A SHORT TEST FOR EASILY SOLUBLE PHOSPHATE IN SOILS¹

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INTRODUCTION

A large amount of work has been done on the digestion of soils with weak solvents, especially acids, in attempts to separate the easily soluble plant-food constituents from the less-soluble portions. The one kind is generally called easily soluble, available or active, and the other dormant, non-available, or, preferably, potential plant-food. This has come about as a result of efforts made to devise somewhat arbitrary methods for measuring that portion of the plant-food in soils which can be utilized by crops at the present time as distinguished from the remainder which in all probability will become more slowly available in the future.

HISTORICAL

Numerous methods have been devised for the above purpose and frequently the results obtained by different forms of procedure do not agree; consequently, there has arisen considerable discussion among scientific workers interested in this subject as to the value of such methods for the purpose intended, which has divided them into two general groups. One comprises those who believe that, on account of it being impossible to duplicate soil conditions in the laboratory by the use of arbitrary chemical methods, no credence can be placed in work of this character. To support this they cite the fact that the results obtained by such methods often vary widely among themselves. There are also some among this class who not only take the position that these methods have no significance, but maintain that all chemical methods, including those for the estimation of total plant-food constituents, give no reliable data for indicating the productive capacity of soils. The other group, while conceding the above to be partly true, nevertheless contend that some of the methods, although they may have their limitations, do give results of a certain value when used by workers with sufficient experience who know how to interpret them in connection with other data that may be obtained. For this reason they continue to employ them in their work.

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It is not necessary to discuss the relative merits of these opposite views or to include a summary of all the work that has been done, as the references in the literature pertaining to this subject are very numerous. For this reason only those which refer to certain matters discussed here will be mentioned.

During the progress of the work given in tables 2 to 5, an article came to the attention of the writer describing some work by Russell and Prescott of the Rothamsted Experiment Station (5), who have made rather extensive experiments on the determination of phosphorus in weak acid digestions of soils. Their work has been confined to a study of this element and among other things includes its adsorption by the soil and the effect of time of digestion and different strengths of various acids on the results obtained. While their experiments are somewhat different from those employed in this work, it is of interest to note that their results show that a short digestion with some acids, especially nitric, not exceeding a certain strength, puts more phosphorus into solution than does one for a longer time. This they prove to be due to adsorption. On account of the different methods of procedure and the limited number of soils used in their work, the conclusions reached regarding the behavior of nitric acid have been only partly confirmed here. An excellent summary of some previous investigations on adsorption phenomena in soils is given by these writers and in a resumé by Prescott (4).

Of all the methods that have been proposed for the estimation of easily soluble constituents in soils, probably the most widely used in this country are those involving the digestion of the soil with weak acids, either 0.2*N* hydrochloric or nitric, for a limited time at a definite temperature. The former was first adopted by the Association of Official Agricultural Chemists as a provisional method and included in their *Methods of Analysis* (3, p. 18).

Probably the first work with 0.2*N* nitric acid as a solvent for plant-food in soils was done by Dr. A. M. Peter, at this station, and the results were so favorable that it was proposed for trial to the association. Later, a committee appointed for the purpose of editing and revising their methods of analysis recommended it as a tentative method to replace the weak hydrochloric digestion (1, p. 27). It should be stated, however, that the method as recommended differs materially in some respects from the procedure as carried on here.

The last committee appointed by the association for the revision of methods has failed, for some unaccountable reason, to include any method for the above separations; consequently none appears in the latest revised *Methods of Analysis* of the association (2).

EXPERIMENTAL

In the early part of 1916, the writer, while engaged in some work on the digestion of soils with 0.2*N* nitric acid and other weak solvents, observed that adsorption apparently had considerable influence on the results obtained,

especially in the phosphorus determinations. It was found at that time (in some unpublished work) that in the 0.2*N* nitric acid digestion, when equal volumes of the acid were used on different amounts of soil, the smaller weight of soil showed more phosphorus in solution after the digestion, in proportion to the quantity of soil used, than was to be expected. An explanation which suggested itself at the time was that where the smaller mass was used, its adsorptive capacity was not satisfied under the conditions of the experiment, but when a larger amount of the same was in contact with the richer solution of phosphate, this requirement was more nearly met. It is possible, however, that the smaller mass of soil allows its particles to be exposed more completely to the relatively larger volume of acid and that this partly accounts for the discrepancies. The results obtained are given in table 1.

TABLE 1
Phosphorus dissolved by 1500 cc. of 0.2 N HNO₃ from different weights of soil, after 5 hours' digestion, being shaken every 30 minutes*

SOIL NUMBER	PHOSPHORUS OBTAINED FROM		RATIO
	7.5 gm. of soil	150 gm. of soil	
	gm.	gm.	1:20
56525	0.0003	0.0010	1:3.3
56526	0.0010	0.0059	1:5.9
56528	0.0008	0.0020	1:2.5
56530	0.0003	0.0009	1:3.0
14604	0.0327	0.4303	1:13.2
14606	0.0005	0.0026	1:5.2
56527	0.0003	0.0011	1:3.7
56529	0.0002	0.0005	1:2.5
56531	0.0003	0.0016	1:5.3
56532	0.0002	0.0004	1:2.0

* These digestions were made at room temperature, as were all others described in this paper.

A large excess of free nitric acid was present at the end of the digestion, in every experiment. That the solvent power of the acid for phosphate was not satisfied is apparent from the large amount of phosphorus taken up from soil no. 14604, as compared with that dissolved from the other soils. It should be borne in mind that the neutralizing power of our soils for 0.2*N* nitric acid, under the conditions of these experiments, is practically negligible except when carbonate is present. Assuming that the action was only that of an acid on the soil with no subsequent fixation of the dissolved phosphate, and that the soil surfaces exposed were somewhat in proportion to the amounts of soil used, the ratio of the phosphorus dissolved would be expected to be more comparable to the relative amounts of soil present. The ratio of the latter was constant (1:20) while that of the former varied considerably in individual soils, the maximum being 1:13.2 and the minimum 1:2. In other

words, the last ratio indicates in these particular soils where the larger amounts were used, only about one-tenth of the quantity of phosphorus in solution that would be expected, provided no adsorption or other interfering factors had intervened.

After the foregoing experiments were completed, attempts were made to determine if adsorption in soils was exerting any considerable influence on the amounts of plant-food constituents obtained in the weak acid digestion. In order to demonstrate this point, digestions were made of the same amounts of soil for short and longer periods of time and it was found that short digestions—for instance, 5 minutes—in 0.02*N* HNO₃, with shaking every minute, gave higher results with some soils as regards certain constituents, especially phosphorus, than the prescribed 5-hour period, with shaking every 30 minutes. In fact, in some soils, by placing them on folded filters, pouring the acid on and allowing it to filter immediately, the results compared favorably with the longer contact. This, however, did not hold true with all soils.

The above work comprises part of an investigation that is being carried on by the writer in an effort to obtain, if possible, more definite knowledge concerning the effect of weak acid digestions of our soils regarding certain constituents, namely phosphorus, potassium, calcium and silica. That such digestions, especially for short intervals of time, do have a certain value for indicating the productive capacity of a soil, is believed for the following reasons. First, the short, weak acid digestion largely eliminates the time action of the acid on the soil and this is one objection that has been raised against all methods of this character; second, the error due to adsorption by the soil, especially if it is of a gradual nature, is partly eliminated; third, the soil silicates are not appreciably attacked, as shown by the silica obtained in the digestion and finally a comparison of the short and the long digestions shows that about the same amount of calcium, a large bulk of the potassium and, in many cases, as much phosphorus is obtained in the former. The inference is that the amounts so obtained are more loosely held and in all probability are combined with the organic matter or humus portions of the soil rather than with the mineral silicates. As stated before, however, this work is being continued and a publication will probably be issued in the near future.

During the last few years the number of soils sent in by our farmers for tests as to their fertilizer requirements has been increasing to such an extent that short qualitative tests have been resorted to in answer to these demands. One of the tests generally made on such samples is for total phosphorus, for the reason that it is one of the most deficient elements in our soils. Unfortunately, however, the test for easily soluble phosphorus requires more time and quantitative work. Therefore it was thought that if some short test could be developed for finding out something about the amount of this element taken up in weak acid, it would be of some benefit in arriving at the need of the soil for soluble phosphate, when considered in connection with the total amount present.

As the short digestion gave as good results with a large number of soils as the longer, the following test, among others, was tried:

Ten grams of air-dried soil was added to 25 cc. of 0.2*N* HNO₃ and the whole shaken every minute for 5 minutes. The solution was filtered until clear into a $\frac{3}{4}$ by 6 inch test tube, 1 or 2 cc. of 60 per cent NH₄NO₃ solution was then added, and 5 cc. of ordinary molybdate solution as prescribed in the official method for fertilizers (3, p. 2). The contents of the test tube were heated to about 60°C., shaken several times and allowed to stand about 30 minutes at room temperature.

A possible objection may be raised against a method of this kind. Since it is only a qualitative test, how are the results obtained by it to be described to an inexperienced worker so that he can interpret them when made? In answer to this it might be stated that those given in the following tests were made by the writer without any previous experience and none have been changed or discarded.

The results marked "large," "fair," "moderate," "very moderate," etc., stand in decreasing order of the amount of ammonium phospho-molybdate obtained. These terms are only approximately defined and in some instances may be partly interchangeable without seriously vitiating the interpretation of the test. Although difficult to describe exactly, the term "moderate" indicates that the yellow precipitate, after settling by gentle tapping of the tube, occupied an area in the rounded tip from 0.5 to about 0.75 cm. in diameter.³ After a little experience no great difficulty will be found in the relative terms to be applied for purposes of comparison.

After the short tests were all made, the results were compared with the quantitative results from 5-hour digestions, so that no preconceived judgment might be formed about the terms to be applied. As stated above, none were changed or discarded.

The 0.2*N* HNO₃ digestion was made by digesting the soil in the proportions of 1 gm. of soil to 10 cc. 0.2*N* HNO₃ for 5 hours at room temperature, shaking every 30 minutes. It was then filtered and an aliquot evaporated to dryness. More HNO₃ was added to oxidize the organic matter, it was again evaporated and the last traces of HNO₃ eliminated by evaporation with HCl. The residue was dried on a steam bath to dehydrate SiO₂, taken up with HCl and H₂O, filtered and the phosphorus determined with ammonium molybdate by precipitating at 40°C. in a small volume and allowing the yellow precipitate to stand at the above temperature for an hour or so and finally over-night at room temperature. It was then filtered and determined volumetrically.

The total phosphorus was determined by the magnesium-nitrate method with slight modifications (1, p. 25).

The large majority of determinations of total and 0.2*N* HNO₃-soluble phosphorus were made by the writer. The remainder were made by different

³ The average diameter of 10 precipitates of this character was 0.64 cm.

analysts in the regular soils work. Where comparisons have been made of virgin with the corresponding cultivated soil, the work was carried on at the same time. The numbers for these are bracketed in the tables.

The soils used in the work have been divided into four general groups. While the classifications are somewhat arbitrary and not definite in all cases, they are satisfactory for the purpose. The results obtained by the short test and the comparisons with the other determinations are given in tables 2, 3, 4 and 5.

TABLE 2

Determinations on soils in which the amounts of total and easily soluble phosphorus are small

SOIL NUMBER	CHARACTER	TOTAL P	SHORT TEST	P DETERMINED AFTER 5 HOURS' DIGESTION IN 0.2 N HNO ₃
		<i>per cent</i>		<i>per cent</i>
25002}	Cultivated	0.037	None	0.0008
25004}	Virgin	0.050	Trace	0.0015
56447}	Cultivated	0.043	None	0.0010
56449}	Virgin	0.079	Very small	0.0021
56489}	Virgin	0.031	None	0.0006
56490}	Cultivated	0.019	None	0.0006
56491}	Virgin	0.018	None	0.0006
56492}	Cultivated	0.019	None	0.0006
56493}	Virgin	0.049	Trace	0.0014
56495}	Cultivated	0.042	None	0.0008
25662}	Cultivated	0.062	None	0.0009
25663}	Virgin	0.072	Trace	0.0015
56497}	Cultivated	0.047	Trace	0.0015
56499}	Virgin	0.037	Very small, larger than 56497	0.0023
36263	Virgin	0.030	Trace	0.0009
43527	Cultivated	0.048	None	0.0004
56699	Cultivated	0.050	None	0.0011
50117	Cultivated	0.044	Trace	0.0016
56701	Cultivated	0.024	None	0.0006
56702	Cultivated	0.037	Trace	0.0014
43506	Cultivated	0.052	Trace	0.0007
56457}	Cultivated	0.030*	None	Not determined
56458}	Virgin	0.033*	None	Not determined
56485}	Virgin	0.032*	None	Not determined
56487}	Cultivated	0.021*	None	Not determined

TABLE 2—Continued

SOIL NUMBER	CHARACTER	TOTAL P	SHORT TEST	P DETERMINED AFTER 5 HOURS' DIGESTION IN 0.2 N HNO ₃
		<i>per cent</i>		<i>per cent</i>
43526	Cultivated	0.042	Trace	0.0013
56652	Cultivated	0.027	None	0.0005
56740	Cultivated	0.031	None	0.0006
56584	Cultivated	0.022	Very small	0.0016
56741	Cultivated	0.032	None	0.0007
56586	Cultivated	0.055	Very small	0.0012
56525	Cultivated	0.018	None	0.0007
56529	Cultivated	0.020	None	0.0010
56530	Cultivated	0.026	Trace	0.0014
56531	Cultivated	0.020	Very small	0.0013
56532	Cultivated	0.030	None	0.0011
56587	Cultivated	0.041	None	0.0013
56592	Cultivated	0.064	Trace	0.0009
25424	Cultivated	0.053	Very small	0.0014
25459	Cultivated	0.021	None	0.0004
25454	Cultivated	0.024	None	0.0002
25418	Cultivated	0.031	None	0.0006
25224	Cultivated	0.053	Trace	0.0006
17902	Cultivated	0.028	None	0.0002
25020	Cultivated	0.054	None	0.0003
36453	Cultivated	0.040*	Trace	Not determined
36538}	Virgin	0.074	Trace	0.0026
36539}	Cultivated	0.052	None	0.0011
Average of 43 soils.		0.040		0.0010†

* Not included in average.

† That is, 2.5 per cent of the total P.

TABLE 3

Determinations on soils in which the total amount of phosphorus is small, but the amount of easily soluble phosphorus is comparatively large

SOIL NUMBER	CHARACTER	TOTAL P	SHORT TEST	P DETERMINED AFTER 5 HOURS' DIGESTION IN 0.2 N HNO ₃
		<i>per cent</i>		<i>per cent</i>
36694}	Cultivated	0.068	Very small	0.0043
36696}	Virgin	0.091	Very small, more than 36694	0.0051
36792}	Virgin	0.088	Trace, more than 36796	0.0028
36796}	Cultivated	0.067	Trace	0.0017
56549	Cultivated	0.043	Trace	0.0028
56583	Cultivated	0.059	Moderate	0.0043
56700	Cultivated	0.053	Trace	0.0021
56526	Cultivated	0.027	Very moderate	0.0022

TABLE 3—Continued

SOIL NUMBER	CHARACTER	TOTAL P	SHORT TEST	P DETERMINED AFTER 5 HOURS' DIGESTION IN 0.2 N HNO ₃
		<i>per cent</i>		<i>per cent</i>
56528	Cultivated	0.026	Small	0.0022
56588	Cultivated	0.039	Very small	0.0019
25227	Cultivated	0.049	Very small	0.0017
36510	Cultivated	0.070	Moderate	0.0149
36511	Cultivated	0.067	Moderate, less than 36510	0.0114
36687	Cultivated	0.087	Very moderate	0.0079
56188	Cultivated	0.080	Very moderate	0.0060
56501	Cultivated	0.070	Moderate	0.0109
56503	Virgin	0.074	Moderate, more than 56501	0.0125
25796	Cultivated	0.081	Very small	0.0031
25797	Virgin	0.092	Small	0.0074
Average of 19 soils.		0.065		0.0055*

* That is, 8.5 per cent of the total P.

TABLE 4

Determinations on soils in which the total amount of phosphorus is large but the amount of easily soluble phosphorus is comparatively small

SOIL NUMBER	CHARACTER	TOTAL P	SHORT TEST	P DETERMINED AFTER 5 HOURS' DIGESTION IN 0.2 N HNO ₃
		<i>per cent</i>		<i>per cent</i>
*9768	Virgin	0.142†	Very moderate, larger than 9771	0.0062†
9771	Cultivated	0.083	Very small	0.0019
14411	Cultivated	0.165	Trace	0.0036
Same			Trace	
*14412	Virgin	0.199†	Moderate, much larger than 14411	0.0191†
Same			Same	
56959	Virgin	0.188	None	0.0024
56960	Cultivated	0.129	None	0.0018
56742	Cultivated	0.088	None	0.0010
56527	Cultivated	0.077	None	0.0010
56585	Cultivated	0.079	Very small	0.0018
36957	Cultivated	0.111	Trace	0.0009
36775	Cultivated	0.114	Trace	0.0024
25114	Cultivated	0.157	Trace	0.0046
17287	Cultivated	0.142	Very small	0.0028

* These soils belong in table 4 and are included here for comparison with the cultivated.

† Not included in average.

TABLE 4—Continued

SOIL NUMBER	CHARACTER	TOTAL P	SHORT TEST	P DETERMINED AFTER 5 HOURS' DIGESTION IN 0.2 N HNO ₃
		<i>per cent</i>		<i>per cent</i>
25910	Cultivated	0.101	None	0.0008
17285	Cultivated	0.136	Very small	0.0038
43563	Cultivated	0.112	Trace	0.0011
17480	Cultivated	0.094	Trace	0.0015
17481	Cultivated	0.127	Very small	0.0030
36798	Cultivated	0.144	Trace	0.0019
25362	Cultivated	0.103	Trace	0.0006
25228	Cultivated	0.095	Very small	0.0017
25134	Cultivated	0.107	Trace	0.0009
25135	Cultivated	0.107	None	0.0008
25995	Cultivated	0.093	None	0.0007
25311	Cultivated	0.131	None	0.0012
36688	Cultivated	0.134	Small	0.0031
56322	Cultivated	0.138	None	0.0022
56324	Cultivated	0.139	None	0.0016
56325	Cultivated	0.136	None	0.0010
43002	Cultivated	0.122	Trace	0.0014
36574	Cultivated	0.144	None	0.0010
14956	Cultivated	0.174	Trace	0.0014
2306	Cultivated	0.154	Very small	0.0023
Average of 31 soils.		0.123		0.0018†

† That is, 1.5 per cent of the total P.

TABLE 5

Determinations on soils in which the amounts of total and easily soluble phosphorus are large

SOIL NUMBER	CHARACTER	TOTAL P	SHORT TEST	P DETERMINED AFTER 5 HOURS' DIGESTION IN 0.2 N HNO ₃
		<i>per cent</i>		<i>per cent</i>
17483	Cultivated	0.175	Moderate	0.0158
17485	Virgin	0.190	Moderate, less than 17483	0.0122
2305*	Cultivated	0.204	Fair, much larger than 2306	0.0145
2306†	Cultivated	0.154‡	Very small	0.0023‡
56747	Virgin	0.317	Large	0.0780
Same			Same	
56784	Cultivated	0.151	Fair, much less than 56747	0.0234
Same			Same	
56733	Cultivated	0.427‡	Moderate	Not determined
50592	Cultivated	0.620	Large	0.2260

* Cultivated since 1884.

† Cultivated since 1860, same farm. Belongs to table 3 but included here for comparison with 2305.

‡ Not included in average.

TABLE 5—Continued

SOIL NUMBER	CHARACTER	TOTAL P	SHORT TEST	P DETERMINED AFTER 5 HOURS' DIGESTION IN 0.2 N HNO ₃
		<i>per cent</i>		<i>per cent</i>
36790	Cultivated	0.791	Large	0.4200
25203	Cultivated	1.464	Large	0.6180
25204	Cultivated	2.227	Large	0.9600
25112	Cultivated	0.243	Moderate	0.0176
25039	Cultivated	0.205	Moderate	0.0160
50735	Cultivated	0.254	Moderate	0.0165
43407	Cultivated	0.203	Moderate	0.0171
25824	Cultivated	0.207	Moderate	0.0116
56329	Cultivated	0.148	Very moderate	0.0067
56855	Cultivated	0.107	Moderate	0.0111
25664	Cultivated	0.093	Very moderate	0.0072
25295	Cultivated	0.101	Moderate	0.0090
56188	Cultivated	0.080	Very moderate	0.0060
9768	Virgin	0.142	Very moderate	0.0062
14412	Virgin	0.199	Moderate	0.0191
Average of 21 soils.		0.387		0.1196§

§ That is, 30.9 per cent of the total P.

GENERAL DISCUSSION AND CONCLUSIONS

Assuming that the 0.2N HNO₃ method has some value, the question arises, where should the demarcation in the results come when they are to be interpreted as to the probable need of a soil for soluble phosphate. After considerable experience from a study of a large number of our soils, it is the writer's opinion that a soil should show a minimum phosphorus solubility of 0.005 per cent by this method, regardless of the total amount present. Between the range of 0.005 and 0.0075 per cent the use of phosphate may be beneficial, while above this it is probably not required.

In regard to total phosphorus, it might be stated that any soil below 0.08 or 0.10 per cent is in need of phosphate, between these figures and 0.15 per cent it may respond, and above this it is probably not necessary. However, this arbitrary classification as to the total probably depends to a large extent on its availability in the soil.

From an examination of the tables we find, therefore, that the demarcation in the results by the short test is defined by "very moderate" to "moderate." As stated above, these terms are difficult to define in some cases and in these instances may be partly interchangeable. At the time, it was the writer's judgment that the terms "moderate," "fair" or "large" indicated that no phosphate was needed, and "very moderate" that there was a doubt, and below this it was necessary. With but few exceptions it will be found that the results are in agreement with those of the 5-hour digestion. The exception may be due partly to inexperience with the tests at first, making it difficult

to define every one accurately, inasmuch as none were changed or discarded. Again, some of the variations are due to differences in the solubility of the phosphate in short and in longer periods of time, partly caused by subsequent adsorption.

The short test probably could be applied for calcium and potassium if desired, and although no attempts have been made, it is possible that by the employment of suitable apparatus and perhaps some modification in the procedure, an approximate quantitative determination could be made of phosphorus and possibly other elements which could be correlated with the figures obtained in the long digestion.

It will be observed that there are considerable differences in the behavior of soils in the weak acid digestion, and the percentages of the total phosphorus soluble in the weak acid are widely divergent. Assuming the surface 6½ inches to weigh 2,000,000 pounds, the averages obtained in the different groups are as given in table 6.

There is no doubt that the conclusions reached by the 0.2*N* HNO₃ digestion are in accord with what we know about the soils in tables 2 and 4. Those in

TABLE 6

	TOTAL P		0.2 <i>N</i> HNO ₃ SOLUBLE P		PER CENT OF THE TOTAL P SOLUBLE IN 0.2 <i>N</i> HNO ₃
	Per cent	Pounds per acre	Per cent	Pounds per acre	
Table 2.....	0.040	800	0.0010	20	2.5
Table 3.....	0.065	1300	0.0055	110	8.5
Table 4.....	0.123	2460	0.0018	36	1.5
Table 5.....	0.387	7740	0.1196	2392	30.9

table 2 show need of phosphate and respond to phosphate treatment in the field. They represent a large majority of our soils of different types and experimental field results verify the laboratory tests. The phosphate in such soils is low, not available and is probably combined with iron and aluminum. On the contrary, the soils in table 5 represent some of our best; those which are in the blue-grass region. Many of these soils have a large content of calcium phosphate and the better class do not respond to phosphate treatment. Here again the 0.2*N* HNO₃ digestion is in accord and also the short test. The soils in tables 3 and 4 represent those regarding which any method for availability would have decided practical value. The weak acid digestion apparently discriminates between those soils in which the phosphorus exists as calcium phosphate and those where it is combined in other forms such as iron and aluminum and generally considered as not available.

The experimental field results in this state show that on those soils which need phosphate, applications of limestone and acid phosphate or rock phosphate give the best results. As the phosphate in either case is probably maintained in the soil in the form of calcium phosphate which, if true, would

more than likely be shown by the 0.2*N* HNO₃ digestion as indicated by some of the foregoing results, this shows that the weak acid digestion should have some value in practice. In fact, those who have been engaged in the chemical analysis of soils at the Kentucky station in the past, before any experimental fields were established, predicted those areas which would respond to phosphate treatment and it is with some gratification that they now find these predictions confirmed.

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