Laboratory method for determination of organic, inorganic and total phosphate

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The following method can normally be applied in sets of 12-16 soil samples and about 3 sets can be applied per day (36-48 samples/day), if the samples already are crushed into powder.

Principles :

The reserves of phosphate in the soil depends on phosphate present in both organic and inorganic components. The method outlined here is based on the assumption that organic phosphate is stable in medium strong to strong acids. By heating a soil sample to 550°C the organic phosphate is transferred into inorganic phosphate. The organic phosphate can therefore be calculated as the difference between an unheated and a heated sample. The method does not include phosphate bound in the silicate structures. In most archaeo-pedological investigations anyhow this last fraction of phosphate is of no particular importance. The methode presented here is a slightly modified version, of the method used at the Institute of Geography, Copenhagen University.

APPARATUS

Sand bath, 70 °C
Colorimeter
Oven , 550 °C

REAGENTS

12 N H_2SO_4
1000 ppm phosphate standard
Spectroquant phosphorus test set (from E. Merck, Darmstadt, Germany) composing of :

1) ammonium molybdate solution
2) standard ascorbic acid

Procedure :

STANDARDS

Make a standard series with 0.00, 0.25, 0.50, 1.00, 1.50 and 2.00 ppm phosphate concentration. This is done by 1) bringing respectively 0.00, 0.50, 1.00, 2.00, 3.00 and 4.00 ml of a 50 ppm phosphate solution in a 100 ml volumetric flask, and 2) add 1.50 ml 12 N H2SO4 to each of them. 3) fill up quantitatively to 100 ml with demineralized water. 4) shake thoroughly.

Bring exactly 4 ml of each standard solution in separate cuvette's and add subsequently a small spoon of ascorbic acid and 4 drops of ammonium molybdate solution.The latter will provide a blue colour to the liquid. Shake thoroughly again. Measure the blue colour of the whole series at 690 nm (or any wavelength between 600-900 nm). Read the transmission signal.

SAMPLES

Preparation :

Crush 2-3 g of sample material into powder. Weigh 2x 0.500 g soilpowder and transfer the material into two porcelain crucibles. If the phosphate content is expected to be low, it is better to use 2 x 1.000 g of crushed soil material. The one sample is heated at 550 °C for one hour. Let the sample cool for an hour. Following steps are similar for both the heated and the unheated samples.

Extraction :

Add 5 ml 12 H_2SO_4 to each crucible. They are then put for 10 minutes on a sand bath, that already has been brought to 70°C (control with a thermometer). After addition of another 5 ml H_2SO_4 the samples are put for cooling during one hour.

Filtration :

The soil is separated from the extract using a filter. Wet the filter before pouring the liquid into the funnel. the extract is filtered into a 100 ml volumetric flask. Wash several times with small portions of demineralized water to make sure that all the soil has been washed carefully. Fill the flask quantitatvely to 100 ml. Shake thoroughly afterwards. Filter again if the liquid is not completely transparent.

Colorimetry :

Bring 0.400 ml of the filtered extract into a cuvette and add 3.600 ml demineralized water. Add 4 drops of the ammonium molybdate solution, and one small spoon of ascorbic acid (use the special spoon attached to the cover of the ascorbic acid bottle) respectively. Shake thoroughly again. Let the sample stand 15 min for the development of an optimal colour intensity. The colour can now be measured at 890 nm using a colorimeter (in the laboratory of Stoops the wavelength 690 nm is used). The values read is the transmission values.

Calculation :

The transmission values should be transformed to extinction values with the following formula :

$$E=2-\log10(T)$$

E the extinction
T the transmission

Calculate the regression for the standard series and use the coefficient to obtain the ppm values for the samples. Take into account the dilution factor, the extracted volume and the exact weight of your sample to calculate your results in mg/100 g of dry soil.

$$P2O5\ (ppm) = F1*F2*F3*F4*\frac{(\text{calculated actual E of the sample})}{(\text{weight of soil})}$$

F1 is the factor to transform the results from P to P_2O_5
F2 is the factor based on the emission values of the phosphorus standards
F3 is the size of the volumetric flask wherein the extract has been transferred, in ml.
F4 dilution factor when the extract is transferred from the volumetric flask into the cuvette

Exemple of calculation :

Sample 1 burned : 0.512 g of soil :

transmission 76

2-log10 (76) = 0.1192

Sample 1 unburned : 0.506 g of soil :

transmission 80

2-log10(80) = 0.0969

Standard series : factor F 2

ppm	*T*	*E*	*E/ppm*	
0.00	100	0.0000		
0.25	75	0.1249	2.0010	
0.50	58	0.2366	2.1135	
1.00	34	0.4685	2.1344	
1.5	19	0.7212	2.0797	
2.00	11	0.9586	2.0864	mean
			sum 10.4150	2.0830

(2.2914*2.0830*100*10*0.1192)/0.512 = 1111 ppm *Total phosphate*

(2.2914*2.0830*100*10*0.0969)/0.506 = 914 ppm *Inorganic phosphate*

(1111-914) = 197 ppm *Organic phosphate*

Jari Hinsch MIKKELSEN, Gent the 20/03/1997

Note of Jari Hinsch Mikkelsen about the phosphate methods and archaeology :

A wide variety of soil phosphate analyses exist on the “market”. Most analyses provide either data on the total phosphate content, or they measure the plant available phosphate for agronomy purposes. Total phosphate analyses are often expensive and time-consuming, a problem raised by scientists dealing with phosphate in an archaeological context.

In archaeology, phosphate analyses are often needed fast and in large numbers, as an example to detect former settlements. This can be done by testing the soil in a grid net over some hectares of land.

The fastest methods are the so-called “field-spot phosphate testing method”, which are performed immediately on the site. In this type of method the quantity of phosphate is quantified relatively, based on the colour intensity developed after some minutes of contact with the soil sample. The colour development is estimated visually, giving the phosphate content a relative classification value. The quality of the visual estimation is relatively low in comparison with analyses performed in the laboratory. Despite their limitations the field-spot phosphate methods can be useful as a survey method for archaeological structures.

Often archaeo-pedologists are involved when the archaeological site has been located and is under investigation. In such situations, more precise phosphate methods are required.

Our purpose during this “round table discussion one” is to describe such laboratory methods, that satisfy the need for a reliable, fast and cheap method. Our purpose is not to force every laboratory to apply the same method(s), but rather to come to a better understanding of the different methods. What are the possible results to be obtained ? What are the differences between the methodes and when should we apply the one or the other method ? During this meeting some methods were presented. In the coming years we hope not only to receive more data, but also to exchange samples between laboratories using different methods.