
 2  209  -213
 3   49*  -34
 4  240  -254
 5  195   203
 6   98    98
 7   81   -76

    H,5,7
 0  524   558
 1  358  -361
 2   96   104
 3  342  -351
 4  535  -564
 5  117   116
 6  291  -212

    H,6,7
 1   18*  -61
 2  114  -131
 3  246   245
 4   93   105
 5   96    86

    H,7,7
 0  181  -188
 1  134   129
 2   19*  -39
 3   19*    7

    H,0,8
 0  503   527
 1  333   357
 2   19*    9

    H,1,8
 1  143  -158
 2   49*  -32

    H,2,8
 0  111   121
```

taken with Ni-filtered Cu $K\alpha_1$ radiation ($\lambda = 1{\cdot}5405$ Å) using NaCl ($a = 5{\cdot}6399$ Å) as an internal standard. They are listed in Table 1. The cell parameters reported by White *et al.* (1965) are given for comparison. Other relevant items are also given in Table 1.

Single crystals are black; the Moh hardness is about 6. Systematic extinctions are $0kl$: $k+l=2n+1$ and $hk0$: $h=2n+1$, indicating the space group to be either $Pnma$ or $Pn2_1a$. From reflexion statistics using normalized structure factors (Karle & Karle, 1966) the structure was found to be centrosymmetric. Refinement was therefore carried out in the centrosymmetric space group $Pnma$. Fig. 1 gives an idealized drawing of the observed crystal morphology, [001] being the prism axis.

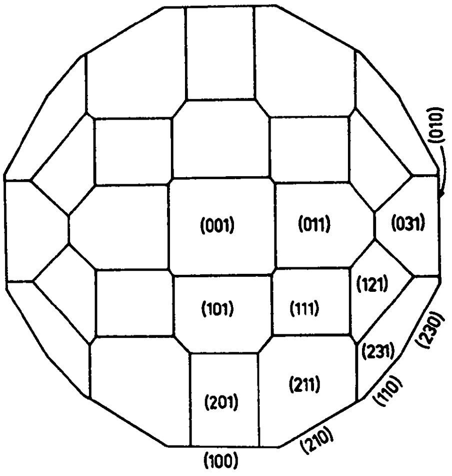

Fig. 1. Observed morphology of an Fe_3BO_6 single crystal (idealized).

Experimental

For X-ray intensity data collection a single crystal was ground to a sphere of radius $R = 208{\cdot}5$ μm in an air-operated crystal grinder. The sphere was mounted on a Nonius CAD-4 four-circle automatic single-crystal diffractometer with κ geometry. A total of 1241 independent reflexions up to $(\sin\theta)/\lambda = 0{\cdot}91$ Å^{-1} was measured with a scintillation counter (NaI:Tl) using Zr-filtered monochromatized Mo $K\alpha$ radiation ($\lambda = 0{\cdot}7107$ Å). Slow drift of the intensity of the primary beam was corrected by measuring the intensities of the standard reflexions 232, 042, and 123 every 20 measurements. The background-corrected integrated intensities were further corrected for Lorentz, polarization and absorption factors. The linear absorption coefficient μ for Mo $K\alpha$ radiation was found to be $114{\cdot}3$ cm^{-1} giving rise to a μR of 2·4. Absorption correction factors as a function of θ were taken from *International Tables for X-ray Crystallography* (1959). Standard deviations were obtained only from counting statistics. 119 reflexions had an integrated intensity less than three times their standard deviation and were omitted as zeros from the structure refinement.

Table 3. *Atomic positional parameters*

		x	y	z
$Fe^{3+}(1)$	8(d)	0·12835 (4)	0·06466 (5)	0·98047 (9)
$Fe^{3+}(2)$	4(c)	0·41246 (6)	$\frac{1}{4}$	0·9749 (1)
B^{3+}	4(c)	0·2177 (4)	$\frac{1}{4}$	0·4106 (9)
$O^{2-}(1)$	8(d)	0·2856 (2)	0·1114 (2)	0·2647 (5)
$O^{2-}(2)$	8(d)	0·4654 (2)	0·0823 (2)	0·7351 (5)
$O^{2-}(3)$	4(c)	0·0813 (3)	$\frac{1}{4}$	0·2736 (7)
$O^{2-}(4)$	4(c)	0·2186 (3)	$\frac{1}{4}$	0·7333 (7)

Table 4. *Anisotropic temperature coefficients* $\times 10^2$

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
$Fe^{3+}(1)$	51 (2)	49 (2)	57 (2)	−1 (1)	−1 (1)	−5 (1)
$Fe^{3+}(2)$	51 (2)	54 (2)	52 (2)	0	6 (2)	0
B^{3+}	46 (13)	64 (14)	32 (13)	0	−5 (11)	0
$O^{2-}(1)$	56 (7)	45 (6)	54 (7)	10 (6)	−2 (6)	1 (6)
$O^{2-}(2)$	61 (7)	59 (7)	61 (7)	3 (6)	3 (6)	−22 (6)
$O^{2-}(3)$	43 (9)	58 (10)	53 (10)	0	−8 (8)	0
$O^{2-}(4)$	73 (10)	56 (10)	42 (10)	0	−4 (8)	0

Table 5. *Interatomic distances* (Å) *and angles* (°)

Ionic charges are omitted for simplicity.

Coordinates of bonding atoms: for Fe(1), Fe(2), B, O(1^{i}), O(2^{i}), O(3^{i}), and O(4^{i}) see Table 3.

O(1^{ii})	x	y	$1+z$
O(1^{iii})	$\frac{1}{2}-x$	$\bar{y}$	$\frac{1}{2}+z$
O(1^{iv})	x	$\frac{1}{2}-y$	$1+z$
O(1^{v})	x	$\frac{1}{2}-y$	z
O(2^{ii})	x	$\frac{1}{2}-y$	z
O(2^{iii})	$\frac{1}{2}-x$	$\bar{y}$	$\frac{1}{2}+z$
O(2^{iv})	$\bar{x}$	y	$\frac{3}{2}-z$
O(3^{ii})	x	y	$1+z$
O(3^{iii})	$\frac{1}{2}+x$	y	$\frac{3}{2}-z$

Fe(1)O_6 octahedron		Fe(2)O_6 octahedron	
Fe(1)–O(1^{ii})	2·069 (4)	Fe(2)–O(1^{ii})	2·166 (4)
–O(1^{iii})	1·989 (4)	–O(1^{iv})	
–O(2^{iii})	1·935 (4)	–O(2^{i})	1·864 (4)
–O(2^{iv})	1·904 (4)	–O(2^{ii})	
–O(3^{ii})	2·107 (3)	–O(3^{iii})	2·041 (5)
–O(4^{i})	2·132 (4)	–O(4^{i})	2·227 (5)

Refinement of the crystal structure

The crystal structure of Fe_3BO_6 was refined using the atomic coordinates given by White *et al.* (1965) as starting parameters. $Fe^{3+}(1)$, $O^{2-}(1)$, and $O^{2-}(2)$ are located at position 8(d), $Fe^{3+}(2)$, B^{3+}, $O^{2-}(3)$, and $O^{2-}(4)$ at position 4(c) of the space group $Pnma$ (No. 62). The atomic scattering factors for Fe^{3+} and B^{3+} were taken from Cromer & Waber (1965), those of O^{2-} from Baur (1965). $\Delta f'$ and $\Delta f''$ corrections for anomalous dispersion in the neutral atoms of Fe and O (Cromer & Liberman, 1970) were applied. A full-matrix least-squares refinement was performed using the program *CRYLSQ* of the X-ray 70 System (Stewart, Kundell & Baldwin, 1970). Weights were taken automatically as the reciprocals of the standard deviations of the observed structure factors. No additional weighting scheme was applied. In the case of

Table 5 (*cont.*)

O(4$^{\rm i}$)—Fe(1)–O(1$^{\rm ii}$)	81·14 (17)	O(2$^{\rm i}$)—Fe(2)–O(4$^{\rm i}$)	88·33 (15)
O(4$^{\rm i}$)—Fe(1)–O(3$^{\rm ii}$)	81·84 (15)	O(4$^{\rm i}$)—Fe(2)–O(2$^{\rm ii}$)	
O(4$^{\rm i}$)—Fe(1)–O(1$^{\rm iii}$)	97·40 (15)	O(2$^{\rm i}$)—Fe(2)–O(1$^{\rm ii}$)	95·52 (15)
O(4$^{\rm i}$)—Fe(1)–O(2$^{\rm iv}$)	92·67 (18)	O(1$^{\rm iv}$)–Fe(2)–O(2$^{\rm ii}$)	
O(1$^{\rm ii}$)–Fe(1)–O(3$^{\rm ii}$)	68·66 (17)	O(2$^{\rm i}$)—Fe(2)–O(3$^{\rm iii}$)	94·52 (15)
O(1$^{\rm ii}$)–Fe(1)–O(1$^{\rm iii}$)	96·61 (14)	O(3$^{\rm iii}$)–Fe(2)–O(2$^{\rm ii}$)	
O(1$^{\rm ii}$)–Fe(1)–O(2$^{\rm iii}$)	97·92 (15)	O(4$^{\rm i}$)—Fe(2)–O(1$^{\rm ii}$)	76·93 (15)
O(3$^{\rm ii}$)–Fe(1)–O(2$^{\rm iii}$)	90·76 (16)	O(4$^{\rm i}$)—Fe(2)–O(1$^{\rm iv}$)	
O(3$^{\rm ii}$)–Fe(1)–O(2$^{\rm iv}$)	93·74 (18)	O(1$^{\rm ii}$)–Fe(2)–O(3$^{\rm iii}$)	99·36 (16)
O(1$^{\rm iii}$)–Fe(1)–O(2$^{\rm iii}$)	90·23 (15)	O(3$^{\rm iii}$)–Fe(2)–O(1$^{\rm iv}$)	
O(2$^{\rm iii}$)–Fe(1)–O(2$^{\rm iv}$)	85·96 (16)	O(1$^{\rm ii}$)–Fe(2)–O(1$^{\rm iv}$)	65·56 (13)
O(1$^{\rm iii}$)–Fe(1)–O(2$^{\rm iv}$)	101·04 (15)	O(2$^{\rm i}$)—Fe(2)–O(2$^{\rm ii}$)	100·29 (17)
O(4$^{\rm i}$)—Fe(1)–O(2$^{\rm iii}$)	172·37 (16)	O(2$^{\rm i}$)—Fe(2)–O(1$^{\rm iv}$)	158·04 (15)
O(1$^{\rm ii}$)–Fe(1)–O(2$^{\rm iv}$)	161·93 (15)	O(1$^{\rm ii}$)–Fe(2)–O(2$^{\rm ii}$)	
O(3$^{\rm ii}$)–Fe(1)–O(1$^{\rm iii}$)	165·22 (17)	O(4$^{\rm i}$)—Fe(2)–O(3$^{\rm iii}$)	175·55 (20)

BO_4 tetrahedron

B–O(1$^{\rm i}$)	1·504 (6)	O(1$^{\rm i}$)–B–O(3$^{\rm i}$)	103·58 (37)
–O(1$^{\rm v}$)		O(3$^{\rm i}$)–B–O(1$^{\rm v}$)	
–O(3$^{\rm i}$)	1·494 (9)	O(1$^{\rm i}$)–B–O(4$^{\rm i}$)	115·50 (36)
–O(4$^{\rm i}$)	1·439 (9)	O(4$^{\rm i}$)–B–O(1$^{\rm v}$)	
		O(1$^{\rm i}$)–B–O(1$^{\rm v}$)	102·44 (46)
		O(3$^{\rm i}$)–B–O(1$^{\rm v}$)	114·58 (55)

individual isotropic thermal motion a residual $R = \sum||F_o|-|F_c||/\sum|F_o|$ of 0·054 was obtained. Refinement with anisotropic temperature coefficients and isotropic extinction correction with Larson's (1967) method resulted in an extinction factor of 2×10^{-3}, an absolute scale of 2·884, an R of 0·031 with zeros omitted, and $R=0{\cdot}035$ with them included. After the final refinement cycle the shifts of all positional and temperature parameters were less than 1% of the corresponding standard deviations. The difference Fourier map calculated with all reflexions was featureless. The F_o, F_c listing is given in Table 2.

Results and discussion

The final atomic coordinates are listed in Table 3, the anisotropic temperature coefficients in Table 4. The lattice constants given in the first row of Table 1 and the atomic coordinates of Table 3 were used to calculate interatomic distances and angles in the Fe_3BO_6 structure by means of the program *BONDLA* of the X-ray 70 System (Stewart *et al.*, 1970). The results are compiled in Table 5.

The crystal structure of Fe_3BO_6 consists of a slightly distorted hexagonal close-packed arrangement of oxygen atoms. Close-packed oxygen layers are stacked along [001]. A relationship to the olivine structure is obvious. The 'Fe_2BO_4' part of the chemical formula forms 'olivine' regions in the structure which are connected in the [010] direction by means of 'FeO_2' units representing 'brucite' regions. For a more detailed description of the norbergite structure see Taylor & West (1929), but note the different setting of the space group which is given as *Pbnm*!

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