Preparation and Analysis of the Aquo Salts of Ruthenium

by

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Ruthenium,one of the platinum groups of metals,has a specific gravity of 12.2,a melting point of 2450 degrees Centigrade,and a boiling point somewhat above 2700 degrees.Its atomic weight is 101.7 and its atomic number 44.It was discovered in 1844 by Claus.Its double salt,the chloride,was described by him at the same time,it had the formulaassigned by him of K_2RuCl_5.He called attention to its similarity to the corresponding rhodium salt,K_2RhCl_5.Some time later he obtained another salt which he believed to be K_2RuCl_6.In 1872 Joly was working on this same salt and found that no hydrolysis took place.On investigation he found evidence of only five chlorine atoms.It was evident that Claus had analyzed only for potassium,ruthenium and chlorine,for when he heated some of the salt in hydrogen he obtained ammonium chloride and considered the formula to be K_2RuCl_5NO.There is a difference of five in the atomic weight,due to the presence of the -NO and the absence of one chlorine,it is r readily seen how the mistake could have been made since the heavy weight of the molecule was the couse of overlooking the two additional elements present.

The next upset came in the attempt to obtain ruthenium in the bivalent form.If some of the supposedly $RuCl_2$ is oxidized in the presence of alcohol and CsCl sone crystals of $Cs_2RuCl_5.H_2O$ are obtained.The potassium or rubidium salts may be obtained in the same way.These salts are called the aquo salts.A few years later Antony obtained some $K_2RuCl_5.H_2O$,which like the other could be dehydrated, but gave a red solution while the other gave a pink one.At a later date Aoyama,a chemist in Japan,obtained a third salt of the same character.At first it was thought that these compounds were inorganic isomers.Kraus took up the study of them along with Remy,Briggs,

and Gutbier.They came to the conclusion that the alpha salt had the formula K_2RuCl_5OH in which the ruthenium is tetra valent.In the other compound the ruthenium is tri-valent.At the present time this is the only solution that has been found to the problem.After much work on Claus'first salt,Remy suggested the formula $(K_2RuCl_5)_2O$ Charronat suggested the formula K_2RuCl_5OH while Antony suggested the formula $K_2RuCl_5H_2O$.The Charronat formula is the one accepted today,one of its advantages is that it has the coordination number of six.There is little difference in the weights of any of these salts and it is difficult to get an accurate analysis of any of them.The hydrogen method of analysis will be described later,as can be seen there are many chances of error,and this lessens the accuracy of the determination.The other method of determining the amount of water directly is not much more accurate due to the small amount of water present.In the water method,both water and chlorine are given off.The presence of the chlorine interferes with an accurate determination of the water.If calcium chloride is used for weighing the water directly,a slight change in temperature causes the $CaCl_2$ to change weight and thus ruin the determination.This change and thus the change in weight of the apparatus may occur at any point,even while the calcium chloride tube is on the balance.It has been gound that the ordinary pentachloro ruthenite can not be chlorinated directly to the hexachloro ruthenate.In 1899 the latter salt was described by Antony and Lucchesi.They obtained it from potassium ruthenate,K_2RuO_4,by treating a solution of the latter with concentrated hydrochloric acid,the ordinary pentachloro ruthenite is expected by this reaction.On the following year the monohydrate of potasssium pentachloro ruthenite was obtaine Miolati and Tagiuri,by precipitation from the acid solution of the

trichlorides by potssium chloride in the cold.Howe described the cesium and rubidium pentachloro ruthenites which crystallize as monohydrates.

In 1901 Howe obtained the monohydrate by boiling an acid solution of K_2RuCl_5 with alcohol.The solution slowly changed from dark red-brown to rose and the rose prisms crystallized out,they were called the aquo chloro ruthenate by Howe and they have the formula $K_2RuCl_5.H_2O$.The corresponding ammonium,rubidium,and cesium salts have been described,as well as the potassium and rubidium aquo aquo bromo ruthenates,The monohydrate of the alpha series was found by Lind and Bliss to be two thirds hydrated,the formula being $Ru(OH)_2Cl$ but the ruthenium is quadrivalent.The corresponding aquo or beta series is one third hydrated the formula being $Ru(OH)Cl_2$.In both cases the solutions must be dilute.Aoyama has more recently obtained what he considered a third isomer having the formula K_2RuCl_5;He formed this by heating K_2RuCl_6 in am atmosphere of HCl gas at 540-560 degrees Centigrade,the gas must be dried before using.He called this the gamma salt,but on repeating the work Gutbier and Niemann considered that it was identical with the dehydrated beta series. The solutions of the dehydrated aquo salt and the dechlorinated hexachloro salt are not at first identical with those of the aquo salt but gradually change over from the typical pure yellow solution to the red color of the aquo solution.Perhaps the most distinguishing feature of the aquo salt is that its solution is immediately darkened by chlorine or bromine fumes.The chloro ruthenate is formed by this reaction,and in practice advantage is taken of this to prepare the above named salts.A rapid stream of chlorine is led through a cold saturated solution of the aquo salt in an excess of hydrochloric acid.The solution immediately darkens to deep brown

with a yellowish cast and the chloro ruthenate precipitates out in minute black octahedra,which have a greenish sheen after drying. The poassium,ammonium,rubidium,and cesium salts have all been prepared in the same way.They have the following formulae respectively: K_2RuCl_6,$(NH_4)_2RuCl_6$?Rb_2RuCl_6,and Cs_2RuCl_6.Several of the bromo ruthenates have been prepared in a similar way.Charonnat deserves a great deal of credit for clearing up the composition of the compound that was formerlu supposed to be the ordinary pentachloro ruthenate.He,along with several other authorities,has conclusively shown that the true formula is K_2RuCl_5OH in which the ruthenium is quadrivalent.It might be considered a hexachloro ruthenate that has been one fourth hydrolized.When K_2RuCl_5OH.is recrystallized from a very concentrated solution oh hydrochloric acid K_2RuCl_6,the hexachoro ruthenate is obtained,but this yields the penta chloro hydroxy ruthenate on recrystallization from a dilute hydrochloric acid solution (less than 6 N.).

The following is a summary of the chloro salts of ruthenium that have been well established:

A. $M_2\overset{vi}{Ru}O_2Cl_4$(M is H,Rb,Cs);the hydrogen salt formed by the action of gaseous hydrogen chloride on RuO_4;the rubidium and cesium salts by the action of rubidium or cesium chloride in a very concentrated hydrochloric acid solution on RuO_4.

B. $M_2\overset{iv}{Ru}Cl_6$(M is NH_4?K,Rb,Cs);formed by the action of chlorine on $M_2RuCl_5H_2O$;by the action of very dilute cold hydrochloric acid on K_2RuO_4 melt;by the recrystallization of M_2RuCl_5OH from concentrated hydrochloric acid.

B(a)$Cs_2RuCl_6.H_2O$;formed by the action of CsCl on a solution of RuO_4 in hydrochloric acid.Efforts to obtain the pure rubidium and potassium salts were unsuccessful.

C. $M_2\overset{iv}{Ru}Cl_5OH$ (M is NH_4,K,Rb,Cs);formed by the action of as alkaline chloride on a solution of RuO_4 in hydrochloric acid (except in very concentrated);when a solution of the ruthenate is treated with hydrochloric acid;when a solution of any oxide,except one precipitated from the aquo salt,is treated with an alkalie chloride, the oxide being dissolved in hydrochloric acid;when a hexachloro ruthenate is recrystallized from hydrochloric acid except when the latter is very concentrated.

D. $M_2\overset{iii}{Ru}Cl_5 \cdot H_2O$ (M is NH_4,K,Rb,Cs);formed by the reduction of any of the salts of quadrivalent ruthenium in hydrochloric acid by alcohol,oxalic acid,or other organic substance,stannous chloride,or potassium iodide;also by the spontaneous oxidation in the air of the blue bivalent ruthenium solution.These are the only crystalline salts of trivalent ruthenium,and are properly called the pentachloro-ruthenites.The term aquo ruthenite distinguishes it from the pentachloro ruthenites,which have now become the pentachloro hydroxy ruthenates.

E. $M_2\overset{iii}{Ru}Cl_5$;formed by the action of dehydrating agents on the aquo salts,or by dechlorination of the hexachloro ruthenates(Aoyama's gamma and beta salts).These have not been obtained in a crystalline form and therefore can not be regarded as definite salts with a coordination number of five,but are of the same character as K_2SO_4 and $Al_2(SO_4)_3$ in dehydrated alum.

For the purpose of gaining practice in the technique of hand-
lying ruthenium several unknown samples were analyzed, the results
of these analysis are given in a condensed form (all the actual
weighings and calculations are tabulated at the end of this paper).

Unknown number 1

        Sample A ----------.329 % Ru.

        Sample B ------------.264 % Ru.

In the running off of this sample no account of the chlorine
evolved was made , the experiment being for the method of the Ru
treatment.

Unknown number 2

        Sample A ----------.1625 % Ru

        Sample B ----------.7745 % Ru

        Sample A ----------.007675 gms. Cl (with Ru)

        Sample B ----------.004354

These results seem to differ rather widely, the cause being unknown
but probably due to experimental error.

Unknown number 3 The samples used were crystals of K_2RuCl_5 or
$K_2RuCl_5.H\,O$ and K_2SnCl_6, probably solid solutions. Account was taken
of the stannic oxide which was left after distilling the Ru.

        Sample A ----------.3092 % Ru

        Sample B ----------.3640 % Ru

        Sample A ---------- 56.25 % SnO_2

        Sample B ---------- 45.91 % SnO_2

        Sample A ----------.4625 % Cl

        Sample B ----------.1154 % Cl

The work for the next period of time was that of distilling Ru
from various residues in preparation to starting the actual research.
A quantity of ruthenium was obtained in the proper form and this was
analyzed for ruthenium content, the results are given below:

Sample number	Gms.Ru/cc.	Gms.Cl/cc.
A	.00570	.00412
B	.00545	.00493
C	.00560	.00473

The lack of agreement was probably due to source of chlorine in the apparatus,apparently in the sulfuric acid.The total volume of solution was approximately 175 cc.

The ruthenium used in preparing the rubidium pentachloro hydroxy and the aquo salts of ruthenium was both the metal and the residues left from other work done on the same metal. A small amount of ruthenium was extracted from filter papers by igniting them and dissolving the ruthenium residue in sodium hypochlorite, in which all the residues were dissolved, as was the metal. Sodium ruthenate is formed by the reaction. If there is not enough of the sodium hydroxide present some of the volatile ruthenium tetroxide willescape and can be detected by the odor of new mown hay which is caused by the evolution of ozone accompanying the above. The reaction is;

$$3NaOCl + NaOH + Ru = Na_2RuO_4 + 2NaCl + HCl$$
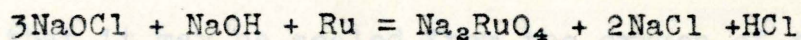
The solution is then placed in a goose-necked pyrex retort or distilling flask with the neck bent down at right angles so that the ruthenium tetroxide distilled over may be led into a flask containing a small amount of concentrated hydrochloric acid. A stream of chlorine was led into the flask and the solution gradually warmed to boiling, the RuO_4 distills over and was collected in the hydrochloric acid contained in the flask, which was placed in a beaker of cold water to prevent loss of ruthenium. The tetroxide may be collected in very cold water or in a flask surrounded by a mixture of ice and salt, but the loss by volatilization is much greater than when collected in concentrated hydrochloric acid. In the latter case the amount lost is negligable and the recovery may be carried out quantitatively. Several lots of ruthenium were distilled over in this way and the resulting solutions combined and evaporated down to give a more concentrated solution of ruthenium. The ruthenium thus recover in HCl changes over until there is an equilibrium established between H_2RuCl_6 and H_2RuCl_5OH. The color of the hypo solution gradually changes to colorless as the amount of ruthen-

ution gradually changes to colorless as the amount of ruthenium in it decreases. The tetroxide has a golden yellow color as it distills over. The concentrated ruthenium solution was then placed in a bottle for further use.

The concentration of the ruthenium was determined by evaporating a two milliliter sample of the solution to dryness in a fused quartz boat. The ruthenium was then reduced to the metallic state by heating in an atmosphere of hydrogen in a cylindrical electric combuation furnace, the boat was placed in a pyrex glass tube somewhat longer than the furnace and larger at one end than at the other. The smaller end is bent down at right angles to the main body of the tube so that it may be sealed from the oxygen of the atmosphere by placing it under the surface of a small amount of water or silver nitrate contained in a beaker. The tube was then placed so as to seal the small end from air and the boat inserted so that it was in the center of the furnace. A stream of hydrogen was then passed through the tube until the air had all been swept out. To ascertain this a glowing spark or splinter of wood was held over the water, if the hydrogen exploded with its familiar noise the heat was turned on and the boat and ruthenium heated until they and the tube had a red glow. The heat was then cut off and the apparatus allowed to cool, the stream of hydrogen being continued until the apparatus was perfectly cold. Hydrogen from a cylinder was used for this purpose, but it was necessary to purify it by passing it first through a wash bottle containing alkaline potassium permanganate solution to remove all organic impurities, then through two wash bottles containing concentrated sulfuric acid to completely remove moisture from the gas. The ruthenium is completely reduced to the metal at the temperature used, by the hydrogen, the chlorine being converted into HCl. It may be collect-

ed in silver nitrate as silver chloride and weighed as such on a
Gooch filter.The boat was weighed clean and then again with the ru-
thenium metal in it,the increase in weight being the amount of ruth-
enium contained in two milliliters of the solution,one half of this
amount being the quantity in one milliliter.To find the total amount
of ruthenium contained in the solution,the total volume was multi-
plied by the amount of ruthenium contained in one milliliter.Dupli-
cates were run on the determination,the results being given below.

From the total amount of ruthenium calculated above,the amount
of rubidium chloride necessary to convert all the ruthenium into
the rubidium pentachloro hydroxy ruthenate,Rb_2RuCl_5OH,was added
after being dissolved in the least possible amount of water.Both
solutions were hot at the time of mixing.Some of the rubidium pen-
tachloro hydroxy ruthenate crystallized out on cooling.The solution
was then evaporated down successively until five or six crops of
crystals were obtained,and about half of the ruthenium had been
precipitated out.The different crops were collected on separate fil-
ters and dried.

The remainder of the ruthenium solution was then refluxed with
alcohol to convert it into the aquo salt,at the beginning the solu-
tion had a brownish color but gradually changed in color until a
rose solution was obtained.The solution of the aquo salt thus ob-
tained was then treated in the following manner,the solution was
evaporated successively until all the Ru was crystallized in five
or six crops.Unless the salts were on the desired form and of the
highest purity they were put in solution in the highest concentration
of hydrochloric that would allow filtering,and recrystallized from
this.In some it was necessary to recrystallize the salt five or six
times.The crystals were collected in crops on filters and dried.

The purpose of this work was to determine the water or the -OH content of the rubidium pentachloro hydroxy and the aquo salts of ruthenium, and also to acquire the technique of handling ruthenium, especially the analysis of the soalts for the water content. This analysis was done by analyzing for each of the other substances or elements present; Ru, RbCl, and Cl (with the ruthenium). The total weight of these substances or elements was subtracted from the weight of the sample, in the case of the aquo salt giving the weight of the water present, and in the case of the pentachloro hydroxy salt the weight of the -OH groups, or the total percentage of the other elements or compounds present may be subtracted from one hundred percent, thus giving the percent of hydrogen and oxygen present.

A sample of the dried salt was weighed into a fused quartz boat and heated in the combustion furnace as described above. Instead of only ruthenium being left in the boat the rubidium chloride is also left. The increase in weight is the ruthenium plus the rubidium chloride contained in the sample. The rubidium chloride was dissolved in the least amount of water possible, the ruthenium filtered out on ashless filter paper, the paper ignited in a weighed boat, the ruthenium reduced in hydrogen as before (in case any oxidation took place in igniting) and weighed as such, the rubidium chloride was evaporated in a weighed porcelain crucible and weighed as such. The chlorine was determined by passing the HCl formed through a silver nitrate solution, silver chloride precipitated and weighed as such on a Gooch filter.

The percentage of ruthenium in the sample was determined by dividing the weight of the metal obtained by the weight of the sample and multiplying by one hundred, the rubidium chloride similarly, and the chlorine (with ruthenium) by multiplying the weight of silver chloride by the gravimetric factoraand one hundred and divid-

ing by the weight of the sample.The results are given below for all the salts analyzed.

Calculations:

Unknown number 1

```
Wt.of watch glass --------------- 4.8598
 "   "    "       "   +Sample A ------6.2994
                      Sample A -----1.4396


Wt.watch glass-------- ----------- 4.8598
      "        "  + Samle B --------- 6.7120
                   Sample B -------- 1.8522


Wt.boat A + Ru in sample A ------ 4.1014
Wt.boat A ------------------------44.0976
             Ru in sample A ------ 0.0038


Wt.boat B +Ru in sample B ------- 3.5681
Wt.boat B ------------------------- 3.5620
             Ru in sample B ------ 0.0061
```

A. $\dfrac{.0038 \times 100}{1.4396}$ % = .264 % Ru

B. $\dfrac{.0061 \times 100}{1.8522}$ % = .3291 % Ru

Unknown number 2

```
wt.watch glass ------------------- 9.0475
 "     "       "   + Sample A -------- 9.9092
                    Sample A ---- 0.8617


Wt.watch glass ------------------- 9.0475
 "      "        "  + Sample B ------- 10.3412
                     Sample B------ 1.2937


Wt.boat A -------------------------- 4.3225
 "    "    "   + RuCl₃ A.------------ 4.3284
                  RuCl₃ in A.   0.0059


Wt.boat B -------------------------4.3834
 "    "    "  + RuCl₃ B --------------4.4037
                  RuCl₃ in B -- 0.0203


Wt.boat A + Ru ------------------- 4.3239
                                   4.3225
                  Ru in A ----- 0.0014
```

Wt.boat B + Ru ------------------ 4.3239
                                  4.3824
                   Ru in B --- 0.0100

A. $\dfrac{0.0014}{0.8617}$ x 100 = .1625 % Ru in sample A

B. $\dfrac{0.0100}{1.2937}$ x 100 = .7745 % Ru in sample B

Wt. of Pt crucible ---------------15.1025
 "   "   "   "   + AgCl A ---- 15.1335
              AgCl in sample A ??- 0.0310

Wt,of Pt.crucible --------------- 15.1335
 "   "   "   "   + AgCl in B -- 15.1756
            AgCl in sample B -- 0.0421

$\dfrac{Cl}{AgCl} = \dfrac{4}{.031}$ = .2474 gns.Cl in $RuCl_3$

$\dfrac{.007675}{.0059}$ x 100 =   no.check in % Cl due to absorbed chlorine in
                    H_2SO_4 used in purifying hydrogen.

$\dfrac{.0421 \times .2474 \times 100}{0.0203}$ = 50.11 % Cl in $RuCl_3$ in sample B.

Unknown number 3

Wt.of sample A	.5494	Wt.of boat A	4.3225
Wt of sample B	.9717	Wt.of boat B	4.0843
Wt.Of crucible A.	8.6630	Wt.of Pt.crucible A	15.1969
Wt.of Crucible B	8.8276	Wt.of Pt.crucible B	15.1515

Wt.crucible A + stannic oxide   8.9718
                                     8.6630
              Stannic oxide A   0.3088

Wt.crucible B + stannic oxide   9.2774
                                     8.8276
              Stannic oxide B   0.4498

$\dfrac{0.3088 \times 100}{.5494}$ = .3092 % SnO_2 in sample A

$$\frac{0.4498 \times 100}{.9717} = 45.91 \% \; SnO_2 \; \text{in sample B}$$

```
Wt. boat A + Ru        4.3242
                       4.3225
       Wt. Ru A        0.0017
```

```
Wt. Boat B + Ru        4.08774
                       4.0843
       Wt. Ru B        0.00344
```

$$\frac{0.0017 \times 100}{.5494} = 0.3092 \% \; Ru \; \text{in sample A}$$

$$\frac{0.00344 \times 100}{.9717} = 0.3640 \% \; Ru \; \text{in sample B}$$

```
Wt. Pt. crucible + AgCl  A.      15.2072
                                 15.1969
               Wt. AgCl A        0.0103
```

```
Wt. Pt. crucible + AgCl  B       15.1969
                                 15.1515
               Wt. AgCl B        0.0454
```

$$\frac{.0103 \times .24738 \times 100}{.5494} = .4625 \% \; Cl \; \text{in sample A (with Ru)}$$

$$\frac{.0454 \times .24738 \times 100}{.9717} = .1154 \% \; Cl \; \text{in sample B (with Ru)}$$

Unknown number 4

```
Wt. of boat A               4.3650
"    "    "   A + sample     4.5348
             Sample A        0.1698
```

```
Wt. of boat A + Ru          4.4179
                            4.3650
        Ru in sample A      0.0529
```

```
Wt. of boat B + Ru          4.3230
"     "    "   B + sample   4.4467
             Sample B       0.1237
```

```
Wt. of boat B + Ru          4.3620
                            4.3230
         Ru in sample B     0.0390
```

Unknown number 4 (continued).

$$\frac{.0529}{.1698} \times 100 = 31.20 \% \text{ Ru in A.}$$

$$\frac{.0399}{.1237} \times 100 = 31.53 \% \text{ Ru in B.}$$

Wt.of pt.crucible	15.9630
Wt.of pt.crucible and AgCl A	16.9494
	0.9864

Wt.of Pt.crucible	15.5167
Wt.of Pt.crucible and AgCl B	15.9630
	0.4463

$$\frac{.4463 \times .24739 \times 100}{.1237} = 89.21 \% \text{ Cl in B}$$

$$.9864 \times .24739 \times 100 = 140.0 \% \text{ Cl in A}$$

At this point chlorine was found to be coming from the sulfuric

acid.

Wt.of boat	4.3639
Wt.of boat and sample C	4.6727
Sample C	.3088

Wt. of boat and Ru in C	4.4548
	4.3639
Ru in C	.0909

$$\frac{.0909 \times 100}{.3088} = 2946 \% \text{ Ru in C.}$$

Wt.of Pt.crucible and AgCl C	16.8850
	16.1531
AgCl C	.7319

$$\frac{.7319 \times .24739 \times 100}{.3088} = 58.63 \% \text{ Cl in C.}$$

Determination of Concentration of Ruthenium in the Solution.

(2 cc. evaporated to dryness in boat and then reduced with

hydrogen each time)

Wt. of boat and Ru A 4.3337
                      4.3225
Grams of Ru in 2 cc.   .0112
Grams of Ru in 1 cc A .0056

Wt. of boat and Ru B 4.3754
                      4.3645
Grams of Ru in 2 cc.   .0109
Grams of Ru in 1 cc B .00545

Wt. of boat and Ru C 4.3339
                      4.3225
Grams of Ru in 2 cc.   .0114
Grams of Ru in 1 cc C .0057

Wt. of Pt. crucible & AgCl A 15.2914
                             15.2577 $\dfrac{.0337 \times .24738}{2}$ = .00414 Gms.
                    AgCl A   .0337   Cl/cc.

Wt. of Pt. crucible & AgCl B 15.2577
                             15.2176 $\dfrac{.0401 \times .24738}{?}$ = .00493 Gms.
                               .0401   Cl/cc.

Calculations for the amount of RbCl to be added to the known amount of Ru to convert it into Rb_2RuCl_5OH. 175 cc. of solution.

175 cc. x .00555 = .971 gms. of Ru

$\dfrac{2\,RbCl}{Ru} = \dfrac{X}{.971}$ X = 2.299 gms. of RbCl to be added.

For a second solution of 190 cc. containing .00867 gms. Ru/cc.

$\dfrac{190 \times .00867 \times 241.8}{101.7}$ = 3.923 gms. RbCl to be added to convert

the Ru into the Rubidium pentachloro

hydroxy ruthenate.

Analysis of aquo salt

Lot one   (sample A)

Weight of boat A ------ -------------------------------4.3222

Weight of boat A and salt----------------------------4.5876

Weight of salt ------------------------------------- .2754

Wt.of boat,RbCl,and Ru(after H reducing)----------4.5222

Weight of boat--4.3222

Weight of RbCl and Ru------------------------------- .2000

Weight of boat and Ru--------------------------------4.3799

Weight of boat---------------------------- -----------4.3222

Weight of Ru---------- ------------------------------.0577

Weight of Pt crucible--------------------------------17.1509

Weight of Pt crucible and AgCl----------------------17.4194

Weight of AgCl-- .2685

Wt of crucible A -----------------------------------11.5764

Weight of crucible A and RbCl----------------------11.7159

Weight of RbCl-------------------------------------- .1395

$\frac{.0577}{.2754}$ x 100 --------------20.95 % of Ru

$\frac{.1395}{.2754}$ x 100 --------------50.65 %   RbCl

$\frac{.2685}{.2754}$ x 100 x -.24738----- 24.12 % Cl

20.95
50.65
24.12
95.72

100.00
95.72
4.28 % water

Lot one (Sample B)

Weight of boat B --4.0818

Weight of boat B and sample--------------------------- 4.3962

Weight of sample -------------------------------------- .3144

Weight of boat B ,RbCl and Ru ----------------------4.3011

Weight of boat B --4.0818

Weight of RbCl and Ru----------------------------------- .2193

Weight of boat and Ru -------------------- -------4.1508

Weight of boat --4.0818

Weight of Ru -- .0690

Weight of Pt crucible -------------------------------17.4194

Weight of Pt crucible and AgCl----------------------17.7024

Weight of AgCl-- .2830

Weight of crucible B ---------------------------------11.2961

Weight of crucible B and RbCl------------------------11.4307

Weight of RbCl-- .1346

$\dfrac{.0690}{.3144}$ x   100   ------------------- 21.95 % Ru

$\dfrac{.1346}{.3144}$ x   100   ---------------------42.81 % RbCl

$\dfrac{.2830}{.3144}$ x   100 -x .24738-------------- 22.27 % Cl

<div style="text-align:right">

22.27
21.95
42.81
87.03

100.00
87.03
12.97 %

Water
</div>

Lot two (Sample A)

weight of boat A --4.3221

Weight of boat A and sample ----------------------------4.5984

Weight of sample -- .2763

weight of boat A, RbCl and Ru--------------------------4.5163

Weight of boat A ---------------------------------------4.3221

Weight of RbCl and Ru --------------------------------- .1942

Weight of boat and Ru ---------------------------------4.3802

Weight of boat ---------------------------------------4.3221

Weight of Ru--- .0581

Weight of Pt crucible ------------------------------- -17.7024

Weight of Pt crucible and AgCl----------------------18.0213

Weight of AgCl--------------------------------------- .3189

Weight of crucible-----------------------------------11.5766

weight of crucible and RbCl-------------------------11.7128

Weight of RbCl-------------------------------------- .1362

$\dfrac{.0581}{.2763}$ x 100 -------------------- 21.03 % Ru

$\dfrac{.1362}{.2763}$ x 100 -------------------- 49.29 % RbCl

$\dfrac{.3189}{.2763}$ x 100 -x-.24738 ----------- 28.53 % Cl

21.03
28.53
49.29
98.85

100.00
98.85
1.15 %

Water

Lot two (Sample B)

Weight of boat B --4.0801

Weight of boat B and sample ------------------------------4.1818

Weight of sample-- .1017

Weight of boat B,RbCl and Ru------------------------------4.1522

weight of boat B --4.0801

Weight of RbCl and Ru -------------------------------------- .0721

weight of boat B and Ru--4.1021

Weigh t of boat B --4.0801

Weight of Ru -- .0220

Weight of Pt crucible --18.0213

Weight of Pt crucible and AgCl----------------------------18.1441

Weight of AgCl-- .1227

Weight of crucible---11.2953

Weight of crucible and RbCl ----------------------------11.3371

Weight of RbCl-- .0418

$\dfrac{.0220}{.1017}$ x 100 ----------------------- 21.42 % Ru

$\dfrac{.0418}{.1017}$ x 100 ------------------------------40.70 % RbCl

$\dfrac{.1227}{.1017}$ x 100 -x-.24738------------ 29.55 % Cl

```
29.55
40.70
21.42
-----
91.67
```

```
100.00
 91.67
------
  8.33 %
```

Water

<u>Lot three</u>   (Sample A)

Weight of boat A---4.3221

Weight of boat A and sample-----------------------------------4.4065

Weight of sample--- .0844

Weight of boat A,RbCl and Ru ---------------------------------4.3814

Weight of boat A ---4.3221

Weight of RbCl and Ru--- .0593

Weight of boat A and Ru---------------------------------------4.3416

Weight of boat A ---------------------- -------------------4.3221

Weight of Ru -- .0195

Weight of pt cruvible ---18.1440

Weight of Pt crucible and AgCl---------------------------------18.2398

Weight of AgCl--- .0958

Weight of crucible --11.5759

Weight of crucible and RbCl-----------------------------------11.6171

Weight of RbCl--- .0412

$\dfrac{.0195}{.0844}$ x 100 --------------------------23.10 % Ru

$\dfrac{.0412}{.0844}$ x 100 --------------------- 48.82 % RbCl

$\dfrac{.0958}{.0844}$ x 100 x .24738 ---------- 28.73 % Cl

These results add over 100% not allowing for water

Lot three (Sample B)

Weight of boat B --4.0802

Weight of boat B and sample-------------------------------------- 4.1647

Weight of sample --- .0845

Weight of boat B,RbCl and Ru -----------------------------------4.1373

Weight of boat B ---4.0802

Weight of RbCl and Ru ---.0571

Weight of boat and Ru--4.0993

Weight of boat --4.0802

Weight of Ru -- .0191

Weight of Pt crucible --18.2398

Weight of Pt crucible and AgCl----------------------------------18.3351

Weight of AgCl------------------------------ ------------------------ .0953

Weight of crucible-- 11.2951

Weight of crucible and RbCl --------------------------------------11.3346

Weight of RbCl -- .0395

$\frac{.0191}{.0845}$ x 100 ----------------------------22.60 % Ru

$\frac{.0395}{.0845}$ x 100 --------------------------- 46.75 % RbCl

$\frac{.0953}{.0845}$ x 100 x .24738 -------------------27.90 % Cl

27.90
46.75
22.60
————
97.25

100.00
97.25
————
2.75 %

water

no.	2RbCl-Ru	Ru	RbCl	RhCl-Ru	Cl	Ru,RbCl,Cl	H_2O
A_1	.2000	.0577	.1395	.1972	.06642	.26362	.0118
	72.62	20.95	50.65	71.61	24.12	95.72	4.285
A_2	.2193	.0690	.1346	.2036	.07001	.2736	.0408
	69.75	21.95	42.81	64.76	22.27	87.02	12.98
B_1	.1942	.0581	.1362	.1943	.07888	.2732	.0031
	70.29	21.03	49.29	70.32	28.53	98.88	1.148
B_2	.0721	.0220	.0418	.0638	.03035	.0942	.0075
	70.21	21.42	40.70	62.12	29.55	91.72	7.303
C_1	.0593	.0195	.0412	.0607	.0237	.0844	.0000
	70.26	23.10	48.82	71.92	28.73	100	0000
C_2	.0571	.0191	.0395	.0586	.02357	.0822	.0023
	67.57	22.60	46.75	69.35	27.90	97.28	2.722

Calculation of the molecular weight of the loss in the analysis of the rubidium aquo salt of ruthenium.

1) $\dfrac{343.5 \times .0118}{.2000}$ -----------------20.28

2) $\dfrac{343.5 \times .0408}{.2193}$ ----------------64.09

3) $\dfrac{343.5 \times .0031}{.1942}$ ----------------- 54.85

4) $\dfrac{343.5 \times .0075}{.07421}$ --------------- 34.79

5) $\dfrac{343.5 \times .0023}{.0571}$ ------------- 13.86