THE CHEMICAL AND PHYSICAL INVESTIGATION OF SOILS.[1]

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I DESIRE to place before this Congress, without going into analytical details which will be set forth in a special report to the Association of Official Chemists, the general results of my long-continued studies on direct soil examination by physical and chemical means that have led me to attach to such examinations a far greater practical importance than is now usually assigned to them by the consensus of opinion both in Europe and America.

I premise that I was led into these investigations more particularly as a consequence of my almost continuous residence in the newer, thinly-settled or unsettled portions of this country, where the question of the fitness of lands for general agriculture, and their special adaptations are burning questions. which are there constantly pressed upon the attention of whosoever occupies a public position in connection with agricultural colleges or experiment stations. My researches have, therefore, borne essentially on virgin soils, or at least such as had been cultivated only for a short time and had received not even incidental fertilization.

With material so different from that which in Europe and in the eastern United States have led most agricultural chemists to consider direct soil examination, and especially chemical soil analysis, as of little practical value, it is not surprising that I should have been led to conclusions differing somewhat widely from those commonly received; so that, while still agreeing that in the case of soils long fertilized, chemical analysis can give but little information as to the immediate producing power, I am satisfied that it can in the case of virgin soils be made of the most direct and vital use to the farmer and intending settler.

The substitute recommended for direct soil examination, after the sweeping condemnation of soil analysis some fifty years ago, was and still is, to put the question to the soil by cultivation and fertilization with the simples, potash, phosphoric acid, and nitrogen; and to deduce therefrom the needs of the soil. It

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[1] Read before the World's Congress of Chemists, August 24, 1893.

goes without saying that this is but unpalatable advice to give to a settler in new regions; while even to the farmer in the older countries the carrying-out of a cogent experiment is by no means so easy a task as the precept implies. Moreover, the latter leaves room for great uncertainty in respect to seasonal differences; and the physical peculiarities of the soil are therein left totally out of consideration, although these are just as frequently in fault as the chemical composition, and the farmer is by no means usually to be credited with the ability to determine these points.

In my view, therefore, the examination of soils for the benefit of the agricultural population is one of the most needful, and in the newer states most urgent tasks set before the experiment stations; provided, of course, that the results can be successfully applied to practice.

My attention has from the outset been strongly drawn to the accuracy and certainty with which experienced farmers will, in a region familiar to them, judge of the quality and adaptations of land by its natural vegetation; more particularly by the forest growth. It is obvious that if we could interpret correctly the physical and chemical qualities of soils that have determined the choice of certain plants that occupy them as a result of secular co-adaptation by the survival of the fittest, we should have as definite and much more cogent data than can be supplied us at best by the brief culture experiments we can make. This thought suggested itself to me in noting the invariable recurrence of certain plants on soils showing obvious physical and chemical peculiarities, such as "stiffness" or "lightness" on the one hand, and richness in lime or humus on the other. I therefore determined to test the idea by actual, systematic examination of the soils in all their relations; and it is upon the results of this study, and of their *comparison with actual agricultural practice*, that my views in the premises are based. It need hardly be recalled to mind that, such relations once established, the conclusions flowing therefrom can be legitimately, and as experience shows, fruitfully, projected into cases in which the actual indications of natural vegetation are not available.

The first point requiring attention in such researches is, naturally, the observation of the soils in place, and the recording of all the peculiarities whether of occurrence, structure derivation, depth, vegetation, local and general climate, and if possible, cultural experience. The taking of the sample to be examined must, of course, be made to conform to the particular conditions of each case, and not to arbitrarily prescribed precepts, above all things. All accidental, abnormal variations must be excluded by a proper selection of the localities, and by sampling the same soil in several places. That it is thus possible to obtain specimens correctly representing in all essentials a wide extent of country without excessive multiplication of samples, my experience has fully proved. The depth to which the "soil" sample should be taken is ordinarily the lower limit of coloration by humus; but should that depth exceed twelve inches, the presumable maximum depth of tillage, one sample should be taken to that depth only. The subsoil is what lies beneath that line of coloration: it is frequently advisable or necessary to sample the soil mass for each twelve inches down to six feet depth, since the nature of the subsoil is one of the most essential points to be observed in all cases.

The first operation I undertake upon any soil sample is to wash about ten grams of it in a beaker with a water current of definite velocity, while stirring actively with a rod. The residue of sand or gravel is examined, macroscopically and microscopically, for the determination of the chief constituent minerals and of their surface condition, whether sharp or much rounded. We thus gain at once a pretty definite estimate of its relations to its place of origin and of its parent rocks, and therefore of its probable general chemical character, it being presumable that the fine matter is of the same general nature as the coarse.

Then follows the "hand test" of the soil by crushing small lumps between the thumb and forefinger, dry at first; then, after wetting, observing the change of color on wetting, and then kneading, in order to test its contents of plastic clay.

The next step, logically, is the test of the relations of the soil to water or moisture. Since the perviousness to water coming from above is so variable according to the accidental condition and the *modus operandi*, I prefer to determine, as more definite factors, the water capacity and the hygroscopic coefficient, both of which give very definite information in respect to the most essential physical properties of a soil.

In determining the *water capacity* it is quite essential to determine both the *maximum* and the *minimum*, and that in the lowest column of water with which one can operate; I place this at one cm. I use a cylindrical vessel with perforated bottom resembling the "test-lead sieve" of Plattner's blow-pipe chest, of twenty-five or fifty cc. capacity. This is filled with the fine earth, and after weighing placed in a shallow dish with a layer of water. Within about an hour it is saturated to the maximum capacity; it is then weighed, which gives the maximum. Then the wet soil is covered with soil, first air-dry, then with such as has been saturated with moisture, until its weight ceases to decrease by liquid absorption. The last weighing determines the minimum water capacity; the difference between the maximum and minimum indicates the greater or less height to which the soil will raise capillary, liquid water.

The hygroscopic coefficient, which contrary to prevailing doctrines I find to be of great importance to the welfare of plants in the arid regions, must be determined by exposing the soil to a fully saturated atmosphere for at least seven hours in a layer 1 mm. thick. Under such conditions, the amount absorbed remains practically the same within ordinary cellar temperatures, contrary to what (according to Knop's law) happens when the air is only partially saturated. We thus obtain a datum which bears a very definite ratio to the clay and humus contents of the soil and its drought-resisting qualities. But in order to obtain comparable results, the drying of the soil must be done at 200° C., for at 100° soils of high absorptive power will continue to lose moisture for weeks. The drying is therefore done in a paraffin bath. From the drying tube or bulb the quantities for the chemical determinations are weighed out.

In the question of the maximum size of grain to be admitted to the chemical analysis, I have also sought to determine by direct experiment the proper limit; instead of assuming an arbitrary one as is usually done, with the result that the chemists of each country have adopted a different rule, and thus their work is rendered incapable of comparison. Thus the German stations have adopted a two mm. mesh for the fine earth sieve, while the French official rule is "one mesh to the millimeter," leaving the aperture an uncertain quantity because dependent upon the thickness of the wire used. As a matter of fact the aperture is about 0.75 mm. In this country one mm. clear aperture has been adopted by the majority of chemists. Thus it becomes impossible to compare with any degree of certainty the analyses of these different countries.

In 1872, having perfected my apparatus for mechanical soil analysis,[1] I caused an investigation of the question to be made by my assistant, Dr. R. H. Loughridge. The result (see *Am. J. Sci., January, 1874*) was to show that in the very generalized soil worked upon, seventy-five per cent. of the dissolved soil ingredients was contained in the colloidal clay remaining suspended in water after twenty-four hours, in a column 200 mm. high; and that solution by the hydrochloric acid employed practically ceased when the diameter of 0.025 mm. (0.5 mm. hydraulic value) was exceeded. It was thus apparent that the finest practicable mesh might safely be used for the "fine-earth"; and this I found to be at 0.5 mm. aperture, equal to sixty-four mm. hydraulic value. That for direct comparison by percentages it would be better to come down a much smaller grain-size, is doubtless true; but this degree of fineness would involve elaborate preparations, and difficulties that would render such investigations few and far between. I have, therefore used the 0.5 mm. mesh in all soil analyses made since under my direction; and as the sieves used in soil work of the surveys of Kentucky and Arkansas were very nearly of the same character, that work remains comparable with mine. But whatever may be the size chosen by any one, the quantitative determination of the grain-sizes of one mm. and two mm. diameter should be made a rule, in order that allowance may be made for them when comparisons are desired.

The preliminary tests made should determine, in a measure

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[1] I omit any detailed reference to this apparatus and the subject of mechanical soil analysis for the reason that the matter has of late had considerable discussion in journals and is presented in full detail in a report to the Association of Official Chemists.

the quantities which it is desirable to use for analysis; since great inconvenience often arises from handling excessive amounts of the iron-alumina precipitate. With such balances and methods as we command at present, I do not think there exists any need of employing hundreds of grams in any ordinary cases. If in the case of ores the selling price is allowed to be governed by the assay of a few grams, it is difficult to see why in the case of a soil we should do otherwise, provided the sample has been well taken and prepared so as to be representative. If that has *not* been done, the taking of fifty grams instead of five will not help the matter much.

I usually weigh out from two and a-half to three grams for general analysis; from three to five for the (separate) determination of phosphoric acid by the molybdate method. When in a strongly clayey soil there is a great deal of calcium carbonate, so much alumina is usually dissolved as to render the handling even from the above amounts troublesome, and parting is best resorted to.

There has been a great deal of discussion in regard to the strength of the solvent to be used in soil analysis. All admit that carbonated water is too feeble to approximate to the assimilative power of plants; while "Aufschliessung" with hydrofluoric acid goes far beyond. It has appeared to me from the outset that we should, if possible, ascertain the possible maximum solvent effect that can be expected of *any* plant; and from the fact that calcium oxalate is of so frequent occurrence in roots, I have thought that oxalic acid might be regarded as the strongest solvent at their command. Now, as is well known, oxalic acid expels from the corresponding salts both hydrochloric and nitric acids. In general, therefore, it stands on a par with these; and it is from this standpoint that I originally determined to use, as the most convenient solvent, hydrochloric acid of medium strength. I have since had the question tested directly by substituting oxalic for hydrochloric acid in comparative analyses of one and the same soil; and while there were differences due to the difficultly soluble compounds formed by oxalic acid, yet the interpretation of the two analyses would, for practical purposes, have been precisely the same.

A series of experiments made by Loughridge in 1873 on one and the same soil with hydrochloric acid of different degrees of concentration showed that the maximum solvent effect was exerted, not by the strongest acid, but by that of about 1.115 sp. gr. I therefore adopted this as the standard acid for soil analysis. Another series of experiments showed that the action was made practically complete by a digestion on the steam bath for five days; this also was, therefore, adopted as the regulation time for the acid digestion, which is always done in a porcelain beaker covered with a watch glass, only half immersed in the steam so as to permit condensation of the acid volatilized. My practice has been to take ten times as many cc. of acid as grams of soil weighed out.

The analysis is then carried out essentially like that of any silicate, including the determination of the silica soluble in sodium carbonate solution, which is a very important datum in estimating the degree of decomposition and zeolite formation that has occurred in the soil. In most cases the silica so determined permits of being assigned to the alumina dissolved, as kaolinite; but to this there are numerous exceptions, there being often twice as much alumina as can be assigned to the silica set free by the acid. The only possible form in which this excess of easily soluble alumina can be present is that of *aluminum hydroxide*; which must, therefore, be considered as one of the normal constituents, notably in the case of highly calcareous, and especially of "alkali" soils.

The phosphoric acid is determined by the molybdate method in a separate and larger portion of material, which is previously ignited in order to determine the "loss by ignition." This latter item, while serving to make the result of the analysis sum up to 100 or thereabouts, is really of no practical significance: since it is made up, besides the combustible matter, of water of hydration from the kaolinite as well as from the various hydrates present, and in calcareous soils of carbon dioxide, which can not be restored by treatment with ammonium carbonate, but must, if necessary, be determined in a separate portion and properly deducted. The combustible portions of the soil farther consist of two distinct portions, namely, humus ready-formed.

and unhumified vegetable matter; so that even if we were able to segregate the water belonging to the several hydrates, we would still be unable to estimate the true humus contents. This can only be done by the method of Grandeau, *viz.*, treatment of the soil first with weak acid and then, after washing, with weak ammonia water, evaporating the filtrate and igniting the residue. The methods of wet or dry combustion usually prescribed for humus determination yield results varying widely with the season, and incapable of serving as the basis of general conclusions; since it is quite uncertain whether or not the unhumified matter will ever become humus, or will be eliminated (as is largely the case in the arid region) by eremacausis, or (in the humid region) by fermentation.

In the ash of the humus, at least phosphoric acid should always be determined, since the phosphoric acid thus contained is certainly more available to plants than that not dissolved in the Grandeau treatment, although not as fully so as was at first claimed by Grandeau. The bulk of the humus ash is usually silica, which should be weighed, since we thus gain an idea of the amount of other ingredients contained in the "*matiere noire.*" But ordinarily I do not consider a farther prosecution of this analysis as of material practical importance.

The humus percentage gives an approximate idea of the nitrogen store in the soil, the percentage of nitrogen in the "*matiere noire*" being usually three to five per cent. While this is true for the region of summer rains (the "humid"), it seems that in the arid region the nitrogen percentage in the humus is so much greater, that a direct determination alone can serve the purpose. This is readily done by substituting in the Grandeau process a solution of potash or soda for that of ammonia, and after neutralizing the filtrate, evaporating and determining the nitrogen by the Kjeldahl method.

The direct determination of nitrates and ammonia in soils is in my view usually of little interest for general purposes, outside of the arid region, where these compounds remain wholly or partially in the soil and may accumulate to a considerable extent. In the humid region they are usually present in such minute proportions and subject to so much change from day to day, that for the purpose of judging the soils' general resources in nitrogen such determinations are of little use. When made they should, of course, be done by leaching with water and treatment of the filtrate, since we have no method by which the determination can be made in the whole soil without attacking also the nitrogen of the humus.

The determinations mentioned are in my view all that is usually called for in the examination of soils for practical purposes: very commonly, a general knowledge of the soils of a large region may render a goodly proportion of the determinations unnecessary. Thus the soils derived from the great eruptive sheet of Oregon and Washington have proved to be so universally rich in phosphates, that even this important determination frequently becomes superfluous: and almost the same can be said for potash and lime throughout the arid region. In a large number of cases it is only necessary to identify the soil under examination by mineralogical analysis with others of the same type previously examined from the same region; and this identification naturally carries with it the conclusions previously deduced from observation in the field and laboratory, with very little trouble.

It now remains for me to consider briefly what are the conclusions and practical deductions legitimately flowing from such work as I have outlined. I have heretofore published some detailed discussions of this subject, and will therefore not go into illustrative details, for which I refer to those publications.

What practical meaning shall we attach to the percentage data found by analyses made as described above? How much of each of the important ingredients must be present in order that the soil may be considered "rich" or "poor?"

In this general form the question is unanswerable, as is shown by the comparison of even a very few soils noted for great and lasting productiveness. Manifestly, cultural experience alone can answer the question in *any* form; and it is precisely upon such experience that I have based the general rules I shall enunciate—not *ex cathedra*, but purely as the result of extensive *comparisons of the results of chemical analysis of virgin soils with cultural experience.*

(1) There is one invariable rule as regards plant-food percentages, to which in virgin soils I fail to find a single exception. It is that all having high percentages are highly productive, unless physical conditions render them uncultivatable.

(2) The reverse is by no means generally true, for there are soils having what must be considered very low percentages that nevertheless prove both immediately productive and of considerable durability.

At first blush this admission seems fatal to the claims of chemical soil analysis to practical utility. It certainly proves that such analysis is not to be relied upon, *alone*, to determine the quality of a soil; it must be supplemented by other data than the plant food percentages.

When we discuss the cases forming this class of apparent contradiction, we find, first, that the soils so circumstanced are almost invariably rather coarsely sandy ones, and of considerable depth. So soon as we throw out of consideration the great mass of obviously inert material and examine the "fine earth" corresponding to the grain sizes of rich clay soils, our percentages assume quite a different magnitude, particularly when the great depth to which plant roots can exercise their vegetative functions in pervious soils are taken into consideration. Three other conditions must. however, be fulfilled in order to render such soils of low percentages thrifty; namely, the *ratios* of the several ingredients among themselves must not fall below certain values. In following up the investigation of these ratios, the substance which assumes commanding importance is *lime*. Unless lime is present in sufficient proportional amount to insure the presence of a certain proportion of calcium carbonate at all times, the soil will lack thriftiness; and this is true of all soils, whether of high or low percentages. Moreover, it is strikingly true that the percentage of lime required to fulfill this condition is decidedly higher in clay soils than in sandy ones.

The existence or absence of this important condition is in general readily recognizable by the vegetation characterizing calcareous soils; and one of the first points that must strike the observer is that "lime plants" are, almost throughout, those upon which old farmers base their successful locations of good land. But in following up this indication we soon find that we must abandon the definition of calcareous soils usually given in text-books ("soils that effervesce with acids"); for all the advantages of calcareous soils are secured with percentages of lime far below those in which the slightest effervescence can be perceived. Here, then, soil analysis renders the essential service of enabling us to account for the choice made by experienced frontiersmen and land experts: practically, *they are in quest of calcareous lands!*

That "a limestone country is a rich country" is an old adage, easily verified by any one who observes the geological map as he travels. But the adage holds true not only of limestone districts, but also of those which from other causes have a notable proportion of lime in their soils. The most striking example of the latter class is the entire arid region, of America as well as of Asia and Africa. As I have more elaborately shown in a special treatise (A Report on the Relations of Soil to Climate: Bulletin No. 3 of the U. S. Weather Bureau), the presence of a notable amount of calcium carbonate in the soils of arid countries is the necessary result of the failure of the scanty rain-fall to leach out the water-soluble products of the weathering process; foremost among these, calcium carbonate is accumulated even where no limestone formations exist. According to the adage above quoted, then, arid countries ought to be rich countries. History shows this to be true, with the proviso that there is any soil at all; for the slowness of the weathering process in arid climates renders soil formation correspondingly slow. Arid climates, moreover, necessitate irrigation; but where, as in Egypt and India, the needful water is supplied, the soil proves almost inexhaustible. It need hardly be mentioned that alongside of lime, other important ingredients accumulate in the soils of the arid regions; notably, *potash* and *nitrates*.

The good results of the presence of much lime in soils, about which there can be no practical question, is doubtless connected partly with the effects which must be attributed to calcium carbonate in the soil as well as in the laboratory: It acts in the "Aufschliessung" of silicates, and it is doubtless from this cause that (as was mentioned above) we find such large amounts of soluble alumina in calcareous clay soils. Moreover, lime favors *nitrification*, and prevents the formation of injurious *acidity*. That much we can say with certainty; but it has doubtless other important functions, even in a chemical point of view; while as a flocculator of clay it performs a most important physical office, in promoting easy tillage, and circulation of water.

In very sandy soils, as little as 0.10 per cent. of lime may cause the soil to show "lime vegetation;" while in very heavy clay soils, I have known even 0.6 per cent. to be inadequate for this purpose. But apart from the most extreme cases, I find we may safely augur favorably of any soil containing *as much lime as potash.*

The average ratio of the lime percentages of soils in the arid and humid regions I find to be about twelve to one, if we exclude from comparison, on both sides, limestone soils proper. The most general expression I can give to the results of my discussion of the analyses of virgin soils with respect to lime, is that in its presence (in adequate proportions) much smaller percentages of the other plant foods will suffice for high and lasting productiveness; and that much less lime suffices to produce this effect in "light" than in "heavy" soils. It thus appears, then, that the question of the adequacy of plant food percentages is largely dependent upon the proportion of lime, as well as upon the physical character of the soil. The latter point depends essentially, of course, upon the greater facility with which an extended root-development occurs in light than in heavy soils.

I think that the failure of agricultural chemistry to recognize as fully as it deserves the extreme importance of lime in soils, is measurably due to the accidental fact that partly extended limestone formations, partly the prevalence of the mixed glacier drift over the greater part of Europe and Eastern North America, have made calcareous soils so predominant in the regions where agricultural science has been developed, that the exceptional and relatively unimportant non-calcareous areas have escaped attention.

As regards *potash*, the comparisons I have made of hundreds of analyses from the arid and humid regions of the United States. respectively, show that potash, like lime, accumulates to a remarkable extent in the soils of the arid climates, doubtless in the form of zeolitic compounds; the average proportion in the two regions being about one to three in favor of the arid. Coupled with the fact that there, potash salts are often abundant in the soil water, as well as in the waters of the streams serving to irrigate these lands, the conclusion that potash is not likely to be soon called for as a fertilizer in that part of the continent becomes natural, and is abundantly confirmed by all tests thus far made. The invariable presence of an excess of lime in the arid soils, moreover, promises the current setting-free of potash from the soil zeolites.

The general average of potash in the arid soils is from 0.7 to 0.8 per cent.; for the humid soils of the Cotton States. from 0.21 to 0.25 per cent. No wonder that, considering the frequent deficiency in lime in the latter region. the Stassfurt salts are among the first of the effective fertilizers for the Eastern United States.

As regards *phosphoric acid*, the comparison of the soils of the humid and arid regions fails to reveal any constant difference; its presence in larger or smaller proportions being, apparently, dependent entirely upon local geological and petrographic conditions. It is curious that in my investigations of virgin soils I have been led to the identical figure for the minimum percentage of this substance that will still insure profitable culture, that, I find, has been accepted by European investigators; namely, five-hundredths of one per cent. (0.05 per cent.). When no more than this amount is found to be present in any soil, production will soon cease unless phosphatic fertilizers are used; the duration of profitable culture depending materially, however, upon the proportion of lime present. Anything much above one-tenth of one per cent. usually proves, in virgin soils, to be quite a full supply, rendering the use of phosphates ineffective for a number of years.

Owing to the difficulty formerly existing in the *nitrogen* determination in soils, my investigations in this direction have not been as extended, nor the results as conclusive, as in the case of the mineral ingredients. Taking the humus determined according to Grandeau as an approximate gauge of the nitrogen store in soils, it would seem that in the humid region—the Cotton States in particular—less than 0.5 per cent. of humus, estimated to contain say an average of five per cent. of nitrogen, constitutes a deficiency; while in the arid region, as before noted, less than half that amount seems to suffice, rendering the use of nitrate fertilizers ineffective. But owing to the faultiness of the methods employed, the data thus far existing are, as yet, too scanty to serve profitably for the discussion of these points.

In summarizing the outcome of my studies on the relations of the actual productiveness of virgin soils to their plant food percentages, I might say that they support the intrinsically reasonable general thesis that *the amount of available plant food in virgin soils of essentially similar origin is sensibly proportional to the respective totals of such ingredients found by analysis;* provided we take into consideration both the proportions of these ingredients among themselves, and the physical nature of the soil. It is abundantly evident that sandy, pervious soils can, owing to the greater root-development they permit, be fertile with much smaller percentages than those which are difficult of penetration; at the same time, there is an obvious limit beyond which this greater root surface cannot make up for too great a scarcity of one or several ingredients. The test of how far the dilution of a close, fertile soil with pure sand can be carried without detriment to production, is apparently easy to make; and yet these experiments encounter no inconsiderable difficulties in actual execution. Yet their importance is so fundamental that I propose to prosecute them as diligently as circumstances permit, and earnestly hope that others will work in the same line of research.