

LXIV.—*Electrometric Study of the Precipitation of Silicates.*

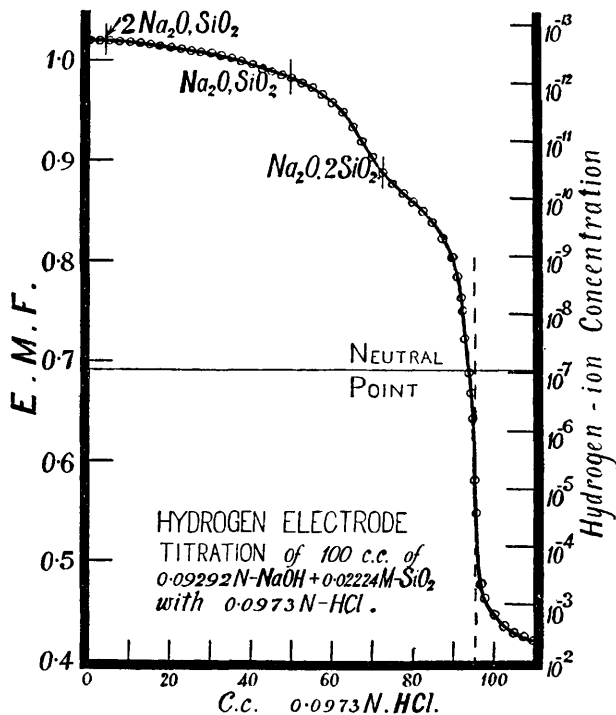
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THE work now described was undertaken in order to gain some insight into the reactivity of silicic acid towards various bases of different strengths. Its reaction with sodium hydroxide was first studied, for, although this subject has received some attention in recent years by measurements of the hydrogen-ion concentrations of solutions of silicic acid in sodium hydroxide (Bogue, *J. Amer. Chem. Soc.*, 1920, **42**, 2575; Joseph and Oakley, *J.*, 1925, **127**, 2814; Harman, *J. Physical Chem.*, 1926, **30**, 1100; Hägg, *Z. anorg. Chem.*, 1926, **155**, 20), there still remains much uncertainty as to the actual manner in which the acid ionises, and, moreover, the results hitherto recorded do not give a complete account of the variation in hydrogen-ion concentration throughout the course of neutralisation at 18°. Joseph and Oakley concluded, from  $p_H$  measurements at 33—38° of solutions of water-glass to which varying amounts of acid or alkali had been added, that the neutralisation curve obtained was "typical of the combination of a strong base with a weak dibasic acid,"  $K_1$  being about  $10^{-10}$ , and  $K_2$  about  $10^{-12}$ , the symbols relating to the first and second dissociation constants, respectively. Hägg also states that, if silicic acid is not colloidal,  $K_1$  of metasilicic acid is of the order of  $10^{-9}$  and  $K_2$  about  $10^{-13}$ . The curve given in Fig. 1 shows the change in hydrogen-ion concentration when 100 c.c. of a solution, 0.09292*N* with respect to NaOH and 0.02224*M* with respect to SiO<sub>2</sub>, were titrated at 18° with 0.0973*N*-hydrochloric acid, using the hydrogen and the normal calomel electrodes in the way previously described (J., 1924, **125**, 1572; 1925, **127**, 2110). The ratios of Na<sub>2</sub>O to SiO<sub>2</sub> present in the solution at the different stages of neutralisation are indicated on the curve. Two inflexions occurred in that portion corresponding to the addition of 2 equivs. of sodium hydroxide to 1 mol. of silicic acid, and the point corresponding to half-neutralisation of metasilicic acid, *i.e.*, Na<sub>2</sub>O,2SiO<sub>2</sub>, falls at  $p_H$  10.4 just at the beginning of the second inflexion. As other workers have shown, the ionisation of silicic acid cannot be represented satisfactorily in terms of two dissociation constants; moreover, the titration curve is not typical of a dibasic acid inasmuch as when  $K_1$  and  $K_2$  are widely different the point corresponding to half-neutralisation should lie somewhere about the middle of the second inflexion—in Fig. 1 it should be near the point corresponding to  $p_H$  11. The curve, however, shows that silicic acid enters into some kind of combination with sodium

hydroxide, and gives some idea of the hydrogen-ion concentrations that are thereby set up. It also shows that the hydrogen-ion concentration imparted by silicic acid to a solution is much too small to have any effect on the methyl-orange end-point, and hence the reason why the alkali present in a silicate solution may be titrated by using such an indicator.

In order to study the reactions (and consequent precipitations) between silicic acid and the alkaline earths, hydrogen-electrode

FIG. 1.

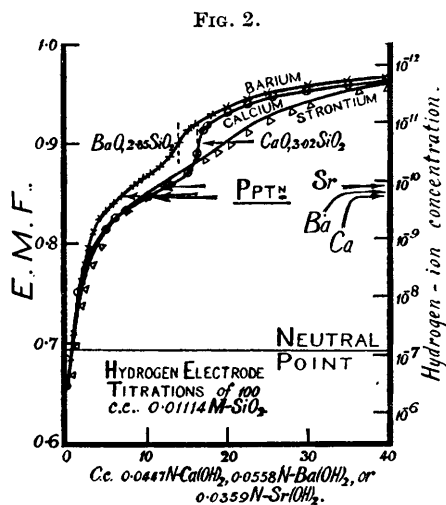


titrations were carried out at 18° of solutions of 100 c.c. of 0.01112M-SiO<sub>2</sub> with each of the following solutions: 0.0447N-calcium hydroxide, 0.0558N-barium hydroxide, and 0.0359N-strontium hydroxide. The solutions were prepared immediately before the titrations by neutralising sodium silicate solutions with hydrochloric acid, using methyl-orange, in order that they should contain the acid in a condition of maximum reactivity and that any diminution by ageing or by precipitation of the silicic acid should be avoided. These very dilute solutions were quite clear. The titration curves

given in Fig. 2 show that precipitation of the silicates did not begin until appreciable amounts of alkaline-earth solutions had been added, and when approximately the same hydrogen-ion concentrations had been attained. Thus the compositions of the solutes when on the point of precipitation were  $\text{CaO}, 5.0\text{SiO}_2$ ;  $\text{BaO}, 5.07\text{SiO}_2$ ; and  $\text{SrO}, 5.54\text{SiO}_2$ ; the respective  $p_{\text{H}}$ 's being 9.74, 9.72, and 9.90. In the case of the calcium and barium titrations marked inflexions occurred between  $p_{\text{H}}$  10 and 11, but in the strontium titration an inflexion is scarcely perceptible. It was found that, in the calcium and barium reactions, these inflexions denoted the points when the silica and the whole of the added alkaline earths had become precipitated; hence the precipitates must have been  $\text{CaO}, 3.02\text{SiO}_2, x\text{H}_2\text{O}$  and  $\text{BaO}, 2.85\text{SiO}_2, x\text{H}_2\text{O}$ . The hydrogen-ion concentrations of the solutions thereafter indicate that some further reaction took place between the precipitates and the additional alkaline-earth solutions. The hydrogen-ion concentrations of the solutions prior to precipitation are comparable with those produced with sodium hydroxide (*vide* Fig. 1), showing that the condition in which the silicic acid is present in both the alkali and the alkaline-earth solutions is similar. It seems reasonable to suppose that the high proportion of silica retained in the solutions must be due to some colloidal micellar formation.

The amounts of silica found in "soil solutions" of any  $p_{\text{H}}$  are very small even when their calcium contents are comparatively high. This seems to be contrary to what might be expected from the foregoing observations. The absorption of carbon dioxide, however, is sufficient to decompose any calcium silicate solutions which may be formed.

The absorption of alkaline earths by silica gel suspended in water was marked by a slow and gradual diminution in hydrogen-ion concentration. The curves showed a "buffer action," the extent of which depended on the amount of silica employed, commencing from  $p_{\text{H}}$  6.5, instead of the sharp change shown in Fig. 2, where the silica was not in great excess. The buffer action of the silica gel



is comparable with that which takes place when slaked lime is added to a soil, the reaction being usually between  $p_H$  6.5 and 9.

Attention was now directed to the action of solutions of sodium silicate on typical metallic-salt solutions. The precipitates thus obtained have received comparatively little attention, and the very few formulæ which have been assigned to them show that the ratio of silica to base is always higher than that required to form the metasilicate. Some investigators have, however, mentioned that their precipitates contained free silica. The difficulties inherent in the separation of a silicate precipitate, often containing free silica, from its mother-liquor in anything approximating to the pure state (of which there is usually no criterion), are so great that it is probable that the few analyses which have been made convey no idea of the reaction which took place. It has been shown above that the acidic property of silicic acid asserts itself only in alkaline solutions, and consequently it would be expected that very little action could take place between it and those hydroxides which are precipitated from acid solutions, unless the substances formed by the interaction were extremely insoluble. The view has been advanced, however, to account for the acidity of "soil solutions," that complex aluminosilicic acids exist in the soil through the interaction of aluminium hydroxide and silica. If any such combination could take place, it is probable that it would have some effect on the hydrogen-ion concentration of the mother-liquor. It was therefore decided to investigate electrometrically the changes in hydrogen-ion concentration when solutions of sodium silicate and of metallic salts are allowed to interact. The sodium silicate employed was prepared from water-glass containing 2.16 mols.  $\text{SiO}_2$  to 1 mol.  $\text{Na}_2\text{O}$ , and was 0.1020*N* with respect to sodium hydroxide. The impurities therein, including carbon dioxide, were so small as to have no measurable effect on the *E.M.F.* measurements. The solutions used are given in Table I, together with the  $p_H$  at which precipitation began, the amount of reactant which had to be added, and the  $p_H$  at which the respective hydroxides are precipitated, these values having been taken from the author's papers (J., 1925, 127, 2110, 2120). 100 C.c. of salt solution were used for each titration.

The titration curves are plotted in Fig. 3, in which the beginning of precipitation is marked by arrows. The amount of sodium silicate required in each reaction such that the alkali was in a quantity equivalent to the acid radical in 100 c.c. of salt solution was 39 c.c. The curves lying beyond 39 c.c. illustrate the effect of increasing amounts of sodium silicate on the hydrogen-ion concentration of the mother-liquor. The equation

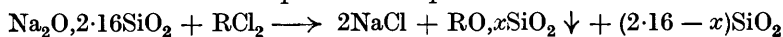
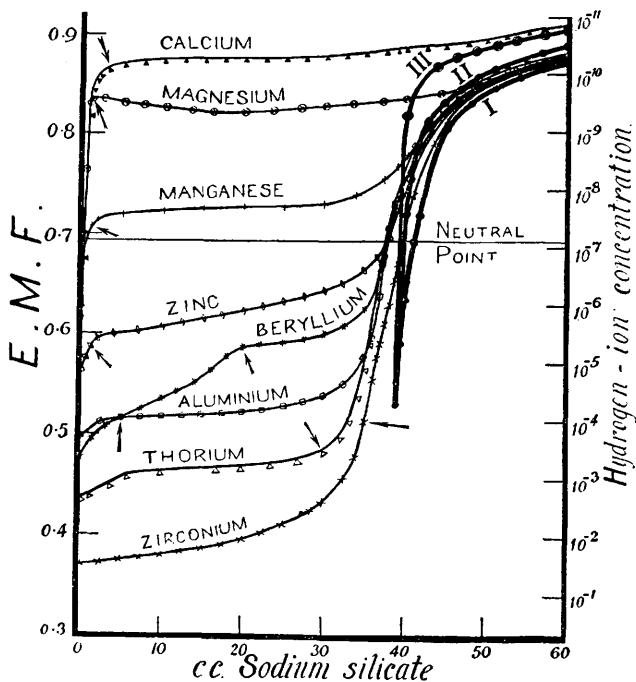


TABLE I.

Solution titrated.	pH.	Precipitation began at		Hydroxide precipitation at pH.
			c.c. of sodium silicate.	
0.01M-ZrCl <sub>4</sub> .	3.98		35.0	1.86
0.01M-ThCl <sub>4</sub> .	3.50		30.0	3.50
0.0067M-Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> .	4.04		5.0	4.14
0.02M-BeSO <sub>4</sub> .	5.31		20.0	5.69
0.02M-ZnSO <sub>4</sub> .	5.25		1.0	5.20
0.02M-MnCl <sub>2</sub> .	7.35		1.0	8.41
0.02M-MgSO <sub>4</sub> .	9.50		1.0	10.49
0.02M-CaCl <sub>2</sub> .	10.07		3.0	—

FIG. 3.



shows that after the silicate  $\text{RO}, x\text{SiO}_2$  had been precipitated by the 39 c.c., the mother-liquor comprised a sodium salt (chloride or sulphate), which through its very small concentration would have no measurable effect on the *P.D.*, and a certain amount of silicic acid which had escaped precipitation and would exert a buffering effect on the excess of sodium silicate. Some measure of the amounts of the silica which had remained in solution, and therefore of the amounts which had been precipitated with the metallic hydroxides, was obtained by following the changes in hydrogen-ion concentration

when sodium silicate, of the same concentration as that used for the precipitations, was added to solutions having the volume attained when the precipitations had become complete, *viz.*, 139 c.c., and containing the same concentration of neutral sodium salt as the original mother-liquors but different amounts of silica. Thus Curve I (Fig. 3) represents the course taken when sodium silicate was added to 139 c.c. of solution containing 39 c.c. of sodium silicate exactly neutralised (to methyl-orange) with hydrochloric acid; it would therefore have been the curve obtained if the hydroxides alone had been precipitated and all the silica had remained in solution. Curve II shows the variation in hydrogen-ion concentration which would have been produced if the metasilicates of the metals had been precipitated, and the remainder of the silica had been left in solution; whereas Curve III corresponds to the change which would have occurred if all the silica present in the 39 c.c. had been precipitated. Except for the calcium curve, those sections of the curves corresponding to an excess of sodium silicate lie between Curves I and II. Hence the amounts of silica carried down with the various precipitates were somewhat smaller than that necessary for the formation of normal metasilicates. Table I shows that the  $p_H$ 's at which the silicate precipitates of thorium, aluminium, and zinc separated were also those at which the respective hydroxides begin to be precipitated. In the case of the zirconium titration, the silicate precipitate did not appear until  $p_H$  3.98 was reached, as compared with  $p_H$  1.86 for the hydroxide when precipitated from chloride solution. The curve in the acid zone shows that the change in hydrogen-ion concentration was similar to that which occurred in the precipitations of the hydroxide, basic borate, and basic carbonate (J., 1926, 125). Precipitation was delayed until 3.59 equivs. of alkali had been added, whereas in the hydroxide precipitation opalescence appeared with 1.5 equivs. (Britton, J., 1925, 127, 2133). Delayed precipitation was also noted with the basic borate and carbonate—3.4 equivs. of reactant being required in each case, and the  $p_H$ 's being 4.07 and 3.95, respectively. It is remarkable that the silica in the reagent should have impeded the precipitation until an almost identical  $p_H$ , 3.98, had been attained. The other precipitate formed whilst the mother-liquor was still acidic was that of beryllium; this began to form at  $p_H$  5.31, which is slightly lower than the hydroxide  $p_H$ , 5.69. As in previous work with beryllium, the precipitate did not begin to form until 1.01 equivs. of alkali, as sodium silicate, had been added. The difference in the two  $p_H$ 's may be due the fact that the silicic acid sol formed in the course of the reaction became very unstable on approaching the neutral point. The  $p_H$ 's at which the magnesium and manganese precipitates began to form are con-

siderably less than those of the respective hydroxides. The cause probably lies in the insolubility of silicic acid, which cannot therefore retain a base in solution until the hydroxide  $p_H$  is reached. This is not the case with those precipitates which are formed in acid solutions, for then the acid liberated by the hydrolysis of the salts is sufficient to maintain solution until the corresponding hydroxide  $p_H$  is attained, or nearly so. The interaction of sodium silicate and a solution of calcium chloride produced a curve which shows that when the stoichiometrical amount of alkali, as silicate, had been added, the alkalinity of the mother-liquor was greater than that which would have been set up if all the silica in the sodium silicate had been removed from solution by the calcium precipitate and an excess of alkali had been added instead of sodium silicate. In the other reactions investigated, it is probable that, owing to the great weakness of silicic acid, very little or no combination took place between it and the various hydroxides, the silica in the precipitates having been the result of simultaneous precipitations. It is nevertheless surprising that all the silica attached to those portions of the reagents which were responsible for the precipitations was not itself completely precipitated with the hydroxides. It is, of course, possible that the excess of sodium silicate which was subsequently added peptised some of the silica which had originally been precipitated. The formation of silicate precipitates is probably the result of the mutual coagulation of oppositely charged colloids through the removal by neutralisation of the substances which tended to stabilise or to peptise them, as found by Thomas and Johnson (*J. Amer. Chem. Soc.*, 1923, 45, 2532) in the case of the mutual coagulation of a ferric hydrosol, peptised by ferric chloride, and a silica sol, peptised by sodium silicate.

The precipitation of calcium silicate is, however, of quite a different nature in that calcium hydroxide is fairly soluble in water and is, moreover, sufficiently strong a base to react with silicic acid. The alkalinity of the solution was caused by the considerable hydrolysis of the calcium silicate, and the exceptionally high  $p_H$  of the solution after 39 c.c. of sodium silicate had been added was apparently due to the withdrawal by the precipitate of more silica from the reagent, the precipitate thus containing more than 2 mols. of  $\text{SiO}_2$  to 1 mol. of  $\text{CaO}$ .

#### *Discussion.*

The foregoing observations have an important bearing on the vexed question of the cause of soil acidity. In contrast with the large amount of work done to discover the cause of acidity in soils, very little has been done to ascertain what are the hydrogen-ion concentration relationships involved when any two of the con-

stituents of the soil react. Swanson (*J. Agric. Res.*, 1923, 26, 83) has shown that the acidity arises chiefly from the mineral constituents of the soil, the part played by the organic matter being, in general, negligible. The modern view is that the varying acidity of soils is due to the presence of complex aluminosilicic acids in different stages of neutralisation (*Ann. Reports*, 1925, 22, 203). This idea appears to have been based on some work of Bradfield (*Missouri Agr. Exp. Sta. Research Bull.*, 1923, No. 60), who concluded from a comparison of the properties of soil colloids, separated from clay, with those of a mixture of hydrated silica, alumina, and ferric oxide, that the colloidal material consisted either of a definite compound or of a mixture of compounds in which the oxides were chemically bound as aluminosilicates. He considered that the type of titration curve obtained with the hydrogen electrode when a solution of calcium hydroxide was titrated with a clay suspension was that of a weak acid, and he was therefore led to believe that the colloidal material in the soil was itself the acid (*J. Amer. Chem. Soc.*, 1923, 45, 2669). Saint (*Trans. 2nd Comm. Internat. Soc. Soil Sci.*, Gröningen, 1926, 134) has suggested that if a soil be represented as a partly neutralised acid, the amounts of different bases which any particular soil can absorb should be in equivalent proportions, and has advanced evidence to support the view in the case of the alkalis and the alkaline earths when considered separately, the difference between the two sets of figures being attributed to the formation of insoluble alkaline-earth salts. This, however, would be expected from a consideration of the very similar reactions which the alkaline earths and the alkalis enter into with hydrated silica (compare Figs. 1 and 2).

The reaction involving the formation of the aluminium silicate precipitate affords no evidence of the existence of a complex aluminosilicic acid (Fig. 3). It is true that if the changes in hydrogen-ion concentration are followed when such a precipitate is subjected to treatment with solutions of alkalis or alkaline earths, the precipitate exerts a pronounced buffer action, which *primâ facie* might be regarded as indicating that the precipitate was an acid. This buffer action is, however, the result of a superposition of several reactions, *viz.*, (a) the reaction between the aluminium hydroxide and the base to form a soluble aluminate, buffering between  $p_H$  8 and 10 (*vide* Britton, this vol., p. 424), (b) the reaction between the base and the hydrated silica, giving rise to a gradual rise in  $p_H$  from 6 upwards, and (c) an equilibrium between the silicic acid and any soluble aluminate, *e.g.*,  $\text{Ca}(\text{AlO}_2)_2$ , which may have been initially formed, to give calcium silicate,  $\text{CaO}, x\text{SiO}_2, y\text{H}_2\text{O}$ , and hydrated alumina, producing a hydrogen-ion concentration from  $p_H$  6 upwards.



The amount of soluble aluminate actually formed is doubtless a function of the amount of water present. Similarly, if the inorganic colloids of the soil be considered as mixtures chiefly of hydrated alumina and silica, then the effect on the hydrogen-ion concentration of adding slaked lime to the soil would be determined by the reactivity of the two constituents with the lime. The titration curves of Hissink and van der Spek (*Trans. 2nd Comm. Internat. Soc. Soil Sci.*, Gröningen, 1926, 72) with the hydrogen electrode of soil suspensions all show buffer action between  $p_H$  6 and 8.

The curves given in Fig. 3 also account for the presence of various metals in soil solutions of different  $p_H$  reactions. Magistad (*Soil Sci.*, 1925, 20, 181) has found that aluminium appears in soil solutions only when the  $p_H$  is lower than 4.0—4.5 or higher than 8. The limit on the acid side is seen to correspond with the  $p_H$  range at which aluminium hydroxide or silicate is precipitated (Fig. 3), and, as pointed out previously (this vol., p. 424), its reappearance in alkaline solutions is due to the acidic behaviour of the aluminium hydroxide exerting itself. Many chemists have also found alumina in the neutral-salt extracts of sour soils; thus, by the use of potassium chloride or nitrate, sodium chloride, etc., Veitch (*J. Amer. Chem. Soc.*, 1904, 26, 637) concluded that the addition of sodium chloride to a soil did not liberate hydrochloric acid as such but aluminium chloride, and therefore the acidity was only apparent. Knight's (*Ind. Eng. Chem.*, 1920, 12, 343) analysis of a soil extract with potassium nitrate shows that 90% of the titratable acid was in combination with alumina. Rice (*J. Physical Chem.*, 1916, 20, 214) found aluminium in potassium nitrate extracts of which the  $p_H$ 's ranged from 3.5 to 4.5. Niklas and Hock (*Z. angew. Chem.*, 1925, 38, 195) have titrated potassium chloride extracts of soils with sodium hydroxide, using the hydrogen electrode. The first branch of their curve, corresponding to the decomposition of the aluminium salt, lies at about  $p_H$  4 (compare Fig. 3), and the remaining section shows a buffer action up to just above  $p_H$  8. The acidity of the more acidic soils is due to the normal hydrolysis of the aluminium salts formed in the soil.

The  $p_H$  of soil solutions from sour soils after treatment with neutral salts often becomes still lower; thus Crowther (*J. Agric. Sci.*, 1925, 15, 212) found that soils of  $p_H$  4.78 on treatment with neutral salts gave about  $p_H$  4. Soils of  $p_H > 6$  appear to be unaffected by neutral salts, and their titratable or "mineral" acidity becomes negligible (compare Christensen and Jensen, *Trans. 2nd Comm. Internat. Soc. Soil. Sci.*, Gröningen, 1926, 111). The mineral acidity given by acid soils appears to depend on the extent to which neutral salts, when allowed to react upon soils, are able to cause an increase

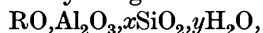
in hydrogen-ion concentration above that requisite for the precipitation of aluminium hydroxide, and thereby to enable alumina to pass into solution as a salt. The fact that the hydrogen-ion concentrations of such soil solutions are nearly the same as that at which aluminium hydroxide is normally precipitated suggests that the soil must contain some aluminium hydroxide in a partly neutralised state (*vide* Britton, J., 1925, 127, 2120). This would, on hydrolysis through contact with water, give a  $p_H$  near that at which aluminium hydroxide becomes precipitable. When a neutral-salt solution is added to such a soil, it is probable that the salt splits up, the basic part combining with the preponderance of silicic acid and being retained by the soil, and the acidic part combining with the partly neutralised aluminium hydroxide to enable some aluminium salt to pass into solution. Thus sodium chloride might be considered to split up into sodium oxide and hydrochloric acid, the sodium oxide attaching itself to the large excess of colloidal silica and thus becoming insoluble, whereas the hydrochloric acid, in reacting with the aluminium hydroxide, causes some aluminium chloride to pass into solution and thereby increases the hydrogen-ion concentration and the titratable acidity. It is significant that this "exchange" acidity disappears almost completely from soils of  $p_H$  6 and above. It is at this point that aluminium hydroxide acquires its optimum insolubility, due to the withdrawal of the small amount of acid radical which it retained on being precipitated. Above this point, it behaves as an acid and consequently is without effect on neutral salts.

Manganese has been found in solutions from certain acid soils. Pollard (private communication) has found it in solutions of  $p_H$  up to 6 from certain sour soils. Rice (*loc. cit.*) found it in potassium nitrate extracts up to  $p_H$  5 of acid soils. Fig. 3 shows that the precipitation of manganese silicate does not begin until just above  $p_H$  7. The  $p_H$  of manganous salt solutions is of the order 5—6, but it must be remembered that, when a manganous salt solution is treated with alkali in a quantity insufficient to start precipitation, the solution rapidly decomposes through oxidation and brown oxide separates. Hence it is unlikely that manganese would be found in a soil solution of  $p_H > 6$ .

Although ferric oxide is often a constituent of soils, very little can pass into the soil solution, except in colloidal form, because it is precipitated from strongly acid solutions (Britton, J., 1925, 127, 2155). It can, however, sometimes be detected in the neutral-salt extracts of sour soils by testing with an alcoholic solution of potassium thiocyanate (Comber's test). Pollard (private communication) found that by adding hydrochloric acid to soils, the

iron content of the aqueous extracts only became perceptible when the solutions had become more acid than  $p_H$  1.5.

The phenomenon of base exchange as exemplified by soils has been attributed to the existence of zeolitic combinations in the inorganic colloidal matter, in spite of the fact that zeolites themselves do not occur in soils. The compositions of most of the zeolites may be expressed by the general formula



where R may represent one atom of a bivalent metal or two of a univalent metal, and  $x =$  or  $> 2$ . For Gans's synthetic permutites  $x$  varied from 3 to 4. They were prepared by the action of alkali-metal aluminate solutions on silicic acid, and not by the action of alkali-metal silicate on aluminium hydroxide. It has been shown (Britton, *Analyst*, 1921, **46**, 363; J., 1925, **127**, 2120; this vol., p. 424) that the amounts of alkali or alkaline earth which dissolve aluminium hydroxide to form the soluble aluminate are approximately the same as those indicated by the formula  $R^IAlO_2$ , viz., either 1 mol.  $R^I_2O$  or 1 mol.  $R^{II}O$  to 1 mol.  $Al_2O_3$ . This happens to be the ratio found in zeolites. Calcium aluminate solution on reacting with silicic acid, as either sol or gel, decomposes between  $p_H$  7 and 9 to give precipitates of calcium silicate (indefinite) and aluminium hydroxide, the aggregate composition of the precipitates being  $CaO, Al_2O_3, xSiO_2, yH_2O$ . Hence, it may be that the "base exchange" exhibited both by zeolites and by soils is due to the similarity of their active constituents.

### Summary.

(1) The reactions between silicic acid and sodium hydroxide or the alkaline earths in solution, and those between solutions of typical metallic salts and sodium silicate, have been investigated by means of the hydrogen electrode.

(2) Although the mode of ionisation of silicic acid cannot be expressed in terms of dissociation constants, its reactions with the stronger bases have a pronounced effect on the hydrogen-ion concentrations of the solutions.

(3) The precipitates obtained by the interaction of a solution of sodium silicate,  $Na_2O, 2 \cdot 16SiO_2$ , and metallic-salt solutions, except that of calcium chloride, contained less silica than that required to form the metasilicates.

(4) The hydrogen-ion concentrations at which these silicate precipitates began to form in solutions of  $p_H$  less than 7 were approximately those at which their corresponding hydroxides separate. The silicate precipitates obtained from alkaline solutions appeared

when the hydrogen-ion concentrations had not been reduced to the extent required by their respective hydroxides.

(5) The results have been discussed in relation to the question of soil acidity.

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