

any one substance is probably due to the further dissociation of the complex ion, which may even become complete, or to the presence of small quantities of complex ions of different form, or, in concentrated solutions, to the variation in the amount of free solvent available. To this breaking-down of the complex ions is probably due also the variation in the speed of migration of the ions with the dilution. The existence of such complexes also accords with the presence of solvent of crystallization in many salts.

It will be observed that this is simply a modification of the ionic theory and takes nothing away from it. In fact, it appears to condition only those portions of the theory which have hitherto failed to hold.

Work is being continued upon these lines in this laboratory, and will be reported upon as soon as possible.

LABORATORY OF PHYSICAL CHEMISTRY.

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COMPARISON OF METHODS FOR THE ESTIMATION OF SOIL ACIDITY.

BY F. P. VEITCH.

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WITHIN the past year and a half two new methods have been proposed for the estimation of the acidity of soils. These are the sodium chloride method devised by Hopkins, Pettit and Knox,¹ and the lime-water method devised by the writer.²

Considerable work has been done with both these methods during the past year, and many of the data obtained have been incorporated in the report of the referee on soils and presented to the Association of Official Agricultural Chemists at the twentieth annual meeting.

Broadly speaking, no more striking proof of the importance of maintaining an alkaline reaction of the soil is needed than is furnished by those soils which have become famous for their

¹ Bull. 73, Bureau of Chemistry, U. S. Dept. of Agr.

² This Journal, 24, 1120 (1902).

persistent fertility under exhaustive cultivation. The loess soils, "regur" of India, "Tschernoseun" of Russia, chalk of England, basalt of the far northwest, prairie of the middle west, "blue-grass" of Kentucky and Tennessee, and the limestone valleys of the east—are soils which are recognized as being the most fertile in their respective localities, and have maintained their pre-eminence in fertility, in some cases, for thousands of years. These soils are all alkaline in reaction. The history of liming furnishes more general evidence upon the value of an alkaline reaction of the soil as one of the chief economic factors in crop production.

In the growing realization of the injury due to acid soils and in the recognition of the wide-spread occurrence of such soils, the attention of chemists has been directed to methods for the recognition, estimation, and study of soil acids. Most methods which have been proposed for this purpose have proved unsatisfactory, analytically, but the two above-mentioned appear worthy of further study.

PARTIAL OR COMPLETE NEUTRALIZATION OF ACIDITY.

Before proceeding to the discussion of these methods, a moment may be given to a consideration of the foundation question which determines the applicability of any proposed method. Briefly stated, it is this: How completely must the apparent acidity of a soil be neutralized that its fertility or the yield of crops may not be directly or indirectly reduced from this cause? If the total acidity of a soil were due entirely to a more or less readily soluble acid or acid salt the problem would be much simplified, but, as will be shown, there is evidence that this is not so, but that we are dealing with several kinds of "acidity" which affect fertility very unequally. It is evident that this question must be answered before a final conclusion can be reached with regard to the most suitable method to be used in estimating acidity.

As a basis for work on this problem, two standards by which to test proposed methods are available. We may aim to neutralize the apparent acidity so completely that finally the soil has a faintly alkaline reaction, or we may aim to neutralize only so much of the apparent acidity as reduces the crop yield of acid sensitive plants. To use any particular plant, red clover for instance, as a standard would necessitate the experimental demonstration of its sensitiveness on many different soils and under

many different conditions. There are so many factors affecting the sensitiveness of any given plant to acids (to say nothing of the widely different resistance of different kinds of plants), such as the nature of the acids, the presence of neutral lime salts, of injurious metallic salts, quantity of available plant food, moisture, activity of bacteria and fungi, etc., that such a standard seems almost impracticable. Nevertheless, in a consideration of the farm practice of any given locality the behavior of the least resistant crop grown may be, at least, tentatively, the economic standard by which to determine the maximum acidity the soil may have.

In the following discussion of the increase in crops, due to more or less complete neutralization of acids, it is to be borne in mind that most crops offer considerable resistance to acid soils. Indeed, the yields of some crops are practically unaffected on land so acid as to be barren of very sensitive plants, particularly when neutral lime salts are present. This is shown by the figures given in Table I and has been demonstrated in a practical way by the work of the Rhode Island Station, where the soils are exceptionally acid, so acid, indeed, as to reduce the yield of most crops. The production, therefore, of maximum yields of such resistant crops on partially neutralized land is not evidence that such partial neutralization is sufficient for the maximum yield of all farm crops, nor that it is the most economic in general farm practice.

TABLE I.—STRENGTH OF ACIDS JUST PERMITTING GROWTH OF SEEDLINGS.

	Lupine. ¹ <i>Lupinus albus</i> .	Peas. ² <i>Pisum sativum</i> .	Corn. ² (<i>Zea mays</i> .)	Pumpkin. ² <i>Cucurbita pijo</i> .	Red clover. ³ <i>Trifolium pratense</i> .	Wheat. ³ <i>Triticum vulgare</i> .
HCl or H ₂ SO ₄	$\frac{N}{6400}$	$\frac{N}{12800}$	$\frac{N}{3200}$	$\frac{N}{6400}$	$\frac{N}{27000}$	$\frac{N}{25000}$ $\frac{N}{18000}$
Acetic acid.....	$\frac{N}{1600}$	$\frac{N}{3200}$	$\frac{N}{800}$	$\frac{N}{20000}$	$\frac{N}{20000}$
Citric acid.....	$\frac{N}{1600}$

¹ Kalenberg and True : *Bot. Gaz.*, 22, 89.

² Heald : *Bot. Gaz.*, 22, 125.

³ Cameron and Breazeale : *J. Phys. Chem.*, 8, 1 (1904).

With reference to these figures it is to be remembered that they do not represent the toxic limit of plants grown under field conditions in which the presence of plant food and other salts, particularly lime salts, raise the toxic limit greatly. It seems possible that the presence of such salts will not change the relative resistance order of these plants, an order which, in general, is in harmony with farm experience.¹

But little work has been done on the amount of lime required to give maximum crop yields. So far as I know, the Maryland Station is the only one experimenting on this point. This work was commenced in 1896 on a poor, flat piece of land which had been out of cultivation for at least ten years. Freshly slaked stone lime was applied at different rates to the freshly plowed ground and harrowed in. No lime has been applied since. The published results follow:²

TABLE II.—EFFECT OF QUANTITY OF LIME ON YIELDS, PER ACRE.

Plot No.	Quantity of lime applied per acre.	Corn 1896, bushels.	Wheat 1897, bush-els.	Hay 1898, pounds.	Total weight in first rotation, pounds.	Corn, 1899.	
						Grain, bushels	Stover, pounds
(1).....	10	23.9	26.0	2502	5496	35.2	1845
(2).....	20	25.6	27.7	2367	5545	28.5	2070
(3).....	none	17.4	22.5	972	3366	30.5	1845
(4).....	30	25.8	28.7	2205	5475	34.8	2160
(5).....	40	27.1	31.9	2322	5862	36.5	2025
(6).....	none	15.3	22.7	1053	3333	23.4	1755
(7).....	50	28.8	31.0	2133	5621	39.8	2115
(8).....	60	29.8	32.5	2286	6024	43.4	2295

There is a fairly regular increase in yields with larger applications of lime, except in the hay crop, which is practically the same on all the limed plots. In all cases there is a marked increase over the unlimed plots.

From 1896 to 1898, inclusive, the surface of the limed plots, to a depth of 3 or 4 inches, was alkaline in reaction, about 1000 pounds of lime being required to render the surface alkaline to a depth of 6 inches. At present the limed plots Nos. 1 and 2 are acid, and the stand of red clover on these plots is much inferior to the stand on the alkaline plots.

¹ The results of Cameron and Breazeale are confirmatory of these statements. *Ibid.*

² Bull. 66, Md. Expt. Station.

In the extended work on acidity conducted at the Rhode Island Station, various quantities of alkalis have been used on a very acid soil, but as the writer has not the data necessary to calculate the completeness of neutralization, little can be said of these experiments, except that incomplete neutralization gave as large yields of the comparatively resistant plants experimented with for two years as complete neutralization gave. At the end of the time other resistant crops gave yields but little greater than on the unlimed pots. The pots limed to alkalinity maintained a high yield for the five years of the experiments and the total produced was twice as great as from the pots to which less alkali was applied.

In the collection of soils used by the Bureau of Chemistry in its vegetation experiments there are quite a number that are acid. On but one of these, however, have we data showing the effect of partial and of complete neutralization. The samples Nos. 67 and 68, Table XI, are from this soil which has naturally a very high apparent acidity, and the bean crops of 1899 and 1900 were almost total failures. Oats on the same soil had not been materially affected until the crop of 1902. In 1901 the soil in pot No. 67 was limed at the rate of 1200 parts per million. According to the sodium chloride method but 1330 parts of lime were required, and by the lime-water method 2000 parts were required. The soil was still acid after this liming (pot No. 67). The crop of cowpeas, grown in 1901, was 50.3 grams water-free material on the limed pot against 1.5 grams on the unlimed pot (No. 68). In the spring of 1902 an additional application of 1200 parts of lime per million rendered the soil strongly alkaline and the yield of cowpeas rose to 204 grams against 1.3 grams on the unlimed pots. The past season (1903) the limed pot (No. 67) grew a good crop of red clover, while on the unlimed soil no plants got above ground.

For the purpose of obtaining more data on the growth of sensitive plants on partially and on completely neutralized soils, we started this spring a series of pot experiments on a number of very acid soils. One pot of each soil was left unlimed, one was limed according to the sodium chloride method, and another according to the lime-water method, the lime being thoroughly incorporated with all the soil in each case. The red clover used in all these experiments was killed at a very early stage by a

fungus in the soil. Another and shorter series started on different soils, and in clay pots kept in another house, gave better results, but were not entirely satisfactory, owing to a lack of water in one pot, and to the incomplete neutralization of another soil (see Table III).

TABLE III.—EXPERIMENTS ON PARTIAL AND COMPLETE NEUTRALIZATION.

	Yield in un- limed pot. Grams.	Yield in pot limed by NaCl method. Grams.	Yield in pot limed by Ca(OH) ₂ method. Grams.
Soil from Smithsonian Institu- tion Grounds. Very acid loam	1.0 ¹	8.0	7.5 ²
	no nodules on roots	few nodules	many nodules
Subsoil typical of Piedmont plateau from North Carolina	1.5	1.5	4.5
	few nodules	few nodules	many nodules
Sand rich in organic matter, Norfolk, Va., A. O. A. C. Sample No. 1.....	4.0	3.0	5.5
Sand rich in organic matter, Norfolk, Va., A. O. A. C. Sample No. 1 ³	8.0	9.0	19.0

The soil in the duplicate pots was treated the same in all respects except as to quantity of lime added. The crop on the unlimed pot of the Smithsonian soil was badly damaged by lack of water, so the yield here is not a fair criterion by which to judge the effect of lime. Owing to an error the soil in pot 3 was not completely neutralized, and apparently the crop yield is still below what it should have been. The soil from the Smithsonian Institution grounds is a very acid loam, from a circular spot about 20 feet in diameter. It has been under observation for about three years, and nothing but sorrel has grown on it during the summer, and crab grass in the fall. Close around this circular spot are patches of red and white clover, where the soil has always been found to be alkaline.

In the second series of pots a red clay subsoil, characteristic of the Piedmont region, was used. This soil, where it crops out, washes badly and, as usually cultivated, is particularly infertile, and it was found necessary to fertilize and inoculate the sample before cropping.

¹ Crop killed for lack of water.

² Soil still acid.

³ Large pots.

In the third and fourth series of pots, soil from Norfolk, Va., (first sample, Table IX) was used. It is a sand mixed with much organic matter, and is sufficiently acid to materially affect the yield of corn. It was believed that from the nature of the soil most of the apparent acidity must be due to organic acids.

In the fourth series it became necessary to fumigate the crops to free them from insects. This was done by covering the car with a tent under which the whole car was fumigated at once. The plants in three of the pots, no-lime, lime by sodium chloride method, and lime by lime-water method not evaporated (see p. 654), were badly wilted, and most of them died in two or three days. The pot limed to alkalinity was uninjured. After waiting three or four days to determine the full effect on the injured plants, all the crops were harvested. While we cannot ascribe these yields entirely to the effect of acidity, we have in them another striking instance of the increased resistance of plants on alkaline soils to unfavorable conditions.

The data which have been presented cannot be regarded as conclusive. The differences in yield between partial and complete neutralization are often not as clear-cut and marked as one would wish. Many valid reasons can be advanced for this, the most plausible of which is that the apparent acidity of a soil is usually due to several different materials having widely different toxic properties. It is hoped, before another year has passed, to present more definite and conclusive data on these points.

Although inconclusive, the majority of the data point to the same practical conclusions, and are in harmony with the history of practical agriculture, the evidence of persistently fertile soils, the researches upon nitrification, physical condition and relation to water, upon fungus diseases and upon the effects of acidity, and it appears that we are justified in accepting this statement as a working hypothesis: "A soil faintly alkaline from carbonate of lime furnishes the best conditions for the economic production of crops, and the use of any method for estimating the acidity or the lime requirements of soils should be based on this hypothesis."

THE SODIUM CHLORIDE METHOD.

This method was proposed at the 19th meeting of the Association of Official Agricultural Chemists¹ by Mr. Hopkins and his asso-

¹ Bull. No. 73, Bureau of Chem., U. S. Dept. Agr.

ciates of the Illinois station. It is "based upon the reaction which occurs when solutions of mineral salts are added to an acid soil. The organic acids of the soil are themselves very difficultly soluble, and it is practically impossible to completely extract them from the soil with distilled water, even though large quantities of water be percolated through the soil; but, when a mineral salt solution is added to the soil, the organic acids (humic acid, etc.) unite with the mineral base, forming neutral humates, etc., and liberating the mineral acid, which, of course, is perfectly soluble, and whose titrating power furnishes a very satisfactory basis for determining the total acidity of the soil. The method consists in shaking 100 grams of soil with 250 cc. of 5 per cent. or normal sodium chloride solution for three hours and titrating 125 cc. of the boiled, clear filtrate with standard alkali, using phenolphthalein as indicator." The result multiplied by 3 equals the acidity in terms of cubic centimeters of standard alkali required to neutralize 100 grams of soil. The factor 3 is used in place of the natural factor 2, because by repeatedly removing one-half of the clear salt solution, restoring volume with 5 per cent. salt solution, shaking and titrating as before, it was found that the total alkali required for 100 grams of soil averaged approximately three times the first titration. (Later work has resulted in changing the factor to 4.)

The older investigators of humus—Knop, Schulze, Detmer¹—found that, as a rule, the "humates" and "humic acid" are much less soluble in salt solutions than in pure water, while only traces are dissolved by acids. Heiden and also Schumacher² found that peat, various preparations of "humic acid" and artificial "humus" removed portions of salts from solutions in which they were placed, but that the removed salts were for the most part readily dissolved by small quantities of water. More recent investigations by Berthelot and André,³ and by Lyashchenko⁴ confirm the older investigations as to the decomposition of mineral salts by humus materials. The fact that potash is partly removed from solution by organic material was brought to the attention of the Association of Official Agricultural Chemists several years ago, and the association

¹ Storer's "Agriculture."

² Johnson's "How Crops Grow."

³ *Bull. Soc. Chim. (Paris)*, 11-12, 15, 771 (1894).

⁴ *Abstract Expt. Sta. Record*, 11, p. 623.

is now struggling with the problem of recovering the total added potash from mixtures of acid phosphates and potash salts.

There can be no doubt that organic matter is able to remove from solution a portion of the mineral salt with which it is brought in contact. None of the standard works on absorption of soils makes mention of the production of free mineral acids; neither theoretical considerations nor a cursory examination of the literature lead one to believe that mineral acids in amounts equivalent to the total organic acids are set free by the action of mineral salt solutions on acid organic material.

Throughout my work with this method numerous tests have been made for free hydrochloric acid in the filtrates from the soil. In but one or two cases, where the presence of water-soluble free sulphuric acid was easily proved, were there any of the sharp characteristic reactions of considerable amounts of free acid. Twenty-five cc. portions of filtrates, requiring 8 or 10 cc. N/20 alkali to neutralize to phenolphthalein, were tested with a drop of methyl orange, and in no case was the acidity greater than that produced by adding 0.2 cc. N/20 acid to 25 cc. of the salt solution. In most cases it was much less. The addition of 0.05 to 0.2 cc. N/20 acid to these filtrates greatly increased the positiveness of their reaction with methyl orange. These results were deemed conclusive proof of the absence of appreciable quantities of free strong acids.

The behavior, on titration, of the boiled filtrate pointed to an explanation of the nature of the reaction between the soil and salt solution. In some work on the solubility of soil constituents in saline solutions I had previously observed that the filtrates were frequently decidedly acid. The presence of considerable quantities of iron, alumina and manganese accounted for this reaction. It seems possible that small quantities of one or more of these bases had been dissolved by the salt solution, and experiment showed that where the apparent acidity was more than 1 or 2 cc. N/20 alkali a precipitate which bore a striking relation to the alkali required was formed during the titration. These precipitates were found to be, usually, chiefly aluminum hydroxide, with smaller amounts of hydroxides of iron and manganese. In two cases it was zinc hydroxide. A number of filtrates were tested for phosphoric acid with negative results. It does not

appear probable that the apparent acidity of the filtrates is due to acid phosphates in solution.

After establishing the value of the alkali in terms of Al_2O_3 by titrating against a known solution of aluminum sulphate (the titration of aluminum salt solutions with alkali is a quite well-known technical process, which, however, is not regarded as very exact owing to the indefinite end-point) under the conditions of the method, a number of precipitates produced in the soil filtrates were filtered off, washed, dissolved in acid, silica separated, and reprecipitated with ammonia, washed, ignited, and weighed. The results, with other pertinent data, are given in Table IV.

From these experiments it seems that there is no setting free of appreciable quantities of hydrochloric acid and that there is practically no reaction between the organic matter and the salt solution, whereby difficultly soluble organic acids are dissolved, but that the acidity of the filtrate (or that acidity which is greater than would be given by water under the same conditions) is due to the solution of alumina or some other acid-salt yielding base. It appears that the hydrated neutral silicates or aluminates are quite strongly attacked by the salt solution, resulting in the replacement of aluminum by sodium, or a breaking-up of the compound, and the consequent formation of an acid solution of aluminum chloride, the titration of which, with alkali, constitutes the apparent acidity as determined by this method. Quite a little silica also goes into solution and is partly precipitated during the titration.

The reaction between salt solutions and soils or minerals in which the base of the solution replaces in approximately equivalent quantities certain of the bases of the soil or mineral attacked, received much attention twenty-five to fifty years ago. I am not aware, however, that a marked solution of alumina by neutral salts has been brought to notice before, although, of course, the analysis of salt-soluble materials has always shown the presence of one or more acid-salt forming bases. Johnson,¹ in discussing the absorptive power of soils, states that Mülder noticed that sesquioxide of iron participates in the displacements resulting from treating soils with saline solutions.

¹ "How Crops Feed," p. 347.

TABLE IV.—SHOWING THE RELATION BETWEEN ALKALI REQUIRED AND DISSOLVED ACID-SALT-FORMING BASES.

Description of sample.	cc. N/20 alkali required by			Mg. bases in 100 cc. alkali filtrate.		cc. N/20 alkali required by		
	100 cc. NaCl filtrate.	100 cc. H ₂ O filtrate.	100 cc. NaCl sol. acids.	Fe ₂ O ₃ , Al ₂ O ₃ , Mn ₂ O ₄ , Mg.	ZnO, Mg.	Fe ₂ O ₃ , etc., calculated as Al ₂ O ₃ .	ZnO.	Not accounted for by Al ₂ O ₃ and ZnO.
Fine sand, rich in organic matter	3.7	2.7	1.0	1.4	1.5	2.7
Rhode Island Station, very acid.....	1.6	1.0	0.6	2.0	2.0	0.0
Kaolin, good grade.....	1.6	0.7	0.9	1.2	1.3	0.3
Kaolin, good.....	4.2	Not det.	..	3.0	3.3	0.9
Vegetation experiments, Bureau of Chemistry, California soil:								
Limed.....	5.3	1.0	4.3	2.2	8.6	2.4	3.6	0.0
Not limed	19.0	3.4	15.6	2.6	29.4	3.0	12.2	3.8
Vegetation experiments, Bureau of Chemistry, Illinois Station.....	2.2	Alkaline	2.2	1.0	1.1	1.1
Carlisle silt loam, subsoil, Illinois	31.5	Not det.	..	30.4	33.4	0.0
Plot No. 6, fertilizer experiments, Pennsylvania Station.....	0.6	Alkaline	..	0.6	0.6	0.0
Plot No 10, P ₂ O ₅ experiments, Maryland Station..	1.2	0.5	0.6	0.6
Sand from propagating house.....	25.7	24.0	1.7	28, Fe ₂ O ₃	21.5	4.2
Collington loam, glauconitic, Prince George County, Md.....	22.6	22.8	23.5	0.0
Loam, District of Columbia, very acid, growth only sorrel.....	22.8	21.8	23.0	0.0
Clay subsoil, Piedmont region	2.8	1.8	2.0	0.8
Fine sand, rich in organic matter, treated with—								
N NaCl	3.6	0.7	2.9	2.3	2.5	1.1
2 N NaCl	5.2	0.7	4.5	3.5	3.9	1.3
N KCl	6.6	0.7	5.9	5.0	5.0	1.1
2 N KCl	8.0	0.7	7.3	6.9	7.6	0.4
Brick clay treated with—								
N NaCl	22.0	0.4	21.6	17.8	19.1	2.9
2 N NaCl	25.3	0.4	24.9	20.0	22.0	3.3
N KCl	27.6	0.4	27.2	20.6	22.2	5.4
2 N KCl	29.6	0.4	29.2	21.6	23.2	6.4

Were we to assume that the action of a mineral salt solution upon a soil affords a measure of the acidity of the soil, it appears reasonable to think that sodium chloride would be no better for this purpose than potassium or calcium chloride. Nor does there appear to be any reason why chlorides are better for the purpose than sulphates or nitrates. Indeed, the originators of the method appear to take this view of the matter, for they say: "Of course other mineral salts, as potassium chloride, serve the purpose equally well, but they are no better than common salt and are considerably more expensive." Bearing in mind the older work on the replacement of bases,¹ as well as more recent investigations² on the same subject, in which it is shown that there are frequently marked differences in the extent of replacement by different bases, it seemed worth while to make a few experiments on this point. In this work a heavy brick clay, such as is quite common in the coastal region of Maryland, and the fine sand, rich in organic matter, were selected. The sample of brick-clay was taken about 4 feet below the surface in the side of a gully, after removing the outer layer. To my certain knowledge the immediately contiguous soil has borne no vegetation for twenty years. It is almost certain that it contained no organic acids, but as it consisted largely of hydrated materials it was anticipated that it would show a high acidity by the sodium chloride method. The other sample was believed to contain chiefly organic acids, and consequently would show little acidity by this method. The various salt solutions used were of approximately equivalent strength. The results are given in Table V.

From Table IV it appears that the precipitate produced by titrating the filtrate is approximately proportional to the apparent acidity and from Table V we see that the apparent acidity will be determined by the salt used (provided the readings are multiplied by the same factor), potassium salts giving higher figures than the corresponding sodium salts. This difference is not great, however.

In investigating the absorptive power of soils, Pellitz³ found that at a certain concentration of the solution the absorption reaches a maximum above which it cannot be raised. Armsby,⁴

¹ Vide: "How Crops Feed."

² Dettrich: *Abs. Expt. Station Record*, 14, 746.

³ *Zitschr. anal. Chem.*, 14, 55, 282.

⁴ *Am. J. Sci. and Arts*, 14, 25.

TABLE V.—NaCl METHOD: ACIDITY BY DIFFERENT SOLVENTS OF SEVERAL STRENGTHS.

Description of sample.	Soil. Grams.	Kind and strength of solvent.	N/20 alkali to 100 cc. of filtrate. Acidity.	
			cc.	Parts per million.
Brick-clay (moist sample).....	200	5 per cent. NaCl..	28.0	980
			29.0	1,015
Do	200	10 per cent. NaCl..	40.0	1,400
Do.....	200	20 per cent. NaCl..	40.0	1,400
Do.....	100	5 per cent. NaCl..	19.2	1,344
			19.0	1,300
Do.....	100	10 per cent. NaCl..	25.0	1,750
			26.0	1,820
Do.....	100	20 per cent. NaCl..	25.5	1,785
			26.5	1,855
Do.....	100	5 per cent. Na ₂ SO ₄	18.8	1,316
Do.....	100	5 per cent. K ₂ SO ₄	22.4	1,568
			23.0	1,610
Brick-clay (air-dry sample).....	100	Normal NaCl	22.0	1,540
			23.0	1,610
Do.....	100	2 Normal NaCl...	25.3	1,771
Do.....	25	Normal NaCl	7.5	2,100
Do.....	50	Normal NaCl	13.5	1,890
Do.....	100	Normal KCl	27.6	1,932
Do.....	100	2 Normal KCl....	29.6	2,072
			3.6	252
Sandy soil, rich in organic matter	100	Normal NaCl....	3.6	252
Do.....	100	2 Normal NaCl...	5.2	364
Do.....	25	Normal NaCl....	1.7	476
Do.....	50	Normal NaCl....	2.5	350
Do.....	100	Normal KCl	6.6	462
Do.....	100	2 Normal KCl....	8.0	560

in working with a hydrous silicate, prepared according to the directions of Way, found that the amount of base absorbed from the solution was never more than two-thirds of the replaceable base in the silicate. He concludes that the extent of the reaction varies with the concentration of the solution, and with the ratio between the volume of the solution and the quantity of soils used.

While it seemed probable, indeed almost certain, that all these conclusions are valid for the apparent acidity shown by the sodium chloride method, it was deemed advisable to make a few experiments on these points. The results, which confirm Armsby's conclusions, are also given in Table V.

The ratio of succeeding titrations to each other when one-half of the salt solution, which has been in contact with the soil, is replaced by a fresh salt solution has also been studied, and a summary of the results is given in Table VI.

TABLE VI.—SHOWING TITRATION RATIOS AND ACTUAL FACTORS.

Soil.	Normal salt solution used.	cc. N/20 alkali required by 125 cc. of filtrate, first titration.	Total cc. N/20 alkali to equal acidity of 50 grams soil.	Average ratio of titrations.	Actual factor by which to multiply first titration.	Acidity parts per million.
Brick-clay	NaCl	30.4	79.4	0.65	2.61	{ 1109 ¹ 1700 ²
	KCl	32.2	81.6	0.61	2.53	{ 1138 ¹ 1800 ²
Sand (rich in organic matter)..... }	NaCl	4.4	18.4	0.91	4.18	{ 257 ¹ 246 ²
	KCl	6.4	26.0	0.85	4.06	{ 367 ¹ 358 ²

These results are again in harmony with absorption work, as well as with the work of the originators of the salt method.

In view of the nature of the reaction and of the results which have been obtained, it is evidently erroneous to attribute such increasing solubility in fresh solutions to absorption phenomena. It is evidently chiefly due to a progressive equilibrium reaction which only ceases when those compounds, which take part in the reaction with the salt solution, have been completely changed in character. It was found that the ratio between successive filtrates is not a constant one, but varies considerably. Hopkins *et al.* found it to vary from 0.62 to 0.8, and the fact that they have recently proposed the factor 4 indicates that they have found a ratio as high as 1.³

The work on the samples given in Table VI resulted in the establishment of the factor 2.60 for the brick-clay and a factor of about 4 for the sample containing much organic matter. These results, with those of Way, Pellitz, and Armsby, quoted above, lead me to think that the factor 4 is too high for soils containing the usual quantity of organic matter. In such soils it is probable that the factor should be less than 3.

Although the end-point in the titration is not particularly sharp, duplicate titrations agree fairly well after some little experience with the method. Toward the close of the titration the pink color produced fades quite slowly, and one may easily make an

¹ Using the actually determined factor.

² Using the factor 4.

³ See note, Bull. 72, Bureau of Chem., p. 117.

error of 1 or 2 cc. of N/20 alkali, if the titration is not continued until the color is faint but permanent. Where the quantity of alkali required is under 5 or 6 cc., duplicate readings will usually agree within 0.3 or 0.4 cc.; where the quantity of alkali required rises to 20 cc. or more, duplicates may differ as much as 1 cc. N/20 alkali.

That is, on faintly acid soils the "limit of accuracy," due to errors of titration, is about 25 parts of acid expressed as CaO per million of soil. On soils of high apparent acidity this error of titration may rise to 70 parts per million of soil. As the ratio of the first titrations to the sum of the successive titrations is not a constant, but varies considerably on different soils, large errors may be introduced by applying the high factor now used to soils upon which the ratio of the titrations is narrow. For example of the erroneous results due to the use of an arbitrary N/20 alkali, the use of the factor 4 makes the apparent acidity 1700, while the true factor 2.61 makes it 1109 parts of CaO per million, an error of 591 parts per million or of about 2000 parts per acre-foot. These are extreme figures, but I do not think that at present we can expect greater accuracy than 100 parts per million of soil even under the most favorable conditions. As an example of the erroneous results due to the use of an arbitrary factor, the results on the sample of coarse sand from the propagating house (Table VII) are interesting; by the lime-water method this sample requires 1000 parts per million of CaO to make it alkaline; by the sodium chloride method, without the factor, it requires 900 parts, while using the factor it requires 1800 parts, which, of course, is wrong.

The method is very simple in execution, and for this reason very attractive to the busy chemist. It is not, however, a method which enables us to determine the total apparent acidity of a soil, nor can we yet say that by it we can determine that acidity injurious to sensitive crops.

By it we can make an approximate determination of the amount of aluminum or other acid-salt forming bases dissolved by sodium chloride under the conditions of the method, and can, of course, calculate the lime required to neutralize such acid salts. But as different salts and different concentrations of the same salts dissolve different quantities of these bases, we cannot assume from

chemical data that lime would act in the same way that normal sodium chloride does, nor that the action of sodium chloride is a measure of the lime requirements of soil.

In this connection it may be noted that a soil limed in accordance with the results of the sodium chloride method is never alkaline unless indeed the lime requirement by this method is as great as the lime requirement by the lime-water method. This is shown in Table VII, where we see that the sample of sandy soil, which required 258 parts of lime by the sodium chloride method, after being limed with that amount, still required 161 parts per million, while the loam soil, after being limed as required, showed an additional lime requirement of 658 parts. Both these soils had grown a crop between the tests, but it cannot be admitted that the acidity had increased at any such rate as this. Faintly alkaline soils, particularly where they contain organic matter, may show considerable acidity by this method, as is shown by samples from pot 35, and the 30 bush CaO plot Maryland Station. This result is undoubtedly due to the action of the sodium carbonate resulting from the reaction between the lime carbonate and sodium chloride.

If it be found, by experience, that the sodium chloride method is a measure of the acidity or lime requirement of soils, then we must conclude that water-insoluble organic acids and acid material play no part in soil acidity, and that an application of lime in such quantities as will approximately neutralize all the acid salts that could be produced by the action of normal chloride solution on the soil, is all that is necessary. With soils containing no organic matter this is, of course, true, as has been shown.

THE LIME-WATER METHOD.

This method is a modification of the method of Tache¹ and is based on the fact that after the affinity of the soil for calcium as bicarbonate is satisfied, the presence of the slightest amount of this salt is manifest by the pink color produced when phenolphthalein is boiled with a portion of the water which has been in contact with the treated soil. It was claimed for the method that it represented the maximum lime requirements of a soil, and from theoretical considerations it was argued that under the conditions of its execution (action of lime as bicarbonate and

¹ *Chem. Ztg.*, 21, 174 (1897).

absence of any considerable excess of this), absorption, attack of non-acid organic matter and of non-acid silicates, aluminates, and silica was probably reduced to a minimum and were practically negligible reactions.

The work since done has changed some of these opinions and strengthened others.

Beyond a doubt the method shows the maximum lime requirements of soils, that is, it shows the amount of lime required under the most favorable conditions of distribution, to make them alkaline in reaction.

Several modifications of the original method have been tried, primarily to reduce the work now required.

The results obtained by all modifications are shown in Table VII, in comparison with the results by the sodium chloride method:

The most serious objection to the method, from the analyst's standpoint, is the number of determinations which must frequently be made before alkalinity is reached. With the view of reducing the work a modification, in which the evaporation to dryness of the mixed soil and lime-water is omitted, was tried.

As will be seen, this modification gave a lower total apparent acidity than the original method on all but one sample. In some cases this difference is great, 800 parts per million in one case, while in others it is but little greater than the limit of error of the method.

It cannot be said from the results that the differences are lower on soils containing large amounts of organic matter than on soils containing an average amount of organic matter, although one would expect this to be the case. On soils containing practically no organic matter, such as kaolin, Carlyle silt loam and brick clay, the difference between the two modifications is much less.

It seemed possible that the drying at 100° of the soil, which has been treated with lime-water, may result in a greater attack of non-acid soil constituents by even so weak a solution as that given by calcium bicarbonate than will take place in the field under atmospheric conditions.

If this is so, the modified method would probably give results nearer the true lime requirements than the original method. To

TABLE VII.—ACIDITY EXPRESSED AS PARTS OF CaO PER MILLION OF SOIL.

Description of sample.	NaCl method.	Ca(OH) ₂ method.			
		Dried on bath.		Dried in air; stood 16 hours.	Not dried; stood 16 hours.
		Stood 16 hours.	Stood 40 hours.		
Sandy soil rich in organic matter, Norfolk, Va.....	258	4,000	3,400	3,900	3,500
Sandy soil rich in organic matter after cropping.....	252	3,900	3,200	>3,400	2,100
Sandy soil rich in organic matter, limed NaCl method 200 parts per M	161	3,800	2,700
Sandy soil rich in organic matter, limed 1,700 parts per M.....	49	2,400	1,500	<1,900	800
Loam, very acid, growth sorrel.....	1,365	2,200	2,500
Loam, very acid, growth sorrel, after cropping	1,631	2,400	2,000	<2,000	2,000
Loam, very acid, growth sorrel, after cropping, limed 1,200 parts....	658	2,100	1,200	1,600
Loam, very acid, growth sorrel, after cropping, limed 2,250 parts per M	91	1,200	600	<1,000	600
Pot No. 67, P ₂ O ₅ experiments, CaO series, Rhode Island Station.....	112	1,000	600	> 800	800
Pot No. 67, vegetation experiments, Bureau of Chemistry, California } soil, limed 1,200 parts.....	370	900
Pot No. 68, vegetation experiments, Bureau of Chemistry, California } soil, unlimed	1,330	2,000	2,000	1,900	1,600
Pot No. 35, vegetation experiments, Bureau of Chemistry, Ill. Station	153	Alkaline
Kaolin No. 1.....	112	100	100	100	100
Kaolin No. 2.....	280	150
Carlyle silt loam, Illinois.....	2,170	2,600	<2,400	2,600	2,400
Coarse sand from propagating house.....	1,800	1,000
Collington loam, subsoil glauconitic, Maryland.....	1,470	1,100
Brick clay, 4 feet below surface, Maryland.....	1,610	1,400	<1,300	1,200
CaO plot, 10 bushels per acre, 0 to 6 inches, Maryland Station.....	196	500	<400	300
CaO plot, 20 bushels per acre, 0 to 6 inches, Maryland Station.....	63	300	200
CaO plot, 30 bushels per acre, 0 to 6 inches, Maryland Station.....	28	Alkaline
CaO plot, 0 bushels per acre, 0 to 6 inches, Maryland Station.....	406	600	<500	400
Plot No. 4, "soil test" plot, Rhode Island Station, taken 1894.....	518	1,800	<1,700	1,700

test this point portions of various soils were treated with lime-water as usual and allowed to dry in the air, being protected from dust or fumes in the meantime.

The results are given in the table under the heading "Air-dried." The results thus obtained agree remarkably well with those obtained by drying on the steam-bath, showing that the reaction at 100° is but little greater than takes place under field conditions. Two exceptions are to be noted where the apparent acidity by drying in the air is much less than by drying on the bath. I have not had time to make further examination of these cases.

On the whole, it seems advisable for the present to follow the method strictly as proposed, as such procedure will give results that agree best with field conditions and, at the same time, be the most concordant.

In the practice of the lime-water method it has been found that the length of time the treated and dried soil is allowed to stand in contact with water has a considerable effect on the apparent acidity as determined by this method. When solution is allowed to go on from forty-eight to seventy-two hours, the pink color with phenolphthalein is developed with less added lime-water than where the solution is allowed to stand sixteen hours or less.

Some soils which are acid when allowed to stand sixteen hours give an alkaline filtrate when allowed to stand twenty-four to forty-eight hours longer. The results of long standing are also given in Table VII.

I cannot say definitely why the time of standing should produce such differences in apparent acidity in the case of many soils. We may assume, with safety, I think, that the result is not due to the failure of calcium carbonate to go into solution in partially carbonated water in the shorter time. Indeed, calcium carbonate is instantly sufficiently soluble in ordinary distilled water to give a strong reaction with phenolphthalein when the filtrate is boiled for a few moments. The same has been found to be true of many naturally alkaline and of limed soils.

The above-mentioned result appears to be due rather to the slower solubility and hydrolyzation of the neutral lime compounds that are formed with the added lime-water, or to the solution and hydrolyzation of neutral compounds already present in the soil.

It has long been believed that the bases of the soil go into solution partly as bicarbonates, and the Rogers Brothers¹ have shown, long since, that many of the common minerals, particularly the zeolites and feldspars, when reduced to fine powder and treated with carbon dioxide-free water, gave a more or less strong reaction with phenolphthaleïn.

Both of these classes of minerals are believed to be present in most soils, but from the nature of the origin of soils the more soluble forms are not likely to be present in considerable amounts, nor are the more insoluble forms likely to be present in a finely divided state. Hence the solution of such minerals would be expected to be slow and the development of an alkaline reaction on long standing, with smaller amounts of lime-water, might be thus accounted for.

In the same way the soil which, on short standing, appears neutral or acid, may, on longer standing, become alkaline, the solution and hydrolyzation of its own bases in a large amount of water resulting in the neutralization of its acidity. It is possible that the influence of a favorable season on the stand of red clover on faintly acid soils may be thus partly accounted for; the apparent acidity of the soil has been reduced by the increased solution of the bases present.

Just what procedure to adopt here I find somewhat difficult to decide. Tentatively, it seems advisable to follow the results given by the shorter time, as it is likely that in this case the alkalinity is due to the solution of calcium carbonate, while with the longer time it appears to be due to decomposition of neutral salts, such as the silicates, aluminates, and perhaps "humates," etc.

How great may be the attack of the bicarbonate upon the non-acid organic matter of the soil I cannot say. Doubtless it is considerable, but as the affinity of this material for lime must be satisfied before the soil solution is alkaline, such attack must be regarded as constituting part of the apparent total acidity of soils.

Experiments have shown that there is but little of soluble organic acids, or acids whose lime salts are soluble, as the lime dissolved from the treated soils by water is but little more than that dissolved from the untreated soils, not more than is contained in 2 cc. of standard lime-water.

¹ *Am. J. Sci. and Arts*, 5, 5 (1845).

On the other hand, it does not appear that the combinations of lime and non-acid organic matter are very rapidly attacked by the organisms of the soil. For example, the sample of fine sand, rich in organic matter, which had nearly ten times as much lime added as called for by the sodium chloride method and about one-half as much as called for by the lime-water method, was still strongly acid after growing a crop of clover, and five months after liming. It still required as much lime as would, with that already added, bring it up to its original lime requirements.

With soils from which the supernatant fluid is clear and practically colorless, duplicates by this method agree well; in no case should they differ by more than 2 cc. of standard lime-water per factor weight of soil (200 parts per million); with soils which give a colored fluid, close duplication is more difficult, and where more than 20 cc. of lime-water are required per factor weight, duplicates may differ at a maximum as much as 3 cc. of standard (300 parts per million).

In comparing the results by the two methods we see they differ least, usually, on soils containing no organic matter and that on such soils, if we may so call them, the results by the sodium chloride method may be higher than by the lime-water method. That is, the apparent acidity in terms of lime is greater than the amount of lime actually required to neutralize it.

As the organic matter increases in the soil the results become more divergent, the sodium chloride showing a much lower acidity than the lime-water method.

As the originators of the sodium chloride method have found field applications of lime in quantities indicated by it to give satisfactory results, a word in explanation of the conclusion thus indicated may be in order.

It has been shown on soils containing but little organic matter that the sodium chloride method gives as high or higher acidity than the lime-water method. In such cases applications of lime in accordance with the sodium chloride method must completely neutralize the acidity of the soil.

With soil on which the lime-water method shows a very much higher acidity the effect of the smaller application of lime may be accounted for by the following facts: The writer has found that lime applied, even in excessive quantities, neutralizes very

slowly the acids of the unstirred subsoil. Indeed, for practical farm purposes we may say that such neutralization is negligible. Hence, as the applied lime is confined to the cultivated surface layer (3 or 4 inches of ordinary harrowed ground), it may often happen that this layer has been rendered alkaline by the applied lime which was expected to neutralize the acidity to a depth of 6 inches or more.

It is true that even heavy applications of lime cannot be perfectly distributed in the field and maximum yields may not be reached because of this. In comparison of large and small applications of lime this fact may be of importance, and it may happen that, owing to imperfect distribution, the full effect is not secured even when enough lime is applied to neutralize the total apparent acidity of the soil, and larger applications may appear more advantageous. The data at hand do not justify a conclusion on this point.

The writer believes, however, that applications of lime, in quantities sufficient to neutralize the acidity of the surface 6 inches of soil, as determined by the sodium chloride method, will not always be sufficient to give satisfactory crops of red clover and alfalfa, even though the lime may be mixed with only the surface 3 inches. He believes that until we have more positive data it would be the wisest practice to apply lime in sufficient quantities to render the cultivated soil slightly alkaline in reaction, and consequently to use a method which will indicate this quantity.

NATURE OF SOIL ACIDITY.

Our knowledge of the nature of "acidity" in soils is very general. Most soils, even those which would be reckoned as very acid by any of the various methods which have been proposed for determining soil acidity, contain but little water-soluble acids or acid salts. Practical farm experience has strongly indicated that the acids which may be removed from soils by simple treatment with water are not the total harmful acids which they may contain. It has been assumed that very difficultly soluble organic acids as "humic," "ulmic," etc., and their acid salts, the "humates," "ulmates," etc., are chiefly responsible for the harmful acidity of soils, and it has been the object in devising methods for determining soil acidity to reach such compounds. The figures given

in Table VII are indicative of the fact that such compounds are not the only ones which go to make up soil acidity.

Further, they show that there is no close relation between the "acidity" as determined by either of the methods and the toxicity of such acidity. Thus the sample of coarse sand, which has an acidity of 1800 parts per million by the sodium chloride method and of 1000 parts by the lime-water method, will, under no condition, produce a growth of the ordinary farm crops. All of the other soils given will grow small crops of the more resistant plants. The soils from California, from Rhode Island, the Carlisle silt loam, and the Collington loam yield fair crops. It is evident, therefore, that acidity, as determined by these methods, cannot be of the same nature in all the soils.

Until we have more definite evidence as to the nature and effect of the various compounds which are reckoned as constituting a part of the total apparent acidity of soils, we may tentatively and for purposes of further study class acidity under two general heads.

Active or actual acidity, due to relatively soluble organic and inorganic acids and acid salts; and *inactive or negative acidity*, reaching, in some soils actual neutrality, as determined by the usual indicators. This latter condition, constituting a part of the absorptive power of soils, appears to be due to the presence of certain constituents, such as the easily attacked hydrated or colloidal silicates, and many non-acid organic compounds, which have a strong affinity for sodium, potassium, calcium, and magnesium. On solution of these bases by the soil water, or on their addition in alkaline compounds they combine immediately with these practically neutral constituents, which thus prevent the reaction of the soil from becoming alkaline until the above-mentioned affinity is satisfied. This condition, while harmful to sensitive plants, is certainly not so harmful as the condition due to free acids or acid salts. Further than this, the reaction which takes place between certain of these soil constituents and added chlorides, sulphates, etc., produces positively acid salts, as we have seen from the reactions of the sodium chloride method. There can be but little doubt that it is due partly, at least, to the acidity thus produced that the injury arising from the use of chlorides and ammonium sulphate on acid or neutral soils, is to be ascribed.

It is the negative acidity, due to inorganic materials, plus the water-soluble free acids and acid salts, that is estimated by the sodium chloride method, while the total active and inactive acidity is estimated by the lime-water method. In other words, this method determines what has been called the "total apparent acidity." I do not believe that for the most economical results it is sufficient to neutralize only the active acidity or only the inactive acidity, even though such procedure should leave the soil practically neutral, for a soil so treated is able to neutralize such bases as may be set free by weathering, while the acid produced by weathering, biological processes, and added fertilizers renders the soil again positively acid.

One exception may, perhaps, be made to this general statement. Where the apparent acidity is chiefly due to free organic acids a partial neutralization may be sufficient to give the soil an alkaline reaction in a short time, because, as has been shown by several observers, the calcium salts of such acids are rapidly attacked by fungi and bacteria; the acid radical is destroyed while the lime, being set free, neutralizes another portion of the acids.

Some experimental work has been done in order to obtain additional information as to the nature of the inorganic compounds which neutralize lime-water. It has been found that ignited acid-insoluble residues from soils do not combine with lime-water to any extent under the conditions of the lime-water method, 12 grams of ignited material requiring but 1 cc. of lime-water to give an alkaline reaction.

Ignition of soils also destroys their affinity for lime; indeed, as would be expected, the ignited residue from some soil, rich in organic matter, is alkaline. It is well known that hydrated silica in water takes up large quantities of lime-water, but when 2 grams of hydrated silica (air-dry) are mixed with 1 cc. of lime-water, evaporated and treated as in the lime-water method, a pale pink color is developed when the liquid is concentrated to 10 to 15 cc.; no greater color is developed, if 5 or 6 cc. of lime-water had been added to the silica.

This same slight color has been so often observed when working with soils, that it is usually ascribed to the presence of calcium silicate rather than calcium carbonates.¹

¹ This view is strengthened by recent work of Keiser and Forder (*Am. Chem. J.*, 31, 153), who have found that calcium silicates and aluminates are sufficiently hydrolyzed by short standing in water to give an alkaline solution.

If it is true that crops are most profitably produced only when the soil is completely neutralized or faintly alkaline, the separation and estimation of the several components of the total apparent acidity is not of much practical importance. On the other hand, if it is not necessary to completely neutralize the total apparent acidity, it becomes necessary to devise methods for the estimation of the harmful acidity, and determine to what it is due.

As a beginning along this line we may estimate the water-soluble acids and acid salts; we may estimate the negative acidity due to easily attacked silicates by means of the sodium chloride method, using a proper correcting factor. We may determine the total apparent acidity by the lime-water method, and by subtracting the acidity determined by the sodium chloride method we have acidity due to insoluble organic matter.

These estimations have been made here with fairly satisfactory agreement with the known character of the soil.

As several small, but important, changes have been made in the lime-water method, the improved method is appended.

To determine the reaction of a soil:

About 10 grams of soil are treated with 100 cc. of distilled water in a Jena flask and allowed to stand over night. Fifty cc. of the supernatant liquid are carefully drawn off and boiled with a few drops of phenolphthalein in a covered Jena beaker until the appearance of the pink color or to a volume of about 5 cc. with no development of color. The pink color shows the soil to be alkaline, while no color shows it to be acid or neutral.

To determine the degree of acidity: To three portions of soil, each consisting of as many grams as the standard lime-water contains milligrams of lime (CaO) per cubic centimeter, add 50 to 60 cc. of distilled water and different amounts of standard lime-water. For example, to the first add 10 cc., to the second 20 cc., and to the third 30 cc. of lime-water. Dry down at once on the steam-bath, transfer to stoppered Jena flasks with 100 cc. of distilled water, allow to stand over night, with occasional shaking, draw off 50 cc., place in a Jena beaker, add a few drops of phenolphthalein solution, and boil until the appearance of the pink color, or in case no color is developed, to a volume of about 5 cc. Then with two portions of treated soils, one of which has been rendered alkaline by the lime-water and the other of which is still acid,

as guides, prepare three fresh portions of 10 grams each and add lime-water as before, except that the amount added to a dish differs from that added to another by 1 or 2 cc. Dry, take up with 100 cc. of water, allow to stand, draw off, and treat exactly as before. The smallest amount of lime-water which gives the characteristic pink color is taken as the acidity equivalent of the soil. Each cubic centimeter of standard lime-water is equivalent to an acidity of 0.01 per cent. expressed as calcium oxide.

It is essential that the distilled water used be free from alkalies and acid.

THE USE OF BASIC ALUMINUM ACETATE AS A PRESERVATIVE IN SAUSAGE.

BY ED. MAC-KAY CHACE.

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IN THE course of the usual work in this laboratory upon imported foods, the writer some time since received a sample of imported canned sausage which was preserved with a salt of aluminum. As aluminum compounds are not among those usually employed as preservatives in canned meats, an investigation was started in order to ascertain the extent of their use, the amount used, etc.

Up to the time of the present writing they have been found in several samples of imported canned sausage, all from the produce of two manufacturers, however. They have not been found in any samples of imported smoked, or domestic canned or smoked sausage.

The two manufacturers in whose goods aluminum was found acknowledge the use of the basic acetate of aluminum in small quantities. The detection of the addition of aluminum is easily carried out inasmuch as it is not a normal constituent of the ash of flesh.¹

The following method was found to work satisfactorily: About 25 grams of the ground sausage are ashed (complete ashing is not necessary) and the ash dissolved in strong hydrochloric acid. Sodium hydroxide is then added in excess and the whole boiled, the precipitate and insoluble ash filtered off, the filtrate made

¹ See the works of Bunge and Halliburton on "Physiological Chemistry."