STANDARDIZATION OF VOLUMETRIC ACID AND ALKALI SOLUTIONS.*

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The U. S. P. VIII recognizes but one substance for the standardization of volumetric acid and alkali solutions—potassium bitartrate. This salt after being purified, as per directions of the Pharmacopœia, is employed to standardize the alkali, which then serves to standardize the acid.

This method is a very good one and leaves little to be desired with reference to accuracy.

Notwithstanding this fact, we constantly hear complaints concerning the method, mainly due to the time and trouble necessary to properly prepare the bitartrate, with the result that many students and pharmacists are inclined to employ some other method of standardization.

To determine the relative accuracy of other commonly used methods as compared with the pharmacopœial one the following work was undertaken.

The Bitartrate Method was employed by following the directions on page 532 of U. S. P. VIII for the purification of the salt and the method for standardizing V.S. KOH on page 552.

The H_2SO_4 V.S. was standardized by measuring 25 cc. into a 100 cc. Erlenmeyer flask and titrating in boiling solution with the above KOH solution employing phenolphthalein indicator. The factor for the H_2SO_4V.S. was found to be 1.0391.

The Ammonium Sulphate method was carried out by accurately weighing 2 small beakers, adding 25 cc. of the H_2SO_4 V. S., an excess of redistilled ammonia water, evaporating to dryness on a water bath, then heating in an air oven at 110 C° for periods of 20 minutes until the weight became constant, when the weight of ammonium sulphate was calculated to sulphuric acid. To avoid contamination with silica, at the time of making the test, strong ammonia was placed in a test tube and the gas distilled into distilled water.

The factor for the H_2SO_4 V.S. was found to be 1.0385.

The Sodium Carbonate Method was carried out by employing Merck's reagent anhydrous sodium carbonate, heating in a platinum dish a few minutes, quickly transferring to a stoppered weighing bottle and after cooling weighing off a portion for analysis, which was dissolved in water and titrated against the V.S.H_2SO_4 using methyl orange indicator.

The factor for the H_2SO_4 V.S. was found to be 1.0409.

The Oxalic Acid Method was carried out by weighing off portions of Merck's reagent oxalic acid crystals, dissolving in water and titrating against the potassium hydrate solution, using phenolphthalein indicator.

This potassium hydrate solution was then employed to standardize the acid

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using 25 cc. of the latter, titrating in hot solution, employing phenolphthalein indicator.

The factor of the H_2SO_4 V.S. was found to be 1.0398.

The Silver Chloride Method was carried out by taking 25 cc. of HCl V.S., about 200 cc. distilled water, adding an excess of one drop of silver nitrate solution, heating to boiling, allowing to stand until granular and filtering and washing on Gooch Crucibles, which after drying on the hot plate were weighed. From the weight of silver chloride the quantity of HCl in the solution was calculated.

This V.S. HCl was then run against the KOH solution, titrating in hot solution and this KOH run against the V.S. H_2SO_4 in hot solution, phenolphthalein being the indicator employed.

The factor for the H_2SO_4 V.S. was found to be 1.0367.

The Barium Sulphate Method was carried out by precipitating the H_2SO_4 in 25 cc. quantities of the V.S. H_2SO_4, the barium chloride solution which was boiling being added from a pipette drop by drop, to the boiling H_2SO_4 solution which had been diluted with water.

The material was allowed to stand on the hot plate until the solution became clear and then filtered through Gooch Crucibles, which after being washed, dried and heated were weighed as barium sulphate from which the quantity of H_2SO_4 was calculated.

The factor for the H_2SO_4 V.S. was found to be 1.0440.

All the determinations were run in duplicate and yielded remarkably close checks.

The following table shows at a glance the results by the various methods:

Potassium Bitartrate Method	(U. S. P. VIII)	Factor 1.0391
Ammonium Sulphate	"	..... 1.0385
Sodium Carbonate	"	..... 1.0409
Oxalic Acid	"	..... 1.0398
Silver Chloride	"	..... 1.0367
Barium Sulphate	"	..... 1.0440

$$6 \mid 6.2390$$

$$1.0398$$

With the exception of the barium sulphate figure the results agree closely, but notwithstanding the fact that this is a trifle higher it has been included in making up the average to derive the factor.

Numerous determinations made on sulphuric acid solutions always yielded a higher result by this method than by others.

This sulphuric acid was originally standardized on June 4, 1912, and the factor which was the average of all the methods was 1.0388.

Considerable fungus growth being present in the solution the work detailed in this paper was begun and carried out on November 12, 1913, the factor which was the average of all the methods being as above noted 1.0398 the difference between the two figures after about 17 months being 1.0398—1.0388=.001, which proved that the age of the solution and the growth in it had not affected it.

As pointed out by Clark, (Proc. Amer. Pharm. Ass'n, 1910, page 978) a volu-

metric solution whose factor has not changed more than four points in the third decimal place in titrating 25 cc. of one solution against 25 cc. of another solution is regarded as not having changed its strength.

These results indicate that any of the above methods may be used with perfect safety for the standardization of volumetric acid and alkali solutions although our personal preference is for the ammonium sulphate, sodium carbonate, and potassium bitartrate methods.

I would take this opportunity to acknowledge my indebtedness to my assistant, Dr. I. Swartz, for his work in carrying out some of the duplicate determinations.

SOME FACTS AND DEMONSTRATIONS ON LLOYD'S REAGENT AND ALCRESTA ALKALOIDS.*

DR. GUSTAV REHFELD, ST. LOUIS.

I am very glad that you afforded me an opportunity to address you to-night and I hope to be able to interest you.

I feel that Lloyd's Reagent and the Alcresta Alkaloids will prove to be, in the near future, a matter of great importance in the fields of exact and applied sciences.

It has occurred many a time in the past, that apparently trifling circumstances were instrumental in revolutionizing the fields of human endeavor. Every one of you is familiar with the cause that gave the first impulse to the evolution of the laws of gravity, how the swinging of a candelabrum affected the science of physics, how an accidental arrangement of lenses fostered the invention of the telescope and the microscope, how the insignificant popping of the lid of a tea kettle brought about the wonderful development of steam power. It is unnecessary to enumerate any further; you know that most discoveries had their starting point in just such every day occurrences; millions and millions of times they happened and millions and millions of times they passed unrecognized until some one somewhere caught the revelation and thereby enriched human knowledge generally, opening up new view points and thus engraved his name indelibly on the pages of historical record.

I feel that the discovery of Dr. John Uri Lloyd is destined to do just such a thing, to change our viewpoints considerably, to make our knowledge more exact in a field that is not altogether easy of treatment and which will, thereby, benefit mankind generally, as it affects chemistry, medicine and pharmacy.

I wish to say to you though, that some time will elapse before the far reaching results, which we expect to get, will be realized. However, enough facts have been established to leave no doubt whatever, that this discovery will affect chemical research in alkaloids; that, already, it has given the medical profession a most valuable addition in materia medica, and to the pharmaceutical profession a means of rendering intensely bitter substances absolutely tasteless.

A few years ago Dr. John Uri Lloyd, of Cincinnati, discovered that a very

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