spirit, and lastly wood oils, which contain large quantities of furfurol and no acetone. The wood oils are separated by passing the last portions of distillate into water. The rectified wood spirit contains about 80 % of methyl acetate, and when saponified gives a very pure methyl alcohol.

One cord of well-seasoned wood will afford 1200 to 1400 lbs. of liquid products. A cord of oak yields 60 to 70 lbs.of glacial acetic acid.

Dr. Squibb further remarked that there was no process commercially practicable for obtaining pure acetic acid from pyroligneous acid, or acetate of lime, but only from acetate of soda. Mr. Parker remarked that he had tried a process for making acetic acid by heating wood fibre with steam, under a pressure of 60 lbs. at.275° C.and confirmed Dr. Squibb's observations on the deterioration of the woody fibre, and also the impracticability of making pure acetic acid from pyroligneous acid.

Dr. Grothe remarked that in distilling acetone with bleaching powder, he had obtained chloroform simultaneously with calcium acetate.

Dr. Doremus brought up the question of giving reprints of papers to members. After some discussion Mr. Casamajor moved that "those members who desire reprints of their papers shall notify the committee on papers and publication, who may make arrangements to have them supplied at the expense of the authors." Carried.

After which the society adjourned to September 1st, 1882.

Arthur H. Elliott,
Rec. Sec. pro tem.

XVI. ACETATE OF LIME—ITS MANUFACTURE AND ANALYSIS.

By Stillwell & Gladding.

Acetic acid and its compounds hold a very important place in the arts and manufactures. They are extensively used in many branches of industry, as, calico-printing and dyeing, in the treatment of gums, in the manufacture of paints and varnishes, etc. Acetic acid is well known in the form of vinegar.

It may be formed in three ways: *(a)* by acetous fermentation, chiefly used for the making of vinegar; *(b)* by destructive distillation of wood, yielding crude pyroligneous acid and by further treatment, acetates of iron, lime, lead, soda, etc.; (*c*) by the distillation of various metallic acetates with sulphuric or hydrochloric acid, yielding the pure acid of commerce.

We will confine ourselves for the present to the second method of obtaining this acid—by the destructive distillation of wood:

"Wood consists essentially of woody fibre or cellulose, a small amount of mineral matter and sap and a variable amount of hygroscopic water. The woody fibre constitutes about 95 per cent. of dry wood, and is composed of, in 100 parts: carbon, 44.45; hydrogen, 6.17; oxygen, 49.38. The sap consists chiefly of water, holding organic and inorganic matters partly in solution and partly suspended. The inorganic constituents of the sap (the ash left after the incineration of the wood) are the same in all kinds of wood. In practice it is assumed that wood leaves about 1 per cent. of ash; but there is a difference for certain portions of the tree, the trunk yielding about 1.23 per cent. of ash, the branches and knotty parts 1.34 and 1.54, and the roots 2.27 parts of ash respectively.

Wood dried at 266° Fah.—at which temperature all the hygroscopic water is driven off—is composed of:

50 parts of carbon (inclusive of one part ash).
50 " chemically combined water."

In the process of dry distillation the hygroscopic moisture of the wood is first driven off. As the heat increases, acetic acid and water are formed by the decomposition of the wood, then follow the tar and volatile oils and at a still greater heat carbonic acid, carbonic oxide and marsh gas appear. The remainder of the carbon is left in the retort in the form of charcoal.

The following table from Wagner's Chemical Technology, shows at a glance the numerous and complex products which are formed during the destructive distillation of wood:

Wood.	1. Real wood. 2. Hygroscopic water.	*a.* Illuminating gas.	Acetylen. Carbonic oxide. Elayl. Carbonic acid. Benzol. Marsh gas. Naphthalin. (?) Hydrogen.
		b. Tar.	Benzol. Oxyphenic acid. Naphthalin.(?) Cresylic acid. Paraffin. Phlorylic acid. Reten. Empyreumatic resins. Carbolic acid. Creosote.
		c. Pyroligneous acid	Acetic acid. Aceton. Propionic acid. Wood spirit.
		d. Wood charcoal.	

Woods differ greatly in the amount of water which is present in them. The following table by Schuebler and Hartig shows the amount of water present in some freshly cut woods:

Hornbeam	contains	18.6	per cent.	of water.
Willow,	"	26.0	" "	"
Ash,	"	28.7	" "	"
Birch,	"	30.8	" "	"
Oak,	"	34.7	" "	"
Pine,	"	39.7	" "	"
Red Beech,	"	39.7	" "	"
Elm,	"	44.5	" "	"
Larch,	"	48.6	" "	"
White Poplar,	"	50.6	" "	"

Different woods yield different results, hence the wood selected must be chosen with reference to the nature of the products desired. If the charcoal is to be used for making powder, alder, willow and dogwood are used ; if for heating, oak and beech ; if for pyroligneous acid alone, birch, thorn, and apple. Oak holds the first place and beech the second when we take into account all the products obtained—charcoal, acetic acid, naphtha, and tar.

The following table, by Stolze, shows the various products obtained from different woods :

336 lbs. wood.	Charcoal.	Acid Liquor.	Sp. Gr.	Dry Acetate of Lime.
Beech	84	180	1.029	25
Walnut	72	150	1.018	14½
Birch, cut 3 years	70	120	1.031	13
Oak	91	190	1.022	24
Ash	90	190	1.024	22
Apple	70	200	1.017	18
Wych Elm	70	180	1.018	16
Maple	77	145	1.018	20

The average production of three works in Paris gave from 4,000 pounds of beech and oak : Charcoal, 1,014 lbs. ; Acid liquor, 2,335 lbs., sp. gr., 1.027,=dry acetate of lime, 416 lbs.; tar, 330 lbs.

When wood is distilled in close cylinders, the charcoal obtained is about 25 per cent. of the weight of the harder woods, as oak and beech, or about three-fifths of the total carbon present ; the liquid part is about 53 per cent., containing about one-fifth of the carbon; the gases contain the remaining one-fifth of the carbon. Of the liquid products, acetic acid equals about 3 or 4 per cent of the harder woods, the naphtha about 1 per cent. and the tar 7½ per cent.

The time occupied in firing a charge is from twelve to sixteen hours; the latter time is preferable, since a slow carbonization yields a larger product. The amount of charcoal obtained depends largely on the length of time occupied. The following table, by Karsten, gives the results of some experiments upon air-dried timber :

SPECIES OF WOOD USED.	CHARCOAL.	
	By quick charging.	By slow charging.
Young oak	16.54	25.60
Old "	15.91	25.71
Young beech	13.12	25.22
Old "	13.65	26.45
Young alder	14.45	25.65
Old "	15.30	25.65
Young birch	13.05	25.05
Old "	12.20	24.70
Young deal	14.25	25.25
Old "	14.05	25.00
Young fir	16.22	24.72
Old "	15.35	24.75
Young pine	15.52	26.07
Old "	13.75	25.95
Lime	13.30	24.60

According to an English authority, the yield which should be obtained can be approximately calculated from the following data. One ton of wood, delivered at the works, will yield 2½ to 3 cwt. of bark, and, if fairly dry, 5 cwt. best charcoal, 1½ to 2 cwt. of best brown acetate, or 1½ of grey, 1¾ to 2 gallons of "miscible" naphtha at 60° over proof, or 2½ gallons of "solvent" naphtha at 45.°

Many improvements in the methods for wood distillation have been preposed from time to time, especially with reference to the utilization of wood in a finely divided state, such as spent residues from tanning and dyeing operations, saw-dust, &c. With properly constructed apparatus, the yield of acid is nearly as large as with fresh wood, but the charcoal, owing to its finely divided state, is almost worthless.

The use of hydrochloric acid for making a superior quality of acetate of lime is as follows: A clear solution of brown acetate is made and evaporated to one-half its bulk, removing the impurities as they rise to the surface. When evaporated, hydrochloric acid is added until litmus paper is just reddened. The resinous products are thus separated and the volatile bodies driven off on further evaporation. About 5lbs. hydrochloric acid to every 41 gallons of liquid, after the naphtha has been expelled, are usually sufficient. The solution is then boiled down to dryness, giving a brown acetate of a remarkably pure quality.

This use of hydrochloric acid is not general in America. It has this disadvantage, that it leaves in the finished product a certain amount of chloride of calcium. When an acetate thus made is distilled with sulphuric acid to obtain the acetic acid the chlorine is carried over as hydrochloric acid and condenses with the acetic acid. We have tested many samples of American make of acetate of lime and find but very few which contain any chlorine at all: in every case where it existed in more than traces, the acetate had been made from shell-lime and hence the chlorine was present in the form of salt.

The lime used for making acetate should be as pure as possible and free from magnesia and silica. Both retard the slaking. The presence of 1 to 2 per cent. of magnesia is not specially injurious; at 10 per cent. the lime is perceptibly deteriorated and at 20 per cent. and upward it is too poor for use. Lime made from oyster shells is sometimes used. It contains more or less of salt. which is therefore carried through the various operations of manufacture and appears in the finished product. The salt may vary in amount from one per cent. to six or even more in the acetate of lime. Its presence is injurious when *sulphuric acid is used for distillation* as previously mentioned under the use of hydrochloric acid in the manufacture. It is also liable to cause serious mistakes in the chemical analysis of acetate of lime when analyzed by distillation, unless specially guarded against. We shall refer to this later on when we come to speak of methods of analysis. A sample of stone lime which gave an acetate of low strength, was found to contain 5.49 per cent. of magnesia; another lime giving an acetate of a better quality, was found to contain but 1.35 per cent. of magnesia.

Having thus glanced at the materials which enter into the manufacture of this important article of commerce, we are prepared to discuss the different kinds of acetate of lime now manufactured and to call special attention to a few points which have much to do with its successful production.

Some years ago, three kinds of acetate of lime were made, the black, the brown and the grey: but with improved methods of manufacture the black acetate has disappeared and but two kinds, the brown and the grey are now in use. The black acetate contained on an average about ten per cent. less acetate of lime than the brown.

In order to show the composition of representative samples of the brown and the grey acetates of lime, we give below two analyses, each of which is an average of twenty-six samples, taken at random from samples analyzed by us during the last three months. The reaction of both samples is alkaline, from the presence of caustic lime.

	Brown.	Grey.
Moisture, at 100° C.,	6.40	4.07
Insoluble, sand, &c.,	1.13	0.87
*Lime	23.95	29.29
Magnesia	0.62	0.15
†Acetic acid, anhydrous,	42.42	53.34
Tar, coloring matter, &c	25.48	12.28
	100.00	100.00

*By *English Commercial Test.*		
Equiv. to acetate of lime	67.37	82.64
†By *Distillation.*		
Equiv. to Glacial acid	49.51	62.32
" " Acetate of lime	65.19	82.05

The following tables of analyses include all the samples which have been analyzed by us during the last two years, ending April 30, 1882. The average of the brown acetates would be slightly increased if it included samples of a few makes of high-grade brown acetate ; but as these lots are usually sold without analysis they do not enter into the average given here. The analyses given below are a fair average of the general class of brown and grey acetates of lime now in the market.

Brown Acetate of Lime ;—*By English Commercial Test.*

No. of samples.	Moisture at 100° C.	Acetate of Lime.	Acetate of Lime (on dry basis.)
179	7.13	67.53	72.71

Grey Acetate of Lime ;—*By English Commercial Test.*

No. of samples.	Moisture at 100° C.	Acetate of Lime.	Acetate of Lime (on dry basis.)
84	5.33	81.01	85.57

Grey Acetate of Lime ;—*By Distillation.*

No. of samples.	Moisture at 100° C.	Glacial Acid.	Acetate of Lime.	Glacial Acid (on dry basis.)	Acetate of Lime. (on dry basis)
22	6.22	62.60	82.42	66.75	87.88

This table of grey acetates, analyzed by distillation, is composed of exceptionally high-grade samples. They are very light in color, containing but little tar and yielding an amount of acetate of lime above the general average of the grey limes quoted above. By the "English Commercial Test" the average of these twenty-two samples would show about 89.00 per cent. acetate of lime on dry basis.

The presence of *chlorine*,—either as *chloride of calcium* from the use of hydrochloric acid in the manufacture, or as *chloride of sodium* from the use of shell-lime——is not usual in American acetates of lime. Out of twelve samples, taken at random from nearly sixty samples analyzed by us during the last three months, ten samples showed no trace of chlorine, one contained a trace only and one had about two per cent. of chlorine. Both the latter samples were made from shell-lime.

In order to make an acetate of lime of as high a grade as possible, three results should be sought :

First : The removal of the *tar*.

Second : The finished product should contain as little *water* as possible.

Third : An undue *elevation of temperature* during the drying of the acetate should be most carefully avoided.

In an article which contains so large a percentage of a valuable ingredient as does acetate of lime, the presence of even one per cent. of impurity causes a loss of 0 7 to 0.9 per cent. acetate of lime ; that is, the one per cent. of useless matter, be it insoluble matter, tar or water, takes the place which might be occupied by valuable acetate of lime.

For example : Two lots of brown acetate of lime are taken, each containing originally 9 per cent. of water and 66 per cent. of acetate of lime. One lot remains unchanged ; the second, by exposure, dries so that it contains 6 per cent. of water. The acetate of lime is therefore raised to 68.17 per cent. The total weight of the second lot would be less than it was at first, but the amount of acetate of lime in one hundred pounds, that is, stated as a percentage would be raised to 68.17. Both lots taken each as a whole would contain exactly the same total amount of acetate of lime. One ton of 2,000 lbs. weighed out from the first lot, which remained unchanged, would contain 1320 lbs. acetate of lime ; but one ton of 2,000 lbs. weighed out from the second lot, which had dried, would contain 1363.4 lbs. acetate of lime.

So of two lots of grey acetate of lime, each containing at first 9 per cent. water and 80 per cent. acetate of lime. By drying to 6 per cent. water the second lot would contain 82.63 per cent. acetate of lime in each one hundred pounds of the dried-out acetate.

Since acetate of lime when dry rapidly absorbs moisture, a sample containing only 2 or 3 per cent. of water might absorb as much more by exposure in a damp place, and the amount of acetate of lime would be proportionally diminished when reckoned into percentage on one hundred pounds. The total weight of the lot would be increased, but the actual amount of acetate of lime in each one hundred pounds of the dampened lot would be less than in one hundred pounds of the lot when first made.

This brings us to the third requisite needed for a high-grade article, the *Drying of the acetate of lime.* It is here that care is indeed needed. A rise of temperature of only 50° Fah. above the safety point begins at once to decompose the acetate of lime and the product rapidly loses in strength with further increase of temperature. We have been unable to find any special reference to this important point in the manufacture of this article. It is indeed stated in books that great care is needed in the drying and that the acetate is easily injured by heat, being decomposed at about 460° Fah. But we have met with no record showing the rate of decomposition produced by length of time or increase of temperature. We have made a long series of experiments on this point, because of its importance, in connection especially, with the manufacture of acetate of lime. We sought to ascertain the temperature at which the decomposition begins, and also to see what influence, if any, the impurities possess in hastening such decomposition. We used for this purpose a so-called chemically pure, white acetate of lime,—which had lost by efflorescence some of its water of crystallization, a commercial brown acetate of lime—the average of twenty-six samples, the full analysis of which has been already given, and a commercial grey acetate. An analysis, by distillation, was first made. Then weighed quantities of the samples were submitted to heat for different lengths of time as detailed below, and the residues were then distilled and the acetic acid determined. The results are calculated to a dry basis for better comparison.

A preliminary trial with a sample of acetate of soda, showed that it was not at all decomposed by an exposure for one hour at 550° Fah.

ACETATE OF LIME—PURE.

	Glacial Acid.	Acetate of Lime.
Sample originally contained,	74.53%	98.13%
Heated for 1 hour, at 500° Fah.	74.35	97.89
" " " " 550° "	73.32	96.54
" " " " 600° "	72.08	94.90

The acetate after heating at the high temperatures above given, was strongly alkaline, from caustic lime. At 500° Fah. no change took place in the color; at 550° Fah. it was in places very slightly browned; at 600° Fah. it was but little darker in color than at 550° Fah. Chemically pure acetate of lime contains 75.95 per cent. of glacial acid, on a dry basis.

	BROWN ACETATE.		GREY ACETATE.	
	Glacial Acid. per cent.	Acetate of Lime. per cent.	Glacial Acid. per cent.	Acetate of Lime. per cent.
Sample originally contained,	52.89	69.64	67.09	88.33
Heated for ½ hour, at 400° Fah.,	52.77	69.48	67.09	88.33
" " " " 450° "	50.15	66.03	66.17	87.12
" " " " 500° "	50.15	66.03	64.86	85.40
" " " " 550° "	48.83	64.29	63.40	83.47
Heated for 1 hour, at 400° Fah.,	51.01	67.16	67.09	88.33
" " " " 450° "	50.16	66.04	65.89	86.75
" " " " 500° "	49.65	65.37	64.09	84.38
" " " " 550° "	48.83	64.29	63.09	83.07
" " " " 600° "	——	——	61.94	81.55

We have here, we think, a clue to at least one of the causes which sometimes yield an acetate of lime far lower in strength than was expected by the manufacturer. We see by the above table that the greater part of the loss occurs during the first half hour of exposure at 500° Fah., that it is three times as great in the brown acetate as in the grey, and that for every additional 50° Fah., the loss will average about 1.40 per cent. of acetate of lime. In a substance so easily decomposed by a high temperature as is acetate of lime, we cannot expect that the results obtained will be in exact ratio to the increase of time or temperature. The figures are given just as obtained from the analyses. Several of them, over 500° Fah. are the results of duplicates, the greatest difference being one half of one per cent. for an hour's heating.

The limit of safety is 400° Fah., for the grey acetate, and 400° Fah. for one half an hour for the brown acetate. This temperature will brown white paper by an exposure of fifteen or twenty minutes. An increase of fifty degrees up to 450° Fah., would not be readily noticed by the operative, but its results would surely be found in the poorer quality of the article produced.

When brown or grey acetates of lime are distilled in a current of steam the distillate is always more or less colored. But after exposure to a temperature of 450–500° Fah. for half an hour or so, the distillate is perfectly colorless. The tarry matters are no longer carried over, but this advantage is gained at a loss of a certain part of the acetate by decomposition, as shown above.

Many samples of acetate we analyze contain pieces or crusts which appear to have been melted or partly burned; some lots contain a large amount of such pieces and their presence must diminish to a certain extent the percentage of real acetate present. A sample of the grey acetate referred to in the table above, was heated for an instant until partly melted and the glacial acid was found to be 65.46 per cent. a reduction of 1.63 per cent., equivalent to 2.14 per cent. acetate of lime.

SAMPLING.

In drawing samples for analysis it is important to obtain a fair average of the lot. One bag in every ten is the usual number sampled; if the lot is small, one bag in every five or six is sampled, care being taken to obtain a fair proportion of both large and small pieces. The samples are taken from the bags by meams of a large cylindrical tryer and put at once into a rubber bag to protect them from change, either by absorbing or losing moisture. The average sample thus obtained is taken to the laboratory and prepared for analysis by thorough mixing and sifting until the final average is reached. The moisture of the sample, as drawn, is determined and is reported in the certificate of analysis.

The final average prepared for analysis may have lost or gained moisture during the operations of grinding, &c., hence it is necessary to make a correction of the analytical results abtained, in order to report the correct amount of acetate of lime present.

For example: a sample of brown acetate prepared for analysis contained 7.04 per cent. moisture, and 70.33 per cent. acetate of lime. But the sample as drawn from the bags contained 9.74 per cent. water; hence the result of 70.33 per cent. is corrected to 68.28 per cent based as the 9.74 per cent of water actually present in the goods. A sample giving 66.86 per cent. acetate of lime, with

7.00 per cent. of water in the prepared sample was corrected to 67.66 per cent. based on 5.90 per cent. water present in the original sample. Some samples containing large amounts of tar or moisture need a preliminary drying of the sample selected for analysis; hence the correction to be applied is much larger than is usual, where the samples are easily ground and sifted.

It sometimes happens that samples are received for analysis, which come in paper boxes or wrappers. Such a sample will very seldom represent the actual condition of a lot of acetate as shipped from the factory. The paper box or wrapper offers but little obstruction to a loss of moisture by drying. Hence the sample received is very likely to give a much better result than will be obtained when the goods are received, and sampled from the bags for analysis.

The amount of moisture present should always be stated in the certificate of analysis of acetate of lime, because it is an important factor, showing the condition of the goods at the time of sampling. This determination of moisture must be made on the sample as drawn, before it has been subjected to any treatment which will at all affect the amount of moisture present.

METHODS OF ANALYSIS.

1.—*Based on the Amount of Soluble Lime salts present.*

The value of an acetate of lime depends entirely on the amount of glacial acetic acid present. Two methods of analysis are in use at the present time; one determines the amount of lime salts soluble in water, and by calculation the amount of lime so found is converted into acetate of lime, or glacial acid, as the case may be. This method is based on the supposition that all the soluble lime salts present are acetates; but this is not so. Acetates of lime almost invariably contain caustic lime in slight amount, and if the lime has been overheated it is present in still greater quantity. Again, organic salts of lime are always present. With improved methods of manufacture, the amount of these has diminished of late years, and the difference between the amounts of acetate of lime found by distillation of the acetic acid, and that found from the amount of soluble lime is much less than formerly. The better the sample of acetate, the less is the difference between the results given by the two methods.

In the year 1872 (see *American Chemist*, vol. II, p. 324, and vol. III, p. 8), this question of methods of analysis came up. It is unnecessary to state here the objections raised by certain English chemists against the process of distillation, which was advocated by some American chemists, since the objectors to a distillatory process had no good foundation upon which to rest. But as the general custom of the trade was at that time based upon the method of analysis by means of the soluble lime salts present, we have before and since that date used the term "*English Commercial Test*," to designate analyses made in this manner, and so state the results on our reports of analysis. Whenever an analysis is made by distillation it is so stated.

2.—*Based on the Distillation of the Acetic Acid.*

Three acids may be used in the process of distillation—hydrochloric, sulphuric or phosphoric.

a.—When *hydrochloric acid* is used, a part of it is carried over with the acetic acid and must be estimated and a correction made. It possesses these two advantages: that it does not act upon the organic matter present, and that the solution of chloride of calcium will permit of the distillation being carried to a low point without danger of error. The distillation is made in a retort connected with a condenser, and the total acids present in the condensed liquid are estimated, and the proper correction applied for the amount of hydrochloric acid found to be present.

b.—The use of *Sulphuric acid* has three disadvantages.

First.—As it becomes concentrated in the retort during the process of distillation, it acts upon the organic matter present, forming sulphurous acid, which is carried over with the acetic acid and included in the estimation of the total acid power of the distillate. Thus the amount present would be calculated as acetic acid, and the result thereby increased unduly. To prevent such action, recourse is had to distillation in a current of steam. (See American Chemist, vol. vi. p; 294.)

Second.—The sulphate of lime formed by the addition of sulphuric acid to the solution of the acetate of lime, is troublesome on account of the bumping which takes place during the distillation. This is partly prevented by the use of a current of steam.

Third.—When a sample of acetate of lime contains *chloride of calcium* or *chloride of sodium*, the chlorine is carried over in the form of hydrochloric acid and neutralizes its equivalent amount of the standard soda, used to receive the acid distillate. The amount thus distilled must be estimated and the correction made. To prevent this, the chlorine present is precipitated by the addition of sulphate of silver to the solution of acetate before distillation begins.

c. *Phosphoric Acid* is the best acid for use in the process of estimating acetic acid by distillation. It has three advantages.

First.—It does not act on the organic matter.

Second.—During the distillation, the liquid in the retort is not suffered to fall below 15 c.c. in bulk. Under such circumstances, phosphoric acid does not decompose any chlorides of calcium or sodium which may be present in the acetate, thus requiring no addition of sulphate of silver. Only the merest trace of chlorine is carried over, as shown by experiments with a sample of acetate containing nearly 5 per cent. of chloride of sodium and also by trial on a known amount of pure chloride of sodium. This unexpected result merits attention, since in this respect phosphoric acid differs so decidedly from sulphuric acid.

Third.—It forms a perfectly clear solution with the acetate of lime. By the use of a large amount of glacial phosphoric acid, at east five times the weight of acetate of lime taken for analysis, the phosphate of lime first formed is redissolved to a clear solution. The distillation proceeds quietly and uniformly without bumping and the amount of liquid in the retort is easily kept at any desired point. The use of a current of steam is necessary both for ease of manipulation and accuracy of results.

After a thorough trial of hydrochloric and sulphuric acids, we discarded both in favor of phosphoric acid and for some years past have used it to our full satisfaction. It is important that the phosphoric acid used should contain no nitric or other volatile acids, which would increase the results obtained above the truth. Each new lot of phosphoric acid should be examined for such impurities before use. If it be suspected that any phosphoric acid has been carried over during the distillation, it is easily detected in the distillate by the use of molybdate of ammonia.

The process of distillation, if carefully and intelligently done, is very accurate. Duplicates made by this method agree with each other fully as closely as do those made by the estimation of the soluble lime. For example: In January last, a sample of grey acetate was analyzed by distillation in our laboratory, with a result of 78.22 per cent. acetate of lime. Three months later, the same sample was reanalyzed by another man in our laboratory, using fresh standard solutions, and 78.20 per cent. of acetate was obtained. This sample contained 3.16 per cent. of common salt, shell-lime having been used in its manufacture. See also the table of analysis quoted above under "Drying of the Acetate of Lime." With care and experience in this method of analysis by distillation, it is not difficult to obtain duplicates which agree to two tenths of one per cent. of acetate of lime; usually the difference is less than that. A sample of grey acetate analyzed recently, was distilled with sulphuric acid and a duplicate with phosphoric acid. The results agreed exactly; the latter distillation, however requiring less attention than the former, for reasons given above under processes of distillation.

The indicator used for titration is a few drops of a solution of phenol-phthalein—one gramme in 250 c.c. of a mixture of equal parts or water and alcohol. We have found this indicator far preferable to either litmus or cochineal; of course, whichever indicator is used, the same must be used both for the standardizing and the actual analysis.

To show the need of some uniform and accurate method of analysis, we refer to a note in Allen's Commercial Organic Analysis, vol. I. p. 205. He finds from experiments made in his own laboratory on the *same sample* of acetate of lime, that results were obtained varying from 47.4 per cent. to 57.6 per cent. of acetic acid. In our own practice for many years previous to the publication of this book we found differences fully as great between results by distillation and by the various commercial processes; but, as we said before, at the present time the differences are not nearly so great as formerly.

In conclusion we wish to lay strong emphasis upon the facts, that, inasmuch as the process of distillation is the only one which gives the real amount of glacial acid present, and, inasmuch as it is an imitation of the actual manufacturing process for obtaining acetic acid from its acetates, it is the most reliable and should be adopted. This position we have maintained for many years, and we notice that, especially in the case of grey acetate, buyers are more and more insisting on the test by distillation. The price should be based on the unit of glacial acid, just as in fertilizing materials, the prices are based upon the units of phosphoric acid nitrogen and potash.