PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

Regular Meeting, Dec. 8, 1882.

The meeting was called to order at 8.40 P. M. Prof. A. R. Leeds in the chair.

The minutes of the previous meeting were read and approved.

The board of directors had no report. The treasurer stated that we had a balance of about $200 in the treasury.

The librarian and curator had no report. The committee on endowment fund, stated that the labor of raising the fund was for the present suspended, there being a larger fund in the possession of the society than was needed for the publication of the original papers presented at the present time.

The committee on papers and publications then stated that it had no formal report to present, but trusted that the Society would judge them by the work they had done.

The committee further stated that the November number of journal is in type, and that it will probably be issued the coming week.

The next in order was the election of officers for the ensuing year.

For President.—The three following gentlemen received the greatest number of votes: A. R. Leeds, E. R. Squibb, J. C. Booth.

Prof. Leeds declined to be a candidate, on the ground that it was of the greatest importance to the interests of the Society, as a national body, that the honorary office of President should be filled by a non-resident member.

It was then moved that the Society proceed to vote for the two other gentlemen standing highest on the list. This being seconded was duly carried.

Prof. J. C. Booth receiving the greatest number of votes, was duly elected President.

For Vice Presidents.—The following gentlemen received the greatest number of votes:

1. James H. Stebbins, Jr., } Three Local Vice-Presidents.
2. A. R. Leeds, }
3. C. F. Chandler, }
4. Arno Behr,
5. P. Schweitzer.
6. N. T. Lupton.

Mr. Casamajor then moved that the resolution preventing members who have read papers before the Society, from publishing them through any other source except the Journal of the American Chemical Society, for the space of thirty (30) days, be rescinded. This being seconded, was duly carried.

The following gentlemen were then elected to fill the remaining offices of the Society:

Corresponding Secretary—P. Casamajor.

Recording Secretary—Thomas S. Gladding.

Treasurer—T. O'C. Sloane.

Librarian—G. A. Prochazka.

Curators—William Rupp.

Committee on Papers and Publications—E. Waller, C. A. Doremus, L. H. Friedburg.

Committee on Nominations.

Board of Directors.—It was then moved and seconded that Drs Alsberg and Geyer, be elected Directors, in lieu of C. F. Chandler and James H. Stebbins, Jr.

James H. Stebbins, Jr.,	Geo. A. Prochazka,
A. R. Leeds,	E. Waller,
C. F. Chandler,	M. Alsberg,
P. Casamajor,	H. Morton,
T. S. Gladding,	Wm. E. Geyer,
T. O'C. Sloane,	Wm. M. Habirshaw,

H. Endemann.

Mr. Casamajor then moved that this meeting be declared adjourned till the usual night for converzatione. This being seconded was duly carried. After which the meeting adjourned.

James H. Stebbins, Jr.,
Recording Secretary.

At the adjourned meeting, held Dec. 15th, 1882, no quorum being present, official business could not be transacted. A paper on the manufacture of tartaric acid was read by Dr. L. H. Friedburg. Mr. Percy Newman was nominated by E. Waller, Jas. H. Stebbins, Jr., and A. H. Elliott.

ON THE MANUFACTURE OF TARTARIC ACID.

By L. H. Friedburg, Ph. D.

In this country tartaric acid is hardly manufactured for its own sake but its preparation is unavoidably attached to the manufacture of cream of tartar. Here the starting points for tartaric acid are sablons, waste liquids and residues of different kinds, which render an analytical control troublesome, so that partly because of this, partly because of great dilution, the raw material is treated more or less empirically, after the known and often described methods * with chalk and chloride of calcium or gypsum.

Abroad, the manufacture of tartaric acid is not everywhere a mere appendix to cream of tartar manufacturing, but forms an independent branch of manufacture. In such cases the raw material consists either of argols or of dry sablons or of lees. A careful analytical test has to be made before treating either of these mother substances, and the manufacture has to be carried on with the greatest care in order to avoid loss.

Until very recently these tartaric acid factories worked generally after the old plan as indicated above, viz : treatment with chalk and chloride of calcium or gypsum.

But it has to be recorded, and I will briefly do so in the following pages that a very neat and new, patented process, which according to my own experience is commendable, is now also in use.

This process is based on the decomposition of the mother substances, as named above, by slaked lime instead of chalk.

This preparation has been hidden in the European patents ‡ § under the heading, "Methods of obtaining the potassium in the form of hydrate, while making tartaric acid out of argols."

This heading is practically speaking untrue, because, as we shall see later on, the potassium is not finally gained as hydrate, though this is in the course of treatment formed and then transformed into sulphate or chloride.

The chemical process, which takes place in decomposing the bitartrate of potash in any mother substance into tartrate of lime,

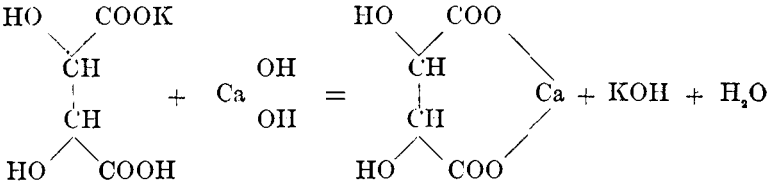
*Bericht ueber die Entwickelung der chem. Industrie; A. W. Hofmann. Vol. II, page 418, etc.

†Journal of the Society of Arts ; Robert Warington, Vol. xxiv, No. 1217, page 366, etc.

‡Die chemische Industrie ; Dr. Emil Jacobsen ; 1879, pages 86 and 238.

§Berichte d. deutschen chemischen Gesellschaft ; 1879, page 1366.

by means of slaked lime, is very simply conveyed through the following equation :



The practical difficulty which stood for so long a time in the way of realizing this decomposition for manufacturing purposes, was the difficulty of making the products of decomposition easily filterable. This the patentees have really overcome, and the method of working is smooth and goes like clock work. Slaked lime, freed from coarse pieces is taken in necessary quantities and a milk, not too thin prepared therefrom. This is heated to boiling, and argols, etc., in necessary quantity, are very gradually and in a state of finest powder, introduced into the boiling mass. The charging finished, boiling has to continue for two hours, the condensing steam being enough to keep the mixture in a concentrated form. Hydrate of potash and neutral tartrate of lime are formed in this way. The nitrogenous organic impurities of the raw materials are by the combined action on them, of lime and hydrate of potash, decomposed so as to form ammonia gas which is volatilized with the steam.

Boiling done, which takes place in an iron tank, the mixture is diluted by enough cold water and then the potash is neutralized by either muriatic or sulphuric acid. The process is finished with the help of litmus paper. Here the ammoniacal exhalations are to be considered, so as not to disturb the reaction.

The decomposition as described above takes place under constant stirring by means of an iron stirrer run by machinery.

After the formation of either chloride or sulphate of potash, the mass is still more diluted with cold water and stands best over night, stirring going on continuously, filtration then taking place the next morning. Here filter presses are used to great advantage. It is advisable not to use too high pressure, so as to get a soft cake, which can more easily be washed out, in order to get rid of the mineral potash salts. These latter are either boiled down, as long as the strength of the solution makes it pay, or they might* be treated with chloride of lime [bleaching powder] and thus trans-

*In case chloride of potassium was formed.

formed into chlorate of potash, which I advise manufacturers to try.

The cakes of brown tartrate of lime, which begin to exhale putrid odors by standing too long in a warm place, (in summer time six hours standing often will show this result) have speedily to be decomposed by sulphuric acid. This decomposition takes place in a wooden, lead lined tank, with heavy wooden stirrer moved by steam. The decomposition takes place in the cold and its completion is determined easily by methyl violet test paper.* No analysis has to be made here, if good paper is at hand, which allows one to guide the reaction so as to get the necessary or the excess of sulphuric acid wished for.

The brown solution of tartaric acid is filtered through filter-presses into wooden receivers.

It is not advisable to evaporate this acid down to the point of crystallization, because it contains impurities enough to spoil the mother liquors at a too early stage. If the course of manufacturing demands a readier transformation of raw material into money, this crude acid solution might be concentrated in the leaden pans to the right concentration for crystallizing or for precipitation by the stirring process, which we shall deal with on another page.

It is preferable to reprecipitate this acid as tartrate of lime, finishing the reaction with chalk and using litmus test paper.

The tartrate of lime thus obtained is filtered on a vacuum filter or by centrifugal power. Of course washing takes place, though slightly. This tartrate of lime is crystalline, light greenish-yellow, keeps perfectly well for any length of time required without decomposing.

It is decomposed in an apparatus similar to the one used for decomposition of the first brown tartrate of lime, by sulphuric acid, in the cold and the reaction finished with the aid of methyl violet test paper. The filtration of the very white gypsum thus obtained cannot be done through filter presses but has to take place on a vacuum filter, very thorough washing being required.

The tartaric acid solution thus obtained ought to stand between 12° and 14° Bé. It is ejected into the lead pans, evaporated at 80° C to the necessary density, by which dissolved gypsum is precipitated, run into the crystallizing boxes and let stand for crystallization. The crop of brown crystals is redissolved to a liquid of the

*Journal of the American Chemical Society; T. O'Connor Sloane, Vol. IV, Nos. 1-4, page 31, etc.

density 25° Bé and treated with bone black, which has been purified by muriatic acid, (so as not to leave a trace of phosphates,) at a medium temperature and under stirring.

The discolored liquid is run through a filter-press and thence into special lead pans. It is evaporated down to about 39° to 40° Be and run into lead boxes for crystallization.

The crystallization being a comparatively slow process, this liquid may be run into a proper tank with stirrer, stirred for several hours, thus yielding a crop of small crystals right away.

Either crystals are washed and dried in centrifugals, by using steam for washing.

The liquid running off from the first crystallization yields after evaporation another crop of white crystals. Then it becomes a brown mother liquor.

The mother liquors of the brown crystals can, under careful attention, be carried along through the sixth or seventh crystallization. Then the predomination of sulphuric acid and impurities does not allow further crystallization.

The mother liquors at that stage are diluted to a proper density, the greater part of the sulphuric acid removed by addition of slacked lime milk and the filtered liquid has then principally to be freed from iron salts and from phosphate of alumina.

The iron is easily expelled by taking care to keep it in the form corresponding to the protoxide, the presence of the phosphate of alumina makes it necessary to treat the liquids boiling with milk of lime, thus precipitating phosphate of alumina and forming an acid tartrate of lime, which is soluble. It has to be filtered hot and is decomposed by an addition of sulphuric acid, thus yielding very pure solutions of tartaric acid.

If a transformation of the acid thus gained, into bitartrate of potash should be wished for, which hardly would be prudent, the simplest way of arriving at this end would be the following :

The solution is divided into two equal parts, one-half *saturated* by caustic or carbonate of potash, so as to form a neutral tartrate of potash and then the other half added for the precipitation of the bitartrate.

New York, 15th December, 1882.

ON THE ACTION OF PHTHALIC ANHYDRIDE UPON GALLIC ACID.

By George A. Prochazka.

Mr. Stebbins* appears to have entirely misunderstood the gist of my remarks at the November meeting.

More than a year ago I experimented upon the action of phthalic anhydride upon gallic acid, with results similar to those of Mr. Stebbins. My object was to find a more economical method for the production on a large scale of gallein (and coerulëin) than given in the books. The substitution of gallic acid in place of pyrogallol readily suggested itself. Subsequently I found in a snyopsis by Ch. Lauth of the report on dye-stuffs, of the jury of the Paris International Exhibition of 1878, (Monit. Teint. 1878) that gallein was prepared by heating together phthalic anhydride and pyrogallic or *gallic* acid. My own results had been anticipated. My own results were never published. In making my remarks at the November meeting it was not my object to substitute a doubtful claim of originality, substantiated by experiments hid under the bushel, in place of Mr. Stebbins, but to call attention to the fact that he had been anticipated as early at least as 1878 by those European manufacturers, who had utilized the reaction in question. Mr. Stebbins does not appear to have been aware of this fact, which through a small notice had become known only to the initiated few. The very careful and detailed experiments (much more exhaustive than my own) of Mr. Stebbins, merit the widest attention, and the public owes to him a debt of gratitude for their publication.

*Journal Am. Chem. Soc. IV., 248.

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