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The Crystal Structures of $KICl_2$ and $KICl_2.H_2O$

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The crystal structures of potassium dichloroiodide and potassium dichloroiodide monohydrate have been determined by X-ray diffraction. Both structures were refined from three-dimentional diffractometer data by least-squares methods to give $R=5·7\%$ and $R=6·0\%$ respectively. $KICl_2$ crystallizes in space group $P2_1/c$ with $a=8·507$ (5), $b=10·907$ (5), $c=12·126$ (5) Å, and $\beta=107·82$ (4)°; $Z=8$. $KICl_2.H_2O$ crystallizes in space group $P2_1/m$ with $a=8·022$ (5), $b=9·611$ (5), $c=4·354$ (3) Å, and $\beta=97·03$ (4)°; $Z=2$. In the anhydrous compound the two independent dichloroiodide ions are nearly linear and symmetric with average I–Cl bond lengths of 2·55 Å. In the hydrate the dichloroiodide ion is linear and symmetric by virtue of lying across a center of symmetry and has an I–Cl bond length of 2·55 Å. Each water molecule is weakly hydrogen-bonded to two chlorine atoms, each 3·39 Å from the oxygen atom.

Introduction

Trihalide ions are sometimes linked in the crystal by weak multicenter bonds, as in CsI_2Br (Carpenter, 1966). It is of interest to examine the relative roles of multicenter bonding and hydrogen bonding in crystals where both are possible. The structures of only two hydrated polyhalide compounds have been reported, that of $KICl_4.H_2O$ (Elema, de Boer, & Vos, 1963) and that of $HICl_4.4H_2O$ (Bateman & Bateman, 1972). Both exhibit weak hydrogen bonding (O–H$\cdots$Cl) and possible indications of bonding between chlorine atoms. A more direct comparison of multicenter bonds and hydrogen bonds should be afforded by the structures of the hydrated and anhydrous versions of the same compound. For this reason we chose to investigate $KICl_2$ and $KICl_2.H_2O$.

The preparation of 'potassium dichloroiodide' was first described by Wells, Wheeler & Penfield (1892). In the course of nuclear quadrupole resonance studies on the ICl_2^- ion (Cornwell & Yamasaki, 1957; Yamasaki & Cornwell, 1959), it became clear that both hydrated and anhydrous forms of $KICl_2$ exist. In both cases the data led the authors to propose for the ICl_2^- ion a bonding scheme based on *p* atomic orbitals. At about the same time Allison & Cheesman (1958) also showed, by physical and powder diffraction methods, that both $KICl_2$ and $KICl_2.H_2O$ exist.

Experimental

Sample preparation

Anhydrous $KICl_2$ was prepared by the method described by Wells (1901) for the preparation of $CsICl_2$; crystals formed upon cooling a hot aqueous solution of KNO_3, I_2, and HCl. The crystals were initially thought to be hydrated because they formed in aqueous solution and because Cremer & Duncan (1931) were able to obtain the anhydrous compound (only as a powder) by a strictly dry procedure. The true nature of our crystals was revealed by the structure determination.

Crystals of $KICl_2.H_2O$ were prepared according to the original methods of Wells, Wheeler & Penfield (1892) by bubbling chlorine through an aqueous solution of KCl and I_2.

Both compounds tended to lose iodine on standing, but decomposition could be prevented by coating them with a halocarbon grease (Kel–F).

Unit cell and space group

Crystallographic data for the two compounds are set out below.

	$KICl_2$	$KICl_2.H_2O$
System	Monoclinic	Monoclinic
a (Å)	8·507 (5)	8·022 (5)
b	10·907 (5)	9·611 (5)
c	12·126 (5)	4·354 (3)
β (°)	107·82 (4)	97·03 (4)
$a:b:c$ (X-ray)	0·7800:1:1·1118	0·8347:1:0·4530
$a:b:c$ (optical)	—	0·8319:1:0·4544
Absences	$h0l$, $l=2n+1$	
	$0k0$, $k=2n+1$	$0k0$, $k=2n+1$
Space group	$P2_1/c$	$P2_1/m$
Z	8	2
d_x (g cm^{-3})	2·91	2·53
d_m	2·87 (5)	2·44 (5)
μ(cm^{-1}), Mo $K\alpha$	75·3	62·0
Habit	parallelpiped bounded by {100}, {010}, {001}, {110}	parallelpiped bounded by {001}, {110}

Unit-cell and space-group data were obtained from preliminary Weissenberg and precession photographs and from 15 2θ values carefully measured with a Picker four-circle diffractometer. The optical axial ratios for the hydrate were measured by Penfield (Wells, Wheeler & Penfield, 1892). Systematic absences determined the space group of $KICl_2$ uniquely. For the hydrate the centrosymmetric alternative was chosen arbitrarily and was confirmed by the success of the refinement. Densities were measured by flotation in a mixture of CCl_4 and $CHBr_3$, in which the crystals slowly dissolved.

Intensity data

Three-dimensional intensity data for each crystal were collected with a Picker automatic four-circle diffractometer in the θ–2θ scan mode. Monochromation was obtained by means of a zirconium filter and pulse height analysis. Nonzero intensities were measured for 1249 independent reflections from the $KICl_2$ crystal, for 542 reflections from $KICl_2.H_2O$. A reflection was regarded as too weak to measure if the apparent intensity was less than twice its estimated standard deviation. Since the dimensions of the $KICl_2$ crystal were only about 0·15 × 0·10 × 0·11 mm, no absorption correction was applied. Since the $KICl_2.H_2O$ crystal was larger, 0·26 × 0·33 × 0·33 mm, the intensities were corrected for absorption by means of the *GONO* 9 program (Hamilton, 1956); transmission coefficients ranged from 0·21 to 0·31.

Structure-factor magnitudes were calculated in the usual way. The standard deviation in F^2 was estimated for both compounds by the expression

$$\sigma(F^2)=(1/\mathrm{Lp})[C+8{\cdot}25+(t_c/2t_b)^2(B_1+B_2+16{\cdot}5)+(0{\cdot}07C)^2+[0{\cdot}07(B_1+B_2)]^2]^{1/2}$$

where Lp is the Lorentz–polarization factor, C is the total integrated count obtained in time t_c, and B_1 and B_2 are the two background counts each obtained in time t_b. The scaler on our instrument introduces a small systematic error by dropping the last digit of a count instead of rounding it off; to remove the systematic part of this error each recorded value was multiplied by ten and 4·5 was added. The terms 8·25 and 16·5 take account of the remaining random error from this source.

Structure determination and refinement

Both structures were determined from Patterson and difference maps, calculated with the *FORDAP* program (Zalkin, 1965), and were refined by least-squares methods with the program *BULS*, a local variation of the classic *ORFLS* program (Busing, Martin & Levy, 1962).

$KICl_2$

From a three-dimensional Patterson function the two independent iodine atoms were located. The coordinates and isotropic temperature factor parameters of the iodine atoms were refined by two cycles of least-squares adjustment. At this stage $R=32\%$ and $R_w=39\%$ based on all nonzero $|F|$ values. An electron-density difference map then showed clearly the chlorine and potassium atoms. Further cycles of refinement of all atoms converged to $R=5{\cdot}7\%$ and $R_w=6{\cdot}6\%$ with anisotropic temperature factors. A final difference map showed no peaks larger than one-tenth the heigth of the potassium atom on the earlier map.

Final positional and thermal parameters, with their estimated standard deviations in parentheses, are listed in Table 1. The observed and calculated structure factors are compared in Table 2.

$KICl_2.H_2O$

With two formula units in the assumed space group $P2_1/m$, the iodine, potassium, and oxygen atoms are required to be on either inversion centers or mirror planes. The Patterson map could be interpreted in terms of iodine atoms at centers, potassium atoms on mirror planes, and chlorine atoms in general positions; the oxygen atom was not yet apparent. Least-squares refinement of the coordinates and isotropic thermal parameters of the known atoms yielded $R=11{\cdot}3\%$, $R_w=14{\cdot}7\%$ for reflections greater than 2σ only. A sub-

Table 1. *Atomic parameters for* $KICl_2$ *and* $KICl_2.H_2O$

Fractional atomic coordinates (× 10^4) and temperature factor parameters (× 10^4) in the expression

$$\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)].$$

Standard deviations in the last place are given in parentheses.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
$KICl_2$									
I(1)	1345 (1)	− 180 (1)	3847 (1)	126·9 (20)	78·4 (12)	50·1 (8)	25·4 (13)	36·0 (10)	0·6 (8)
I(2)	−2843 (1)	940 (1)	845 (1)	100·6 (19)	61·2 (11)	67·1 (10)	− 9·7 (12)	20·3 (11)	7·3 (8)
Cl(1)	3625 (6)	1425 (5)	4306 (4)	126 (8)	108 (6)	98 (5)	−80 (6)	36 (6)	1 (4)
Cl(2)	− 965 (6)	−1768 (5)	3363 (4)	169 (8)	89 (5)	64 (4)	9 (6)	35 (5)	0 (3)
Cl(3)	−3165 (6)	1905 (5)	2687 (4)	176 (9)	79 (5)	90 (5)	−24 (6)	41 (5)	−25 (4)
Cl(4)	−2402 (6)	−99 (5)	− 923 (4)	155 (8)	105 (5)	62 (4)	10 (6)	30 (5)	12 (4)
K(1)	2821 (5)	3021 (4)	4111 (4)	156 (8)	82 (4)	73 (4)	5 (5)	28 (4)	−7 (3)
K(2)	−4569 (6)	− 700 (5)	3060 (4)	125 (7)	143 (6)	90 (4)	−10 (6)	15 (5)	29 (4)
$KICl_2.H_2O$									
I	0	0	0	136·8 (20)	75·1 (14)	331·8 (72)	− 4·2 (8)	22·5 (23)	0·7 (1)
Cl	3034 (4)	− 405 (4)	2358 (7)	145 (4)	102 (3)	603 (18)	13 (3)	−20 (7)	0 (1)
K	−4620 (5)	2500	2259 (8)	2337 (8)	122 (5)	378 (18)	0	27 (9)	0
O	3159 (16)	2500	−3129 (27)	214 (23)	1145 (15)	679 (76)	0	49 (33)	0

sequent difference map showed the oxygen atom on a mirror plane. Several cycles of least-squares refinement, with anisotropic temperature parameters, converged to a structure with $R=6{\cdot}0\%$ and $R_w=7{\cdot}4\%$. An attempt to locate the hydrogen atoms by means of a difference map based on low-angle reflections, $(\sin\theta)/\lambda \le 0{\cdot}305$, was unsuccessful.

The final parameters are listed in Table 1. The calculated and observed structure factors are presented in Table 3.

Table 2. *Observed and calculated structure factor magnitudes for* $KICl_2$, *multiplied by*10

K	L	FOBS	FCAL
******H= 3******			
0	2	716	718
0	4	2580	2910
0	6	2227	2344
0	10	936	993
0	12	349	337
1	2	795	783
1	3	1483	1538
1	4	317	307
1	5	676	673
1	6	315	309
1	7	1001	1057
1	9	538	555
1	11	554	580
1	13	673	689
2	0	2003	2130
2	1	222	217
2	2	771	746
2	3	1274	1276
2	4	1765	1858
2	5	1353	1408
2	6	733	731
2	7	562	595
2	8	519	546
2	9	541	560
2	10	715	748
2	11	349	369
2	12	364	313
3	1	623	608
3	2	2243	2420
3	3	450	454
3	4	968	970
3	5	1330	1352
3	6	217	199
3	7	366	360
3	8	1117	1142
3	9	777	632
3	10	294	260
3	12	269	237
3	13	431	497
4	0	241	247
4	2	291	256
4	3	1338	1411
4	4	864	798
4	5	173	214
4	8	945	1034
4	9	585	536
4	10	303	231
4	11	474	497
4	12	456	460
4	13	259	262
5	1	1362	1387
5	2	1263	1274
5	3	713	731
5	4	539	530
5	5	1067	1071
5	8	589	577
5	9	448	456
6	0	575	576
6	1	867	901
6	2	282	275
6	3	1467	1527
6	4	541	554
6	5	213	203
6	6	253	237
6	7	414	447
6	8	709	717
6	9	383	364
6	12	307	304
7	1	1115	1139
7	4	635	638
7	5	1121	1123
7	6	531	558
7	7	236	172
7	8	493	526
7	9	267	259
7	10	321	303
8	1	725	762
8	2	473	474
8	3	965	1017
8	4	512	538
8	6	277	257
8	7	573	613
8	8	328	312
9	1	402	378
9	4	217	221
9	6	452	486
9	7	356	367
10	0	999	1079
10	2	253	249
10	3	412	442
10	4	431	492
10	6	687	699
10	7	390	430
11	1	444	442
11	2	399	404
11	3	229	147
11	4	259	220
11	5	462	452
11	6	417	377
11	7	409	384
12	0	352	396
12	2	245	118
12	4	247	215
******H= 1******			
0	-12	419	433
0	-10	295	280
0	-8	1826	1836
0	-6	1039	968
0	-4	3414	3936
0	-2	1976	1934
0	0	850	795
0	2	2370	2753
0	4	594	568
0	6	967	957
1	-11	778	742
1	-10	353	355
1	-8	611	588
1	-7	1235	1190
1	-6	972	895
1	-5	416	383
1	-4	479	446
1	-3	1191	1067
1	-2	192	154
1	-1	668	618
1	0	862	839
1	1	883	811
1	4	408	395
1	5	1781	1755
1	6	778	756
1	9	976	943
1	11	512	509
1	12	227	219
1	13	244	256
2	-12	502	532
2	-11	683	692
2	-10	339	306
2	-9	314	252
2	-8	1246	1222
2	-7	412	377
2	-6	452	384
2	-5	1079	1103
2	-4	795	632
2	-3	1256	1253
2	-2	1383	1255
2	-1	929	841
2	0	237	163
2	1	2035	2314
2	2	1404	1481
2	3	1221	1194
2	4	310	296
2	5	687	675
2	6	914	912
2	7	1118	1136
2	8	410	419
2	9	466	430
2	10	436	425
2	11	257	293
2	13	374	384
3	-13	462	448
3	-12	334	317
3	-11	465	459
3	-10	619	509
3	-9	746	755
3	-8	257	178
3	-7	943	915
3	-6	1587	1502
3	-5	834	841
3	-4	568	578
3	-3	1326	1209
3	-2	1113	1032
3	-1	749	652
3	0	1519	1543
3	1	2504	2769
3	2	812	874
3	3	893	928
3	4	1528	1613
3	5	1311	1381
3	6	436	450
3	7	1253	1132
3	9	550	562
3	11	258	268
4	-13	426	402
4	-12	294	318
4	-10	512	455
4	-7	415	426
4	-5	220	57
4	-2	918	823
4	-1	1251	1139
4	0	1448	1477
4	1	345	347
4	3	522	534
4	4	1671	1793
4	5	214	205
4	6	1093	1117
4	7	1103	1146
4	8	633	647
4	9	275	202
4	10	942	985
4	11	322	359
5	-13	543	504
5	-11	433	432
5	-10	450	378
5	-9	889	855
5	-7	1505	1538
5	-6	979	865
5	-4	340	320
5	-3	2911	2877
5	-2	369	340
5	-1	929	799
5	0	850	860
5	1	1016	1042
5	2	396	440
5	3	1446	1533
5	4	798	839
5	5	251	193
5	6	239	247
5	7	427	470
5	9	446	453
5	10	318	272
6	-11	249	282
6	-10	359	347
6	-9	553	543
6	-8	324	289
6	-7	863	797
6	-6	359	310
6	-5	1106	1040
6	-4	278	216
6	-2	495	463
6	-1	1571	1440
6	0	1167	1216
6	1	548	578
6	2	507	546
6	3	600	615
6	4	1150	1238
6	5	607	640
6	6	437	460
6	8	489	495
6	10	747	777
7	-9	697	640
7	-8	441	439
7	-7	719	728
7	-5	449	437
7	-4	267	231
7	-3	1714	1618
7	-1	671	622
7	0	389	419
7	1	1193	1288
7	2	995	1090
7	3	644	656
7	4	285	308
7	6	592	590
7	7	480	523
7	8	527	560
8	-11	492	485
8	-9	511	537
8	-8	282	285
8	-7	485	459
8	-6	227	165
8	-5	1274	1261
8	-2	692	642
8	-1	1371	1292
8	1	708	741
8	2	263	222
8	3	373	366
8	5	591	624
8	7	282	250
8	10	370	282
9	-6	286	302
9	-2	501	452
9	0	392	420
9	1	304	325
9	2	781	865
9	4	521	527
9	5	395	422
9	6	693	735
9	8	607	628
9	9	262	310
10	-8	344	296
10	-7	234	203
10	-6	252	198
10	-5	566	530
10	-4	574	599
10	-2	476	455
10	-1	654	664
10	1	277	348
10	2	548	614
10	3	331	426
10	5	301	361
10	6	281	219
11	-6	331	303
11	-4	383	339
11	-1	483	482
11	0	423	445
11	3	363	363
11	5	411	363
12	-2	322	280
12	-1	350	311
12	3	285	334
******H= 2******			
0	-14	415	370
0	-12	851	831
0	-10	1507	1514
0	-8	519	484
0	-6	2616	2702
0	-4	1341	1204
0	-2	1776	1658
0	0	1581	1568
0	2	306	269
0	4	212	131
0	6	548	520
0	8	415	400
0	10	632	614
1	-12	423	420
1	-10	428	442
1	-8	627	602
1	-7	524	477
1	-6	473	438
1	-5	325	262
1	-4	1488	1360
1	-3	1866	1775
1	-2	438	410
1	0	746	688
1	1	2497	2843
1	2	1235	1185
1	3	828	875
1	4	1103	1184
1	5	858	938
1	7	1547	1718
1	8	407	450
1	10	270	257
1	11	978	961
2	-14	313	337
2	-12	739	713
2	-10	959	928
2	-9	653	661
2	-7	1203	1135
2	-6	2122	2082
2	-5	472	441
2	-4	1428	1326
2	-3	1604	1512
2	-2	2691	2682
2	0	1320	1239
2	1	354	386
2	2	497	444
2	3	1034	1121
2	4	859	840
2	5	1345	1392
2	9	829	833
2	11	329	333
3	-12	277	217
3	-11	312	312
3	-10	354	346
3	-8	916	858
3	-6	468	433
3	-5	721	675
3	-4	1421	1279
3	-3	1919	1800
3	-2	1401	1282
3	-1	906	887
3	0	631	634
3	1	1469	1552
3	2	358	358
3	3	1430	1505
3	4	913	921
3	5	728	753
3	7	810	794
3	11	394	363
4	-8	238	250
4	-6	419	391
4	-4	1109	1008
4	-3	385	350
4	-2	1591	1473
4	-1	1045	1048
4	0	426	431
4	1	550	562
4	2	2000	2134
4	3	1263	1352
4	4	578	563
4	5	301	295
4	6	1071	1088
4	8	682	685
4	9	591	595
5	-11	863	851
5	-10	294	274
5	-9	752	748
5	-8	607	588
5	-7	1442	1530
5	-6	339	296
5	-5	1131	1053
5	-4	1055	945
5	-2	702	662
5	-1	1477	1506
5	0	651	675
5	1	806	830
5	3	246	286
5	5	455	445
5	8	237	217
5	11	223	139
6	-10	381	351
6	-9	442	454
6	-8	378	352
6	-7	323	396
6	-6	215	181
6	-5	439	407
6	-4	1070	1006
6	-3	593	557
6	-2	241	227
6	-1	432	417
6	0	403	424
6	2	1221	1326
6	5	362	307
6	6	967	956
6	7	250	253
6	8	733	698
6	10	275	288
7	-12	266	226
7	-11	483	455
7	-9	232	180
7	-7	550	593
7	-6	435	371
7	-5	560	556
7	-4	513	480
7	-3	249	220
7	-2	1021	964
7	-1	365	352
7	0	545	568
7	1	267	210
7	3	375	411
7	4	883	868
7	5	387	368
7	6	485	482
7	7	263	187
7	8	423	436
7	9	248	196
7	10	427	428
8	-10	433	398
8	-9	771	763
8	-7	583	630
8	-6	557	592
8	-5	692	671
8	-4	528	480
8	-3	940	893
8	-2	486	476
8	-1	255	256
8	0	450	474
8	1	592	578
8	3	458	493
8	4	352	334
8	6	275	294
9	-6	557	532
9	-5	258	189
9	-3	422	410
9	-2	607	612
9	0	1021	1092
9	1	572	629
9	2	277	260
9	3	494	525
9	4	1056	1074
9	5	422	392
9	6	363	349
9	7	510	470
9	8	365	396
10	-9	376	367
10	-6	508	489
10	-5	413	369
10	-4	323	277
10	-3	465	459
10	-2	281	226
10	0	563	600
10	1	532	595
10	3	253	220
11	-5	560	510
11	-4	508	474
11	-3	299	252
11	-2	270	249
11	1	526	555
11	2	313	316
11	3	257	285
11	5	239	124
12	-5	326	271
12	-4	283	273
12	-1	470	500
12	0	357	370
12	3	391	361
******H= 3******			
0	-14	705	716
0	-12	588	569
0	-10	982	1064
0	-8	651	667
0	-6	401	406
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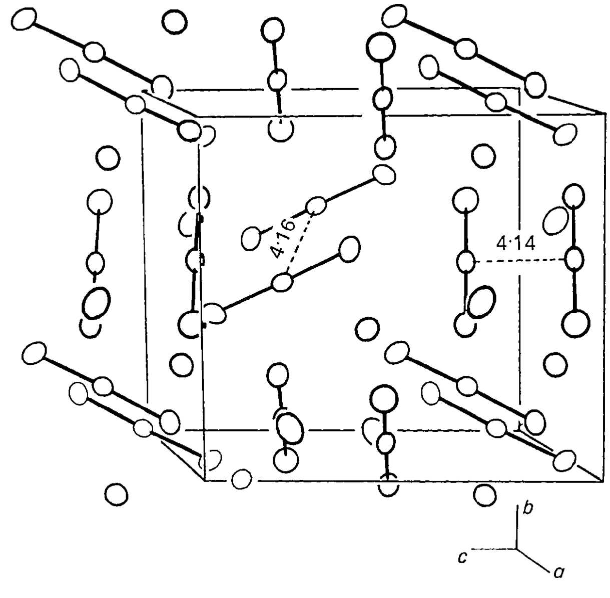

Fig. 1. Packing diagram of $KICl_2$, showing pairs of parallel ICl_2^- ions.

Discussion

The crystal structures of $KICl_2$ and $KICl_2.H_2O$ are illustrated in Figs. 1 and 2, respectively; interatomic distances and angles are given in Tables 4, 5, and 6.

Table 5. *Intermolecular approaches in* $KICl_2$ (Å)

Symmetry code No symbol	x	y	z
i	$-x$	$-y$	$1-z$
ii	$1+x$	y	z
iii	$-x$	$-\frac{1}{2}+y$	$\frac{1}{2}-z$
iv	$-1-x$	$\frac{1}{2}+y$	$\frac{1}{2}-z$
v	$-1-x$	$-y$	$-z$
vi	x	$\frac{1}{2}-y$	$-\frac{1}{2}+z$

I(1)···I(1^{i})	4·139 (4)	I(2)···I(2^{v})	4·156 (4)
I(1^{i})···Cl(2)	4·092 (5)	I(2^{v})···Cl(4)	4·162 (5)
Cl(1)···K(1)	3·281 (8)	Cl(3)···K(1)	3·157 (7)
Cl(1)···K(2^{i})	3·145 (7)	Cl(3)···K(2)	3·168 (9)
Cl(1)···K(2^{ii})	3·381 (8)	Cl(3)···K(2^{iv})	3·208 (9)
Cl(2)···K(2)	3·195 (7)	Cl(4)···K(2^{v})	3·159 (7)
Cl(2)···K(1^{i})	3·240 (8)	Cl(4)···K(1^{vi})	3·219 (8)
Cl(2)···K(1^{iii})	3·234 (8)	Cl(4)···K(1^{iii})	3·145 (7)

Table 3. *Observed and calculated structure factor magnitudes for* $KICl_2.H_2O$, *multiplied by* 10

H	K	FOBS	FCAL
••••••L= 0••••••			
1	0	436	393
2	0	522	636
3	0	916	1051
4	0	682	932
5	0	164	153
6	0	493	526
7	0	413	432
8	0	130	131
9	0	174	176
1	1	104	96
2	1	115	123
3	1	140	121
4	1	134	122
5	1	141	133
6	1	104	110
7	1	65	65
0	2	751	944
1	2	874	1033
2	2	396	326
3	2	875	1084
4	2	559	565
5	2	350	343
6	2	431	424
7	2	388	377
8	2	150	155
9	2	139	141
1	3	416	355
2	3	171	139
3	3	302	279
4	3	384	379
5	3	126	119
6	3	54	55
8	3	72	69
9	3	75	72
0	4	965	1278
1	4	444	398
2	4	656	640
3	4	702	667
4	4	584	599
5	4	264	244
6	4	425	431
7	4	313	358
8	4	124	123
1	5	331	274
2	5	226	197
3	5	90	27
4	5	103	90
5	5	84	77
6	5	170	163
7	5	110	107
0	6	363	361
1	6	676	682
2	6	446	418
3	6	553	506
4	6	390	334
5	6	396	364
6	6	259	259
7	6	209	198
8	6	160	165
1	7	220	207
2	7	113	97
3	7	156	148
4	7	252	212
5	7	75	64
7	7	58	49
0	8	421	422
1	8	320	309
2	8	467	466
3	8	278	244
4	8	315	289
5	8	265	261
6	8	220	230
1	9	101	92
2	9	73	70
0	10	109	108
1	10	325	330
2	10	264	276
3	10	179	174
4	10	163	156
••••••L= 1••••••			
-9	0	73	79
-8	0	311	311
-7	0	342	344
-6	0	324	323
-5	0	374	369
-3	0	355	361
-2	0	662	764
1	0	515	510
4	0	241	216
5	0	554	555
6	0	464	448
7	0	252	248
8	0	109	121
9	0	234	247
-6	1	49	50
-5	1	64	62
-4	1	92	94
-3	1	309	308
-2	1	329	333
-1	1	117	130
0	1	37	55
1	1	291	264
2	1	429	422
3	1	304	296
4	1	49	55
5	1	43	42
7	1	56	44
-9	2	101	112
-8	2	247	216
-7	2	446	456
-6	2	179	161
-5	2	488	505
-4	2	670	738
-3	2	614	696
-2	2	317	271
0	2	821	893
1	2	289	281
2	2	944	1042
3	2	761	771
4	2	368	350
5	2	387	357
6	2	562	581
7	2	137	128
8	2	177	182
-8	3	75	72
-7	3	73	72
-5	3	151	151
-4	3	111	114
-1	3	271	268
0	3	472	453
1	3	311	302
2	3	56	52
4	3	149	138
5	3	161	156
6	3	51	49
7	3	62	67
-8	4	243	238
-7	4	254	252
-6	4	318	333
-5	4	320	318
-4	4	626	677
-3	4	381	382
-2	4	694	632
-1	4	707	713
0	4	693	697
1	4	591	574
2	4	671	625
3	4	690	658
4	4	301	295
5	4	448	455
6	4	318	303
7	4	234	236
8	4	130	134
-9	5	77	71
-7	5	40	48
-6	5	73	73
-5	5	151	162
-4	5	36	40
-3	5	304	320
-2	5	328	327
0	5	175	175
1	5	107	97
2	5	408	391
3	5	327	312
5	5	137	140
6	5	41	42
7	5	96	96
8	5	72	63
-8	6	128	129
-7	6	257	258
-6	6	204	214
-5	6	338	354
-4	6	333	340
-3	6	488	520
-2	6	358	358
0	6	547	516
1	6	445	434
2	6	558	526
3	6	404	385
4	6	375	380
5	6	266	261
6	6	295	294
7	6	135	143
-7	7	38	48
-6	7	36	30
-5	7	121	124
-4	7	35	50
-3	7	51	58
-2	7	35	48
-1	7	143	148
0	7	266	262
1	7	150	144
3	7	48	53
4	7	81	85
5	7	122	121
7	7	54	60
-6	8	240	262
-5	8	193	206
-4	8	259	257
-3	8	261	297
-2	8	422	449
-1	8	263	251
0	8	366	360
1	8	446	455
2	8	289	271
3	8	344	277
4	8	284	238
5	8	246	261
6	8	111	108
-5	9	53	63
-4	9	43	27
-3	9	102	113
-2	9	113	114
0	9	45	52
1	9	47	36
2	9	131	133
3	9	131	121
-4	10	105	110
-3	10	237	276
-2	10	214	244
-1	10	189	194
0	10	233	237
1	10	279	300
2	10	209	202
3	10	155	148
-1	11	46	37
0	11	55	49
1	11	58	38
••••••L= 2••••••			
-9	0	132	131
-8	0	325	319
-7	0	211	201
-6	0	266	255
-5	0	582	625
-4	0	483	502
-3	0	374	378
-2	0	684	718
-1	0	983	1143
0	0	101	85
1	0	912	905
2	0	748	770
3	0	475	457
4	0	272	247
5	0	604	637
6	0	230	230
7	0	136	128
8	0	250	253
-7	1	59	58
-6	1	106	109
-5	1	122	120
-4	1	105	96
-3	1	89	82
-2	1	125	131
-1	1	171	169
0	1	132	111
2	1	79	72
3	1	61	54
4	1	49	42
5	1	61	59
6	1	86	76
7	1	57	49
-9	2	162	169
-8	2	263	248
-7	2	219	221
-6	2	271	273
-5	2	530	546
-4	2	481	486
-3	2	333	318
-2	2	843	935
-1	2	624	554
0	2	614	619
1	2	589	515
2	2	833	913
3	2	270	234
4	2	456	467
5	2	451	441
6	2	274	272
7	2	139	139
8	2	227	222
-8	3	39	30
-5	3	144	146
-4	3	261	261
-3	3	167	163
-2	3	83	79
-1	3	100	96
0	3	149	140
1	3	276	257
2	3	57	57
3	3	204	202
4	3	205	200
5	3	46	40
6	3	68	63
-8	4	242	236
-7	4	203	201
-6	4	241	244
-5	4	433	439
-4	4	414	420
-3	4	405	407
-2	4	503	496
-1	4	837	805
0	4	252	241
1	4	739	685
2	4	513	512
3	4	447	441
4	4	275	267
5	4	454	436
6	4	137	125
7	4	110	113
-7	5	44	47
-6	5	146	141
-5	5	47	46
-4	5	43	47
-3	5	139	134
-1	5	242	235
0	5	34	35
1	5	172	161
2	5	110	116
3	5	63	61
4	5	72	74
5	5	43	43
6	5	131	134
7	5	61	65
-7	6	147	139
-6	6	221	231
-5	6	274	275
-4	6	339	346
-3	6	324	345
-2	6	473	445
-1	6	366	314
0	6	564	596
1	6	311	324
2	6	451	454
3	6	276	273
4	6	356	364
5	6	203	213
6	6	254	276
-6	7	36	32
-5	7	93	83
-4	7	160	166
-3	7	92	99
-2	7	70	76
-1	7	43	48
0	7	74	72
1	7	173	170
2	7	43	47
3	7	137	132
4	7	141	139
6	7	71	65
-6	8	140	142
-5	8	154	179
-4	8	243	252
-3	8	311	330
-2	8	216	213
-1	8	373	360
0	8	271	292
1	8	371	359
2	8	211	192
3	8	306	326
4	8	200	204
5	8	141	176
-2	9	73	74
-1	9	44	44
1	9	43	57
2	9	51	44
-3	10	203	228
-2	10	144	148
-1	10	143	138
0	10	302	320
1	10	164	164
2	10	167	161
••••••L= 3••••••			
-8	0	116	138
-7	0	232	148
-6	0	313	312
-5	0	436	444
-4	0	164	158
-3	0	634	639
-2	0	522	503
-1	0	444	423
0	0	455	425
1	0	721	700
2	0	361	355
3	0	243	262
4	0	504	503
5	0	270	246
6	0	153	161
7	0	175	185
-6	1	46	45
-5	1	38	28
-4	1	42	41
-3	1	110	104
-2	1	96	87
-1	1	59	44
0	1	76	71
1	1	167	164
2	1	203	191
3	1	127	121
4	1	67	34
-8	2	142	195
-7	2	124	123
-6	2	372	172
-5	2	324	310
-4	2	334	347
-3	2	355	340
-2	2	720	723
-1	2	291	264
0	2	476	460
1	2	651	623
2	2	378	372
3	2	270	272
4	2	431	431
5	2	318	328
6	2	93	83
-7	3	59	57
-5	3	67	67
-4	3	35	34
-1	3	137	131
0	3	232	228
1	3	151	148
4	3	92	90
5	3	113	113
-7	4	193	191
-6	4	238	231
-5	4	354	352
-4	4	191	198
-3	4	495	493
-2	4	374	363
-1	4	423	412
0	4	340	372
1	4	509	491
2	4	434	426
3	4	270	273
4	4	367	365
5	4	194	197
6	4	159	178
-6	5	77	69
-5	5	72	74
-4	5	37	24
-3	5	155	154
-2	5	115	113
0	5	53	54
1	5	92	94
2	5	221	218
3	5	133	139
4	5	67	69
-6	6	220	216
-5	6	194	203
-4	6	249	313
-3	6	220	218
-2	6	405	349
-1	6	240	231
0	6	338	345
1	6	328	317
2	6	292	294
3	6	257	264
4	6	209	214
5	6	201	213
-5	7	57	63
-3	7	62	64
-1	7	91	90
0	7	146	154
1	7	83	80
4	7	63	58
-4	8	177	191
-3	8	252	254
-2	8	166	161
-1	8	298	306
0	8	231	242
1	8	197	200
2	8	221	226
3	8	210	221
-2	9	49	41
1	9	39	38
••••••L= 4••••••			
-6	0	242	234
-5	0	205	202
-4	0	266	261
-3	0	405	393
-2	0	339	339
-1	0	200	197
0	0	529	530
1	0	264	264
2	0	251	265
3	0	262	256
4	0	324	340
5	0	75	79
-6	1	55	54
-5	1	62	54
-4	1	34	40
-3	1	53	56
-2	1	91	92
-1	1	107	103
0	1	55	60
4	1	36	[illegible]
-6	2	257	249
-5	2	165	165
-4	2	279	273
-3	2	405	398
-2	2	224	223
-1	2	342	343
0	2	362	353
1	2	355	378
2	2	143	135
3	2	362	378
4	2	207	206
-5	3	72	69
-4	3	117	110
-3	3	65	65
0	3	83	87
1	3	94	103
3	3	70	65
4	3	70	63
-5	4	200	198
-4	4	217	223
-3	4	308	292
-2	4	306	304
-1	4	201	199
0	4	396	393
1	4	215	224
2	4	248	264
3	4	201	198
4	4	247	261
5	4	36	25
-3	5	40	31
-2	5	106	110
-1	5	119	118
1	5	66	59
3	5	47	44
-4	6	190	197
-3	6	226	229
-2	6	192	196
-1	6	272	276
0	6	187	194
1	6	244	262
2	6	145	163
0	7	56	55
••••••L= 5••••••			
-3	0	143	143
-2	0	214	223
-1	0	224	224
0	0	270	282
1	0	111	120
0	1	36	33
1	1	48	56
-2	2	119	118
-1	2	283	291
0	2	202	202

Table 4. *Bond lengths and angles*

The superscripts are defined in Tables 5 and 6.

$KICl_2$		$KICl_2.H_2O$	
I(1)-Cl(1)	2·545 (5) Å	I—Cl	2·553 (4) Å
I(1)-Cl(2)	2·550 (6)	O-H···Cl	3·386 (13)
I(2)-Cl(3)	2·558 (6)	O-H···Cl^{iii}	3·386 (13)
I(2)-Cl(4)	2·546 (6)		
Cl(1)-I(1)-Cl(2)	179·17 (23)°	Cl-I-Cl^{i}	180°
Cl(4)-I(2)-Cl(3)	177·05 (15)	Cl···O···Cl^{iii}	111·4 (4)

Table 6. *Intermolecular approaches in* $KICl_2.H_2O$ (Å)

Symmetry code

No symbol	x	y	z
i	$-x$	$-y$	$-z$
ii	$1+x$	y	z
iii	x	$\frac{1}{2}-y$	z
iv	x	$1+y$	z
v	x	y	$1+z$
vi	$1-x$	$-y$	$-z$
vii	$1-x$	$-y$	$1-z$

$Cl\cdots Cl^{iii}$	5·583 (3)	$I\cdots O$	3·877 (10)
$Cl^{iii}\cdots Cl^{iv}$	4·027 (11)	$I\cdots O^{i}$	3·877 (10)
$Cl\cdots Cl^{vii}$	3·751 (8)	$I\cdots O^{v}$	4·368 (13)
$Cl\cdots Cl^{vi}$	4·044 (9)	$I\cdots I^{v}$	4·354 (20)
$Cl\cdots Cl^{v}$	4·354 (20)	$K^{ii}\cdots O$	2·797 (15)
$O^{v}–H\cdots Cl$	3·386 (10)	$K\cdots O^{v}$	2·797 (15)
$O^{v}–H\cdots Cl^{iii}$	3·386 (10)	$K\cdots Cl^{vi}$	3·216 (8)
$O^{v}\cdots Cl^{vii}$	3·639 (11)	$K\cdots Cl^{vii}$	3·234 (7)
$O^{v}\cdots Cl^{v}$	3·710 (12)	$K\cdots Cl$	3·370 (6)

The dichloroiodide ion

In both compounds the ICl^- ion is exactly or very nearly linear and symmetric. In $KICl_2$ none of the four independent I–Cl distances is significantly different from their average, 2·550 Å. The independent Cl–I–Cl angles, 179·2 (2) and 177·1° (2), show statistically significant but minor deviations from linearity. In $KICl_2.H_2O$ the ion is exactly linear and symmetric, since it lies on a center of symmetry. The I–Cl distance is 2·553 (4) Å, not significantly different from the average value in $KICl_2$. These I–Cl distances are similar to those found in other examples of the same ion: 2·55 Å in $N(CH_3)_4ICl_2$ (Visser & Vos, 1964), average value 2·58 Å in piperazinium bis(dichloroiodide), $C_4H_{10}N_2.2HICl_2$ (Rømming, 1958). In the present examples the average I–Cl distance is 0·22 Å longer than the sum of the covalent radii of iodine and chlorine atoms. These facts are consistent with the idea that the three atoms are linked by a three-center four-electron bond (Rundle, 1962).

The packing

The structure of $KICl_2$ exhibits pairs of parallel dichloroiodide ions arranged in a herringbone pattern (Fig. 1). Each pair consists of two ions related by a center of symmetry. The pairs formed from the two crystallographically independent ions are remarkably similar (see Fig. 3 and Table 5). Within these pairs the iodine atoms are separated by 4·139 (4) and 4·156 (4) Å, slightly less than the van der Waals separation of 4·30 Å; no other distances are shorter than the van der Waals value. Adjacent pairs are well separated. Apparently the anions are drawn together in parallel orientation by van der Waals interaction, in spite of electrostatic repulsion, and then shifted lengthwise so that the iodine atoms of one fits into the constriction at the I–Cl bond of the other.

The arrangement of potassium ions around each of the two independent dichloroiodide pairs is shown in Fig. 3 and corresponding interatomic distances are given in Table 5. Each chlorine atom has three neighboring potassium ions. Around the anion Cl(1)–I(1)–Cl(2) the average $Cl\cdots K$ distance is 3·22 Å around Cl(2) and 3·27 Å around Cl(1). Around the anion Cl(3)–I(2)–Cl(4) the average $Cl\cdots K$ distance is 3·18 Å around both chlorine atoms. The similarity in the environment at each end of each anion implies a similar electrostatic potential in each case, as is usual for symmetrical trihalide ions (Migchelsen & Vos, 1967).

In $KICl_2.H_2O$ there are strips of parallel dichloroiodide ions running along the **c** direction (see Fig. 2). The $I\cdots I$ approach is 4·35 (2) Å, approximately equal to the van der Waals separation and somewhat longer than the corresponding distance in $KICl_2$. Nevertheless, the orientation of adjacent anions along these strips is similar to that of a pair of anions in $KICl_2$. Within a sheet of such strips extending along the **b** direction, the strips are alternately pivoted clockwise and counterclockwise about **c**. The water molecules and potassium ions fit into the spaces thus created.

The electrostatic environment around each dichloroiodide ion is symmetrical by virtue of the ion lying on a center of symmetry. Significant interatomic distances are given in Table 6.

Each oxygen atom lies on a mirror plane, with three pairs of mirror-related chlorine atoms nearby, as shown in Fig. 2. The shortest $O\cdots Cl$ distance, 3·39 (1) Å, is shown dotted in Fig. 2; the next shortest are more than 0·2 Å greater. Each of the shortest distances represents a weak hydrogen bond, although it is about 0·3 Å longer than a strong $O–H\cdots Cl$ bond such as that in $HCl.2H_2O$ (Lundgren & Olovsson, 1967). It is comparable with the reported hydrogen-bond lengths 3·35 Å in $HICl_4.4H_2O$ (Bateman & Bateman, 1972) and 3·42 Å in $KICl_4.H_2O$ (Elema, de Boer & Vos, 1963). As the latter authors pointed out, the increased length relative to that involving simple chloride ions is the result of the lesser charge on the chlorine atom. The

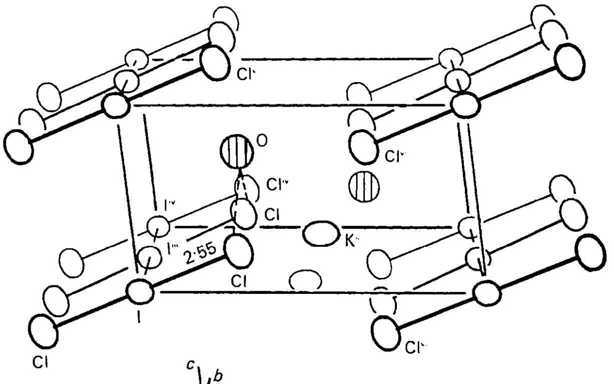

Fig. 2. Packing diagram of $KICl_2.H_2O$. The oxygen (shaded) and potassium atoms lie on mirror planes at $y=\frac{1}{4}$ and $\frac{3}{4}$. The dotted lines show the hydrogen bonds between the oxygen atom of the water molecule and the chlorine atoms.

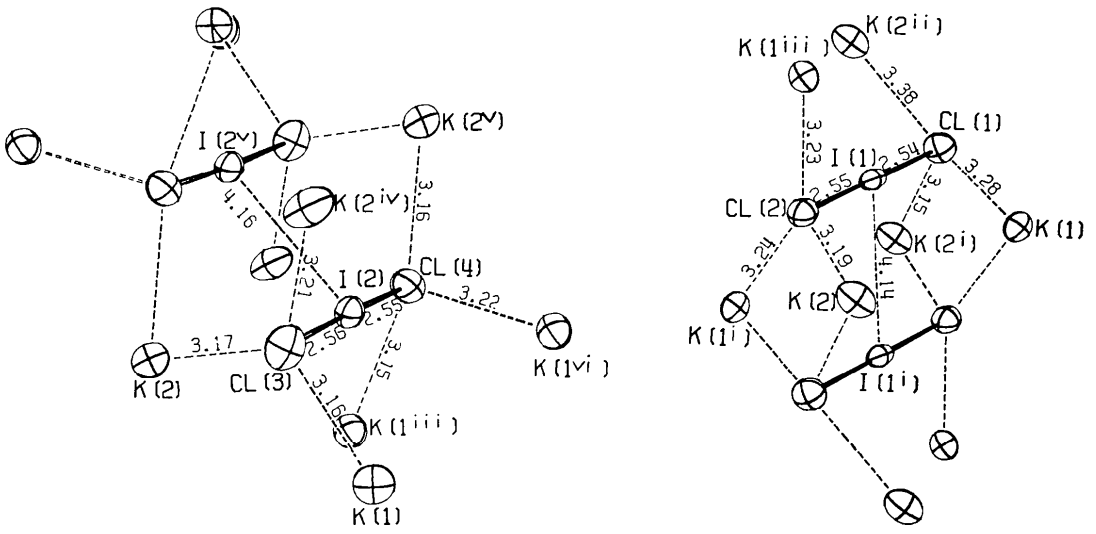

Fig. 3. Arrangement of K^+ ions around the ends of the two independent ICl_2^- ion pairs in $KICl_2$.

angle Cl···O···Cl between two hydrogen bonds is 111·4°, which is reasonable for a hydrogen-bonded system. The existence of some hydrogen bonding was confirmed by the infrared absorption spectrum of a Nujol mull recorded with a Perkin-Elmer 257 spectrometer. The spectrum showed a broad band at 3150 cm^{-1} with width at half height of 270 cm^{-1}; such a band indicates hydrogen bonding (Hamilton & Ibers, 1968).

Although evidence is found for multicenter bonds linking trihalide ions in other examples, as mentioned in the introduction, there is no indication of such a feature in either of these structures. This illustrates the reluctance of a halogen as electronegative as chlorine to serve as the central atom in a multicenter bond.

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