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Structural Chemistry of Copper and Zinc Minerals. II. Stereochemistry of Copper(II) and Iodine(V) in Bellingerite, $3Cu(IO_3)_2.2H_2O$

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Bellingerite, $3Cu(IO_3)_2.2H_2O$, is triclinic, space group $P\bar{1}$, with $a_0=7{\cdot}256\,(2)$, $b_0=7{\cdot}950\,(2)$, $c_0=7{\cdot}856\,(2)$ Å, $\alpha=105{\cdot}10\,(2)$, $\beta=92{\cdot}95\,(2)$, and $\gamma=96{\cdot}95\,(2)°$, $Z=1$. The heavy-atom positions were determined from a three-dimensional Patterson map. The crystal structure was determined by the heavy-atom method and refined by the method of least-squares, with anisotropic temperature factors, to an R of 0·038 for 3156 reflections measured on an automatic single-crystal diffractometer. The standard error in Cu–O and I–O bond lengths is ±0·005 Å and in O–Cu–O and O–I–O angles ±0·02°. The crystal structure of bellingerite is a three-dimensional framework consisting of a corner-sharing tetragonally distorted $Cu(1)O_6$ octahedron, a $[Cu(2)_2O_8(H_2O)_2]$ octahedral dimer and three independent pyramidal iodate groups. Cu(1) at a symmetry center is bonded to four oxygens at 1·936 Å (× 2) and 1·967 Å (× 2) approximately in a square plane, while two oxygens at 2·528 Å (× 2) complete the octahedron. Cu(2) has three oxygen atoms at 1·942, 1·946 and 1·973 Å and a water molecule at 1·950 Å approximately in a square plane, while one oxygen at 2·456 Å and a water molecule at 2·483 Å form the apices of the distorted octahedron. Two Cu(2) octahedra form a dimer by sharing an octahedral edge. The pyramidal $I(1)O_3$ group, with point-group symmetry $3m$, has three oxygens at 1·815, 1·823 and 1·824 Å from the iodine atom, with O–I–O angles 96·0, 95·7 and 97·3°. I(1) is weakly bonded to three further oxygens at 2·755, 2·676 and 2·796 Å. The $I(1)O_6$ coordination polyhedron is a distorted octahedron. I(2) is bonded to three oxygens at 1·817, 1·815 and 1·825 Å, the O–I–O angles being 99·1, 99·4, and 95·1°. Four further oxygens form weak bonds at 2·737, 2·957, 3·050 and 3·172 Å. The $I(2)O_7$ polyhedron can be described as a square pyramid with two domes below the equatorial plane. I(3) is bonded to three oxygens at 1·801, 1·823 and 1·795 Å, with O–I–O angles 96·7, 101·0 and 101·1°. I(3) is weakly bonded to two further oxygens at 2·771 and 2·873 Å. The $I(3)O_5$ polyhedron is a distorted trigonal bipyramid. Both $I(2)O_3$ and $I(3)O_3$ groups approximate very closely to the point-group symmetry m. Both H atoms belonging to the water molecule are involved in strong hydrogen bonds (both $O\text{–}H\cdots O=2{\cdot}663$ Å).

Introduction

In part I of this series, we described the structure of veszelyite, $(Cu,Zn)_2ZnPO_4(OH)_3.2H_2O$ (Ghose, Leo & Wan, 1974), which has a novel type of octahedral sheet structure. In this paper we describe the structure determination of a copper iodate mineral, bellingerite, $3Cu(IO_3)_2.2H_2O$ from Chuquicamata, Chile. The only other copper iodate mineral is salesite, $CuIO_3(OH)$ also from Chile, whose crystal structure has been described by Ghose (1962). Synthetic analogs of these two copper iodate minerals have been prepared by Granger & de Schulten (1904).

In addition to the stereochemistry of the cupric ions, the structure of bellingerite provides an opportunity to study the stereochemistry of three crystallographically independent iodate groups, which are not restricted by any point-group symmetry imposed by the crystal symmetry. The stereochemistry and bonding of iodate groups is currently of interest [see Alcock (1972*a*) for a review] in terms of the significance of the I–O contacts between 2·5 and 3·5 Å in addition to the three short pyramidal I–O bonds (~1·82 Å).

Crystal data

Bellingerite, $3Cu(IO_3)_2.2H_2O$, light-green prismatic crystals, triclinic;

a_0=7·2560 (23) Å	α=105·096 (16)°
b_0=7·9503 (15)	β= 92·945 (22)
c_0=7·8559 (17)	γ= 96·952 (21)
Cell volume	432·73 (19) Å^3
Space group	$P\bar{1}$
Cell content	$3Cu(IO_3)_2.2H_2O$
D_m	4·89 g cm^{-3}
D_c	4·932 g cm^{-3}
μ(Mo $K\alpha$)	135·72 cm^{-1}
λ(Mo $K\alpha_1$)	0·70926 Å

The cell dimensions listed above have been derived from a least-squares refinement of 15 reflections with 2θ values between 50 and 60° measured on an automatic single-crystal diffractometer using Mo $K\alpha_1$ radiation. The cell dimensions are in good agreement with those proposed by Berman & Wolfe (1940), except that *b* and *c* have been interchanged to maintain a right-handed coordinate system. The crystal morphology indicated the centrosymmetric space group $P\bar{1}$, which has been confirmed by the structure determination.

Experimental

A small bellingerite single crystal, checked for quality by precession photographs, was ground into a sphere with a Bond-type sphere grinder (Bond, 1951). X-ray transmission Laue photographs indicated a surface coating of powdery material, which developed by surface flow during grinding. The surface coating was removed by immersing the crystal in a dilute HCl solution for a short time. The crystal sphere, with a diameter of 0·21 (1) mm, was mounted on a Syntex $P\bar{1}$ single-crystal diffractometer in an arbitrary orientation. The orientation matrix was found automatically. The intensities of all reflections with 2θ values below 65° (a total of 3156) were measured by the $2\theta:\theta$ method, with Mo $K\alpha$ radiation monochromatized by reflection from a graphite 'single' crystal, and a solid-state detection system. The variable-scan method was used, the minimum scan rate being 1° min^{-1}. The intensities were corrected for Lorentz, polarization and absorption effects. For intensity I less than $0·7\sigma(I)$, where $\sigma(I)$ is the standard error of measurement, I was set equal to $0·7\sigma(I)$, regardless of whether I was positive or negative. A three-dimensional Patterson synthesis was calculated with the Fourier program incorporated in the X-RAY 67 system.

Determination and refinement of the structure

The cell content of $3Cu(IO_3)_2.2H_2O$ in the space group $P\bar{1}$ requires that one of the two copper atoms, Cu(1), must occupy a symmetry center, which was arbitrarily chosen at the origin. Hence in the Patterson map an image of the structure exists in its true position, modulated by the electron density of Cu(1). This image was retrieved by systematically considering Cu(1)–I vectors

Table 1. *Bellingerite*, $3Cu(IO_3)_2.2H_2O$: *positional and thermal parameters* (*with standard deviations in parentheses*)

Fractional coordinates and anisotropic temperature factors are × 10^5.

Form of the temperature factor: $\exp\,[-\{\sum_{i=1}^{3}\sum_{j=1}^{3} h_ih_j\beta_{ij}\}]$.

	x	y	z	B_{eq} (Å^2)	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cu(1)	0	0	0	1·505 (16)	772 (18)	691 (16)	577 (15)	258 (14)	87 (13)	90 (13)
Cu(2)	39635 (11)	57637 (10)	35154 (10)	1·406 (12)	819 (13)	554 (11)	637 (11)	192 (9)	272 (10)	236 (9)
I(1)	60934 (5)	13645 (5)	86911 (5)	1·179 (7)	685 (7)	451 (5)	498 (6)	92 (4)	151 (4)	145 (4)
I(2)	85790 (5)	15515 (5)	39779 (5)	1·269 (7)	679 (7)	498 (6)	558 (6)	89 (4)	157 (4)	132 (4)
I(3)	18703 (5)	40381 (5)	85862 (5)	1·348 (8)	643 (7)	595 (6)	553 (6)	109 (4)	139 (4)	66 (4)
O(1)	52811 (72)	8006 (61)	19016 (64)	1·694 (68)	1005 (88)	561 (63)	712 (70)	20 (59)	23 (63)	149 (54)
O(2)	57102 (70)	17900 (62)	65384 (62)	1·607 (65)	976 (84)	572 (65)	658 (68)	112 (58)	40 (59)	176 (54)
O(3)	83655 (68)	6418 (74)	83178 (65)	1·859 (72)	682 (76)	1142 (86)	739 (72)	329 (65)	205 (59)	259 (64)
O(4)	79211 (70)	37298 (66)	48187 (70)	1·797 (72)	912 (83)	664 (72)	949 (79)	277 (62)	448 (65)	223 (61)
O(5)	8513 (74)	18993 (65)	51917 (65)	1·810 (70)	1085 (91)	642 (67)	702 (73)	31 (62)	147 (64)	125 (57)
O(6)	92966 (70)	19106 (65)	18930 (63)	1·670 (66)	856 (81)	829 (71)	622 (67)	147 (60)	262 (59)	215 (57)
O(7)	41073 (68)	49592 (68)	80969 (68)	1·795 (71)	753 (76)	840 (75)	913 (78)	132 (60)	476 (62)	303 (62)
O(8)	80763 (66)	45422 (65)	91725 (60)	1·578 (64)	731 (75)	789 (70)	555 (64)	87 (58)	247 (55)	48 (54)
O(9)	24170 (76)	20405 (68)	90261 (72)	1·934 (73)	1044 (91)	715 (72)	920 (80)	47 (64)	239 (67)	302 (62)
O(10) (H_2O)	37102 (70)	34132 (62)	38730 (67)	1·739 (69)	936 (84)	550 (65)	904 (78)	69 (58)	299 (63)	206 (58)

(x_i,y_i,z_i), which occur halfway between I–I vectors ($2x_i,2y_i,2z_i$) from iodine atoms related by inversion. These iodine positions were confirmed by considering interatomic vectors between crystallographically different iodine atoms (x_i-x_j,y_i-y_j,z_i-z_j). Next, the Cu(2) position was determined from a consideration of Cu(2)–I vectors and confirmed by the Cu(1)–Cu(2) vector. Two cycles of isotropic least-squares refinement using the heavy-atom positions only reduced R to 0·19. Three-dimensional Fourier and difference-Fourier syntheses, based on the structure factors calculated with the heavy-atom positions, yielded the positions of the re-

Table 2. *Bellingerite*, $3Cu(IO_3)_2.2H_2O$: *observed and calculated structure factors*

0,0,L		
1	491	457
2	121	97
3	883	-870
4	2049	-2183
5	321	323
6	514	527
7	636	657
8	844	893
9	81	-71
10	73	-58
11	399	-397
0,1,L		
-11	122	107
-10	107	108
-9	629	-655
-8	329	332
-7	119	129
-6	599	622
-5	1301	1344
-4	224	-206
-3	128	-98
-2	1331	-1326
-1	582	-564
0	203	171
1	795	804
2	1983	2083
3	281	278
4	546	557
5	348	-346
6	1152	-1208
7	421	443
8	111	-49
9	557	578
10	673	678
11	222	-207
0,2,L		
-11	43	44
-10	340	-347
-9	60	34
-8	331	398
-7	82	-82
-6	1151	1204
-5	262	-287
-4	264	-222
-3	487	484
-2	1097	-1060
-1	1563	1473
0	397	400
1	49	-63
2	729	759
3	367	-355
4	428	434
5	288	290
6	116	116
7	208	205
8	268	-272
9	243	246
10	38	-5
0,3,L		
-11	481	487
-10	433	-450
-9	440	-452
-8	387	-405
-7	93	-65
-6	952	962
-5	1047	1091
-4	1221	1195
-3	201	-158
-2	921	-927
-1	912	-790
0	1514	-1476
1	1493	1501
2	1044	998
3	748	772
4	954	985
5	1019	-1041
6	134	-139
7	92	-88
8	114	-90
9	604	615
10	93	84
0,4,L		
-11	336	344
-10	342	355
-9	682	689
-8	934	-956
-7	352	-380
-6	499	-483
-5	566	-577
-4	1949	2039
-3	1098	1038
-2	857	836
-1	827	772
0	1760	-1694
1	558	-538
2	502	-466
3	538	533
4	1028	1054
5	653	670
6	354	351
7	590	-597
8	285	-285
9	248	-248
0,5,L		
-11	52	-35
-10	175	158
-9	328	342
-8	132	131
-7	314	327
-6	377	-383
-5	89	-87
-4	201	-189
-3	338	-330
-2	1443	1454
-1	265	-275
0	981	993
1	298	263
2	965	-930
3	395	383
4	408	-391
5	178	154
6	837	881
7	35	-16
8	312	331
9	230	-232
0,6,L		
-11	46	-53
-10	694	714
-9	55	53
-8	146	-57
-7	136	150
-6	783	-807
-5	349	366
-4	229	208
-3	306	334
-2	903	861
-1	236	-182
0	239	208
1	411	-386
2	53	-5
3	274	296
4	38	-60
5	477	511
6	195	-182
7	55	51
8	201	190
0,7,L		
-11	287	-298
-10	78	85
-9	285	282
-8	1003	1076
-7	202	180
-6	79	-44
-5	380	-417
-4	1313	-1299
-3	530	507
-2	327	359
-1	622	614
0	1169	1202
1	400	-430
2	117	-85
3	328	-348
4	485	-493
5	427	444
6	184	195
7	482	492
0,8,L		
-10	191	-211
-9	456	-453
-8	401	387
-7	259	286
-6	564	539
-5	508	556
-4	183	-200
-3	317	-290
-2	647	-674
-1	186	66
0	237	220
1	650	696
2	759	791
3	284	-289
4	82	-82
5	475	-498
6	340	-346
0,9,L		
-9	256	247
-8	19	-4
-7	174	-180
-6	404	427
-5	265	-273
-4	319	317
-3	423	436
-2	472	-499
-1	324	347
0	315	-327
1	32	25
2	444	463
3	41	48
4	283	290
5	77	-51
0,10,L		
-8	151	-144
-7	144	140
-6	463	495
-5	199	187
-4	328	354
-3	411	-445
-2	194	-170
-1	72	-89
0	142	-95
1	541	595
2	35	54
3	95	81
0,11,L		
-7	76	-56
-6	144	-149
-5	135	146
-4	775	791
-3	81	102
-2	188	171
-1	313	-310
0	431	-472
1	70	-26
1,-12,L		
3	58	-70
4	246	-236
1,-11,L		
-1	192	213
0	468	484
1	131	-135
2	248	245
3	211	-207
4	256	-269
5	157	156
6	83	-72
7	239	234
1,-10,L		
-3	241	-253
-2	590	-611
-1	117	-75
0	93	86
1	585	624
2	777	809
3	132	-118
4	120	107
5	571	-583
6	359	-398
7	302	334
8	18	-21
1,-9,L		
-5	26	-9
-4	625	657
-3	428	439
-2	355	-353
-1	60	42
0	738	-755
1	115	-147
2	377	420
3	434	426
4	701	765
5	137	83
6	158	-146
7	477	-511
8	330	-325
9	239	227
1,-8,L		
-6	22	-4
-5	29	3
-4	268	257
-3	156	-138
-2	464	455
-1	17	15
0	262	-296
1	519	577
2	663	-719
3	368	421
4	369	360
5	193	-177
6	741	773
7	179	-143
8	161	-175
9	63	52
10	342	-348
1,-7,L		
-7	58	-25
-6	365	-354
-5	273	270
-4	821	868
-3	183	160
-2	535	578
-1	371	-394
0	476	-461
1	187	-149
2	135	-102
3	611	595
4	284	268
5	433	453
6	97	94
7	374	-393
8	264	274
9	317	-331
10	228	234
11	238	246
1,-6,L		
-8	411	409
-7	38	15
-6	704	-739
-5	251	-250
-4	598	-620
-3	547	568
-2	1280	1334
-1	329	336
0	1056	1070
1	698	-708
2	1030	-1020
3	46	20
4	422	-421
5	1339	1060
6	793	849
7	212	191
8	124	93
9	594	-628
10	215	-214
11	64	-83
1,-5,L		
-9	266	-268
-8	392	402
-7	449	460
-6	207	211
-5	345	355
-4	537	-531
-3	604	-615
-2	183	-189
-1	408	431
0	1318	1295
1	458	492
2	782	754
3	949	-927
4	547	-594
5	113	153
6	350	-390
7	1010	1081
8	518	514
9	89	92
10	238	242
11	574	-575
1,-4,L		
-9	159	153
-8	437	455
-7	276	-301
-6	380	411
-5	193	-208
-4	521	-511
-3	1127	1123
-2	562	-545
-1	741	682
0	626	670
1	907	-932
2	1040	1074
3	171	131
4	109	128
5	409	404
6	433	-436
7	119	94
8	40	3
9	372	377
10	175	182
11	21	26
1,-3,L		
-10	544	-560
-9	150	-152
-8	515	516
-7	529	570
-6	1064	1056
-5	41	48
-4	501	-525
-3	762	-745
-2	760	-769
-1	1315	1321
0	86	37
1	1505	1680
2	214	195
3	645	-619
4	507	510
5	958	-975
6	103	93
7	766	806
8	48	-41
9	769	833
10	103	-101
11	228	-234
1,-2,L		
-10	316	-318
-9	37	-5
-8	827	-861
-7	90	-95
-6	740	766
-5	241	211
-4	1793	1911
-3	289	271
-2	553	-558
-1	422	-587
0	1816	-1818
1	889	887
2	1466	1606
3	1534	1606
4	973	955
5	970	-1001
6	536	-560
7	924	-963
8	209	224
9	632	660
10	361	360
11	580	587
1,-1,L		
-11	137	130
-10	137	129
-9	330	340
-8	91	-74
-7	158	159
-6	578	-601
-5	171	182
-4	397	405
-3	118	106
-2	1963	2073
-1	522	-496
0	465	-459
1	165	185
2	1596	-1722
3	1317	1370
4	976	1011
5	481	470
6	1018	1076
7	665	-702
8	307	-315
9	293	-303
10	130	-94
11	426	435
1,0,L		
-11	100	-105
-10	383	386
-9	216	-224
-8	535	-557
-7	424	446
-6	301	-288
-5	554	549
-4	1254	1268
-3	390	-379
-2	764	769
-1	607	-600
0	78	3
1	161	193
2	706	721
3	470	493
4	214	-193
5	585	608
6	201	-213
7	49	27
8	423	437
9	352	-374
10	237	243
11	67	67
1,1,L		
-11	232	203
-10	760	777
-9	339	350
-8	392	403
-7	564	-578
-6	931	-935
-5	144	155
-4	513	-529
-3	1774	1872
-2	1734	1713
-1	250	-211
0	1063	1009
1	2149	-2388
2	871	-892
3	750	789
4	317	305
5	1664	1758
6	459	479
7	163	-166
8	243	-251
9	512	-527
10	56	35
11	100	87
1,2,L		
-11	291	-294
-10	118	104
-9	61	-78
-8	1048	1120
-7	558	574
-6	102	-19
-5	150	-164
-4	1495	-1483
-3	394	347
-2	193	211
-1	2069	2089
0	1611	1543
1	239	221
2	71	-25
3	1629	-1700
4	733	-745
5	377	393
6	214	210
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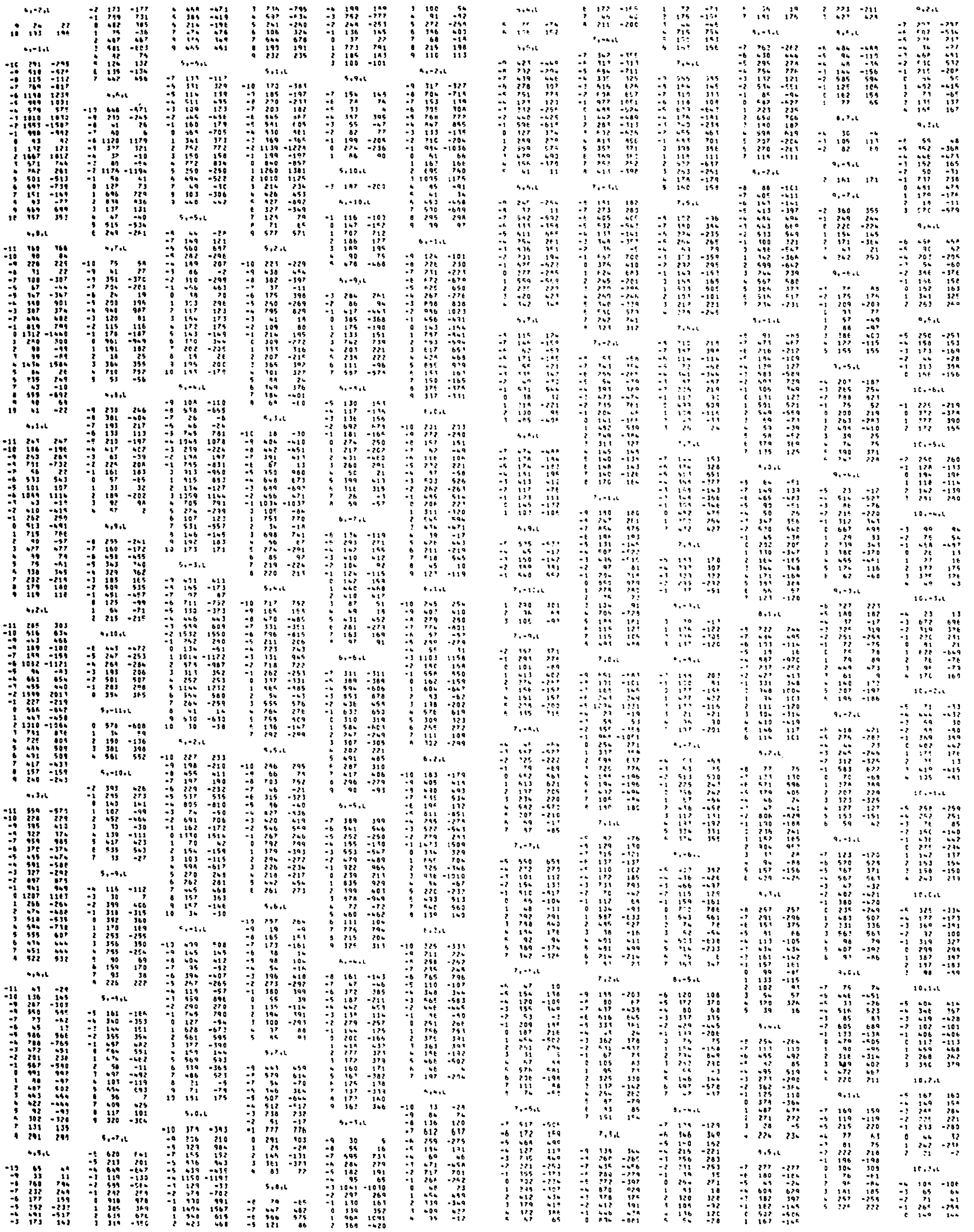
4	250	-264
5	185	188
3,9,L		
-9	321	-325
-8	153	158
-7	600	619
-6	232	250
-5	350	351
-4	281	-271
-3	434	-458
-2	208	-193
-1	120	-141
0	529	571
1	248	236
2	395	427
3	18	-27
3,10,L		
-7	178	-180
-6	120	108
-5	276	299
-4	174	-201
-3	324	352
-2	126	-142
-1	195	-183
0	204	193
1	162	-154
3,11,L		
-4	43	-6
-3	74	-85
-2	366	-365
4,-11,L		
-1	27	16
0	151	155
1	305	324
2	26	-11
3	148	141
4	286	-306
5	117	-100
6	123	104
4,-10,L		
-3	77	-81
-2	448	473
-1	45	-43
0	448	475
1	107	85
2	347	-332
3	221	204
4	313	-300
5	133	108
6	161	148
7	40	20
4,-9,L		
-4	201	-215
-3	362	-346
-2	478	-526
-1	95	-67
0	540	528
1	377	421
2	821	824
3	41	-6
4	426	-458
5	64	-38
6	591	-618
7	269	288
8	373	374
4,-8,L		
-6	229	246
-5	426	430
-4	117	143
-3	726	728
-2	304	-294
-1	456	-498
0	159	-142
1	579	-613
2	676	711
3	643	661
4	533	552
5	186	182
6	422	-438
7	358	-371
8	268	-263
9	244	248
4,-7,L		
-7	88	-79
-6	201	196
-5	97	-98
-4	140	134
-3	230	248
-2	68	-64
-1	575	562
0	350	-392
1	38	40
2	190	209
3	419	-436
4	718	762
5	112	95
6	121	130
7	346	354
8	454	-453
9	24	5
10	65	-53
4,-6,L		
-8	355	-360
-7	421	-431
-6	357	365
-5	134	-127
-4	927	939
-3	534	559
-2	118	-126
-1	101	21
0	769	-807
1	93	-79
2	225	208
3	416	459
4	624	633
5	93	-70
6	265	264
7	269	-256
8	109	-183
9	236	236
10	174	-182
4,-5,L		
-8	114	112
-7	73	32
-6	811	-830
-5	494	-522
-4	270	279
-3	85	39
-2	1568	1656
-1	684	652
0	220	-177
1	415	406
2	1508	-1575
3	125	-170
4	375	421
5	310	301
6	1138	1213
7	90	82
8	246	-237
9	332	-341
10	363	-357
4,-4,L		
-9	179	190
-8	31	-16
-7	800	847
-6	85	68
-5	56	10
-4	232	-245
-3	843	-814
-2	47	-17
-1	453	500
0	1121	1115
1	786	862
2	33	-5
3	156	-155
4	1103	-1213
5	246	266
6	264	272
7	396	422
8	863	890
9	200	-203
10	109	-107
4,-3,L		
-9	61	0
-8	273	291
-7	45	30
-6	325	-317
-5	615	609
-4	393	-379
-3	270	248
-2	544	547
-1	625	-645
0	1118	1192
1	67	-59
2	84	87
3	453	495
4	354	-377
5	251	263
6	35	20
7	121	-62
8	253	242
9	51	18
10	302	303
4,-2,L		
-10	119	-114
-9	299	-305
-8	587	604
-7	777	824
-6	518	537
-5	306	308
-4	883	-893
-3	523	-512
-2	682	-654
-1	648	640
0	1196	1283
1	288	304
2	939	1065
3	665	-717
4	482	-489
5	196	178
6	338	-359
7	728	773
8	358	367

Table 2 (*cont.*)



maining 10 oxygen atoms. The full-matrix least-squares program *RFINE* (Finger, 1969) was used for the structure refinement. The structure factors (F_o) were weighted $F_o/3\sigma^2(F_o)$, where $\sigma(F_o)$ is the standard error of the measurement of F_o derived from the counting statistics. The scattering factors of the non-ionized atoms Cu, I, and O were taken from Cromer & Mann (1968). Dispersion corrections were made according to Cromer & Waber (1965). Three cycles of least-squares refinement using isotropic temperature factors, followed by three more cycles using anisotropic temperature factors, reduced R to 0·038 (unweighted) and 0·057 (weighted) for 3156 reflections. The conventional R value and the weighted R value, R_w, are defined as:

$$R=[\sum(|F_o|-|F_c|)/\sum|F_o|]$$

and

$$R_w=[\sum w(F_o|-|F_c|)^2/\sum w|F_o|^2]^{1/2},$$

where w is the weight of the observed structure factor. After completion of the least-squares refinement, a three-dimensional difference-Fourier synthesis was computed to locate the hydrogen atoms; however, this attempt was unsuccessful.

The atomic positional and thermal parameters are listed in Table 1, a list of observed and calculated structure factors is given in Table 2; the bond lengths and bond angles with standard deviations are listed in Table 3, and the thermal ellipsoids with their standard deviations are listed in Table 4. The standard deviations in bond lengths and bond angles as well as those of thermal ellipsoids have been calculated with the *ERROR* program (Finger, 1972). The average standard deviation of the Cu–O and I–O bond lengths is ±0·005 Å and in O–Cu–O and O–I–O bond angles ±0·02°.

Table 3. *Bellingerite*, $3Cu(IO_3)_2.2H_2O$: *interatomic distances* (Å) *and angles* (°) (*with standard deviations in parentheses*)

Cu(1) octahedron

Cu(1)-O(3)	1·936 (5) (×2)	O(3)–Cu(1)–O(6)	89·3 (2)
Cu(1)-O(6)	1·967 (5) (×2)	O(3)–Cu(1)–O(9)	82·8 (2)
Cu(1)-O(9)	2·528 (5) (×2)	O(3)–Cu(1)–O(6′)	90·7 (2)
O(6)—O(9)	3·286 (7) (×2)	O(3)–Cu(1)–O(9′)	97·2 (2)
O(9)—O(6′)	3·117 (7) (×2)	O(6)–Cu(1)–O(9)	93·1 (2) (×2)
O(3)—O(6)	2·744 (7) (×2)	O(6′)–Cu(1)–O(9)	86·9 (2) (×2)
O(3)—O(9)	2·984 (7) (×2)		

Cu(2) octahedron

Cu(2)–O(2)	1·942 (5)	O(2)—Cu(2)–O(4)	89·4 (2)
Cu(2)–O(4)	1·946 (5)	O(4)—Cu(2)–O(10)	86·9 (2)
Cu(2)–O(7)	1·973 (5)	O(10)–Cu(2)–O(7)	87·6 (2)
Cu(2)–O(10)	1·950 (5)	O(7)—Cu(2)–O(2)	95·9 (2)
Cu(2)–O(8)	2·456 (5)	O(8)—Cu(2)–O(2)	81·4 (2)
Cu(2)–O(10′)	2·483 (5)	O(8)—Cu(2)–O(4)	96·6 (2)
O(2)—O(4)	2·736 (7)	O(8)—Cu(2)–O(10)	104·7 (2)
O(4)—O(10)	2·679 (7)	O(8)—Cu(2)–O(7)	86·0 (2)
O(10)–O(7)	2·716 (7)	O(10′)–Cu(2)–O(2)	88·6 (2)
O(7)—O(2)	2·907 (7)	O(10′)–Cu(2)–O(4)	86·9 (2)
O(8)—O(2)	2·896 (7)	O(10′)–Cu(2)–O(10)	85·4 (2)
O(8)—O(4)	3·305 (7)	O(10′)–Cu(2)–O(7)	91·4 (2)
O(8)—O(10)	3·502 (7)	O(2)—Cu(2)–O(10)	173·2 (2)
O(8)—O(7)	3·041 (7)	O(4)—Cu(2)–O(7)	174·4 (2)
O(10′)–O(2)	3·115 (7)	O(8)—Cu(2)–O(10′)	169·4 (2)
O(10′)–O(4)	3·072 (7)		
O(10′)–O(10)	3·032 (7)		
O(10)–O(7)	3·209 (7)		
Cu(2)–Cu(2′)	3·2765 (15)		

I(1) polyhedron

I(1)—O(1)	1·815 (5)	O(1)–I(1)—O(2)	96·0 (2)
I(1)—O(2)	1·823 (5)	O(2)–I(1)—O(3)	95·7 (2)
I(1)—O(3)	1·824 (5)	O(3)–I(1)—O(1)	97·3 (2)
Mean	1·821	Mean	96·3
I(1)—O(1′)	2·755 (5)	O(1)–O(2)–O(3)	60·7 (2)
I(1)—O(8)	2·676 (5)	O(2)–O(3)–O(1)	59·6 (2)
I(1)—O(9)	2·796 (6)	O(3)–O(1)–O(2)	59·7 (2)
O(1)–O(2)	2·703 (7)	Mean	60·0
O(2)–O(3)	2·704 (7)	O(1)–I(1)—O(1′)	76·2 (2)
O(3)–O(1)	2·732 (7)	O(1)–I(1)—O(9)	75·7 (2)
Mean	2·713	O(1)–I(1)—O(8)	173·5 (2)
O(1)–O(1′)	2·912 (10)	O(1′)–I(1)—O(2)	158·7 (2)
O(1)–O(9)	3·401 (7)	O(1′)–I(1)—O(9)	75·6 (2)
O(1′)–O(9)	2·933 (7)	O(1′)–I(1)—O(3)	104·8 (2)
O(9)–O(3)	2·984 (7)	O(1′)–I(1)—O(8)	110·3 (2)
O(1′)–O(2)	3·510 (7)	O(9)–I(1)—O(3)	172·8 (2)
O(8)–O(1′)	4·457 (7)	O(9)–I(1)—O(2)	83·3 (2)
O(8)–O(9)	2·792 (7)	O(9)–I(1)—O(8)	104·3 (2)
O(8)–O(3)	3·033 (7)	O(2)–I(1)—O(8)	77·6 (2)
O(8)–O(2)	2·896 (7)	O(3)–I(1)—O(8)	82·4 (2)

Table 3 (*cont.*)

I(2) polyhedron

I(2)—O(4)	1·817 (5)	O(4)–I(2)—O(5)	99·0 (2)
I(2)—O(5)	1·815 (5)	O(5)–I(2)—O(6)	99·4 (2)
I(2)—O(6)	1·825 (5)	O(6)–I(2)—O(4)	95·1 (2)
Mean	1·819	Mean	97·9
I(2)—O(1)	2·737 (5)	O(4)–O(5)–O(6)	58·1 (2)
I(2)—O(2)	2·957 (5)	O(5)–O(6)–O(4)	60·7 (2)
I(2)—O(5′)	3·050 (5)	O(6)–O(4)–O(5)	61·2 (2)
I(2)—O(9)	3·172 (5)	Mean	60·0
O(4)–O(5)	2·763 (7)	O(1)–I(2)—O(6)	77·5 (2)
O(5)–O(6)	2·775 (7)	O(1)–I(2)—O(2)	76·0 (2)
O(6)–O(4)	2·688 (7)	O(2)–I(2)—O(5)	108·4 (2)
Mean	2·742	O(4)–I(2)—O(1)	85·6 (2)
O(1)–O(2′)	3·510 (7)	O(4)–I(2)—O(2)	64·9 (2)
O(2)–O(5)	3·646 (7)	O(5′)–I(2)—O(6)	118·8 (2)
O(6)–O(1)	2·941 (7)	O(5′)–I(2)—O(1)	104·8 (2)
O(4)–O(1)	3·168 (7)	O(5′)–I(2)—O(2)	85·8 (2)
O(4)–O(2′)	2·736 (7)	O(5′)–I(2)—O(5)	72·8 (2)
O(5)–O(1)	4·588 (7)	O(4)–I(2)—O(5′)	145·8 (2)
O(5)–O(2′)	3·927 (7)	O(5)–I(2)—O(1)	174·6 (2)
O(5)–O(5)	3·053 (10)	O(6)–I(2)—O(2)	147·7 (2)
O(5)–O(6)	4·242 (7)		

I(3) polyhedron

I(3)—O(7)	1·801 (5)	O(7)—I(3)—O(8)	96·7 (2)
I(3)—O(8)	1·823 (5)	O(8)—I(3)—O(9)	101·0 (2)
I(3)—O(9)	1·795 (5)	O(9)—I(3)—O(7)	101·0 (2)
Mean	1·806	Mean	99·6
I(3)—O(5)	2·771 (5)	O(7)—O(8)—O(9)	60·6 (2)
I(3)—O(8)	2·873 (5)	O(8)—O(9)—O(7)	58·2 (2)
O(7)—O(8)	2·707 (6)	O(9)—O(7)—O(8)	61·2 (2)
O(8)—O(9)	2·792 (7)	Mean	60·0
O(9)—O(7)	2·776 (7)	O(5)—I(3)—O(8)	165·8 (2)
Mean	2·758	O(5)—I(3)—O(9)	83·7 (2)
O(5)—O(8)	4·561 (7)	O(5)—I(3)—O(7)	95·5 (2)
O(5)—O(9)	3·133 (7)	O(8′)—I(3)—O(5)	92·0 (2)
O(5)—O(7)	3·446 (7)	O(8′)—I(3)—O(8)	73·8 (2)
O(8′)–O(5)	4·062 (7)	O(8′)—I(3)—O(9)	111·9 (2)
O(8′)–O(9)	3·915 (7)	O(8′)—I(3)—O(7)	146·8 (2)
O(8′)–O(8)	2·942 (10)		

Hydrogen bonds

O(10)–O(1)	2·663 (7)	O(1)—O(10)–O(5)	106·2 (2)
O(10)–O(5)	2·663 (7)	Cu(2)–O(10)–Cu(2′)	94·6 (2)
O(10)–Cu(2)	1·950 (5)	Cu(2)–O(10)–O(1)	122·9 (2)
O(10)–Cu(2′)	2·483 (5)	Cu(2)–O(10)–O(5)	126·1 (2)
		Cu(2′)–O(10)–O(1)	97·7 (2)
		Cu(2′)–O(10)–O(5)	100·1 (2)

Cu–I distances		I–I distances (< 4·2 Å)	
Cu(1)–I(1)	3·3608 (4) (×2)	I(1)–I(1′)	3·6443 (7)
Cu(1)–I(2)	3·3138 (4) (×2)	I(1)–I(2)	4·0125 (5)
Cu(1)–I(3)	3·7867 (4) (×2)	I(1)–I(3′)	3·7965 (5)
Cu(2)–I(1)	3·2087 (8)	I(1)–I(3)	3·9442 (5)
Cu(2)–I(2)	3·3128 (8)	I(2)–I(2′)	3·9840 (7)
Cu(2)–I(3)	3·5245 (8)	I(2)–I(3)	4·1181 (5)
Cu(2)–I(3′)	3·9125 (9)	I(3)–I(3′)	3·8072 (7)

Description of the structure

Stereochemistry of the cupric ions

The isolated Cu(1)O_6 group is a tetragonally distorted octahedron (Fig. 1). O(3) (×2) and O(6) (×2) atoms form a rectangle at distances of 1·936 Å (×2) and 1·967 Å (×2) respectively, while O(9) is the apical oxygen at 2·528 Å (×2).

The $[Cu(2)O_4(H_2O)_2]$ octahedron is also tetragonally distorted; O(2), O(4), O(7) and O(10) (H_2O) form a rectangle with Cu–O distances 1·942, 1·946, 1·973 and 1·950 Å respectively, while O(8) and O(10′) complete the octahedron at distances of 2·456 and 2·483 Å respectively. The copper atom deviates slightly from the rectangular plane towards the apical O(8) atom, as indicated by the O(2)–Cu(2)–O(10) angle of 173·2° and the O(4)–Cu(2)–O(7) angle of 174·4°. The apical oxygens O(8) and O(10′) make an angle of 169·4° at Cu(2). The Cu(2) octahedron shares a bipyramidal edge O(10)–O(10′) with a centrosymmetrically related Cu(2) octahedron, forming a dimeric $[Cu_2O_8(H_2O)_2]$ group (Fig. 1).

Stereochemistry of the iodate groups

The three crystallographically independent iodate groups are all trigonal pyramids (Fig. 1).

I(1) is closely bonded to O(1), O(2) and O(3) at distances of 1·815, 1·823 and 1·824 Å respectively. The O–I–O angles are 96·0, 95·7 and 97·3°. I(1) is weakly bonded to three further oxygens O(1′), O(8) and O(9) at 2·755, 2·676 and 2·796 Å respectively (Fig. 2). The configuration around the I(1) atom can be described as a highly distorted octahedron, where the apical oxygens O(1) and O(8) are closer to one edge O(2)–O(3) of the equatorial plane, rather than being exactly above and below the plane defined by O(1), O(9) and O(2), O(3)

Table 4. *Bellingerite*, $3Cu(IO_3)_2.2H_2O$: *thermal ellipsoids* (*with standard deviations in parentheses*)

			Angle (°) with respect to		
	r.m.s. amplitude		$+a$	$+b$	$+c$
Cu(1)	*r* 1	0·1183	58·6 (5·5)	60·0 (4·4)	64·2 (3·9)
	r 2	0·1353	54·7 (8·0)	68·6 (6·0)	144·0 (8·2)
	r 3	0·1578	128·9 (4·7)	35·5 (4·6)	113·0 (3·4)
Cu(2)	*r* 1	0·1152	59·2 (11·9)	110·1 (9·2)	34·0 (12·1)
	r 2	0·1209	113·1 (9·5)	146·5 (8·4)	88·4 (8·0)
	r 3	0·1599	144·0 (2·3)	81·6 (1·5)	54·2 (2·1)
I(1)	*r* 1	0·1114	65·7 (11·6)	122·2 (10·8)	31·7 (13·4)
	r 2	0·1147	106·7 (6·7)	140·6 (7·1)	104·7 (8·5)
	r 3	0·1387	154·2 (1·7)	95·4 (1·2)	61·9 (1·8)
I(2)	*r* 1	0·1167	56·4 (7·5)	78·9 (5·3)	48·6 (7·0)
	r 2	0·1212	79·0 (5·1)	174·3 (3·4)	71·4 (6·2)
	r 3	0·1410	142·6 (2·4)	102·7 (1·4)	51·4 (2·4)
I(3)	*r* 1	0·1132	58·4 (2·0)	70·8 (1·1)	53·7 (1·5)
	r 2	0·1325	33·1 (4·2)	126·2 (3·7)	97·8 (3·0)
	r 3	0·1444	109·9 (2·8)	138·6 (2·0)	44·7 (3·1)
O(1)	*r* 1	0·1270	99·7 (12·0)	16·7 (18·8)	88·5 (19·0)
	r 2	0·1447	85·4 (16·6)	71·2 (19·4)	175·7 (20·4)
	r 3	0·1653	154·3 (12·9)	103·1 (11·5)	97·2 (14·4)
O(2)	*r* 1	0·1283	84·3 (13·1)	12·7 (12·9)	106·3 (35·7)
	r 2	0·1378	85·3 (16·6)	91·5 (35·0)	163·5 (35·0)
	r 3	0·1602	163·4 (13·4)	91·2 (9·6)	98·8 (13·7)
O(3)	*r* 1	0·1175	143·9 (12·2)	102·5 (8·0)	110·6 (10·6)
	r 2	0·1492	68·7 (15·5)	71·4 (9·2)	160·2 (15·6)
	r 3	0·1860	104·1 (7·2)	12·0 (7·2)	94·8 (8·6)
O(4)	*r* 1	0·1141	130·9 (16·1)	112·6 (11·2)	114·3 (13·3)
	r 2	0·1421	74·2 (15·8)	159·9 (10·9)	58·4 (12·6)
	r 3	0·1873	130·8 (10·0)	92·0 (7·2)	38·3 (9·8)
O(5)	*r* 1	0·1345	91·8 (19·2)	39·4 (38·4)	66·4 (42·0)
	r 2	0·1430	113·2 (45·7)	58·8 (56·2)	149·9 (53·5)
	r 3	0·1738	150·9 (11·8)	111·2 (11·0)	73·3 (10·5)
O(6)	*r* 1	0·1170	57·3 (12·8)	94·8 (8·0)	38·1 (12·7)
	r 2	0·1554	101·8 (58·4)	160·7 (64·4)	78·4 (39·3)
	r 3	0·1600	149·5 (82·2)	86·7 (72·8)	57·1 (81·0)
O(7)	*r* 1	0·1005	139·1 (9·2)	82·0 (6·0)	127·0 (9·3)
	r 2	0·1559	100·5 (9·1)	159·6 (10·7)	84·5 (9·4)
	r 3	0·1839	127·7 (13·9)	95·3 (12·2)	34·9 (13·8)
O(8)	*r* 1	0·1062	56·7 (12·0)	82·4 (5·7)	45·8 (10·2)
	r 2	0·1434	39·8 (26·7)	126·5 (22·0)	106·3 (19·3)
	r 3	0·1677	65·6 (15·0)	37·1 (11·5)	123·7 (16·4)
O(9)	*r* 1	0·1320	115·0 (18·4)	32·4 (17·9)	127·3 (20·1)
	r 2	0·1576	116·9 (26·1)	120·3 (13·7)	110·3 (17·7)
	r 3	0·1766	142·5 (22·2)	101·8 (15·8)	50·9 (22·3)
O(10) (H_2O)	*r* 1	0·1239	107·8 (24·6)	13·1 (17·6)	111·1 (23·5)
	r 2	0·1382	128·5 (29·4)	107·5 (18·8)	121·7 (25·6)
	r 3	0·1779	132·5 (12·6)	103·1 (8·4)	40·4 (12·8)

(Fig. 2). The $I(1)O_6$ group forms a dimer with a centrosymmetrically related $I(1)O_6$ group sharing the O(1)–O(1′) edge.

I(2) is closely bonded to O(4), O(5) and O(6) at distances of 1·817, 1·815 and 1·825 Å respectively, the O–I–O angles being 99·0, 99·4 and 95·1°. Four further oxygens, O(1), O(2), O(5) and O(9), form weak bonds at 2·737, 2·957, 3·050 and 3·172 Å respectively (Fig. 2). If we disregard the farthest oxygen O(9), the coordination polyhedron is again a highly distorted octahedron. O(1), O(6), O(5) and O(2) form an equatorial plane, while O(4) and O(5′) form apical oxygens, which are closer to the edge, O(2)–O(5) of the equatorial plane. If we include the farthest oxygen O(9) within the coordination sphere, the coordination polyhedron becomes a square pyramid with two further oxygens below the equatorial plane making two domes with the corners of the equatorial plane. Two $I(2)O_7$ groups, related by inversion, form a dimer by sharing the edge O(5)–O(5′).

The $I(3)O_3$ group has O(7), O(8) and O(9) at distances of 1·801, 1·823 and 1·795 Å respectively from the iodine atom. The O–I–O angles are 96·7, 101·0 and 101·1°. The last two angles are significantly larger than

Table 5. *Iodine*(V)*–oxygen bonds in crystalline iodates*

Compound	Coordination number of iodine		Distances (Å)		Coordination polyhedron	Reference
			O–I	I···O		
$LiIO_3$	6		1·809 (8) (×3)	2·892 (9) (×3)	Distorted octahedron	DeBoer *et al.* (1966)
$NaIO_3$	6		1·80	2·65	Distorted octahedron	McGillavry & Van Eck (1943)
			1·80	2·81		
			1·83	2·81		
NH_4IO_3	6		1·765 (8)	2·830 (8)	Distorted octahedron	Keve *et al.* (1971)
			1·806 (8)	2·778 (9)		
			1·836 (12)	2·819 (11)		
$RbIO_3$	6		1·807 (3) (×3)	2·763 (×3)	Distorted octahedron	Alcock (1972*b*)
$CuIO_3(OH)$	6		1·78	2·50	Distorted octahedron	Ghose (1962)
			1·82 (×2)	2·69 (×2)		
α-HIO_3	6		1·780 (10)	2·879 (10)	Distorted octahedron	Garrett (1954)
			1·816 (10)	2·767 (9)		
			1·899 (11)	2·503 (11)		
$Ce(IO_3)_4.H_2O$	6	I(1)	1·81	2·93	Distorted octahedron	Ibers (1956)
			1·83	3·00		
			1·84	2·99		
	6	I(2)	1·82	2·78		
			1·82	2·56		
			1·83	2·99		
	6	I(3)	1·82	2·78	Distorted octahedron	Ibers (1956)
			1·83	2·56		
			1·86	2·99		
	6	I(4)	1·82	2·73		
			1·83	3·10		
			1·86	2·51		
$Ce(IO_3)_4$	8		1·78 (9)	2·90 (9)	Antiprism	Cromer & Larson (1956)
			1·84 (9)	2·68 (9)		
			1·83 (9)	3·07 (9)		
				3·25 (9)		
				3·28 (9)		
$Zr(IO_3)_4$	8		1·81 (2)	2·94 (2)	Antiprism	Larson & Cromer (1961)
			1·84 (2)	2·55 (2)		
			1·85 (2)	2·83 (2)		
				2·94 (2)		
				3·11 (2)		
$Ca(IO_3)_2.6H_2O$	6		1·78 (3)	2·85 (2)	Distorted octahedron	Braibanti *et al.* (1971)
			1·90 (2)	2·86 (7)		
			1·85 (3)	2·89 (3)		
$Sr(IO_3)_2.H_2O$	6		1·786 (8)	3·168 (6)	Distorted pentagonal bipyramid	Manotti Lanfredi *et al.* (1972)
			1·806 (9)	2·853 (11)		
			1·825 (6)	3·219 (8)		
				2·846 (11)		
$3Cu(IO_3)_2.2H_2O$	6	I(1)	1·815 (5)	2·755 (5)	Distorted octahedron	Present work
			1·823 (5)	2·676 (5)		
			1·824 (5)	2·796 (6)		
	7	I(2)	1·817 (5)	2·957 (5)	Irregular	
			1·815 (5)	2·737 (5)		
			1·825 (5)	3·050 (5)		
				3·172 (5)		
	5	I(3)	1·801 (5)	2·771 (5)	Distorted trigonal bipyramid	
			1·823 (5)	2·873 (5)		
			1·795 (5)			

the average O–I–O angles within the $I(1)O_3$ (96·3°) and $I(2)O_3$ (97·9°) groups. In contrast to I(1) and I(2), I(3) forms only two weak bonds to O(5) and O(8) at distances of 2·771 and 2·873 Å respectively. The five-fold coordination around I(3) can be considered as a distorted trigonal bipyramid; O(5), O(8) and O(9) form a triangle, while O(7) and O(8′) form the apical oxygens (Fig. 2). The $I(2)O_5$ group also forms a dimer by sharing the edge O(8)–O(8′) with another centrosymmetrically related $I(3)O_5$ group.

If the symmetry of the IO_3 pyramidal groups only is considered, the $I(1)O_3$ group is very nearly an ideal trigonal pyramid with point symmetry $3m$. On the other hand, $I(2)O_3$ and $I(3)O_3$ groups deviate slightly but

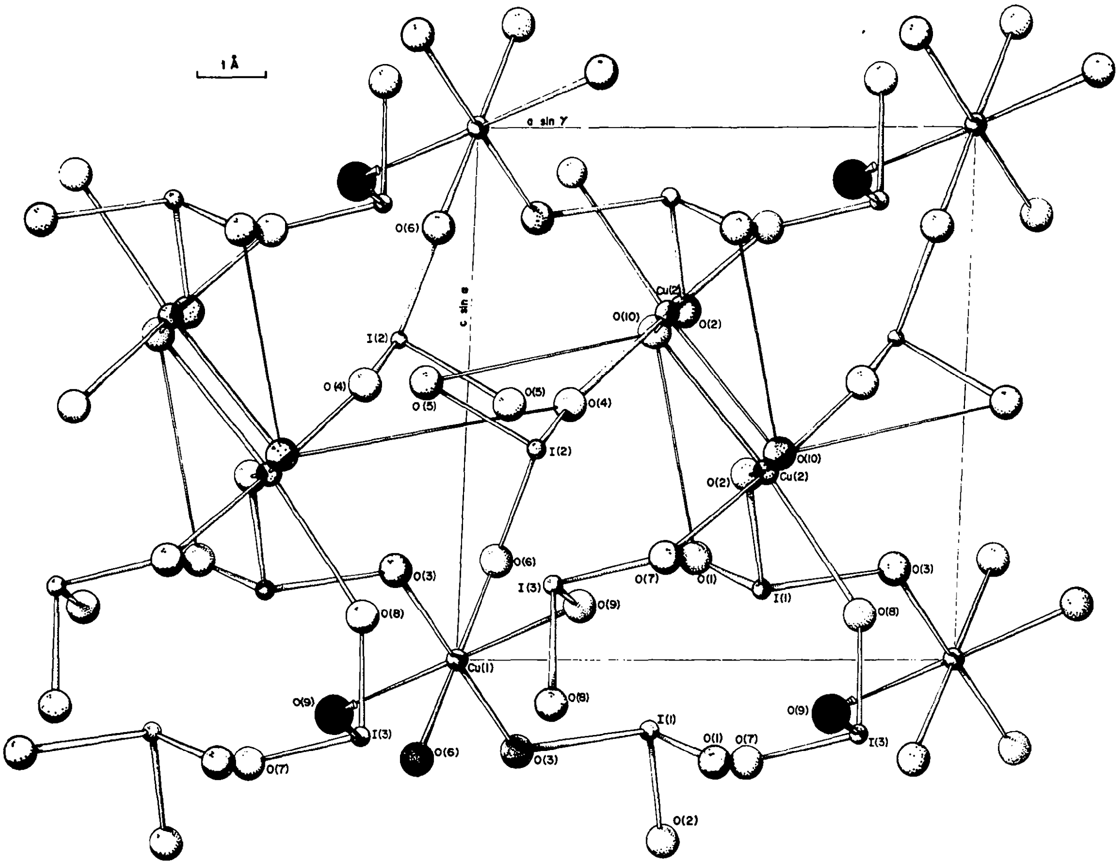


Fig. 1. A view of the bellingerite structure along the *b* axis. The bonds connecting O(10) (H_2O) with O(1) and O(5) are hydrogen bonds.

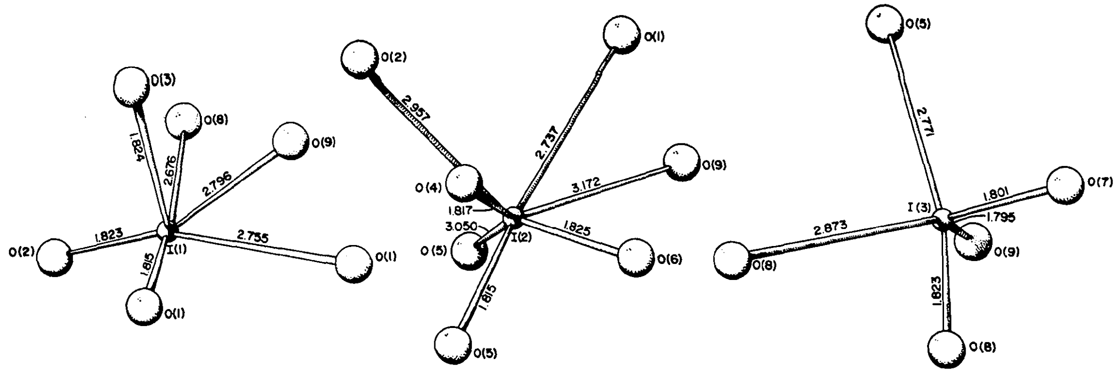


Fig. 2. Stereochemical configuration of $I(1)O_6$, $I(2)O_7$ and $I(2)O_5$ groups.

significantly from regular trigonal symmetry (particularly in O–I–O angles by as much as 4°) and approximate closely to the point-group symmetry *m*.

Configuration of the water molecule and hydrogen bonds

The water molecule O(10) is closely bonded to Cu(2) at 1·949 Å and to Cu(2′) at 2·483 Å. It has two close approaches to O(1) and O(5) both at 2·663 Å, which indicate strong hydrogen bonds (Fig. 3). The O(1)–O(10)–O(5) angle (106·2°) is very close to the H–O–H angle in a free H_2O molecule (104·5°). Hence it is expected that the H atoms will be located almost directly on the O(10)–O(1) and O(10)–O(5) directions. Around O(10), the configuration of the two hydrogen bonds, as well as of the bonds to Cu(2) and Cu(2′), is tetrahedral.

Three-dimensional framework

The crystal structure of bellingerite can be considered as a three-dimensional framework consisting of a corner-sharing Cu(1) octahedron, a Cu(2) octahedral dimer and three pyramidal iodate groups. The isolated Cu(1) octahedron shares corners with two oxygens each from groups $I(1)O_3$, $I(2)O_3$ and $I(3)O_3$. The Cu(2) octahedron shares four corner oxygens with iodate groups, O(2) with $I(1)O_3$, O(4) with $I(2)O_3$ and O(7) and O(8) with two separate $I(3)O_3$ groups.

Alternatively, if we consider the weak I–O bonds, the structure of bellingerite may be considered as a close packing of $I(1)O_6$, $I(2)O_7$ and $I(3)O_5$ groups, the interstices of which are filled by Cu(1) and Cu(2) ions and water molecules.

Discussion

Charge balance and bonding in the iodate groups

Usually, two of the three oxygens belonging to each iodate group are bonded to two separate copper atoms. Thus in the $I(1)O_3$ group, O(2) and O(3) are bonded to Cu(2) and Cu(1) respectively, while O(1) is only closely bonded to I(1). If we consider the close approaches to neighboring I(1) (2·744 Å) and I(2) (2·737 Å) atoms as weak bonds, as well as the H bond it receives from the water molecule O(10), the O(1) oxygen atom becomes charge-balanced.

Likewise within the $I(2)O_3$ group the charge balance of the O(5) oxygen, which is only bonded to iodine, is achieved through weak bonds to I(3) (2·771 Å) and I(2) (3·050 Å) and a H bond from O(10) (2·663 Å).

The $I(3)O_3$ shares O(7) with Cu(2), O(9) with Cu(1) and O(8) with another Cu(2) atom. However, the Cu(2)–O(8) bond (2·345 Å) is weak. For charge balance, O(8) is weakly bonded to I(1) (2·676 Å) and I(3) (2·873 Å). The configuration of the four bonds each around O(1), O(5) and O(8) is distorted-tetrahedral.

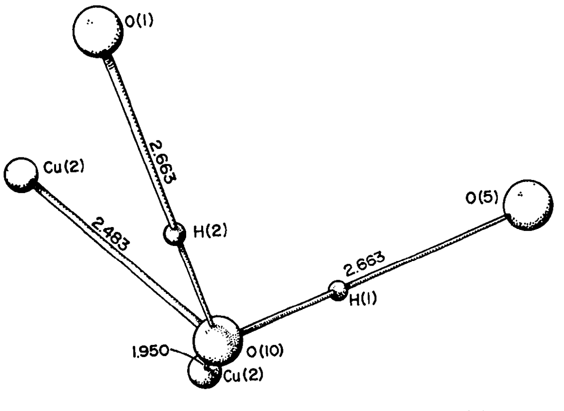


Fig. 3. Configuration of the hydrogen bonds (with assumed hydrogen positions) and Cu–O bonds around O(10) (H_2O).

Coordination number of iodine(V) *and the geometry of the iodine–oxygen coordination polyhedron*

In considering the iodine–oxygen interaction, I–O distances below 3·5 Å (van der Waals contact) would normally indicate weak bonds. In bellingerite, however, the shortest Cu(2)–I(1) distance is 3·2087 Å, which cannot be considered as a bonding interaction. Furthermore, there are a number of Cu–I distances between 3·2 and 3·5 Å (Table 3). Accordingly, we accept 3·20 Å as the practical limit for the consideration of weak I–O bonds. If this limit is extended to 3·5 Å, I(1), I(2) and I(3) become 9-, 8-, 6-coordinated respectively.

If we consider I–O contacts below 3·2 Å as weak bonds, the coordination number of iodine(V) in iodates investigated to date (Table 5) ranges from five to eight. The coordination polyhedron is a trigonal bipyramid for fivefold coordination, a distorted octahedron for sixfold coordination, a pentagonal bipyramid or irregular for sevenfold coordination and an Archimedean square antiprism for eightfold coordination. To the best of our knowledge, fivefold trigonal bipyramid coordination for idodine(V) is found for the first time in bellingerite.

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The Crystal Structure of the 1:1 Complex of Acetamide with 5,5-Diethylbarbituric Acid (Barbital)

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The 1:1 complex of acetamide and barbital ($C_2H_5NO.C_8H_{12}N_2O_3$), m.p. 116°C, is orthorhombic with space group $P2_12_12_1$. Lattice translations are a = 10·615 (2), b = 10·568 (2), c = 11·243 (2) Å. The crystal density (1·283 g cm $^{-3}$) agrees with the calculated density (1·281 g cm^{-3}) for four molecules of each component in the unit cell. The crystal structure has been determined from 1500 integrated intensities measured on a computer-controlled diffractometer with nickel-filtered Cu $K\alpha$ radiation. The final R index is 0·044. The crystal structure is isomorphous with the urea/barbital complex. The acetamide complex has slight differences in barbital conformation as well as in the relative translations and orientations of the component molecules, but is similar in having two strong hydrogen bonds (N···O, 2·84 Å) in which barbital is donor and the oxygen of the second component (acetamide) is acceptor.

Introduction

This crystal structure determination is one of a series involving NH···O=C hydrogen bonded complexes of drug-active barbiturates with various other amides. The crystal structure of the 1:1 complex of 5,5-diethylbarbituric acid (barbital, Fig. 1) with acetamide is of interest because it is isomorphous with the corresponding urea complex (Gartland & Craven, 1974). Thus the acetamide methyl group which does not hydrogen bond, replaces a urea amino group which does hydrogen bond, with only minor perturbation of the crystal structure.

Experimental

Transparent, prismatic crystals of the complex were grown from a saturated solution of barbital and acetamide in a solvent mixture of propanol and cyclohexane (4:1). The crystal density was determined by flotation in a mixture of benzene and carbon tetrachloride. Intensity data and unit-cell dimensions were measured on a four-circle computer-controlled diffractometer (Enraf-Nonius CAD-4) using nickel-filtered Cu $K\alpha$ radiation (λ = 1·5418 Å). The crystal data for the isomorphous complexes of barbital with acetamide and urea are given in Table 1.

Table 1. *Crystal data for the isomorphous* (1:1) *complexes barbital/acetamide and barbital/urea*

	Barbital/acetamide (This work)	Barbital/urea (Gartland & Craven, 1974)
	$C_8H_{12}N_2O_3.C_2H_5NO$	$C_8H_{12}N_2O_3.CH_4N_2O$
	Orthorhombic, space group $P2_12_12_1$	
m.p.	116°C	146–150°C
a	10·615 (2) Å	10·302 (5) Å
b	10·568 (2)	10·181 (2)
c	11·243 (2)	11·627 (3)
V	1261·2 Å^3	1219·5 Å^3
D_{meas}	1·283 g cm^{-3}	1·320 g cm^{-3}
D_x	1·281	1·330

1500 independent reflections were measured in the range $\theta \le 75°$. The crystal, which had dimensions 0·3 × 0·2 × 0·2 mm, was mounted so that there was an angle of 15° between the crystal axis a and the φ axis of the goniostat. Reflections were scanned in the ω–2θ mode at different rates to obtain a minimum net count of 5000 within a specified maximum scan time (90 s). The background counts were taken at each of the scan limits for $\frac{1}{4}$ of the scan time. The 2θ scan width in degrees was 1·2 + 0·4 tan θ. There were 86 reflections for which the integrated intensity (I) was less than $2\sigma(I)$ as calculated from the counting statistics. These