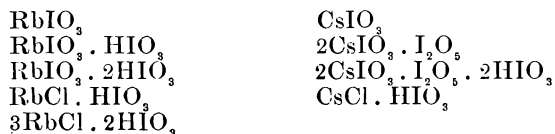


ART. XVIII.—*On some Alkaline Iodates*; by H. L. WHEELER.  
*With Crystallographic Notes*; by S. L. PENFIELD.

WHILE work on the compounds of iodine trichloride with alkaline chlorides\* was in progress in this laboratory, it was noticed in making  $\text{KCl} \cdot \text{Cl}_3\text{I}$ ,  $\text{RbCl} \cdot \text{Cl}_3\text{I}$  and  $\text{CsCl} \cdot \text{Cl}_3\text{I}$  that white crystals were often formed under certain conditions. These compounds proved to be  $\text{KCl} \cdot \text{KIO}_3 \cdot \text{HIO}_3$ ,  $\text{RbCl} \cdot \text{HIO}_3$  and  $2\text{CsIO}_3 \cdot \text{I}_2\text{O}_5$ . Since they were not analogous, although formed under similar conditions, and since the rubidium and caesium salts have not been described, an investigation of them was undertaken. Attempts to prepare these compounds by other methods led to the discovery of several other iodates. The new compounds that have been prepared are as follows:



\* This Journal, xliv, 42.

The compound which separated from the solution of the potassium pentahalide has already been described, but since this is a new method of preparation and since there are conflicting statements concerning its state of hydration, it has been re-investigated.

The results of the investigation of the rubidium salts show that the normal iodate is the only one of the series that can be recrystallized unaltered from an aqueous solution. In the case of the caesium compounds, the normal iodate and the salt  $2\text{CsIO}_3 \cdot \text{I}_2\text{O}_5$  are not decomposed by water. The other caesium iodates give  $2\text{CsIO}_3 \cdot \text{I}_2\text{O}_5$  when recrystallized from water and not the normal iodate, thus showing an interesting difference between the rubidium and caesium compounds.

It is the tendency of the acid rubidium iodates to separate in a higher state of hydration than the corresponding caesium compounds.

It is also an interesting fact that the formation of the compounds of normal chloride and iodic acid was not observed on mixing the constituents. In the case of rubidium, products were obtained which proved to be  $\text{RbIO}_3$ ,  $\text{RbIO}_3 \cdot \text{HIO}_3$  or  $\text{RbIO}_3 \cdot 2\text{HIO}_3$  according to the concentration of the solutions and the excess of  $\text{RbCl}$  or  $\text{HIO}_3$ . On the other hand by adding hydrochloric acid to a solution of rubidium iodate, if the acid is dilute  $\text{RbIO}_3 \cdot 2\text{HIO}_3$  is formed, if concentrated the iodate is completely decomposed. Similar experiments under taken with caesium chloride and iodic acid, did not give the peculiar double compound  $\text{CsCl} \cdot \text{HIO}_3$  but resulted, in each case, in the formation of  $2\text{CsIO}_3 \cdot \text{I}_2\text{O}_5$ .

#### *Method of Analysis.*

After the substances were prepared for analysis as described in detail beyond, the halogens were determined by first reducing the solution of iodate with sulphur dioxide then precipitating with silver nitrate in the presence of nitric acid. This precipitate was then heated in a stream of chlorine, thus combining the test for chlorine and its determination in one operation. In the filtrate from the silver precipitate the alkali metal was determined as sulphate after the removal of the excess of silver by means of hydrogen sulphide. Oxygen was determined in a separate portion by precipitation with silver sulphate, drying the precipitate at  $100^\circ$  and then determining the loss on ignition. Duplicate halogen determinations were then made in this residue. In the case of the compounds containing the group  $\text{I}_2\text{O}_5$ , where an error would be introduced if the oxygen was determined in this manner, the substance itself was ignited and the oxygen calculated from the loss.

The presence of water in these compounds was determined by directly weighing it in a calcium-chloride tube, the substance being ignited in a combustion tube containing a mixture of lead chromate and lead oxide.

*Normal rubidium iodate,  $RbIO_3$ .*—This compound was made by adding one molecule of iodine pentoxide in either strong or dilute aqueous solution, to a solution of one molecule of rubidium carbonate. If the solutions are strong, the iodate separates as a sandy precipitate, but if they are hot and dilute it separates on cooling in small grains or as a crystalline crust. At 23° 100 parts of water dissolve 2.1 parts of the salt. The compound, after filtering on the pump, washing with a little water and drying on paper, gave the following results on analysis:

	Found.	Calculated for $RbIO_3$ .
Rubidium,	32.17	32.82
Iodine,	48.50	48.75
Oxygen,	20.59	18.43

The salt when heated decrepitates strongly, melts, gives off oxygen but no iodine, and the residue is rubidium iodide. Hydrochloric acid readily dissolves it in the cold to a faint yellow-colored solution which increases in color on standing. On warming, chlorine is evolved and the solution turns bright yellow from the formation of iodine trichloride. If boiled with strong hydrochloric acid,  $RbCl \cdot ClH^*$  is formed which separates on cooling.

The formation of normal rubidium iodate was also observed: when a hot dilute aqueous solution of iodine trichloride was treated with rubidium carbonate. The compound thus obtained gave 48.43 per cent of iodine on analysis. Also by dissolving the acid iodate in a strong hot solution of rubidium chloride and allowing the mixture to crystallize. This was identified by a rubidium determination which gave 32.58 per cent. In general the iodates of rubidium all give this body when they are dissolved in hot water and the solutions left to crystallize. The products obtained in this manner decrepitated on heating and did not give off iodine. A rubidium determination in the substance obtained from  $RbCl \cdot HIO_3$  gave 32.76 per cent; from  $3RbCl \cdot 2HIO_3$ , 32.22 per cent.

*Acid rubidium iodate,  $RbIO_3 \cdot HIO_3$ .*—This was obtained by mixing warm solutions of one molecule of iodine pentoxide and two molecules of rubidium chloride. The compound generally separates on cooling as a heavy crystalline powder. It is difficultly soluble in cold water. Hot water dissolves it more readily and on cooling, the normal iodate separates. It

\* This Journal, xliii, 475.

is insoluble in alcohol. The crystals were filtered on a pump and washed with a little cold water and then pressed on paper. An analysis of these dried at 100° gave the following results, the oxygen being determined by difference.

		Calculated for $\text{RbIO}_3 \cdot \text{HIO}_3$ .
Rubidium,	20.13	19.58
Iodine,	58.12	58.19
Oxygen,	21.46	21.99
Hydrogen,	.29	.23

The reaction which takes place in the preparation of this compound is probably according to the following equation :



The hydrochloric acid thus liberated reacts on a part of the iodic acid, chlorine is evolved and the solution becomes yellow. When heated it does not decrepitate, but melts to a yellow mass, gives off water, then iodine and finally froths with the evolution of oxygen. The residue consists of rubidium iodide.

*Diacid rubidium iodate,  $\text{RbIO}_3 \cdot 2\text{HIO}_3$ .*—For the preparation of this compound 5 grams of  $\text{RbIO}_3$  were dissolved in 50 cc. of water with the aid of heat, then 13 grams of iodine pentoxide in 50 cc. of water were added, the mixture boiled down to half its volume and allowed to cool. The body separates as a heavy crystalline powder. It is difficultly soluble in cold water. When dissolved in hot water and the solution left to crystalline  $\text{RbIO}_3$  separates. The product obtained as stated above was separated from the mother liquor by filtering on the pump, washed with a little cold water and dried at 100°.

	Found.		Calculated for $\text{RbIO}_3 \cdot 2\text{HIO}_3$ .
Rubidium,	13.93	14.13	13.96
Iodine,	61.91	62.48	62.20
Oxygen,	23.74		23.51
Hydrogen,	.42		.33

This compound does not lose water at 100°. When heated it does not decrepitate, but melts, gives off water, then iodine and oxygen, leaving a residue of rubidium iodide. The compound was also obtained by adding 10 cc. of hydrochloric acid sp. gr. 1.1 to 5 grams of  $\text{RbIO}_3$  in 20 cc. of water. The mixture was warmed until all the  $\text{RbIO}_3$  dissolved. It gave a faint yellow solution which slowly deepened in color. On standing, a well crystallized product of the compound under consideration was obtained containing 14.13 per cent of rubidium and 62.19 per cent of iodine.

The addition of a saturated solution of rubidium chloride to syrupy iodic acid produces a precipitate which dissolves again in the excess of iodic acid. When more rubidium chloride is added, the whole being kept over a lamp, a point is reached where a precipitate begins to form in the hot solution. This is the compound in question. It was identified by a rubidium and an iodine determination. This gave 14.17 per cent of rubidium and 61.83 per cent of iodine.

*RbCl . HIO<sub>3</sub>*.—This salt can be made by simply allowing a saturated solution of  $\text{RbCl} \cdot \text{Cl}_3\text{I}$  to stand for some hours, when large colorless prisms form, attached to the plates of  $\text{RbCl} \cdot \text{Cl}_3\text{I}$ . The solution, after removing the crystals, warming to dissolve the pentahalide and pressing chlorine in again, does not yield a further deposit of the substance. This is explained by the fact that so much hydrochloric acid is formed in the solution that the formation of this compound is prevented. The crystals remain unaltered on exposure to the air. On treatment with cold water they are decomposed, losing their luster and becoming white. The solution has an acid reaction towards litmus. The hot saturated solution of this compound gives the normal iodate on cooling. The material for analysis was mechanically separated from adhering  $\text{RbCl} \cdot \text{Cl}_3\text{I}$  and dried in the air.

	Found.		Calculated for $\text{RbCl} \cdot \text{HIO}_3$ .
Rubidium,	28.88		28.78
Iodine,	42.29	42.62	42.76
Chlorine,	12.09	12.13	11.95
Oxygen,	16.33		16.16
Hydrogen,		.26	.33

This salt can also be prepared by adding a strong aqueous solution of rubidium hydrate to a strong solution of iodine trichloride in water. This gives at first a precipitate of the compound  $3\text{RbCl} \cdot 2\text{HIO}_3$ , and the solution left at rest for a few days gives the large well developed crystals of  $\text{RbCl} \cdot \text{HIO}_3$  unmingled with  $\text{RbCl} \cdot \text{Cl}_3\text{I}$ . These were identified by their crystalline form.

On warming the crystals with hydrochloric acid  $\text{RbCl} \cdot \text{Cl}_3\text{I}$  is formed, probably according to the following equation:



and the  $\text{RbCl} \cdot \text{Cl}_3\text{I}$  on further heating gives  $\text{RbCl} \cdot \text{ClI}$  with the liberation of chlorine. When the substance is heated it melts, gives off water, chloride of iodine, and oxygen the residue consists of rubidium chloride and iodide. A determination of the halogens in this residue gave 3.52 per cent of chlorine and 53.66 per cent of iodine.

$3RbCl \cdot 2HIO_3$ .—This compound, which is analogous to the sodium compound  $3NaCl \cdot 2NaIO_3 \cdot 9H_2O$  described by Rammeisberg\* and also to the salt  $3NaI \cdot 2NaIO_3 \cdot 19H_2O$  obtained by Penny† or  $3NaI \cdot 2NaIO_3 \cdot 20H_2O$  according to Marignac,‡ except that it contains no water of crystallization, was prepared by two methods. It was obtained by the addition of a hot, strong aqueous solution of rubidium hydroxide to a strong solution of iodide trichloride, the latter being in excess. The mixture was then filtered hot and on cooling, a mass of fine needles separated. The mother liquor on standing yielded the large crystals of  $RbCl \cdot HIO_3$ . The needles are stable in the air and at  $100^\circ$ . From the hot saturated aqueous solution of the compound the normal iodate separates on cooling.

The formation of this compound was also observed on adding a strong solution of rubidium carbonate to a hot saturated solution of  $RbCl \cdot Cl_3I$ , the latter being in excess. The colorless slender transparent needles thus obtained generally separate in groups radiating from a point on the surface of the yellow crystals of  $RbCl \cdot Cl_3I$ . After separating the colorless crystals mechanically from the pentahalide they were air-dried on paper and then analyzed, while the material obtained according to the previous method was dried at  $100^\circ$ .

	From RbOH and $ICl_3$ .		From $Rb_2CO_3$ and $RbCl \cdot Cl_3I$ .		Calculated for $3RbCl \cdot 2HIO_3$ .
Rubidium,	35.41	34.58	35.78		35.87
Iodine,	35.27	36.00	35.87	35.81	35.52
Chlorine,	14.99	14.82	15.26	15.16	14.90
Oxygen,		13.15		13.64	13.43
Hydrogen,		.29		.30	.28

When heated, the substance does not decrepitate but melts, gives off chloride of iodine and the residue consists of a mixture of rubidium chloride and iodide. A sample of this residue gave on analysis 9.68 per cent of chlorine and 38.91 per cent of iodine.

*Normal Cæsium iodate*  $CsIO_3$ .—This was prepared by adding a moderately strong aqueous solution of iodic acid to a strong solution of cæsium carbonate, care being taken to have the carbonate in excess. When all the iodic acid had been added, the solution was boiled. On cooling, a crystalline mass separated consisting apparently of small cubes. At  $24^\circ$ , 100 parts water dissolve 2.6 parts of the salt. It is insoluble in alcohol. The body was prepared for analysis by filtering on the pump, washing with cold water and then pressing on paper and drying at  $100^\circ$ .

\* Pogg. Ann., xlv, 548; cxv, 584.

† Ann. Ch. Pharm., xxxvii, 202. ‡ Jahresb., 1857. 124: Ann. Min., V, ix, 1.

			Calculated for CsIO <sub>3</sub> .
Cæsium,	43·08	43·53	43·18
Iodine,	40·84		41·23
Oxygen,	15·74		15·59

This was also obtained in attempts to prepare a cæsium salt corresponding to  $3\text{RbCl} \cdot 2\text{HIO}_3$  by adding cæsium hydrate or carbonate in moderately strong aqueous solution to a strong solution of iodine trichloride in excess, when it at once separated in the form of a white sandy precipitate, which under the microscope was seen to consist of transparent grains of indefinite form. Unless the iodine trichloride is nearly saturated with the carbonate,  $\text{CsCl} \cdot \text{Cl}_3\text{I}$  or  $\text{CsCl} \cdot \text{ClI}^*$  is obtained mixed with the iodate. An iodine and oxygen determination in the air-dried salt gave 40·55 and 40·83 per cent of iodine and 15·67 per cent of oxygen. When this iodate is heated it does not give off iodine but melts and evolves oxygen. The residue is cæsium iodide.

$2\text{CsIO}_3 \cdot \text{I}_2\text{O}_5$ .—This substance can be prepared in pure condition and in large quantity by mixing a moderately dilute aqueous solution of two molecules of cæsium chloride with one molecule of iodine pentoxide dissolved in a little water. Any precipitate that may have been produced is dissolved by the aid of heat and more water if necessary. On cooling, the compound separates as a sandy powder. This can be washed with water or recrystallized from hot water without decomposition. It can also be recrystallized from dilute solutions of iodic acid. At 21° 100 parts of water dissolve 2·5 parts of this salt. It is insoluble in alcohol. The material for analysis was air-dried after pressing on paper.

	Found.	Calculated for $2\text{CsIO}_3 \cdot \text{I}_2\text{O}_5$ .
Cæsium,	27·93	28·00
Iodine,	53·42	53·47
Oxygen,	18·69	18·53

This compound invariably separates along with the crystals of  $\text{CsCl} \cdot \text{Cl}_3\text{I}$ , when the latter is prepared in the absence of hydrochloric acid. The yield is not very large. It is thus obtained in the form of small rounded white nodules which on close inspection are seen to occur in pairs, the two nodules being on opposite sides of a thin layer of the pentahalide. They were mechanically separated from the pentahalide, no water being used to wash the compound when prepared for analysis. The following results are sufficient for its identification.

\* This Journal, III, xliii, 17, and xliiv, 42.

Cæsium .....	29.11
Iodine .....	50.21
Oxygen .....	18.99
Chlorine .....	3.24

This compound was also obtained by the following methods. By mixing 6 grams of  $\text{CsIO}_3$ , 20 cc. of water and 10 cc. of  $\text{HCl}$  sp. gr. 1.1. The mixture was boiled, it became yellow and chlorine was evolved. When cooled the substance separated as a crystalline crust. It was identified by a determination of cæsium which gave 28.40 per cent.

The compounds  $2\text{CsIO}_3 \cdot \text{I}_2\text{O}_5 \cdot 2\text{HIO}_3$  and  $\text{CsCl} \cdot \text{HIO}_3$  give this body when their hot saturated solutions are cooled. A cæsium determination in the products thus obtained gave 27.94 and 28.12 per cent respectively.

When this body is treated with hydrochloric acid, sp. gr. 1.1, the solution becomes yellow, evolves chlorine on warming, and when concentrated on the water bath yields on cooling well crystallized  $\text{CsCl} \cdot \text{ClI}$ . Analysis gave 50.63 per cent of cæsium chloride, calculated for  $\text{CsCl} \cdot \text{ClI}$  50.90 per cent.

When heated in a closed tube it gives no sign of water, gives off iodine then melts with the evolution of iodine and oxygen. The residue consists of cæsium iodide.

$2\text{CsIO}_3 \cdot \text{I}_2\text{O}_5 \cdot 2\text{HIO}_3$ .—This body was obtained by adding 5 grams of  $2\text{CsIO}_3 \cdot \text{I}_2\text{O}_5$  to a boiling solution of 25 grams of iodine pentoxide in sufficient water to form a syrup. Water was then added and the precipitate thus produced proved to be the compound in question. Thus produced it separates as a finely divided amorphous precipitate which can be dried in the air or at  $100^\circ$  without losing water. It is difficultly soluble in water and when crystallized from an aqueous solution gives  $2\text{CsIO}_3 \cdot \text{I}_2\text{O}_5$ . An analysis of the substance dried at  $100^\circ$  gave

	Found.	Calculated for $2\text{CsIO}_3 \cdot \text{I}_2\text{O}_5 \cdot 2\text{HIO}_3$ .
Cæsium,	19.71	20.43
Iodine,	57.68	58.52
Oxygen,	20.41	20.89
Hydrogen,	.12	.16

Water determinations in samples dried in the air on paper gave 1.45 and 1.38 per cent; theory requires 1.44.

When the substance is heated it gives off water and iodine, then oxygen, the residue consisting of cæsium iodide.

$\text{CsCl} \cdot \text{HIO}_3$ .—This was obtained in an attempt to increase the yield of  $2\text{CsIO}_3 \cdot \text{I}_2\text{O}_5$  by adding a rather small quantity of cæsium carbonate to a hot saturated solution of  $\text{CsCl} \cdot \text{Cl}_3\text{I}$ , when on cooling and allowing the mixture to stand, colorless,



flat, transparent prisms separated on the yellow crystals of  $\text{CsCl} \cdot \text{Cl}_3\text{I}$  previously formed. These colorless prisms were picked out from the solution dried on paper and separated mechanically, as far as possible from any adhering  $\text{CsCl} \cdot \text{Cl}_3\text{I}$ . These on analysis gave the following results.

	Found.		Calculated for $\text{CsCl} \cdot \text{HIO}_3$ .
Cæsium	38.09		38.60
Iodine	36.08	36.29	36.86
Chlorine	11.69	11.82	10.31
Oxygen	13.85		13.94
Hydrogen	.30		.29

The crystals remain unaltered on exposure to dry air but on treating them with water they immediately become opaque. On recrystallizing from water they give  $2\text{CsIO}_3 \cdot \text{I}_2\text{O}_5$ . When the substance is heated it gives off water and iodine chloride, melts, and gives off oxygen, the residue consisting of chloride and iodide of cæsium. When it is warmed with hydrochloric acid it undergoes the same decomposition as the corresponding rubidium compound.

$\text{KCl} \cdot \text{KIO}_3 \cdot \text{HIO}_3$ .—This compound has previously been prepared by treating  $\text{KIO}_3$  with hydrochloric acid, or a solution of iodine trichloride with potassium hydrate or carbonate. It has been described by Serullas\* and Rammelsberg† as anhydrous and the formula  $2\text{KCl} \cdot 2\text{KIO}_3 \cdot \text{I}_2\text{O}_5$  was assigned to the salt. Millon‡ from his determination of potash in this salt concluded that the substance contained a molecule of water, but he made no determination of it. Finally, Marignac§ who examined it more carefully, made a determination of the water by drying the substance at  $100^\circ$ , then igniting it in a tube with metallic copper and collecting, and weighing the water by means of a sulphuric acid tube.

The compound obtained from a solution of  $\text{KCl} \cdot \text{Cl}_3\text{I}$  separated in shining transparent prisms, stable in the air. It contained water corresponding to the formula  $2\text{KCl} \cdot 2\text{KIO}_3 \cdot \text{I}_2\text{O}_5 \cdot \text{H}_2\text{O}$  or  $\text{KCl} \cdot \text{KIO}_3 \cdot \text{HIO}_3$ . An analysis of the air-dried salt gave the following results.

	Found.		Calculated for $\text{KCl} \cdot \text{KIO}_3 \cdot \text{HIO}_3$ .
Potassium	16.94	16.83	16.82
Iodine	54.46		54.66
Chlorine	7.72		7.64
Oxygen			20.66
Hydrogen	.20		.22

\* Ann. Ch. Phys., II, xliii, 113.

† Ann. Ch. Phys., III, ix, 407.

‡ Pogg. Ann., xcvii.

§ Jahresb., 1856, 298. Ann. Min., V, ix, 1.

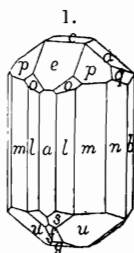
This compound and the one obtained by Marignac are therefore identical.

On ignition it gives off water, iodine chloride and oxygen, the residue consisting of potassium iodide and chloride. An analysis of this residue gave 2.39 per cent. chlorine and 70.87 per cent. iodine.

The author takes occasion here to express his obligations to Professor H. L. Wells for the use of the material in this investigation and for valuable suggestions, also to Professor S. L. Penfield who has kindly furnished the crystallographical descriptions.

*Notes on the Crystalline form of RbCl. HIO<sub>3</sub> and CsCl. HIO<sub>3</sub>;* by S. L. Penfield.

RbCl. HIO<sub>3</sub>.



The form of RbCl. HIO<sub>3</sub> is monoclinic. The crystals are highly modified, doubly terminated prisms, fig. 1. The faces gave fair reflections and the measurements which were chosen as fundamental are marked by an asterisk in the table of angles.

The axial ratio and forms are as follows:

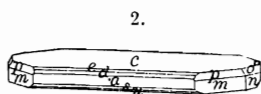
$$\lambda : \bar{b} : \bar{c} = 0.9830 : 1 : 0.7577, \quad \beta = 100 \wedge 0 \text{c}1 = 87^\circ 56'$$

<i>a</i> , 100, $i\bar{i}$	<i>l</i> , 320, $i\bar{2}$	<i>d</i> , 011, $1\bar{i}$	<i>g</i> , $\bar{1}02$ , $\frac{1}{2}\bar{i}$	<i>q</i> , 142, $-2\bar{4}$
<i>b</i> , 010, $i\bar{i}$	<i>m</i> , 110, I	<i>e</i> , 101, $-1\bar{i}$	<i>o</i> , 211, $-2\bar{2}$	<i>s</i> , $\bar{2}11$ , $2\bar{2}$
<i>c</i> , 001, 0	<i>n</i> , 120, $i\bar{2}$	<i>f</i> , $\bar{1}01$ , $1\bar{i}$	<i>p</i> , 111, $-1$	<i>u</i> , $\bar{1}11$ , 1.

Measured.		Calculated.	Measured.		Calculated.
$a \wedge c$ , 100 $\wedge$ 001	=*87° 56'		$a \wedge d$ , 100 $\wedge$ 011	= 88° 29'	88° 21'
$a \wedge e$ , 100 $\wedge$ 101	=*51 5		$a \wedge s$ , $\bar{1}00 \wedge \bar{2}11$	= 38 32	39 47
$c \wedge d$ , 001 $\wedge$ 011	=*37 8		$a \wedge u$ , $\bar{1}00 \wedge \bar{1}11$	= 59 57	59 38
$a \wedge l$ , 100 $\wedge$ 320	= 33 13	33° 13'	$e \wedge p$ , 101 $\wedge$ 111	= 50 28	30 31
$a \wedge m$ , 100 $\wedge$ 110	= 44 7	44 29½	$f \wedge u$ , $\bar{1}01 \wedge \bar{1}11$	= 31 22½	31 24
$a \wedge n$ , 100 $\wedge$ 120	= 62 42	63 1½	$p \wedge q$ , 111 $\wedge$ 142	= 26 36	26 30
$a \wedge o$ , 100 $\wedge$ 211	= 38 19	38 28½	$c \wedge g$ , 001 $\wedge$ $\bar{1}02$	= 21 17	21 20
$a \wedge p$ , 100 $\wedge$ 111	= 57 13	57 14	$c \wedge f$ , 001 $\wedge$ $\bar{1}01$	= 38 26	38 23½

CsCl. HIO<sub>3</sub>.

The form of CsCl. HIO<sub>3</sub> is monoclinic. The crystals, from



the one crop which was examined, were about 5 mm. in length and had the habit shown in fig. 2. They were attached at one end and usually grew in radiating and divergent groups. The faces were not very perfect and only approximate measure-

ments could be made. Those which were chosen as fundamental are :

$$m \wedge \bar{m}, 110 \wedge \bar{1}10 = 90^\circ 12' \quad m \wedge p, 110 \wedge 221 = 24^\circ 37' \quad a \wedge p, 100 \wedge 221 = 49^\circ 53'$$

The axial ratio and forms are as follows :

$$\begin{aligned} \bar{a} : \bar{b} : \bar{c} &= 0.9965 : 1 : 0.7698 & \beta &= 100 \wedge 001 = 89^\circ 53\frac{1}{2}' \\ a, 100, \bar{i}\bar{i} & \quad m, 110, \bar{1} & d, 403, -\frac{1}{3}\bar{i} & \quad s, \bar{1}03, \frac{1}{3}\bar{i} & p, 221, -2 \\ c, 001, 0 & \quad n, 130, \bar{i}\bar{3} & e, 203, -\frac{2}{3}\bar{i} & \quad u, \bar{2}03, \frac{2}{3}\bar{i} & o, 263, -2\bar{3} \end{aligned}$$

The pyramids  $p$  and  $o$  were frequently wanting. The orthodomies  $d$ ,  $e$ ,  $s$  and  $u$  were very constant in their development and gave to the crystals an orthorhombic habit. Owing to the curved and striated character of the faces the symmetry could not be satisfactorily determined by measurement, but the optical properties showed that the crystals were truly monoclinic. In polarized light the tables show an extinction parallel to the ortho-axis and in convergent light one of the optical axes and the acute bisectrix can be seen near the limits of the field. The plane of the optical axes is the clinopinacoid.

These two salts, although entirely different in crystalline habit, are very similar in their axial ratios.

Sheffield Scientific School,  
April, 1892.