The analytical separation of iron and aluminium

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Recommended Citation
Radasch, Henry Erdmann. "The analytical separation of iron and aluminium." MS (Master of Science) thesis, State University of Iowa, 1897.
https://doi.org/10.17077/etd.o9i027pu

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The Analytical Separation of Iron and Aluminium

A Thesis for the Attainment of the Degree Master of Science

By

Henry Edman

State University of Iowa

June 1897
Note.

On pages 66-69 will be found the bibliography and after each separation will be found a number which corresponds to one on those pages.
The Analytical Separation of Iron and Aluminium.

Of all the methods given, up to the last year, we have only a few that may be said to be both satisfactory and speedy. By satisfactory is meant, directness of working and accuracy. It is true that some of the methods are quite indirect but still they lack directness and speed. Within certain limits, accuracy may be sacrificed to directness and speed. Thorough methods give opportunities for loss of material, and some of the more direct ones give opportunities for an increase of material, i.e., through the solution of the silicate of the metal used by the reagents. Although these losses may be avoided to a greater or less extent, they still occur, at times, in spite of the most careful manipulation.

The methods have been grouped under four classes or heads and this classification will be used in the discussion. These heads are as follows:

I. Those methods depending upon a difference of solubility of the precipitates in
an excess of the precipitant.

1. Potassium hydrate method.
2. Soda.
4. Ethylammine.
5. Trimethylamine.
6. Hydrochloric acid and Ether.

II.

Depending upon the ease reduction.

III.

Sulphates.

1. Solvent this sulphate method.
2. Reduction of the ferrous oxide by hydrogen, and (a) Solution of iron in sulphuric acid and determination,
   (A). by ammonia,
   (B). Volumetrically.
3. Volatilisation of iron by hydrochloric acid gas.
4. Electrolytic separation,
   (a). From solutions of organic salts,
   (b). From phosphoric acid solutions.
5. Griesbust method.

III.

Depending upon the basically.

1. Intrósó-3-Hoplitos method.
2. Ferron.sulphide method
   (a) In solutions of organic salts
   (b) Tartaric acid

Miscellaneous:

1. Basic nitrate method
2. Potassium sulpho cyanide method
3. Double cyanide
4. Potassium carbonate

The discussion of the methods will be in order of their classification.
Discussion

Case I.

1. Potassium hydride method (1)

To the hot potassium hydride solution add slowly, with constant stirring, the nearly neutral solution of iron and aluminium salts. Boil until the precipitate is quite clear. Filter and wash the precipitate and dissolve in dilute hydrochloric acid and reprecipitate with ammonia, observing the necessary precautions. Dry, ignite and weigh as the oxide.

To the filtrate add sufficient hydrochloric acid to dissolve up the precipitate that forms and reprecipitate the salt. Minimize as the hydride, and weigh as the oxide.

This method though quite accurate and fairly quick lacks directness. If the ferric hydride precipitate did not retain traces of the carbonate potash (soda) after careful washing, this method would be more valuable. As it is a second precipitation of iron is required. The action of the carbonate alkalies upon the glass vessel that must
be used is no doubt the chief objection. The soln. thus dissolved appears with the alumina and is separated from the latter only with greatest difficulty. For this separation a nickel dish is used as neither glass nor porcelain will answer. Nickel is even better than platinum. As the solution in boiling spirit up the sides of the dish and dries precautions must be taken to avoid this. The action of the air upon the solution is to form the carbonate of alumina which would be precipitated and appear with the iron and be a source of error. For both of these the best thing to be done is to keep the nickel dish well covered during the whole operation.

To prevent the action of the caustic alkali solution upon the fuller flask the hydrochloric acid is placed in the latter and the filtrate of caustic alumina all is allowed to run into it and so the effect upon the flask prevented. This method gives satisfaction if all the precautions are followed, but omission of any is liable to cause errors in the results.
2. Sodium hydrate method (2)

This method is practically the same as the foregoing and is subject to the same sources of error. The caustic alkali should be present in sufficient excess to cause the solution of the aluminium hydrate and to overcome the action of the air. In this case the quantity in excess is not sufficient to cause any great action upon the glass but it is best to filter into the hydrochloric acid.

It is difficult to get chemically pure sodium hydrate; potassium hydrate is used in most cases. A good substitute for the sodium hydrate is the combination of the oxide as this can easily be obtained chemically pure.

3. Barium hydrate method (3)

This method differs slightly from the other two in the latter part of the preparation. The solution of iron and aluminium salts is added gradually and with constant stirring to the hot solution of the barium nitrate. The whole solution is boiled for some time and the precipitate filtered and washed with hot water and dissolved in dilute
sulphuric acid. The precipitate of barium sulphate, should there be any, is filtered off and the remainder treated on the usual way, volumetrically or by estimation, and weighed as oxides.

To the filtrate, sulphuric acid is added until all the barium is separated and the solution treated as the aluminium determined in the filtrate in the usual way.

The method was not described in detail but from its similarity to the other two this method of procedure was thought to be right. From the appearance the method would seem better than the first two but this is not the case. The action upon the glass is not as strong as either sodium or potassium hydrate, but the precipitate obtained is worse than the precipitate obtained with the latter two. The separation of the barium is effected with difficulty and the precipitate is so fine and porous that it passes through the filter paper. This precipitate must be allowed to settle completely before it may be handled and so the time required is considerable.
The first two methods are quickness and succinctness and an inaccurate doubt.

4. Ethylamine method

The method used is not given. Merely the results and a description of the reagent were published in the periodicals to which proc.
ness was had. The reagent is described as a heavy oily liquid nearly as an crushed bones
sium hydroxid.

From its similarity to some other method tried, the method of procedure is possibly as follows:

Tolu nearly 100°C solution add
an excess of Ethylamine and mix thor-
oughly. Allow the precipitate to stand from
8-12 hours and then filter and wash
first, with a solution of Ethylamine (dilute) and then cold water. Dry, ignite and
weigh the precipitate as the oxide.

Precipitate the aluminium am the film
trate in the usual way and weigh it as
the oxide.

To great an excess of precipitant should
be avoided as this is liable to lead to error
in the results, through reduction of ferri
oxide by an excess of carbon. The time
allowed, the precipitate to settle is normally regulated by the excess of the precipitant, which if small will require a longer time. The ignition of the precipitate of iron salts will have to be attended to with great care. It may have a tendency to puff when heated, but at one time, it may swell up into a black voluminous mass upon heating greatly and at this stage, it should be allowed to cool and some running water added to help the oxidation of the carbon. It is best to use two bunsen flames for ignition after the mass is charred. The chief source of error will be in the ignition of the precipitate. If this latter has been conducted properly, the ferric oxide will be a loose, fluffy, red mass.

This method is said to be the most accurate as well as the most costly.

5. Quatetylamine Method.

Quatetylamine method resembles the above very much, and could be governed by the same principles. Its source ferric would be the same and it should be as accurate too.
Blotting.

The iron is determined in the following in the usual way, after evaporating off the ether and the greater part of the excess of hydrochloric acid.

The separation of the aluminium is complete as none could be found in the iron after the first few trials of the method. The errors are in the evolution of the hydrochloric acid gas. If too rapid the solution is liable to bubble over the sides of the vessel, if a very small one is used. Care should be exercised in heating the precipitate as her in the evolution of the water and hydrochloric acid of the aluminium. There is great opportunity for the loss of material by the escaping vapours. This is overcome almost entirely, by the rise of the layer of mercuric oxide.

This method is very rapid and direct and also very accurate. It has the great

fold that the writers claim for it and

will not doubt be used to a great extent. Of small opportunity for errors

if all precautions are observed.
and in this one thing alone it is better than a great many of the methods now in use. The solution of hydrochloric acid and ether saturated with the hydrochloric acid gas is not very pleasant to work with and this is due to its irritating properties.
Class II.

1. Sodium thiocyanate Method. (7)

In this method the solution of iron and aluminium salts is boiled with an excess of sodium thiocyanate until there are no more fumes of sulphur dioxide given off. This usually takes about five hours of boiling. The evaporated water is not replaced. After the sulphur dioxide is driven off potassium hydroxide is added and the solution boiled and the former sulphide filtered and washed. The iron is determined in several ways.

In the filtrate the aluminium is de:
Terned by adding hydrochloric acid enough to dissolve the precipitate that forms and then ammonia. The alum
minium is weighed as the oxide.

This method has the disadvantage of the long continued boiling. The volumetric
method would perhaps be the best for the

determination of the iron. If it is reduced
by hydrogen there is opportunity for loss.
The sulphide may however, be dissolved in hydrochloric acid (white) and

\[ \text{Na}_2 \text{SO}_3 + \text{H}_2\text{SO}_4 \rightarrow \text{Na}_2\text{SO}_4 + \text{H}_2\text{O} + \text{SO}_2 \]
2. Reduction by hydrogen, and

3. Determination volumetrically

4. By volatilisation with hydrochloric acid gas.

The elements are both precipitated as hydrates, ignited and weighed together first.

Then 2.

(2), the precipitate is placed in a porcelain boat and ignited in a current of hydrogen until the weight is constant. The iron is dissolved out and weighed as the oxide by precipitation with volumetrically ammonium and ignition.

13. Volatilised by a current of hydrochloric acid gas and determined by difference.

Instead of determining the iron volumetrically it would be more accurate to determine it volumetrically. There is some opportunity for loss of material by the current of hydrogen or hydrochloric acid gas. The error. Volatilisation of the iron will then help give some trouble as it is always difficult to get rid of the last portion, as the opportunities for error are very great.
3. Electrolytic methods. (9)

(a) From solutions of organic acids.

(b) From phosphoric acid.

(92) Half nearly neutral solution sufficient ammonium oxalate is added to the solution forming the precipitate when the addition of some ammonia. Then an excess of both acetic acid and ammonium oxalate is added, the former about 0.5 grams for each 0.1 gram of ferrous oxide. The solution is warmed to the current passed through. During the deposition, the solution should be kept warm but not boiling. If any of the aluminium should have been deposited the whole deposit must be redissolved in dilute acid and the process repeated without altering the current. The solution should be slightly acid all the time for if alkaline there is a tendency towards the precipitation of ferrous hydride. If the ammonium carbonate at any time should become greater in quantity than the ammonium oxalate, the aluminium is deposited. A current
of about 10-12 cc electrolyte gas is required. This method although quite accurate requires considerable attention.

(b) From phosphoric acid solutions

In this method sufficient pyrophosphate phosphoric acid, sodium pyrophosphate is added to a concentrated bath of metals and ammonium carbonate added to dissolve it and to be in considerable excess. The current is passed through for from 8-12 hours. The solution tested for iron. If all is deposited the iron is washed with cold water and alcohol alcohol distilled at 70-80°C and cooled and weighed.

In this solution the aluminium is determined in the usual way.

This method differs from the above in the attention required, for the above, and for the above, considerably. It is also a more accurate method, especially where the amount of iron and aluminium are about equal. The treatment of the deposited iron is the same for both methods.
4. Guncrust method

The mixed odes are first weighed and then rubbed with guncrust, about an equal volume of sand placed in a porcelain crucible. This mixture is covered with a layer of guncrust heated violently from 5 to 8 minutes over a Bunsen burner. The cold mass is easily separated from the crucible and transferred to a flask with crucible. The crucible is washed with sulphuric acid (2 parts - 1 part) and the washing poured upon the melt. Ammonia is added to the latter and the whole boiled. There is no need to guard against the oxidation of the iron as none takes place. Guncrust acts better than filings as it dissolves more readily. If any arsenic contained in the guncrust or filings, it must be determined. Weighted quantities of the former placed at the bottom of the crucible.

This method is far better most of the others.
Class III.


Evaporate the solution to a small bulk and add enough ammonia so that the slight precipitate that forms dissolves upon the addition of a few drops of hydrochloric acid. Add an equal volume of 50% acetic acid and then the acetic acid solution of the retro-β-naphthal (50% acetic acid being used). The precipitation is affected with cold. Stir thoroughly and allow the solution to stand from 4-8 hours. Filter and wash the precipitate with 50% acetic acid and then cold water until the filtrate is pale straw colored, no residue is left upon evaporation and ignition in a few drops upon a platinum foil. Mix the precipitate with 5 grams of oxalic acid crystals and ignite, gently at first and finally over a blast lamp. The oxide should be a bright red color.

Heat the filtrate to drive off the excess acetic acid and add ammonia in slight excess. Boil off the excess ammonia and allow the precipitate to settle. Filter and wash thoroughly. Ignite and weigh as the
oxide. The latter should be pure white if the separation is complete. If this is not the case, the precipitate after ignition will be tinged with red according to the amount of iron present.

The greatest errors will occur in the ignition of the precipitate.Both contain considerable carbon. The effect upon the flammability of this carbon is not applicable except perhaps in the way of "puffing" and the particles are carried away. In the case of the ferric oxide, unless care is taken, the oxide will be reduced to iron as a brown to metallic iron. The oxalic acid is to prevent the puffing. The better way is to dry the carbonaceous precipitate and separate it from the ferric oxide by precipitation separately. Transfer the precipitate to a crucible and heat very gently at first. As the calcination goes on, the mass swells and blackens and bursts here and there. Let the gases escape. Then, gases burn with a light yellowish flame. By the end of some funny virtues and the oxidation of this black leathery mass is greatly aided. It must be added when the mass is cold for
otherwise there will be a loss by the lively incandescence which causes a puffing and in that is thrown some of the precipitate out of the crucible. This is the only place for error as it is also in the Ethylamine—Tertiaryamine methods. The results are very good.

2. Ferric Sulphide method. 

(a) From Tannic acid solution.

To the solution of iron sulphate add sufficient tannic acid to prevent the precipitation of the iron as alumminium. After the addition of ammonia. Add an excess of ammonium sulphide, allow the precipitate to settle, and then filter. Wash first with water containing a little ammonium sulphide and then cold water. Dissolve the precipitate in about half hydrochloric acid and reprecipitate with ammonia.

To the filtrate add sodium thiosulphate and then hydrochloric acid so that there will be about 1 or more than is required to decompose the ammonium sulphide. Add 2–5 grams of sodium
acetic acid and boil from 1-2 hours without replacing the evaporated water. Allow the precipitate to settle and filter. Wash twice or twice to get rid of most of the tartaric acid and dissolve the precipitate in dilute nitric acid. This solution is then nearly neutralised with ammonia and 2 to 3 drops of phosphoric acid added and also 2-3 grams of ammonium acetate. Boil from 1/2 - 1 hour and filter and wash. Bar trie boiling water throughout for washing the precipitate of aluminium phosphate. Dry, grind, and weigh the precipitate as \( \text{Al}_2 \text{O}_3 \cdot \text{P}_2\text{O}_5 \). The separation of the iron is easily and completely accomplished. The method is tedious, however, as the time required for boiling is considerable. The method has the disadvantage of requiring a second precipitation of the aluminium phosphate. The object of this is to get rid of the tartaric acid. It could not be washed out of the first precipitate completely and would give considerable trouble in the ignition of the precipitate. This double precipitation is carried out with the extreme kindness of the precipitate.
give opportunities for loss of water. The results as far as the iron is concerned, are fair, but when those of the aluminium are taken into consideration, the method cannot be said to be a very desirable one. If the second precipitation were not required it would be a good method as the error in the case of the aluminium would be only in filtration, with precipitation will come through if care is not taken. But in this method is not satisfactory in quickness, directness or accuracy.
Class II.

1. Basic nitrate method.

The hydrous precipitate of bauxite and aluminium is dissolved in a little dilute nitric acid and to the solution about 5 cc of a 20% solution of potassium hydride are added. This is evaporated to dryness upon a waterbath until two more furnaces of nitric acid are given off. The residue is moistened with a little water, about 2 cc, and re-evaporated. The residue of basic nitrates is dissolved in water and warmed gently and to the solution 15-20 drops of a saturated solution of sodium sulphate are added. The precipitate is allowed to settle and then filtered over asbestos or by means of a double filter paper, without the aid of the felts. The precipitate of basic basic nitrate is washed well with hot dilute hydrochloric acid solution and then dissolved in hydrochloric acid (white) and rapidly saturated with ammonia.

To the filtrate a little hydrochloric acid is added and the aluminium debris removed in the usual way.

The solution of the basic nitrate should
The basic ferric nitrate is very fine and
soon chokes the filler unless due care is exercised. It is very fine to
run through and so the filler must
cannot be safely used. If the evaporation
was carried far enough with just place
the separation of the main is very complete.

The method has the advantage that it re-
gures very little attention when once started
and other work may be carried on. It
is also quite accurate in its results. After
one has acquired the knack of separation.

The time required is very long when duplications
are made. Unless the basic nitrate is
well washed the aluminum salt may
adhere to the fibers of the filler faster and
affair with the main. The ferric hydroxide
should also be thoroughly washed not only
to get rid of the macromolecular chlorides but
especially of the potashum salts as
they would increase the weight of the
ferric oxide. Their presence may be
detected by an alkaline reaction of the
oxide.
2. Double cyanide method

The solution is evaporated to small bulk and then treated with a concentrated solution of sodium sulphate to reduce the ferric to ferrous iron. Then it is added slowly to a hot ammoniacal solution of potassium cyanide of twice the volume of the first solution. (The potassium cyanide is in the proportion of 15-20 grams for each 15-3 grams of ferrous oxide). The solution is then boiled for a short time and acetic acid added. The aluminium is then precipitated with ammonium carbonate. The precipitate is filtered and washed with hot water. If the precipitate is not pure white but greenish it must be purified. This is accomplished by digesting both the precipitate in dilute hydrochloric acid 1:4. The cyanide of iron, which is chelate, not a compound resembling sberin blue. This is insoluble in the acid and may be filtered and washed and the aluminium dissolved in the filtrate.

The method of determining the iron was not given by the author.
It is supposed to have given very good results in the analysis. There is some danger in working with the hot ammoniacal solution of potassium cyanide. The method must be rapidly carried through in order to prevent the formation of the cyanide of iron as much as possible.


This consists in adding an excess of potassium sulpho-cyanate to the solution of the iron and aluminium salts and then dissolving the sulpho-cyanate of iron in ether. The solution is shaken with successive portions of ether until the latter is not colored. The iron and aluminium are each determined in their separate solutions.

This method is good only when the amount of iron present is relatively small when compared to the amount of aluminium.
4. Potassium salt method

To the almost neutral solution of iron and aluminium salts add an excess of a solution of neutral potassium salt. Add the latter little by little, and when the solution will after each addition, the salts of iron and aluminium separate out in a reddish brown mass (color depending upon the amount of iron present). Transfer the precipitate to a filter with cold the potassium chloride and dry the precipitate, for a white, fine calcium chloride. Then wash out the hot salt form by means of a solution of petroleum. The aluminium salt is left upon the filter as a pure white mass. This is ignited directly and the aluminium weighed.

To determine the iron boronate, the filtrate is a weighed platinum dish and char and incinerate the residue.

The precipitated salts are very sticky and stick to anything with which they come in contact. They are transferred only with the greatest difficulty.

This is a decided disadvantage. This
almost impossible to get the right kind of Petroleum.

In the above I have given the chief methods of separation of these elements, quantitatively, and have tried to point out the principal errors and the best way to avoid or overcome them. Most of the methods are given in outline, and in many are a great many of the details are omitted. This leaves the working out of the method almost entirely to the student. The best conditions for the separation are sought after and in nearly all the separations they are omitted, perhaps through oversight.

In the following will be given the methods of separation that have been worked out together with any changes that were made and the results obtained.
Separation...
Preparation of Aurone-β-Naphthyl  

The first method of separation tried was that by means of aurone-β-naphthyl. The latter was prepared as follows:

About 50 grams of β-naphthyl were dissolved in 250 cc. of 95% alcohol and boiled upon a water-bath. To this was added, successively, a neutral solution of zinc chloride free from iron. The latter was precipitated by zinc oxide. To the boiling solution was added, another solution of concentrated sodium nitrite, containing about 25 grams of the latter. The addition of this sodium nitrite solution caused a lively effervescence that the solution formed soon took fire. When this was extinguished and the flame put out, the remainder of the sodium nitrite was added and then the boiling continued to complete the reaction. The precipitate was filtered and washed with water, just by mistake. The solution was poured off of the precipitate and alcohol used until the filtrate was pale. When the precipitate was
first transferred to the filter it was dark red, as the zinc salt should be. By the
time that the washing with alcohol was done,
bleed that the precipitate had changed to
a dark green through-out. The wash water
and alcohol were both tested for alkalis
but none found. The zinc chloride solution
gave no reaction for iron, sodium or
ammonia. The precipitate was tested for
manganese but none was found. It was
tested for iron, by igniting a little upon
a platinum foil & reducing, but no residue
was yellow while hot and white when
cold. An iron would have given this
residue, of zinc oxide, a larger red.
Subsequent analysis of the salt (the
determination of the zinc) showed the
percentage of zinc to be a great deal higher
than that of the normal zinc salt. The
percentages were only approximate as
the determination was made by direct evap-
oration of the organic salt. Some of the
zinc was no doubt lost by volatilisation.
Several lots of this zinc nitrate & starch
had been prepared by the two and all
were dried. Another lot was prepared
from the same reagent, and in a nearly the same manner as possible, but the resulting zinc salt was deeper.

The greater part of this zinc salt was added to a solution of sodium hydrosulphite (36 grams of sodium hydrosulphite and 15 cc. of water) and placed under a current of air. From this it might be inferred the action of the carbon dioxide of the air upon the solution. The precipitate was filtered and washed with water, containing a little sodium hydrate and then cold water. If the precipitate was then dissolved in concentrated hydrochloric acid and the brownish precipitate of sulphuric acid washed filtered, washed and dried.
Separation.

The method given is as follows:

To the cold neutral or nearly neutral solution of iron and aluminium salts, add an equal volume of 50% acetic acid and an excess of a solution of nitric-5-naphtal in 50% acetic acid (using about 25 cc of acetic acid). Allow the solution to stand 6-8 hours and then filter and wash the precipitate with a little 50% acetic acid and cold water (until the residue is left upon evaporation of 5 or 6 drops of the filtrate upon a platinum foil). Dry the precipitate with 5 grams formic acid crystals and ignite, gently at first and finally over a blast lamp. If the oxalic acid is not present, the precipitate will puff upon ignition and some material will be lost. It will red colour.

The filtrate is heated to drive off the excess of acetic acid, and ammonia added in slight excess with the excess evaporated off. The dark precipitate is washed, ignited and weighed as aluminium. If the separation has been complete, the alumina will be pure white.
A sufficient excess of the nitros-β-naphthol solution should be added in the first place to ensure the complete separation of the free iron, as is required, then, as stated in the article. This may be due to the fact that the authors (Kniepe and Isslinski) allowed their solutions to stand 6-8 hours, while in the separations 2 hours at the most were allowed. With a considerable excess of the precipitant, no iron could be detected in the solution after 2 hours standing. It is best to allow the nitros-β-naphthol to digest one night in the acetic acid. This gives a beautiful deep red solution. If too great a heat is used in adding the solutions of the nitros-β-naphthol, a black furry mass separates out and the solution is useless. If these precautions are followed no trouble will be experienced from the precipitant.

After the addition of the precipitant, the solution, water is added to make the
amount of acetic acid below 10%. This important point is not stated in the article. At first the results were entirely too low for the iron but the aluminium came out fairly.
well. These results for the iron were probably due to the reduction of the ferric
oxide by a powdered, or powered carbon, present, by the excess of carbon present. The
blast-flame was used for the final igni-
tion. The residue of ferric oxide obtained
were composed of small pieces of brownish col-
our and not of the blackful shade that the
authors described. Two very ordinary
burners were substituted for the blast-flame
and another method of igniting the
precipitate was followed. The precipitate
was dried and separated from the jello and
the latter incinerated and the ash added to
the precipitate in a porcelain crucible. The
crucible was heated gently (without the
addition of nitric acid) and the
mass of precipitate began to swell and blacken
and emit bubbles of gas. These bubbles
burned with a yellow soapy flame. After the precipitate was charred and no more gas given off it was allowed to
cool and a few drops of glaining
sulphuric acid were added. Sufficient time
was allowed for the complete reaction to
and the residue ignited gently at first and
later with the two bromes. If the nitric acid is added while the mixture is warm a
lively condensation ensues, amounting to a sort of puffing which throw-
up particles of the precipitate which are lost. The resulting ferric oxide is of a
reddish brown and is a very loose, fluffy, mass, very easily blown about. After
washing, the precipitate was again moistened with nitric acid and digested. This
process was repeated until the weight was constant.

The aluminium hydroxide precipitates in various shades of green, at first,
but changed to brown upon standing. In nearly all cases the aluminium (after digi-
tation) was quite white. In some cases
the colour was pale green but this did not seem to affect the weight. When digested
both red and iron was contained.

The greatest source of error comes in
the ignition of the ferric oxide. Thus unless
great care is taken, the excess of carbon
acts as a reducing agent and ferric
oxides are obtained. Then again care
must be exercised in the addition of the
nitric acid, so that there will be no loss by furring. The source of the greenish color
of the alumina is not known, but this color did not seem to affect results. The method is
quite accurate and quite direct. The
time required is not inconsiderable
and the method is a highly satisfactory one. The
results obtained were as follows:

**Iron**

<table>
<thead>
<tr>
<th>I.</th>
<th>II.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Amount of iron ammonium alum taken</td>
<td>.4206 gm.</td>
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</tbody>
</table>

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight of sample empty</td>
<td>24.1892</td>
</tr>
<tr>
<td>+ Al₂O₃ t.</td>
<td>24.2016</td>
</tr>
</tbody>
</table>

**Purity**

<p>| | |</p>
<table>
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<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Amount in above</td>
<td>.0544</td>
</tr>
<tr>
<td>Percent found</td>
<td>12.75%</td>
</tr>
</tbody>
</table>

**Alumina**

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Amount in above</td>
<td>.7842</td>
</tr>
<tr>
<td>Percent found</td>
<td>5.97%</td>
</tr>
</tbody>
</table>
### Iron

<table>
<thead>
<tr>
<th>Ammonium Alum Taken</th>
<th>(\text{III})</th>
<th>(\text{IV})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron</td>
<td>57.35</td>
<td>62.03</td>
</tr>
</tbody>
</table>

\[\text{Fe}_2\text{O}_3 + \text{ash}\]

| Ferric oxide | 1162 | 1258 |
| Iron @ 70.01% | 0.813 | 0.881 |
| Amount in above | 0.812 | 0.884 |
| Percent found | 14.09% | 14.09% |

### Aluminate

| Potassium alum taken | 104.77 | 76.71 |

\[\text{Weight of crucible empty}\]

| Ferric oxide | 1138 | 1243 |
| Iron @ 70.01% | 0.682 | 0.686 |
| Amount in above | 0.756 | 0.837 |
| Percent found | 7.53% | 5.82% |

In separations (I) and (II), the results are

from (I) to the iron. The per cent of alum.

ium found in (II) is very close to the theoretical.

From the second up, the results are good.

The first results obtained were so poor

that they are not entered here, but many

trials were not made before the separation

began to work quite well.
Iron.

Iron ammonium alum taken 14.925 76.17

Dext Fumarate empty 15.6091 14.2907

+ Ferri oxalate 15.5297 14.5369

Ferri oxalate 10.14 15.48

Iron @ 70.01% 9.710 10.84

Amount m above 0.5696 10.76

Percent found 14.4070 14.7470

Aluminniun.

Potassium alum taken .9673 1.3463

Dext Fumarate empty 24.2994 23.4944

+ Aluminniun oxal. 24.1865 23.3491

Aluminniun oxal. 11.59 14.33

Aluminniun @ 52.947% 8.520 8.769

Amount m above 8.337 8.766

Percent found 8.7230 5.71%

Theoretical percentage

Iron Aluminniun

14.13% 5.69%

Average of all

13.83% 5.64%

The average results are very good.
Basic Ferric Method

Mixed precipitated hydrous iron and aluminium are dissolved in dilute nitric acid until the solution is 5 or 6 N. Potassium nitrate solution is added. The whole is evaporated to dryness upon a water bath and the residue moistened with the evaporated gain. This process is repeated until no more fumes of nitric acid come off. Then moisten the residue with water and transfer to a breaker and wash. Add 15-20 drops of a saturated sodium sulphate solution and allow the precipitate to settle. Filter while warm and wash the basic ferric nitrate with a hot solution of dilute potassium nitrate. Dissolve the precipitate in dilute hydrochloric acid and reprecipitate aluminium with ammonium.

To the filtrate add a little hydrosulphurous acid and determine the aluminium in the usual way.

Use a small porcelain dish for the evaporation, and as small a bulk of solution as possible. The solution of basic nitrate is then heated deep red for 15 mins.
In some cases the increase of weight was due to the presence of aluminium. This was detected by dissolving the ignited oxidic slate in perchloric acid and boiling the solution with caustic soda. The solution was precipitated as the aluminium perchlorate with solution. The precipitate was filtered and washed and the filtrate saturated with hydrochloric acid. Ammonia was then added and a dark gelatinous precipitate denoted the aluminium.

Finally good results were obtained and the method was abandoned. The time required for the separation is greatly considerably. It has the advantage, though, that it requires no further attention, after once starting it, than to monitor the residue and distilling it. After that the regular work comes. While the evaporation is going on other work may be carried on. To ensure the complete separation, if the raw materials are high, due to weighed ferric oxide in place of ferric nitrate, hydrochloric acid and repeat the separation. By this second treatment every trace of aluminium may be separated. Unless the iron has been completely converted into its
has been properly conducted. The preparation of basic ferric nitrate is very fine that unless proper precautions are taken it will run through. This gives the additional trouble of another filtration. The suction pump should not be used at first and it is best not to use it at all. It may be used after the whole precipitate has been transferred to the filter if care is taken in starting it.

As the fibres of the felt paper have almost dency to retain aluminium salts some experiments were made to see if this was the case. Several of the aluminium solutions were run through the process of separation and filtered through filter paper. The latter were properly washed, dried and ignited and the weight taken. In no case was this weight appreciably greater than the weight of the ashy felt. The weight of the ferric oxide was high at first. This increase in weight was probably due to the presence of alkalis in the precipitate but this is not certain as the reaction of the filtered nitrate was not noted.
Iron

Iron taken

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1001</td>
<td>1001</td>
</tr>
</tbody>
</table>

Weight of crucible empty

22.6779
+ Ferric oxide added. 22.5332
Total 45.2111
Ferric oxide

45.2111

Iron as 70.01% 0.6998 0.6992
Amount in above

1.001 1.001
Percent found

99.70% 99.18%

Aluminium

Aluminium taken

10.38 10.38

Weight Ferric oxide

25.2770 25.5733
+ Alumina

25.1765 25.3768
Alumina

19.75 19.75
Alumina as 62.94% 10.46 10.35
Amount in above

10.38 10.38
Percent found

100.71% 99.71%

Theoretical per cents

Iron 100.00 100.00
Alumina

Average found (III + IV) 99.40 100.23%

The other results gave very high percentages of iron and alumina as good results were obtained in duplicate. The method was abandoned.
Upon retraction it may not all be precipitated by the sodium sulphate, but will appear with the alumina. This is one of the chief sources of errors but it may easily be avoided. Another source is the fineness of the precipitate. If it once passes through it is impossible to get a perfectly clear filtrate, no matter how much the solution is filtered. The results are as follows:

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>105.5</td>
<td>103.0</td>
</tr>
</tbody>
</table>

\[\text{Weight of precipitate} \quad 22.825 \quad 22.3284\]

\[+ \text{Ferric nitrate} \quad 22.7317 \quad 22.1637\]

\[\begin{array}{c}
\text{Ferric oxide} \\
\text{Sum at 70.017} \\
\text{Amount in above} \\
\text{Percent Jansen.}
\end{array}\]

\[\begin{array}{c}
15.45 \\
1081 \\
1647 \\
10285
\end{array}\]

\[\begin{array}{c}
16.17 \\
1131 \\
1047 \\
107.7178
\end{array}\]

<table>
<thead>
<tr>
<th>Alumina</th>
<th>0.888</th>
<th>0.888</th>
</tr>
</thead>
</table>

\[\text{Wt. Ferroible nit} \quad 24.3099 \quad 25.2687\]

\[+ \text{alumina} \quad 24.1421 \quad 25.1030\]

\[\begin{array}{c}
\text{Alumina} \\
\text{Alumina at 52.947} \\
\text{Amount in above} \\
\text{Percent Jansen.}
\end{array}\]

\[\begin{array}{c}
16.78 \\
0.888 \\
0.888 \\
97.4178
\end{array}\]
Lithium Hydrate Method

Add the solution, nearly neutralized with sodium carbonate, slowly and with constant stirring, to the nearly boiling solution of sodium hydrate. Filter and wash the precipitate with hot water and dissolve in dilute hydrochloric acid. Re-precipitate with ammonia and weigh as the oxide.

To the filtrate add enough hydrochloric acid to dissolve the precipitate that forms and precipitate the aluminium with ammonia and weigh as the oxide.

In this later work a standard solution was used and the value is expressed in terms of the iron and aluminium present.

In place of using caustic soda, sodium hydroxide was used. This can be obtained in a more stable, more easily chlorate the caustic soda. 2.3 grams in 100 cc of water were used for each analysis.

The hydrochloric acid was placed in the filter flask and the effects of the excess caustic soda were counteracted. The results thus obtained, first, were above the theoretical value of the soln.
time so that a test of the solution was made. The iron solution was found to
be all right but the value of the alum-
minimum solution was too high as usual. When the results were reduced to the new
value they were good.

As the reaction of the ferric oxide was
not latent, the cause of the increased
weight was not known to be due to alkalies
present. Only in cases where the results
for the ferric oxide were exceptionally
high, were any pieces of aluminium
found.

The chief source of error in the weight
of the aluminium would be the silica diss
olved from the feldspar flasks. This source
was counteracted by heating the hydro-
chloric acid in the flask and allowing
the sodium aluminium to fisk until it. The
dish was kept well covered during the spe-
cial and the opportunities for the solution
finding upon the sides of the dish, or, to
be affected by the carbon dioxide of the
air were reduced to a minimum. Or
less the hydroxylate of ferric hydroxide
(reacted one) is thoroughly washed. The
Potassium salts will appear with the ferric oxide. This may have been the cause of the increase of weight of the ferric oxide. The method does not require so much time yet over time it takes twice as quickly as the separation must be affected as quickly as possible.

Results:

<table>
<thead>
<tr>
<th></th>
<th>I</th>
<th>II</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron A.</td>
<td>10.81</td>
<td>10.81</td>
</tr>
</tbody>
</table>

| Iron Tenable simply | 20.9479 | 24.8532 |
| + Ferric oxide & ash | 7.888   | 5.688   |
| Ferric oxide        | 0.1661  | 0.1644  |
| Iron @ 70.01 %      | 0.1164  | 0.1150  |
| Amount as above     | 0.1078  | 0.1078  |
| Percent found       | 107.68% | 107.68% |

Aluminium Tenable:

|        | 0.888 | 0.888 |

| Iron Tenable simply | 24.3105 | 25.2688 |
| + Alumina oxide & ash | 14.26  | 25.0997 |
| Alumina             | 0.1678  | 0.1691  |
| Alumina @ 52.94%    | 0.0899  | 0.0895  |
| Amount as above     | 0.0888  | 0.0888  |
| Percent found       | 100.10% | 100.18% |

Alumina T. It had a faint red tinge.
Iron taken

C. H. F. Feasible wt.: 22.8869  22.3264
Fe as FeO... 2.245  1.622
Fe as FeS...  1.624  1.622
Iron @ 70.01%... 11.36  11.36
Amount in above...  1.078  1.078
Percent found... 104.99%  104.90%

Aluminum taken...  0.888  0.888
Cordite 2.745  25.2693
+ Aluminum ash...  2.424  1.038
Aluminum... 1.665  1.665
Aluminum @ 52.94%... 0.887  0.878
Amount in above...  0.888  0.888
Percent found... 99.90%  99.88%

The iron in IV showed traces of aluminum.

Average fused: 106.25% 99.88%
Theoretical... 99.96% 100.00%
<table>
<thead>
<tr>
<th></th>
<th>I</th>
<th>II</th>
<th>III</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron Oxide</td>
<td>23.76</td>
<td>21.52</td>
<td>22.67</td>
</tr>
<tr>
<td>+ Feric Oxide</td>
<td>23.76</td>
<td>21.52</td>
<td>22.67</td>
</tr>
<tr>
<td>Ferric Oxide</td>
<td>28.30</td>
<td>28.85</td>
<td>14.84</td>
</tr>
<tr>
<td>Iron @ 70.0%</td>
<td>19.81</td>
<td>20.19</td>
<td>10.09</td>
</tr>
<tr>
<td>Amount in above</td>
<td>20.02</td>
<td>20.02</td>
<td>10.01</td>
</tr>
<tr>
<td>Percent found</td>
<td>99.01%</td>
<td>100.74%</td>
<td>100.57%</td>
</tr>
</tbody>
</table>

**Alumminium**

<table>
<thead>
<tr>
<th>Alumminium Taken</th>
<th>2076</th>
<th>2076</th>
<th>1038</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crucible empty</td>
<td>25.276</td>
<td>24.3172</td>
<td>25.5744</td>
</tr>
<tr>
<td>+ Alumminium + ash</td>
<td>24.8839</td>
<td>23.9236</td>
<td>3.79</td>
</tr>
<tr>
<td>Alumminium</td>
<td>39.24</td>
<td>39.36</td>
<td>19.65</td>
</tr>
<tr>
<td>Alumminium @ 5-19%</td>
<td>20.77</td>
<td>20.42</td>
<td>10.4</td>
</tr>
<tr>
<td>Amount in above</td>
<td>20.76</td>
<td>20.76</td>
<td>10.38</td>
</tr>
<tr>
<td>Percent found</td>
<td>100.55%</td>
<td>100.38%</td>
<td>100.19%</td>
</tr>
</tbody>
</table>
Preparation of Potassium Alkal.

To about 95 grams of oleic acid add two or three drops of phenol phthalein and
then some concentrated caustic potash.

Stir vigorously and add more caustic
potash. If the mass gets too stiff to work
place upon a sand-bath for a while or add
a little alcohol. Better make as soft and
easy to work by the latter method is better.

Add the caustic potash gradually and
work the mixture thoroughly to a very
favorable alkaline reaction. It is then ready
for use. Make some up into a solution.
Separation

To the nearly neutral solution of iron and aluminium salts add a little of the neutral potassium salt solution and shake vigorously. When this is worked up add some more and continue until the solution is colourless. By this time all the iron and aluminium have been converted into the chlorides. Transfer the precipitate to a filter and wash with hot water until the potassium chloride is all removed. By the precipitate over calcium chloride for a little while and then wash with hot petroleum. The iron is almost insoluble in the latter, passes through with the filter, the white aluminium salt remains behind. Ignite the aluminium salt to white ash and weigh as alumina.

To determine the iron in the filtrate, evaporate it to dryness in a weighed platinum dish and weigh as ferric oxide. The petroleum used was a solvent of the aluminium salt also and so would not answer. It was found that the aluminium salt was insoluble in petroleum.
but the iron oxide was not soluble enough.

The preservative is very bad to work with. It is so sticky and adheres to anything with which it comes in contact. This makes it exceedingly difficult to transfer to the filler.

No petroleum was obtained that would answer the purpose. It is hard to obtain the correct one. These hydrocarbons have no definable formula and so what is called petroleum by one author may be another thing in another author's opinion.
Separation in a Tartaric Acid Solution.

To the solution add Tartaric acid until no precipitate is given by the addition of ammonia. Add Ammonium sulphide in excess and allow it to precipitate. Wash and filter it. Wash with water containing a little ammonium sulphide, and then with cold water. Dissolve the precipitated sulphide in dilute hydrochloric acid and determine the iron by means of ammonia.

To the filtrate add sodium phosphate and then hydrochloric acid so that this will be about three-quarters of the latter in excess of that required to decompose the ammonium sulphide. Then add 1-5 grams of sodium acetate and boil from 1-2 hours without replacing the evaporated water. Filter and wash the precipitate once with hot water and dissolve in dilute nitric acid. Nearly neutralise this solution with ammonia and add 2-3 drops of phosphoric acid and then 2-3 grams of ammonium acetate. Boil from 1/2-1 hour. Filter and wash the precipitate thoroughly. Dry, grind, and weigh it as Al₂O₃, O₃. The separation of chromium is complete.
As the sulphide is not assayed readily in the hydrochloric acid, some potassium chlorate was added to aid the solution.

The aluminium precipitate is so fine that some is lost in filtration and as the results are not good, it is also a tedious method. Taking all into consideration, this is a very unsatisfactory method.

Results:

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Alunite in 25°:  
+ Ferric chloride:  
  Ferric oxide: .1426 .1410
  Iron as FeO: .0968 .0987
  Amount from above: .1001 .1001
  Percent found: 99.70% 98.60%

Alumina:
  .1031 .1031

Aluminate:
  23.1346 22.1422
  + Aluminate: 22.7631 21.7615
  Aluminate: .3977 .3987
  Aluminate @ 25.34% .1008 .1010
  Amount in above: .1038 .1038
  Percent found: 97.13% 92.33%
<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron</td>
<td>1001</td>
<td>1001</td>
</tr>
<tr>
<td>Enamelled</td>
<td>22.397</td>
<td>23.128</td>
</tr>
<tr>
<td>+ Fusible oxide</td>
<td>22.786</td>
<td>22.9746</td>
</tr>
<tr>
<td>Fusible oxide</td>
<td>14.31</td>
<td>14.31</td>
</tr>
<tr>
<td>Iron @ 7.00%</td>
<td>1002</td>
<td>1002</td>
</tr>
<tr>
<td>Amount via above</td>
<td>1001</td>
<td>1001</td>
</tr>
<tr>
<td>Percent found</td>
<td>102.097</td>
<td>102.497</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Alumina</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Alumina nitrate</td>
<td>10.31</td>
<td>10.31</td>
</tr>
<tr>
<td>Enamelled</td>
<td>22.3497</td>
<td>23.8391</td>
</tr>
<tr>
<td>+ Fusible oxide</td>
<td>22.943</td>
<td>23.4364</td>
</tr>
<tr>
<td>Fusible oxide</td>
<td>14.057</td>
<td>14.027</td>
</tr>
<tr>
<td>Alumina nitrate @ 23.34%</td>
<td>10.28</td>
<td>10.20</td>
</tr>
<tr>
<td>Amount via above</td>
<td>10.36</td>
<td>10.36</td>
</tr>
<tr>
<td>Percent found</td>
<td>99.167</td>
<td>98.617</td>
</tr>
</tbody>
</table>

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Approximate Percent</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Percent found average</td>
<td>99.72%</td>
<td>98.04%</td>
</tr>
</tbody>
</table>

Results for iron in the average but iron for the Alumina.
To the solution add enough ammonium oxalate so that no precipitate forms upon the addition of ammonia. Add oxalic acid and an excess of ammonium oxalate. Pass a current of 20-30 cc electrolyte gas per minute through. Keep warm during the separation.

When the iron is all deposited, wash the deposit with water and then absolute alcohol and heat to 100°C, cool and weigh.

If any aluminium has been deposited the whole deposit of iron and aluminium must be dissolved in nitric acid and the process repeated. The solution must be added to alcohol always, for if alkaline ferric hydrate is precipitated, if the amount of ammonium carbonate formed exceeds the amount of ammonium oxalate in solution, aluminium will be deposited. chlorine should not be used.

The aluminium is determined in a suitable way.

This method gives good results but requires considerable attention. The results with the chloride form...
IV. From Phosphoric Acid Solutions.

To the nearly neutral solution add sodium
pyrophosphate to precipitate all the iron and
aluminium present and then add ammonia
bicarbonate in excess. The solution should be
perfectly clear before using. The current was
passed through for at least 40 hours and
the deposit was not allowed until then.

The results with this method should be much
better than with the last one. It requires no
attention. The deposit is treated as in the
other method. The aluminium is determined as
Al₂O₃, B₃O₅ which involves a loss of water.

In all other determinations the chloride solution
gave much worse results than those were not
considered. In other separations a nitrate
solution was used.

The results for the iron are fair but
for the aluminium very poor. The amount
used was exceedingly small.
<table>
<thead>
<tr>
<th>Iron</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron taken</td>
<td>0.053</td>
</tr>
<tr>
<td>Weight</td>
<td></td>
</tr>
<tr>
<td>before</td>
<td>10.6744</td>
</tr>
<tr>
<td>after</td>
<td>10.6575</td>
</tr>
<tr>
<td>Iron</td>
<td>0.129</td>
</tr>
<tr>
<td>Amount in above</td>
<td>0.0155</td>
</tr>
<tr>
<td>Percent found</td>
<td>1.6903%</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Aluminum</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum taken</td>
<td>0.0140</td>
</tr>
<tr>
<td>Feasible only</td>
<td>23.1457</td>
</tr>
<tr>
<td>+ Alys 0.5</td>
<td>22.9917</td>
</tr>
<tr>
<td>C = 0.5 0.5</td>
<td>0.0534</td>
</tr>
<tr>
<td>Aluminum @ 25.34%</td>
<td>0.135</td>
</tr>
<tr>
<td>Amount in above</td>
<td>0.140</td>
</tr>
<tr>
<td>Percent found</td>
<td>96.4670</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Theoretical Percent</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Percent found average</td>
<td>103.53</td>
</tr>
</tbody>
</table>

Sources: Aluminum results recorded due to loss of material. No iron analyzed aluminum detected.
Separation with Hydrochloric Acid and Ether

To the solution of small bulk add 10-15 cc of solution of equal parts ether and concentrated hydriodic acid. Stir the solution (not very 25-30 cc) with hydrochloric acid gas to saturation. (Keep the flask cool with running water). Add 5 cc ether to remove free peroxide. Equilibrate the two solutions. Add excess hydrochloric acid gas through to perfect saturation.

Transfer the necrotised aluminaum chloride to an asbestos filter and wash with a solution of hydrochloric acid and ether saturated at 10°C with hydrochloric acid gas. Dry the precipitate ½ hour under cover.

Weigh a layer of pure mercuric oxide and ignite gently at first and finally over a Bunsen flame and weigh as the oxide.

After drying off the ether and excess hydrochloric acid from the precipitate determine the iron in the usual way.

The estimation is complete and only at first will faint traces of iron be found in the alumina and this will chiefly due to the iron of the mercuric oxide. The greatest trouble for error is in the equilibration of the precipitate. There is danger of rehydrolyzing the
freewheel being carried away by the evolved gas or water, so great care must be exercised in the first part of the ignition. This is also guarded against by the use of mercuric oxide. The current of hydrochloric acid gas should also be carefully regulated.

This method is the best one given. It is the quickest and most direct method known and the results are very satisfactory. The opportunities for error are many and mistakes from may be easily guarded against.

The results are on the next page.
### Results:

<table>
<thead>
<tr>
<th>Item</th>
<th>Unit 1</th>
<th>Unit 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formed empty</td>
<td>25.189</td>
<td>22.6756</td>
</tr>
<tr>
<td>+ Volume added, calc.</td>
<td>24.942</td>
<td>22.5526</td>
</tr>
<tr>
<td>Formed added</td>
<td>1.281</td>
<td>1.270</td>
</tr>
<tr>
<td>Form. O70.01 %</td>
<td>0.588</td>
<td>0.588</td>
</tr>
<tr>
<td>Amount in above</td>
<td>0.587</td>
<td>0.587</td>
</tr>
<tr>
<td>Percent found</td>
<td>102.98%</td>
<td>99.80%</td>
</tr>
</tbody>
</table>

### Aluminium

<table>
<thead>
<tr>
<th>Item</th>
<th>Unit 1</th>
<th>Unit 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminium taken</td>
<td>0.309</td>
<td>0.309</td>
</tr>
</tbody>
</table>

### Possible error

<table>
<thead>
<tr>
<th>Item</th>
<th>Unit 1</th>
<th>Unit 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbonated</td>
<td>14.850</td>
<td>17.1516</td>
</tr>
<tr>
<td>- + aluminium, calc.</td>
<td>14.7923</td>
<td>17.0527</td>
</tr>
<tr>
<td>Alumina</td>
<td>0.577</td>
<td>0.573</td>
</tr>
<tr>
<td>Aluminium O52.34 %</td>
<td>0.305</td>
<td>0.3005</td>
</tr>
<tr>
<td>Percent above</td>
<td>0.309</td>
<td>0.309</td>
</tr>
<tr>
<td>Percent found</td>
<td>99.61%</td>
<td>99.83%</td>
</tr>
</tbody>
</table>

M. I. contained aluminium.
<table>
<thead>
<tr>
<th></th>
<th>0.8077</th>
<th>1940</th>
</tr>
</thead>
<tbody>
<tr>
<td>Enamal weight</td>
<td>25.0661</td>
<td>22.6742</td>
</tr>
<tr>
<td>+ Ferrie oxide</td>
<td>34.9827</td>
<td>22.6249</td>
</tr>
<tr>
<td>Ferrie oxide</td>
<td>1.254</td>
<td>247.3</td>
</tr>
<tr>
<td>Disc @ 76.07%</td>
<td>0.8878</td>
<td>1745.4</td>
</tr>
<tr>
<td>Amount un avai.</td>
<td>0.0870</td>
<td>1740.5</td>
</tr>
<tr>
<td>Percent Enamal</td>
<td>190.8770</td>
<td>190.282</td>
</tr>
</tbody>
</table>

**Alumminium**

<table>
<thead>
<tr>
<th></th>
<th>0.809</th>
<th>10618</th>
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</thead>
<tbody>
<tr>
<td>Enamel weight</td>
<td>17.0937</td>
<td>17.0705</td>
</tr>
<tr>
<td>+ Alumina</td>
<td>12.6536</td>
<td>16.9542</td>
</tr>
<tr>
<td>Alumina</td>
<td>0.581</td>
<td>116.3</td>
</tr>
<tr>
<td>Alumminium @ 52.94%</td>
<td>0.6302</td>
<td>0.616</td>
</tr>
<tr>
<td>Amount un avai.</td>
<td>0.309</td>
<td>0.6168</td>
</tr>
<tr>
<td>Percent Enamal</td>
<td>97.83%</td>
<td>97.64%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>----------------</td>
<td>-------</td>
<td>-------</td>
</tr>
<tr>
<td>Iron</td>
<td>1.740</td>
<td>1.740</td>
</tr>
<tr>
<td>Laxative</td>
<td>22.6739</td>
<td>23.4458</td>
</tr>
<tr>
<td>+ Ferrous oxide</td>
<td>22.4255</td>
<td>23.1235</td>
</tr>
<tr>
<td>Ferrous oxide</td>
<td>2.4484</td>
<td>2.4972</td>
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<tr>
<td>Iron 7.00%</td>
<td>17.388</td>
<td>17.244</td>
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<tr>
<td>Amount in oxide</td>
<td>1.6740</td>
<td>1.740</td>
</tr>
<tr>
<td>Percent found</td>
<td>99.93%</td>
<td>100.23%</td>
</tr>
<tr>
<td>aluminum</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Aluminum oxide</td>
<td>0.615</td>
<td>0.618</td>
</tr>
<tr>
<td>Laxative</td>
<td>17.2834</td>
<td>17.3457</td>
</tr>
<tr>
<td>Oxide + alunim.</td>
<td>17.6432</td>
<td>17.2294</td>
</tr>
<tr>
<td>Alumina</td>
<td>1.162</td>
<td>1.164</td>
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<tr>
<td>Aluminum 92.94%</td>
<td>0.665</td>
<td>0.668</td>
</tr>
<tr>
<td>Amount in alunim.</td>
<td>0.668</td>
<td>0.668</td>
</tr>
<tr>
<td>Percent found</td>
<td>99.47%</td>
<td>99.64%</td>
</tr>
</tbody>
</table>

Theoretical Percent
Percent found (average) 100.00% 100.00%

Average results are good.
From the results obtained it will be seen that the best satisfaction was at first obtained from the nitrous \(\text{N}_2\text{O}_3\) chloride, sodium hydroxide, and hydrochloric acid and ether solvents.

The first named gave especially good results for the amounts used. This method is supposed to be the best but probably may have been classed with it by just as mentioned. The number of methods that was not large but on the average they worked fairly well.
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