

## **On the acetates and other compounds of alumina / by Walter Crum.**

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*Supplemental*  
*from the Author.*

ON THE

## ACETATES AND OTHER COMPOUNDS OF ALUMINA.\*

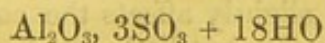
BY WALTER CRUM, F.R.S.

PRESIDENT OF THE PHILOSOPHICAL SOCIETY OF GLASGOW.

THE salt from which most of the products described in the present memoir were produced, is the tersulphate of alumina, now manufactured in large quantity in the north of England, under the name of concentrated alum. It is formed by the direct action of sulphuric acid upon clay, and, after a certain purification, is evaporated and sold in cakes.

The impurity which the commercial article contains in largest quantity is potash alum. To separate that substance, advantage was taken of its insolubility in a saturated solution of the tersulphate. The cake-alum was dissolved in a quantity of boiling water insufficient for its entire solution in the cold. On cooling, the excess of sulphate of alumina, mixed with almost the whole of the potash alum, deposited, and was separated by filtration through calico. The filtered solution was then evaporated and cooled, when the tersulphate was found to crystallize in granular spongy masses. It was then drained and separated as much as possible from the mother liquor by strong pressure between numerous folds of calico. The product was re-dissolved in boiling water, and again crystallized and pressed.

A salt was thus obtained, having little more than traces of the arsenious acid and the potash of the original material, together with a little iron and chloride of sodium. It has the formula



first observed in 1825 by Boussingault in the hair-like substance found among the black schistus of the Andes of Columbia, near Bogota. He afterwards found the same substance in the volcano of Pasto, (north of Quito,) where it is formed by the action of the sulphurous vapours upon the schistus. It exists there in such quantities as to have enabled the Pastos to become the manufacturers of chemical products for the whole country. They dissolve the alum in water, evaporate it to dryness, and form it into spherical masses like camphor, which are altogether free from iron.

The same substance is formed in this neighbourhood from a schistus containing pyrites, in which case it is mixed or combined with sulphate of iron.

\* Read before the Philosophical Society of Glasgow, April 27th, 1853.



The quantity of cake-alum now produced annually at Newcastle and at Sowerby-bridge, according to statistics furnished me by Mr. Wilson of Hurlet, is .....1,500 tons.  
 And of crystallized alum (chiefly ammoniacal) in England, 13,200 "  
           Do.                           do.                           do.                           in Scotland, 4,200 "

Total in Great Britain, .....18,900 tons.

It is estimated by Mr. Wilson that about one-fourth of this quantity is consumed by calico printers, and converted more or less completely into acetate of alumina. There is no other example of a substance so important and so extensively employed in the arts, as acetate of alumina, having been so little attended to by chemists.

#### INSOLUBLE ACETATES OF ALUMINA.

I. *Insoluble Binacetate of Alumina, 5 Hydrate.* An acetate of alumina was produced by mixing together strong solutions of tersulphate of alumina (purified as already described) and of acetate of lead. They were poured slowly together into a vessel surrounded with cold water, and much agitated to reduce the temperature of the mixture. To the filtrate was added sulphide of hydrogen, to precipitate the lead of the sulphate of lead which remained in solution; and acetate of baryta to throw down its sulphuric acid.

The strongest solution formed in this way contained about 5 per cent. of alumina, (from about 6 lbs. acetate of lead in an imperial gallon of the mixture.)

When such a solution (or one containing even 4 per cent. of alumina) is set aside and left at rest at a temperature of 60° or 70° Fahr., it begins after four or five days (without losing much of its transparency) to deposit upon the vessel a crust, which continues for some time to increase in thickness. When the liquid is poured off, and the crust allowed to dry, it separates readily from the vessel in hard plates like porcelain. If the solution be not left strictly at rest, it becomes turbid after some days, and the crust is produced in a more friable state. In cold weather the solution remains unaltered for a much longer time. Heated to redness, the salt becomes black from the decomposition of part of its acetic acid, and in this state it very slightly affects the colour of moistened litmus paper, showing that it retains but little alkali. Dissolved in nitric acid and tested with nitrate of silver, it showed a trace of chlorine, and it contained also a little iron. It was freed from these impurities by processes to be afterwards detailed.

To ascertain the quantity of alumina in one of these salts, it is enough to moisten it with rectified and concentrated sulphuric acid in a platinum crucible, to evaporate the excess of acid over a spirit lamp, and then to subject it to a white heat half-an-hour in a furnace. The sulphuric acid expels acetic acid, and thereby secures the absence of charcoal. It facilitates the disengagement of water; and the powder, which, without it, becomes so pulverulent by calcination as to be with difficulty prevented from escaping, is thereby rendered coherent.

Attempts were first made to determine the acetic acid by the alka-



limeter, taking potash alum of known purity for a standard, and for a comparison of results. The substance being kept in water at a boiling heat during the addition of the alkali, (soda,) and by a careful comparison of the shades of the litmus paper, appeared at first to redden that test, so long as any portion of the salt remained undecomposed. But it was found, after some practice, that at least 5 per cent. of the insoluble salt remained untouched by the alkali after all trace of acid reaction had disappeared in the process, and that within a range of 3 or 4 per cent. the method was not to be depended upon. It gave valuable assistance, however, in this investigation, in the testing of solutions of alumina, with which it gave results much more accurate than with the insoluble salts.

33.24 grains of the substance, dried first in the air, then pulverized and dried further in a stove twenty hours at 100° Fahr., were moistened with 80 grains of rectified sulphuric acid in a platinum crucible, and calcined as above. There remained 8.64 grains. When this residue was treated with water and filtered, the solution showed no trace of sulphuric acid, and was alumina nearly pure = 25.99 per cent. of the original substance.

In another experiment, 32.64 grains, dried four hours in the stove, and treated as above, left 8.4 grains of alumina = 25.74 per cent. The mean is 25.86 per cent. of alumina.

Burned with oxide of copper, 2.985 grains produced 5.526 cub. inches of dry carbonic acid = 2.612 grains, and corresponding to 50.69 per cent. of acetic acid. In another experiment 1.685 grains indicated 50.58 of acetic acid. The mean is 50.63. It was assumed that the remaining 23.51 per cent. was water. The salt was therefore composed of—

Acetic acid, .....	50.63
Alumina, .....	25.86
Water, .....	23.51
	<hr/>
	100.00

The formula,  $\text{Al}_2\text{O}_3, 2\bar{\text{A}} + 5\text{HO}$ , requires—

Acetic acid, .....	51.41
Alumina, .....	25.91
Water, .....	22.68
	<hr/>
	100.00

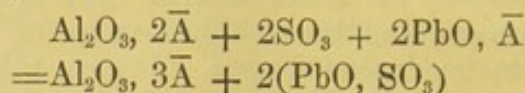
II. *Insoluble Binacetate of Alumina, 2 Hydrate.* If heat be applied to the strong solution of acetate of alumina, described in the previous page, it speedily becomes turbid, and deposits a heavy white powder, which falls readily to the bottom of the vessel. In a couple of days, at 100° Fahr., a considerable quantity of this powder is deposited; but in two or three hours at 160°, and in much shorter time at boiling heat, the whole of the salt is thrown down, and nothing remains in the liquid but acetic acid, excepting a trace of alumina, just distinguishable by carbonate of soda.

The precipitate has a crystalline shining appearance in the moist state, and seems, under the microscope, to consist of small oval particles of uniform



size. It falls into fine powder in drying, after which, when mixed with water, it remains for a long time in suspension. Boiling water does not free it from chloride of sodium, of which it contains about 0.1 per cent.

To remove that impurity, the substance is to be dissolved with the assistance of heat in two equivalents of rectified sulphuric acid, diluted with three times its weight of water. The solution is decomposed by subacetate of lead—



and the filtrate freed from lead and sulphuric acid as before. When heat is now applied to this solution, a precipitate is obtained, in which no trace of alkali or of chlorine can be detected.

A portion of the substance obtained at a boiling heat, and dried 24 hours at 100° Fahr., yielded 29.83 per cent. of alumina. Another portion dried 48 hours gave 30.54 per cent. Burned with oxide of copper, 1.697 grains of the salt, dried at 100° Fahr., indicated 59.87 per cent. of acetic acid. In another experiment 1.605 grains indicated 58.88 per cent. The mean is 59.37. The substance is, therefore, composed of—

Acetic acid, .....	59.37
Alumina, .....	30.18
Water, .....	10.45
	<hr/>
	100.00

The formula,  $\text{Al}_2\text{O}_3, 2\bar{\text{A}} + 2\text{HO}$ , requires—

Acetic acid, .....	59.51
Alumina, .....	29.99
Water, .....	10.50
	<hr/>
	100.00

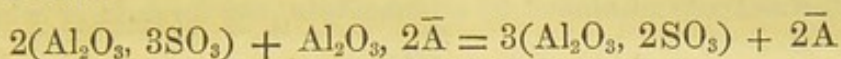
Numerous specimens of this salt were formed by precipitation from solutions of various strengths, at various temperatures below boiling water, and analyzed for acetic acid and alumina. They generally agreed in the proportions of acid and base, but varied in the absolute quantity; indicating a range of from 3 to 5 equivalents in the proportions of water. The two salts which have been described as deposited, one at 60° and the other at a boiling heat, gave results nearly uniform.

When heat is applied to a solution of teracetate of alumina weaker than that which has been described; to one, for example, containing 3 per cent. of alumina, it also yields the insoluble binacetate, but, in that case, not acetic acid alone, but a considerable quantity of acetate of alumina remains in the solution. Solutions containing 2 per cent. of alumina are precipitated by boiling, if they have been kept some weeks, but not if recently prepared. It appears from these experiments, that the presence of free acetic acid favours, in some way, the production of the insoluble binacetate of alumina from a solution of teracetate. It was found, accordingly, that a solution of teracetate containing  $\frac{3}{4}$  per cent. of alumina, and which could not therefore be precipitated by boiling, acquired that property when made to contain acetic acid equal to a solution of teracetate, with 4 per cent. of alumina.

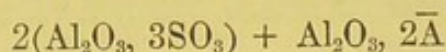


In whichever way deposited, this substance is exceedingly insoluble in water, either cold or hot, and it is equally insoluble in acetic acid. When one part of it is digested for an hour and a-half in two hundred parts of boiling water, it dissolves; and the solution consists partly of soluble binacetate of alumina, and partly of acetic acid and the bihydrate of alumina to be afterwards described.

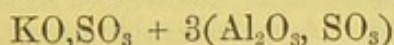
It dissolves in two equivalents of sulphuric acid, or of hydrochloric, or nitric acid, forming bisalts of alumina, and liberating the two atoms of acetic acid. It also dissolves in a strong solution of tersulphate of alumina, with the assistance of heat, forming bisulphate of alumina and free acetic acid.



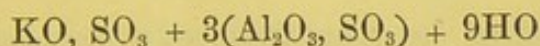
The mixture does not precipitate on the addition of water like the bisulphate alone, and must therefore be changed by water into tersulphate and binacetate of alumina.



A solution of potash alum dissolved this binacetate in the same circumstances, but on the heat being continued for some time, a precipitate was formed anew, which appeared from a qualitative analysis to be the monosulphate of alumina and potash,



the substance which is formed when hydrate of alumina is boiled with alum. The same substance is found native, under the name of alum-stone, at La Tolfa, the seat of the celebrated manufacture of Roman alum, near Civita Vecchia; and was ascertained, by Collet Descotils, to be formed of—



#### SOLUBLE ACETATES OF ALUMINA.

*Soluble Binacetate of Alumina.*  $\text{Al}_2\text{O}_3, 2\bar{\text{A}} + 4\text{HO}$ . Notwithstanding the tendency of a concentrated solution of the ter-acetate of alumina to deposit the insoluble salt, it may be evaporated, with certain precautions, to a dry substance soluble in water. For this purpose it must be spread very thin over sheets of glass, or of porcelain, exposed to a heat not exceeding 100° Fahr., and, as it runs together into drops, like water upon an oiled surface, it must be constantly rubbed with a thin platinum or silver knife. If these precautions are neglected, a mixture is obtained of the insoluble with a soluble acetate.

The soluble salt is thus produced in scales, having the appearance of gum when moistened, and leaving no residue when dissolved in water.

For analysis it was reduced to powder, and dried in the air twenty-four hours, at the temperature of 100° Fahr. The alkalimetric method was first employed, and it indicated 54.8 per cent. of acetic acid; but the more accurate method, by oxide of copper, gave 55.82 per cent. of acid. In experiments for the alumina 26.4 per cent. was obtained. The composition of the substance was thus—



Acetic acid, .....	55.82
Alumina, .....	26.40
Water, .....	17.78
	<hr/>
	100.00

But the solution from which this substance was obtained was a teracetate, and contained for the same quantity of alumina,

Acetic acid, .....	78.58
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It lost, therefore, during gentle evaporation, nearly one-third of its acetic acid, and was reduced almost to a binacetate, the formula of which—

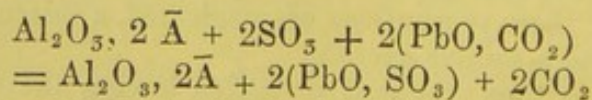
$\text{Al}_2\text{O}_3, 2\bar{\text{A}} + 4\text{HO}$ —requires—	
Acetic acid, .....	52.38
Alumina, .....	26.40
Water, .....	18.49
	<hr/>
	97.27

The excess of acetic acid = 3.44 per cent. over the proportions of a binacetate, was presumed to be free acid adhering to the product. To remove that excess, a portion of it was heated in a water bath, and afterwards treated with water. A considerable quantity of insoluble matter was left, and the solution on being evaporated to dryness, at a gentle heat, produced a soluble acetate, of which 100 parts contained—

of acetic acid, .....	49.55
and of alumina, .....	32.47

corresponding, therefore, in composition, to a sesquiacetate; but it can only be looked upon as an accidental mixture, for every fresh portion that was produced in a similar way gave a different result—the amount of acid depending upon the extent and duration of the heat.

The binacetate of alumina may be produced at once in solution; and as it is the most suitable combination from which to form the dry soluble binacetate, as well as other bodies, I shall describe particularly the manner of obtaining it. Dissolve 24 parts of precipitated binacetate of alumina in 15 of rectified sulphuric acid, and 40 of water. Dilute further with 80 parts of water, and add carbonate of lead (about 44 parts) to precipitate the sulphuric acid.



Filter the solution, and pass sulphide of hydrogen through it until it ceases to precipitate lead, and then add acetate of baryta, so long as it is precipitated by the sulphuric acid of the sulphate of lead which had remained in solution. When in this state, if the mixture be well agitated for half an hour in an open vessel, the excess of sulphide of hydrogen will be removed, and it may be filtered without the danger of the filtrate becoming afterwards milky, from the effects of the sulphide.



A solution of binacetate of alumina is thus obtained, containing about 5 per cent. of alumina, and a minute portion of iron; the last traces of which it was, for a long time, difficult to remove from these acetates. Iron is found in all the solutions, and in both of the insoluble binacetates. Traces of it exist even in the binacetate which has been freed, by a second precipitation, from all its other impurities. It was at last observed that a solution of binacetate of alumina, at 5 per cent., which is strong enough to form, after some time, a crust of the binacetate, deposited its iron along with the first portions of the crust, and left the solution altogether free from that impurity. The teracetate of alumina does not deposit its iron in like circumstances; a difference which may be accounted for from the fact that the binacetate of the sesquioxide of iron is more readily decomposed than the teracetate of that base.

Spread very thinly over a sheet of glass, it evaporates at 60° or at 100° Fahr., without running into large drops so much as the teracetate, and without having an equal tendency to produce the insoluble binacetate. The scales into which it forms in drying are transparent and soluble in water.

Burned with oxide of copper for acetic acid, and calcined for alumina, 100 parts were found to contain—

Acetic acid, .....	55.21
Alumina, .....	31.31

But the alkalimeter had shown, that in the solution the same quantity of alumina had been combined with—

Acetic acid, .....	59.13
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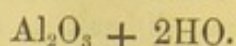
More than 4 per cent. therefore had been lost by evaporation at 100° Fahr.

After some other attempts, I was obliged to conclude that the only way to obtain the dry soluble binacetate in something like atomic proportions, was the unsatisfactory one of evaporating in the air a solution of binacetate, mixed with such a proportion of acetic acid or of teracetate as is found by trial to produce it.

On the question, as to which of the soluble acetates of alumina can be considered as a definite compound, it has already been stated of the teracetate, that when evaporated rapidly enough, and at a heat just low enough to prevent the formation of the insoluble salt, it leaves a product whose composition is nearly that of a binacetate. It may be added, that the solution of teracetate gives off acetic acid as freely in the cold, as if a third part of its acid were free. On making an experiment with two solutions of acetate of lead—one of which was decomposed by sulphuric acid, and the other (which was three times as strong) by tersulphate of alumina—it was found that the aluminous solution gave a smell of acetic acid considerably stronger than that in which the acid was known to be free. It may be doubted, then, whether there exists a combination of acetic acid with alumina corresponding to the tersulphate of alumina. The solution of binacetate has no smell of acetic acid at ordinary temperatures.



## BIHYDRATE OF ALUMINA SOLUBLE IN WATER.



*Hydrate from the soluble Binacetate.*—By the continued action of heat on a weak solution of binacetate of alumina, a permanent separation of the constituents of the salt takes place, although no acid escapes, and no alumina is precipitated. The properties of the alumina are at the same time materially changed.

A solution of binacetate of alumina, diluted so as to contain not more than 1 part of alumina in 200 of water, was placed in a close vessel which was immersed to the neck in boiling water, and kept in that state day and night for ten days. It had then nearly lost the astringent taste of alum, and acquired the taste of acetic acid. The liquid was now placed in a wide and shallow vessel, where it was kept at a uniform depth of a quarter of an inch; and on heat being applied to make it boil briskly over the whole surface, the acetic acid was driven off in about an hour and a half, so as to be no longer sensible to litmus paper. The liquid in this operation should not contain more than 1 part of alumina in 400 of water, and the loss from evaporation is supplied by continual additions of water.

The solution thus obtained is nearly as transparent and limpid as before the loss of its acid. By longer boiling, and particularly by concentration, it becomes more and more gummy; a quality of which it is partially deprived by acetic acid. It is altogether tasteless.

When one grain of sulphuric acid ( $\text{SO}_3$ ) in 1000 grains of water, is mixed with 8000 grains of the solution, containing 20 grains of alumina, the whole is converted into a solid transparent jelly. By pressure in a bag the liquid part of this jelly is readily separated from the solid; whose volume in the compressed state, is not more than  $\frac{1}{60}$  or  $\frac{1}{70}$  of the volume of the jelly. On examination it was found that the solid part of the coagulum contained almost the whole of the sulphuric acid which had produced it—about one equivalent of sulphuric acid to fifteen of alumina.

One atom of citric acid (tribasic) coagulates as powerfully as three atoms of sulphuric acid; and tartaric acid (bibasic) as much as two. Two atoms of oxalic acid are required to produce the same effect as one of sulphuric acid. Of muriatic and nitric acids not less than 300 equivalents must be employed to produce an effect equal to that of one equivalent of sulphuric acid.

Of the other acids which have been tried, the chromic, molybdic, racemic, suberic, salicylic, benzoic, gallic, lactic, cinnamic, butyric, valerianic, carbazotic, camphoric, uric, meconic, comenic and hemipinic acids all coagulate the solution; but their exact power has not been ascertained.

The acetic, formic, boracic, arsenious, and cyanuric acids do not coagulate, at least when moderately concentrated; and, of the opium acids, for a supply of which I am indebted to Professor Anderson, the pyromeconic and opianic acids also do not coagulate.

One grain of potash in 1000 grains of water coagulates 9000 grains of the solution—a proportion which gives about 1 equivalent of potash to 20 of alumina. The mixture has a slight alkaline reaction. Soda,



ammonia, and lime have an equally powerful effect. The coagulum they produce is partially re-dissolved when the alkali is saturated by acetic or by muriatic acid. The salts which are thus formed render the solution somewhat oily.

A boiling solution of potash or soda dissolves the coagulum, and at the same time changes it into the ordinary alumina, which is thrown down in the state of terhydrate when the alkali is saturated by an acid.

Oil of vitriol also dissolves the solid part of the coagulum, and the same substance dried; especially when assisted by heat. Strong muriatic acid, at a boiling heat, does the same, though with greater difficulty; and the products are the ordinary sulphate and muriate of alumina.

Large quantities of the acetic salts may be added before they coagulate the aluminous solution.\* When the solid part of the coagulum produced by a strong solution of acetate of soda was afterwards freed from that salt by pressure, it re-dissolved in pure water, and the solution was again coagulated by a fresh addition of the salt. An experiment with acetate of lime gave the same result.

The nitrates and chlorides coagulate also with great difficulty.

Solutions of sulphate of soda, magnesia, and lime coagulate as readily as a liquid containing the same quantity of sulphuric acid in the free state. On examining one of these mixtures, the sulphuric acid was found in the solid part of the coagulum, as before; and the mixture remained neutral.

A small spoonful taken into the mouth becomes immediately solid from the effect of the saliva.

The digested solution of alumina which has not been deprived of its acetic acid by boiling, requires about twice as much sulphuric acid to coagulate it as does the boiled solution.

One of the most characteristic properties of the digested and altered acetate of alumina is its loss of the power of acting as a mordant. The ordinary acetate, as is well known, forms a yellow opaque precipitate with decoction of quercitron. That which has been thoroughly digested is merely coagulated by that decoction,—the colour of which is but little altered, and the coagulum is translucent. The same effect is produced with decoctions of logwood, brazil-wood, &c.

A quantity of the solution of hydrate of alumina was evaporated to dryness at the heat of boiling water. After being pulverised and again submitted to the same heat, it was moistened with sulphuric acid as before described, and heated to whiteness. It lost 25.67 per cent. of its weight.

*Hydrate from the Insoluble Binacetate.*—It has been already mentioned that when the precipitated binacetate is kept for an hour or two in 200 parts of boiling water, it is changed into the soluble binacetate. It must be constantly agitated during that time. The substance so produced may be converted, like the original solution into the peculiar bihydrate. Thirty to thirty-six hours digestion is sufficient to complete the change, for after that time the solution has no longer any taste of alum, and the power of coagulating with acids does not increase.

\* Hence, in preparing the binacetate of alumina which is to be used in obtaining the bihydrate, it is better to employ an excess of acetate of baryta, than to leave in the solution the slightest trace of sulphuric acid.



6.93 grs. of this hydrate, which had been dried at a steam heat, were moistened with sulphuric acid, which was then gradually expelled by a spirit-lamp, and the residue, when kept forty minutes at a white heat, left 5.20 grs. of alumina, indicating 24.97 per cent. of water. The mean of this and the previous experiment gives for the composition of the hydrate—

Alumina, ..	74.68
Water, .....	25.32
	<hr/>
	100.00

A bihydrate requires—

Alumina, .....	74.06
Water, .....	25.94
	<hr/>
	100.00

#### ACETATES OF SESQUIOXIDE OF IRON.

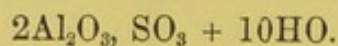
The analogy between the salts of alumina and those of the red oxide of iron, induced me to inquire whether there might not exist allotropic acetates of iron, corresponding with those of alumina.

A solution of sulphate of iron, to which was added half an equivalent of sulphuric acid, was acted upon by strong nitric acid, and the tersulphate so obtained was converted into the teracetate by decomposition with neutral acetate of lead. A binacetate was also produced by decomposing with a mixture of acetate and carbonate of lead. Both solutions had the same intense red colour which is peculiar to the peracetate of iron.

The teracetate of iron, whether concentrated or weak, is little liable to decomposition in the cold. Boiling causes the deposition of a hydrated peroxide, but the decomposition is only partial, and the hydrate difficult to collect.

The binacetate soon begins to let fall an oxide in the cold; and at the heat of boiling water a complete separation takes place. A rich deep-coloured hydrate goes readily down, and the whole of the acetic acid remains in the liquid, which is perfectly colourless. No allotropic acetates of iron, corresponding to those of alumina, appear therefore to exist.

#### BIBASIC SULPHATE OF ALUMINA.



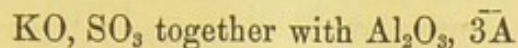
It is well known that when teracetate of alumina is boiled along with sulphate of potash, a gelatinous precipitate is formed, which redissolves when the solution becomes cold. M. Kœchlin-Schouch\* found it to be a subsulphate of alumina.

When alum, dissolved in six times its weight of water, was treated with three equivalents of acetate of lead—enough to decompose its tersulphate of alumina, but not the sulphate of potash, a large propor-

\* Sur le Mordant Rouge, &c. Bulletin de la Société Industrielle de Mulhausen I. 299.



tion (74 per cent.) of the sulphate of potash was found to be carried down by the sulphate of lead formed in the process. To a solution so prepared, the loss was restored by an addition of sulphate of potash, and a mixture formed of—



This solution, diluted so as to contain about 0·3 per cent. of alumina, (from 5 oz. of alum per gallon,) was exposed to heat. At about 90° Fah., the gelatinous precipitate began to form; and after two hours boiling, when scarcely a trace of alumina remained in solution, it was thrown upon a filter of calico, which was kept hot within a steam pan until the filtrate passed through. The collected precipitate was pressed between numerous folds of bleached calico, and brought gradually to the state of a stiff clay occupying not more than  $\frac{1}{40}$  of the volume of the original solution. It was then divided into portions which could conveniently be shaken and thoroughly mixed in bottles with quantities of water equal in all to the original solution. The mixture was filtered as before, at nearly a boiling heat, and pressed between folds of calico; and another repetition having been made of the same process, the precipitate was deprived of every admixture of soluble matter.

A portion being dissolved in nitric acid and filtered, gave an abundant precipitate with nitrate of baryta. Exposed to a white heat, and the residue pulverized and boiled in water, the filtered liquor gave no longer a precipitate with nitrate of baryta, and no indication, or a very doubtful one, of alkali to litmus paper; proving that the sulphuric acid indicated by the first test had been combined, not with potash, but with alumina. Mixed with sulphuric acid the substance gave no smell of acetic acid, and when burned with oxide of copper it yielded no more carbonic acid than could readily be accounted for by the few fibres of cotton wool that were to be distinguished in the liquid. The substance dried into a hard, whitish, semi-transparent matter, easily pulverized.

For analysis 33·93 grains dried at 100° Fahr. were dissolved in muriatic acid, and the solution was exactly decomposed at a boiling heat by 15·25 grains of chloride of barium.\* This indicates 5·867 grains, or 17·29 per cent. of sulphuric acid. 19·715 grains mixed with 16 grains sulphuric acid in a platinum crucible were first heated with a spirit lamp, and then exposed to a white heat. It left 8·51 grains = 43·16 per cent. of alumina.

\* The heat in this case greatly facilitates the arrival at the nearest point of decomposition, particularly by enabling the filter to furnish, immediately, clear portions, from time to time, for testing.

Where nitrate of baryta is employed for decomposing a sulphate, it was observed by Mitscherlich that a quantity of the nitrate was taken down unaltered by the sulphate of baryta. This source of error, which amounts to not less than 5 per cent. of the quantity of sulphuric acid when the experiment is performed in the cold, is entirely obviated at a boiling heat.

Boiling, however, does not render the decomposition of a sulphuric salt by a salt of baryta complete without a considerable excess of the barytic salt, and *vice versa*. The point sought for in the present instance is that at which an equal precipitate is formed on adding an excess of either salt.



Another experiment gave 42.94 per cent. The mean is 43.05. The composition of the substance is therefore—

Sulphuric acid, .....	17.29	SO <sub>3</sub> .....	17.18
Alumina, .....	43.05	2Al <sub>2</sub> O <sub>3</sub> .....	44.16
Water, .....	39.66	10HO .....	38.66
	<hr/>		<hr/>
	100.00		100.00

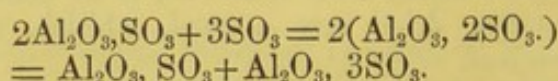
When the solution from alum and sugar of lead was heated before the restoration of its sulphate of potash, a very slight precipitate was formed. The addition of that salt completed the precipitation, but the substance thrown down was more transparent, and dried into a brownish, horny-looking matter. Its analysis, however, gave nearly the same results.

Sulphuric acid, .....	17.23
Alumina, .....	43.51
Water, .....	39.26
	<hr/>
	100.00

But when the solution was boiled for two hours before the addition of the sulphate of potash, a quantity of alumina seems to have gone down with the sub-sulphate, for its analysis gave in 100 parts

Sulphuric acid, .....	13.73
Alumina, .....	50.71

It had an appearance similar to the preceding product. In the moist state the sub-sulphate is soluble in cold acetic acid, as well as in the mineral acids. It dissolves in three atoms of sulphuric acid to form two of the bisulphate, which, on the addition of water is resolved into the tersulphate of alumina, and the insoluble monosulphate.



The same mixture of teracetate of alumina and sulphate of potash, which in a diluted state produces subsulphate of alumina on the application of heat, gives a precipitate of binacetate of alumina when heat is applied to it in a concentrated state. A quantity of sulphate of potash, however, adheres to the insoluble binacetate even after abundant washing.

#### PRECIPITATE FROM ACETATE OF ALUMINA WITH CHLORIDE OF SODIUM.

A solution of teracetate of alumina was produced from 1 pound acetate of lead in 10 pounds water, decomposed by tersulphate of alumina. After purification, common salt was added to it in the proportion of one equivalent to one of tersulphate of alumina. The solution, when heated in the water bath, became of an opaque white from the deposition of a powder so very fine that the mixture passed through the filter almost without change. Neither heat, nor any other application could alter this character. It was left at rest, and after some weeks, the liquid



having become nearly clear, was poured off, and the precipitate, which had contracted into 1-64th of the bulk of the liquid was mixed again with fresh water 11 times its volume. After several weeks more, the precipitate fell again to the bottom of the vessel, when it was mixed with a third portion of fresh water, which again, after four weeks, was decanted, and the precipitate dried in a capsule at 100° Fahr.

On analysis it yielded the following results—I do not give the details of the experiments, not having made out a formula which can be stated in atomic proportions:—

Alumina, .....	44.66
Acetic acid, .....	21.96
Hydrochloric acid, .....	5.51
Water, .....	25.90
	<hr/>
	98.03
Chloride of sodium, .....	1.97
	<hr/>
	100.00

Nitrate of potash forms a precipitate when heated with acetate of alumina, similar in appearance to that from common salt, but it was not particularly examined.

#### CONCLUSIONS.

1. The aluminous solution obtained by decomposing pure tersulphate of alumina with the neutral acetate of lead consists, I believe, of a mixture of binacetate of alumina, with an equivalent of free acetic acid. No true teracetate of alumina appears to exist.

2. When means are taken to evaporate the preceding aluminous solution at a low temperature with sufficient rapidity, a dry substance is obtained, which may be redissolved easily and entirely by water. This is the binacetate of alumina, ( $\text{Al}_2\text{O}_3, 2\text{C}_4\text{H}_3\text{O}_3 + 4\text{HO}$ ), in which the alumina retains all its usual properties.

3. When the first aluminous solution, containing not less than 4 or 5 per cent. of alumina, is left for some days in the cold, a salt is deposited in the form of a white crust, which is an allotropic binacetate of alumina, insoluble in water. Heat effects the same change in the aluminous solution more rapidly, and the new acetate then precipitates as a granular powder. At the boiling temperature the liquid is thus deprived, in about half an hour, of the whole of its alumina, which goes down with two-thirds of the acetic acid; leaving one-third in the liquid.

4. The red acetates of iron treated in a similar manner do not produce corresponding isomeric binacetates. By heating the binacetate of the sesquioxide there is a total separation of acid and base. The facility with which the binacetate is decomposed, even in the cold, furnished the means of freeing the solution of binacetate of alumina of traces of iron which could not otherwise be separated from it.

5. The soluble binacetate of alumina is decomposed by heat, and affords a new and remarkable product. When a dilute solution of that salt is exposed to heat for several days, the whole acetic acid appears



to become free, and the alumina to pass into an allotropic condition, in which, although it remains in solution, it ceases to be capable of acting as a mordant, or entering into any other definite combination. When the acetic acid is expelled by boiling, the alumina, in its altered state, remains alone dissolved in pure water. It is more soluble, however, in acetic acid. The allotropic hydrate of alumina retains two equivalents of water when dried at the heat of boiling water. Its solution is coagulated, more or less powerfully by the mineral, and most of the vegetable acids and their salts; by the alkalies, and by decoctions of dyewoods. The coagulum which is formed by the various acids, is not re-dissolved when they are added in excess. The solid part of the coagulum yields, however, to the continued action of oil of vitriol, especially if assisted by heat, and the result is the ordinary sulphate of alumina. Boiling potash also dissolves it, and changes it into the ordinary terhydrate. Its coagulum with dyewoods has the colour of the infusion, but is translucent and entirely different from the dense opaque lakes which ordinary alumina forms with the same colouring matters.

6. The insoluble binacetate of alumina, when digested in a large quantity of water, is gradually changed into the soluble binacetate; of which a part, however, becomes decomposed during the process into acetic acid, and the allotropic bihydrate of alumina.

7. The precipitate which is formed on the application of heat to a mixed solution of acetate of alumina and sulphate of potash, and which is soluble in cold acetic acid, is a bibasic sulphate of alumina.