70 ± 3 F. and a relative humidity of not less than 90 percent. The length of the curing period may be 7 or 28 days.

TESTING OF SPECIMENS

After specimens are cured, they are

completely immersed in distilled water at about 70 F. for 24 hours. Each specimen 1s then tested for unconfined compressive strength. The rate of deformation during testing is 0.1 inch per minute. The maximum test load causing failure of the specimen 1s taken as its compressive strength.

Soil Stabilization with Resins and Chemicals

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Introductory Remarks by the Chairman: Many federal, state, and private agencies have made essential contributions to the practice and science of soil stabilization. Most noteworthy among these are the Bureau of Public Roads, the state highway departments, the engineering departments of the Army, the Navy, and the Airforce, the Civil Aeronautics Administration, and the Bureau of Reclamation.

It is natural that the quality and quantity of contributions of these and other agencies varied with time, one or another being leading at one particular period. At one time, there was a golden age for the Civil Aeronautics Administration with respect to soil and soil stabilization research when Grieme had assembled a staff of keen and active men. Among these were David S. Jenkins, now director of the Saline Water Conversion Program of the Department of the Interior; George W. McAlpin, now chief soils engineer of the State of New York; and R. C. Mainfort, presently with the Dow Chemical Company.

During this period the CAA not only sponsored at various universities research that was of utmost importance and has by now largely become classical, but organized its own laboratories for further evaluation and supplementation of this research. Mainfort, who remained with the Soils and Pavement Research Section of the CAA until this section was dissolved, has enriched this symposium with a concise and condesned review of the work he directed while associated with the CAA.

•FOR several years various organizations have investigated the possibility of using chemical additives to alter the characteristics of natural soils in order that they might be more successfully used as a structural material. The desired effect might be obtained by bonding, waterproofing, or otherwise modifying the natural soil so that the resulting mixture can withstand the detrimental forces of weather, moisture, and load application. From the standpoint of highway and airport construction, the application of such techniques should permit the economical and efficient utilization of soils for the construction of durable wearing surfaces and base courses.

The improvement of natural soil by the addition of chemical admixtures is generally referred to as "chemical soil stabilization." For the purpose of this report, the term is used to describe any method whereby the engineering properties of natural soils are improved by the chemical or physicochemical interaction between an admixture and a soil and includes the use of such general materials as cement and bitumen.

Most of these soil stabilizers fulfill their function by imparting their own properties to the resulting mixtures. Recent studies, however, indicate that a group of substances, referred to as "trace chemicals," are capable of altering soil properties almost entirely by interaction with the surfaces of the soil particles (1). Such surface-active substances show promise of being effective in quantities in the range of 0.1 to 0.5 percent by weight of the soil.

The overall testing of prospective soil-

stabilizing agents has covered a considerable period of time and has been the work of different investigators. For this reason, and because of the fact that uniform test procedures were not employed, the relative effectiveness of various admixtures can be presented only in a quantitative way.

GENERAL FESTING PROCEDURES

Other than the methods used for evaluating the effectiveness of soil-cement mixtures, there are no recognized standard procedures for testing the acceptability of chemically treated soil samples. Different investigators have used different methods, depending upon their own requirements or particular definition of a successful soil-stabilizing agent.

In many of the earlier investigations, mild exposure tests were used, and in some cases the treated samples were subjected only to a few hours of capillary moisture absorption or immersion. Certain materials tested in this manner appeared to have stabilizing properties which, under more severe laboratory and field exposures, proved to be merely temporary. As additional test data have become available, the trend has been toward more-severe laboratory-testing procedures supplemented by small-scale fieldweathering studies.

Observation of field-weathered samples indicates that a properly stabilized soil should be able to withstand not only moisture absorption but also numerous cycles of freezing and thawing, wetting and drying, or other temperature variations in the presence of moisture. One form of laboratory-testing procedure in which the results compare closely to those obtained by field weathering has been reported by the Civil Aeronautics Administration (2).

An important factor in any laboratory evaluation test is the type and amount of curing allowed a sample prior to its subjection to exposure. The principal methods that have been used are (1) moistcuring, (2) oven-drying to a constant weight at 140 F, (3) air-drying to a constant weight, and (4) partially drying to a predetermined moisture content.

A partial drying of the sample is probably nearer to the field conditions likely to be attained, but this procedure is difficult to control and duplicate in the laboratory. For this reason, air-drying to constant weight or moist-curing are generally used. Materials requiring moisture for proper hardening (e.g. portland cement) should be moist-cured prior to any desired air drying.

Oven drying is advantageous for obtaining a uniform drying rate which can be easily auplicated and is suitable for curing natural soils and those treated with some admixtures. For admixtures such as resins and bitumens, however, the application of heat results in an improvement to the treatment that cannot be duplicated during normal construction procedures.

In order that the general effectiveness of proposed soil-stabilizing agents can be determined, they should be tested with a variety of soil types. The differences in soil composition and their corresponding reaction to chemical treatments have been the largest variables encountered in soilstabilization studies.

In most testing procedures the natural soil obtained from the field is air dried, screened to a selected top size (usually through the No. 4 or No. 10 screen), and classified by standard soil-testing methods. The maximum density and optimum moisture are determined, and treated samples are molded to predetermined density by dynamic or static compaction. For important evaluations of more-promising stabilizers, the densities should be determined with the required admixture in the soil. After molding, the samples are cured and then subjected to laboratory or field exposure. The relative effectiveness of the treatment can be evaluated at the end of the test by an inspection of the sample, measurement of its moisture content, and determination of its compressive strength.

INORGANIC CEMENTING MATERIALS

Several inorganic cementing materials are capable of hardening or otherwise modifying the physical characteristics of soils, but none have been found which will impart appreciable waterproofing characteristics to the resulting structure. Inorganic cementing agents are particularly susceptible to moisture attack during conditions of freezing and thawing. A soil that has been successfully bonded will absorb considerably less moisture than the same untreated soil. This condition is usually a result of the inability of the bonded sample to swell, rather than a result of any waterproofing action of the treatment.

A relatively high percentage of an inorganic cementing agent (10 percent or more) is normally required for properly bonding a soil. Smaller quantities, however, are sometimes effective in improving the structural properties of soils by modifying their physical and chemical characteristics. Portland cement, lime, sodium silicate, and crushed slag are particularly effective in reducing the plasticity of fine-grained soils.

Portland Cement

Portland cement probably has been used more widely and with greater success than any other soil-stabilizing material, and its properties in this respect have been reported by numerous investigators. Although an excellent bonding agent for many soils, portland cement does not impart water-repellent characteristics to the treated soil; and under certain climatic conditions, especially where wide temperature variations prevail, it has not always proved to be a durable admixture. In addition to the highly plastic soils, which are not recommended for cement treatment, there exist certain soils of good physical characteristics which cannot be successfully stabilized with cement (2, 3, 4).

Various additives have been used in an effort to waterproof soil-cement mixtures, but none of these have been significantly effective under severe testing conditions. Resins and salts of stearic and of oleic acid produce temporary water repellency but are not effective under prolonged weathering. Some of these additives reduce the strength of the soil cement. Calcium chloride has been successfully used to improve the susceptibility of certain soils to cement treatment (3).

Even without admixtures cement has proved to be the most-generally satisfactory material for stabilizing fine-grained soils. Additional study that might extend its usefulness appears to be fully warranted.

Lime

A considerable amount of work has been conducted by numerous investigators to determine the suitability of lime as a soilstabilizing agent. Some bonding of soil has been obtained using lime treatments of 6 percent and higher, but the samples are particularly susceptible to the disintegrating action of cycles of freezing and thawing. Lime-treated samples have failed completely during one winter of exposure (2). Numerous additives have been used in an effort to improve the soil-stabilizing characteristics of lime, but none have proved to be particularly effective.

The physical properties of plastic soils are considerably modified by the addition of small quantities of lime. The plasticity is reduced and the soils become more friable and are much easier to mix and mold to uniform density.

Favorable results have been reported in which soils were treated with a combination of lime and flyash (5), Purdue University (6) and the Texas Highway Department (7) have recently conducted valuable studies concerning the soil-modifying properties of lime.

Sodium Silicate

The use of sodium silicate is another method which has received considerable attention for solidifying soils. Good bonding of sandy soils has been attained with treatments of 6 percentor more of sodium silicate, but such treatments will not withstand attack by moisture. Sodium silicate is not effective with fine-grained soils. Earlier studies (8) indicated that the addition of sodium aluminate increased the resistance to moisture attack of samples treated with sodium silicate, but later tests using a variety of soils and moresevere exposure tests have clearly shown this combination to be unsuitable as a soilstabilizing agent.

The most-effective form of sodium silicate found for soil-stabilization purposes is a 40-percent solution, 40 to 42 degrees Baumé, with a ratio of 1 part of Na₂O to 3. 25 parts of SiO₂.

Combinations of sodium silicate with a strong inorganic salt, such as calcium chloride, have been used successfully for stabilizing medium- and fine-sand masses in deep-foundation construction (9). In this method the two chemicals, which are usually applied separately by pressure injection, react within the sand pores to form an impervious gel which, upon hardening, binds the sand particles into a solid mass. Sodium silicate alone, and in combination with other chemicals, has also been used to temporarily harden sandy soils to such an extent that excavation work could proceed without the use of shoring.

Powdered Slag

Powdered slag provides only slight bonding when mixed with soils in quantities up to 20 percent. The addition of small quantities of crushed slag considerably modifies the characteristics of the moreplastic soils. In this respect, the admixture reacts with the soil in the same way that cement or lime reacts; it reduces the plasticity and allows the soils to be more uniformly and easily mixed.

Other Additives

Inorganic soluble salts, such as sodium chloride, calcium chloride, barium chloride, copper sulfate, and barium sulfate, are ineffective for stabilizing fine-grained soils where waterproofing or bonding qualities are required. The plasticity of some soils has been reduced by the addition of calcium chloride, but the permanency of such treatment is questionable. Both calcium chloride and sodium chloride have been used successfully to enable granular roads to retain a moisture content beneficial to stability and to facilitate further densification and increased stability of the road under the action of traffic.

Oxychloride cements, zinc oxide, and plaster of paris do not set up well when mixed with soils in reasonable quantities. Natural and air-entrained cements are not as effective as portland cement.

BITUMINOUS MATERIALS

Bituminous materials have been used extensively for soil stabilization, and their properties in this respect have been thoroughly investigated in the laboratory and field by numerous agencies. A brief summary of the methods used for testing and applying these materials has been compiled by the Highway Research Board (10).

Bituminous materials have not been particularly effective when used with finegrained soils. It is difficult to obtain a thorough mixture of the bitumens and the finer-grained soils, even though mixing methods recommended by the individual producers are followed closely. In an effort to determine the most-effective mixing procedure, bitumens have been added to prepared soil under the following conditions: (1) air-dried soil, (2) soil at optimum moisture, (3) soil above optimum moisture (near liquid limit), and (4) soil at different intermediate moisture contents. None of these variations in mixing procedure appears to improve the resulting stability of samples. More-nearly uniform dispersion is obtained when bituminous materials are added to soil at a moisture content well above optimum and mixed in slurry form. In this case, the mixture should be dried back to near optimum moisture before molding. All bitumens provide a certain degree of waterrepellency to treated soil, but the resulting mixture is particularly susceptible to the destructive action of alternate freezing and thawing.

The stabilizing properties of bituminous materials, particularly MC-2, RC-2and asphalt emulsion, have been improved by the individual addition of small percentages of lime, resorcinol-formaldehyde resins, amiline-furfural resin, and zinc stearate (2).

Bituminous materials have been and are being successfully used to stabilize and waterproof sandy soils.

RESINOUS WATERPROOFING MATERIALS

For the purpose of soil stabilization, resinous waterproofing materials are considered to be those natural or synthetic resins whose chief function is to maintain the moisture content of a soil at or below optimum moisture by preventing entry of water into the treated and compacted mixture. Little or no cementing action is obtained from these materials. Unlike bonding agents, whose effectiveness generally increases with the quantity used, waterproofing agents usually attain maximum effectiveness when applied in small quantities—2 percent or less by weight of the treated soil.

Although imparting desirable characteristics to the soil and providing considerable waterproofing effect under mild exposure, none of the water-repellent resins appears to be a suitable soil-stabilizing agent at the present time. It has been suggested that soil-bacterial activity may have a detrimental effect on the permanency of organic soil stabilizers such as bituminous and resinous materials (11).

Stabinol

Stabinol is composed of 80 percent of portland cement and 20 percent of a complex resinous compound. When used in small quantities, the cement fraction cannot impart appreciable bonding to the treated soil but acts as a carrier for the resinous material and as a possible modifier for the soil to allow more effective use of the resin. A maximum 2-percent treatment usually has been recommended for water-repellent purposes.

Initial laboratory tests of the material were quite promising (12). In further studies involving field testing, moresevere laboratory exposure, and the use of a wider range of soils, Stabinol was not effective unless applied in sufficient quantities to enable the cement fraction to become active as a bonding material. Used in larger quantities, however, Stabinol cannot be considered as strictly a waterproofing material.

Resin 321

The effectiveness of Resin 321, a partially neutralized salt of abietic acid, as a soil-stabilizing material has been previously reported (2, 12, 13). These reports indicate that Resin 321 is an excellent waterproofing agent for certain soils and that its water-repellent characteristics can be utilized for improving the effectiveness of other stabilizing materials. However, the beneficial qualities of Resin 321 are reduced considerably under severe exposure.

Vinsol and NVX

Vinsol and NVX (a neutralized form of Vinsol) are powdered resins which, like Stabinol and Resin 321, function to impart water-repellent characteristics when mixed with soils. Vinsol is superior to NVX but inferior to both Stabinol and Resin 321 (2, 12).

Other Resins

A considerable number of natural and partially neutralized natural results have been investigated. These include Belro, Copal, Kauri, Batavia Damar, and Elemi resins. All of these are slightly beneficial as soil-waterproofing agents but are recommended only for temporary emergency use in areas where they are locally available.

Other resinous materials which have been tested as soil stabilizers are Soilpak (approximately seven parts lime to one of resin), Valite (a sugar resin), commercial powdered rosin, lignin sulfate, and partially neutralized tall oil. None of these are particularly effective.

RESINOUS BONDING MATERIALS

The term "resinous bonding materials," as applied to soil stabilization, refers to those natural or synthetic resinous materials which cement or bond the particles of soils with which they are mixed. Some of these resins are also good water-repellent materials. Approximately 5 percent of the resin has been arbitrarily selected as a maximum treatment in most studies, but testing of higher percentages indicate that their effectiveness increases with the quantity used. Some resinous materials, however, show considerable effectiveness when used in quantities of less than 5 percent.

Although several of the synthetic resins show considerable promise as soil-stabilizing agents, this form of treatment is still in the experimental stage. The present high cost of resins, compared with conventional methods of soil treatment, is an important factor to be considered in their possible use as soil-stabilizing agents. Initial phases of the work with synthetic resins have been reported by several agencies (2, 14, 15, 16).

Aniline-Furfural Resin

The soil-stabilizing properties of the result formed by the interaction in the soil of two parts of aniline and one part of furfural has been thoroughly investigated and reported (2, 14, 17).

Antline and furfural begin reaction upon contact; therefore, it is necessary to add the chemicals to the soil separately. Unless special catalysts are used, the reaction between aniline and furfural in moist soil is slow. Treated samples are easily molded within at least 8 hours after mixing. Samples which were moist cured prior to testing are inferior to those permitted to air dry or oven dry, indicating that complete resinification of aniline and furfural does not take place under moistcure conditions.

Laboratory and field testing indicate that aniline and furfural under air-dry conditions form one of the best soil-stabilizing agents, one which is effective over a wide range of soils. In some tests aniline-furfural-treated soil samples have proved to be superior to those treated with even higher percentages of cement.

Furfural plus aniline sulfate and furfural plus aniline chloride are also effective stabilizers, but to a lesser degree than furfural plus aniline. Two parts of aniline sulfate to one part furfural was the most effective of these combinations.

Aniline-furfural mixtures are the cheapest of the synthetic bonding resins which have been considered for soil stabilization. Aniline is toxic and must be handled with considerable care.

Resorcinol-Formaldehyde Resin

Certain resorcinol-formaldehyde-resin formulations are able to harden soil under moist-cure conditions at room temperature (2). Samples treated with this material and stored in a moist cabinet become quite firm within a few hours after mixing. Air-dried samples are equally as stable as those which are moist cured.

These resins are usually furnished in liquid form and require the addition of 15 percent, by weight of the resin, of an aldehyde hardener to accelerate and complete the resinification.

At least 5 percent of this treatment is required for effective soil stabilization, but higher percentages of treatment produce higher stabilities. Although not an outstanding stabilizing agent, resorcinolformaldehyde resins have shown considerable promise, even under severe laboratory and field exposure.

At the present time this type resin is far too costly for consideration as a primary soil-stabilizing agent. However, it appears to be suitable for improving other more economical admixtures, particularly the bituminous materials. The stabilizing properties of MC-2 and asphalt emulsion have been particularly benefited by the addition of small quantities of this material.

Phenol-Formaldehyde Resin

Several forms of phenol-formaldehyde resin have shown promise as soil stabilizers (2). The outstanding material of this group was a two-stage-type resin, which required the addition of 10 percent of hexamethylenetetramine for obtaining proper set. Five percent treatment was used for most of the tests, although higher percentages were more effective.

This type reacts with the soil in a manner similar to resorcinol-formaldehyde resin, although it is not so effective. It sets well at room temperature during moist curing or air drying. Although quite promising in laboratory tests, all samples failed during field exposure. Numerous catalysts were used in an effort to improve its effectiveness, but none were particularly beneficial. Magnesium stearate, zinc stearate, potassium permanganate, and ammonium chloride were of slight benefit with certain soils.

Urea-Formaldehyde Resins

Several forms of urea-formaldehyde resins have been tested as soil stabilizers (2). The more effective of these are produced in powder form and are precatalyzed so that no activator is required to insure complete reaction. They set up with equal effectiveness under moist- and air-cure conditions.

The addition of urea-formaldehyde resin tends to dry a moist soil and to disperse or fluff the particles. This action greatly reduces the density and increases the optimum moisture obtained by a given compactive effort as compared with untreated soil.

This form of soil treatment was not applicable over a wide range of soil types.

Furfuryl Alcohol Plus Sulfuric Acid

Some stability has been imparted to sandy soils by the resin formed from the interaction of furfuryl alcohol and sulfuric acid (2, 8). In this method of treatment the furfuryl alcohol and the required acid are added to the soil in separate portions of the mix water. Sulfuric acid has been used for most tests, although other strong inorganic acids are also effective informing the resin. The reaction of furfuryl alcohol with strong acid solutions is very rapid, requiring considerable care in hanvarying the concentration of the acid. Promising results were obtained by this method of treatment in preliminary work, using a sandy soil, particularly when the samples were oven dried prior to testing. However, the treatment was found to be only slightly effective with other types of soil under air-dry or moist-curing conditions. Freeze-thaw and field-weathering tests were particularly destructive to samples treated with furfuryl alcohol.

The furfuryl-alcohol treatment is muchmore expensive and far-less effective than that using the aniline-furfural mixture.

Calcium Acrylate Resin

An extensive program of research and development concerning the soil-stabilizing properties of calcium acrylate is at present under way at the Massachusetts Institute of Technology for the Army Research and Development Laboratories (<u>18</u>).

Calcium acrylate is a water-soluble white powder which forms a hard resin when mixed with certain activators. Present recommendations for its use as a soil treatment are 10 percent calcium acrylate plus 1.36 and 1.25 percent sodium thiosulfate and ammonium persulfate, respectively. All of these quantities are based upon the dry weight of the soil. The hardening time of the resin can be controlled by varying the quantities of the activators.

In some respects, particularly from a military standpoint, the action of this material approaches the ideal requirements of a soil stabilizer. It can be added to extremely wet soils and in a matter of minutes transform the mass into a dry solid structure capable of sustaining considerable load application.

When properly mixed, calcium-acrylate-treated samples are quite resistant to laboratory-weathering tests. The ratio of the activators to the calcium acrylate is critical and should be such that the combination does not begin to set before it has been thoroughly mixed with the soil. The treatment appears to be applicable to a wide range of soil types. With certain soils its effectiveness can be improved by the individual addition of small quantities of Resin 321 and calcium stearate.

Tung Oil

The results obtained from the laboratory testing of samples treated with tung oil have been reported (8, 19). Although the data contained in these publications indicate that tung oil is a promising stabilizer with certain soil types when airdried, its use is not economically feasible for normal construction purposes in this country. The test results show that tungoil-treated samples were not particularly effective under severe exposure. Tung oil is superior as a soil stabilizer to all other oils studied, including linseed oil, soya oil, tall oil, and several synthetic drying oils. The addition of calcium oxide improved the effectiveness of tung oil to a small degree.

Other Resins

Several other resins have been investigated for possible use in soil stabilization, but none are particularly effective. These include melamine-formaldehyde, styrene, coumarone-indene, polyterpene, and methyl-methacrylate resins.

CONCLUSIONS

At the present time there appears to be no entirely successful method for stabilizing soils by the use of chemical admixtures. Although several materials are suitable under specific conditions or with particular soils, none have proved to be universally acceptable.

Portland cement is the most-generally effective soil-stabilizing material, but its characteristics in this respect could be improved. A suitable waterproofing additive for soil-cement mixtures has not yet been found.

The resin formed by the interaction of two parts aniline and one part furfural is a very good bonding agent for soil and the most-effective waterproofing material studied so far. Under certain conditions this treatment is effective in quantities as low as 2 percent of the dry weight of the soil.

Calcium acrylate activated by sodium thiosulfate and ammonium persulfate is highly successful in transforming wet, plastic soils into firm, durable structures. Although only in the experimental stages of development, this material appears to have good possibilities as a soil modifier and stabilizer. Like other synthetic resins, however, it is too expensive at the present time to be considered for normal construction purposes.

Other resinous materials have shown some promise as soil stabilizers, but their principal value appears to be as additives to nonconventional and cheaper paving materials. Resorcinol and phenolformaldehyde resins have considerably improved the soil-stabilizing effectiveness of bituminous materials.

Because of the extremely variable and complex chemical characteristics of different soils, it is probable that no universally suitable soil-stabilizing agent will be found. It may be that the most we can expect to develop is a chemical which, although not specifically furnishing bonding or waterproofing characteristics, will otherwise modify natural soil to such an extent that it will be more useful for structural purposes. Development of an economical and durable material which would reduce the plasticity and improve the shrinkage and swelling characteristics of soil would be a valuable contribution to soil science. Such materials as cement. lime, sodium silicate, aniline-furfural, and calcium acrylate have shown considerable promise in this respect.

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Effect of Calcium on the Continuity of Electroősmotic Flow Rate

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Introductory Remarks by the Chairman: Large-scale use of the electroösmotic phenomenon appears to have been mainly in the drainage of fine-grained natural soils. For this, relatively low electric potentials are employed, and interference by chemical and polarization phenomena has been reported to be practically negligible. Chemical phenomena, however, have been utilized in electroösmotic stabilization of friction pile systems and have been observed in laboratory studies on soils in which the exchange ions were varied.

Much experimental work with chemically well-defined soils and employing a wide range of electric potentials remains to be done before electroösmosis can become a dependable tool in soil stabilization. It is gratifying, therefore, that the Engineering Experiment Station of Iowa State College has attacked this problem on a wide front and that Rollins is presenting us with the first results of this attack on the effect of calcium on the continuity of the electroösmotic flow rate.

● THE bearing capacity of a subgrade is a function of moisture content. A large number of pavement failures can be attributed to the loss in subgrade support which takes place when excess moisture accumulates. It follows that any method of controlling or reducing this accumulated moisture falls into the category of soil stabilization.

A number of investigators have shown that moisture content beneath pavements fluctuates throughout the year, increasing during the cold months and decreasing during the warm periods.

The most-critical time, so far as subgrade bearing support is concerned, is during the thawing period in the early spring. If some means were available for removing the excess moisture at that time, it seems reasonable to expect that the performance of the pavement would be considerably improved.

One possible means of removing this excess moisture which appears to have some possibilities is electroösmosis, or the movement of moisture by an electricalpotential gradient.

PREVIOUS WORK IN ELECTROŐSMOSIS IN SOILS

Space does not permit the inclusion of a complete review of all previous investigations in electroősmosis of soils; however Casagrande (2) has reviewed the subject in a recent publication. Unfortunately, in many cases too little attention has been paid to the physicochemical characteristics of the soil, and that makes it impossible to project the results to other areas.

Winterkorn (6) has presented data showing the relative electroösmotic permeabilities for homoionic modifications of a soil involving the usual cations likely to be associated with the electroösmosis of soils. The flow of water for constant amperage was very slow for Ca, Na, and K and contained a high percentage of suspended colloids. Mg and Fe soils gave a regular and clear flow up to approximately one hour, while the current density had to be reduced for A1 and H systems early in the experiment when the resistance of the sample became so large that the power sup-