



STATENS GEOTEKNISKA INSTITUT
SWEDISH GEOTECHNICAL INSTITUTE

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No 27 E

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carbonate content and sulphur content
in soils**

ROLF LARSSON
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PREFACE

This report deals with various methods for determination of organic content, carbonate content and sulphur content especially for geotechnical purposes. The purpose of the work has been to obtain methods with sufficient accuracy and which at the same time are simple and safe and can be performed as routine tests in geotechnical laboratories. The methods recommended are described in appendix.

The report forms part of the research in progress at SGI concerning the characteristics of organic soils, using grants from the Swedish Council for Building Research and the Swedish National Road Administration.

Different methods have been tested for determination of the organic and carbonate contents. These methods originate from the Norwegian Geotechnical Institute in Oslo, the Technical Research Centre in Helsinki and Földmérési Talajvizsgáló Vállalat in Budapest.

The method tested for determination of sulphide content is based on a diploma project by Håkan Svensson at the Department of Chemistry of Linköping University. Valuable ideas concerning determination of the sulphide content have also been provided by Arvid Jacobsson at the Technical University of Luleå and Danaï Georgala at Scandiaconsult.

The more accurate determinations of the organic and carbonate contents that have been used as reference determinations have been performed by the Swedish Laboratory for Agricultural Chemistry. Reference determinations of the sulphur content have been performed by Micro Kemi AB in Uppsala.

In the comparison between the more accurate methods for determination of organic content, several institutions have taken part, these being the Soil Laboratory at the Swedish Geological Survey, the Department of Forest Soils at the Swedish Agricultural University, the Division

of Marine Microbiology at the Department of Botany of Gothenburg University, the Swedish Laboratory for Agricultural Chemistry and the Quaternary Geological Department at Stockholm University.

Linköping August 1987

Rolf Larsson

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Determination of organic content by colorimetric measurement

Determination of carbonate content by measurement of evolved gas pressure

Determination of ferrous sulphide content by wet oxidizing and titration

SUMMARY

An investigation has been made in order to get an estimate of what accuracy can be obtained in routine determinations of organic matter, carbonates and sulphides. The investigation has also been aimed at getting simple and reliable methods for determination of these contents accurate enough for geotechnical purposes.

Comparative tests have been performed with a large number of methods; simple as well as more complicated. In these comparisons has been found that, due to the natural variation in natural soils and the very small samples used in the analyses, the same accuracy can practically be obtained by simple methods to determine the various contents as by more complicated methods.

After tests with various simple methods a recommendation is given to use the colorimetric method for determination of organic matter in soils with low to medium high organic contents. For determination of very high organic contents or determination of the ash content in peats the ignition loss method is recommended.

For determination of carbonates the use of Moum's apparatus or the Chittick apparatus is recommended.

For determination of sulphides a method with wet combustion and titration is recommended.

The recommended methods are described in detail in appendix.

1. INTRODUCTION

For geotechnical purposes, the organic content has mainly been estimated by determination of the loss on ignition. Often under the assumption that the soil is free from carbonates, the loss on ignition has then been corrected with values gained from experience. In Sweden, empirical values originating from Ekström (1927) have been used. In the determinations, visible shells have been removed. For soils with an obvious lime content, the carbonate contents have been determined and corrections have been made accordingly. In sulphide soils, the loss on ignition due to organic content and losses due to the presence of iron sulphide have not been separated, but have been assumed to influence the soil in the same way.

The content of calcium carbonate has been estimated roughly by visual inspection of a sample treated with diluted hydrochloric acid. More accurate determinations have been made with Passon's apparatus, in which the volume of gas developed at treatment of the sample with diluted hydrochloric acid is measured. Estimations of sulphide for geotechnical purposes have not been made to any large extent.

In other sciences dealing with soils, such as agriculture, forestry and geology, where more accurate determinations have been required, a number of different methods have been developed through the years. In some countries, e.g. Hungary, Finland and Norway, this development has led to simpler and better methods for estimation of organic and carbonate content also coming into use for geotechnical purposes.

2. PURPOSE AND EXTENT OF THE INVESTIGATION

For some years a larger investigation concerning the geotechnical characteristics of organic soils has been in progress at SGI. This investigation is being carried out with grants from the Swedish Council for Building Research and the Swedish National Road Administration.

A part of this investigation has been aimed at finding simple and inexpensive methods with sufficient accuracy for estimation of organic, carbonate and sulphide content.

Existing methods have been examined and a number of relatively simple methods have been tested. The results from these tests have been compared with results from more advanced determinations made on the same material in other laboratories. The results from the determinations of organic content have furthermore been compared with estimations by loss on ignition and the corrections and accuracy for this method have been studied. To obtain an idea of the accuracy in determination of organic content that can be obtained by using the more advanced equipments of today, comparative investigations have been made at a number of research institutions, where comparative investigations have also been made concerning determination of carbonate content.

On the basis of the results and experience from these and other investigations, simple methods of determining the various contents are recommended and descriptions and instructions are given in an Appendix.

3. METHODS FOR DETERMINATION OF ORGANIC CONTENT

3.1 General

Determination of organic content has always been of great interest for soil chemists and new methods have been steadily developed since the beginning of the 19th century. Literature regarding this subject is very comprehensive and a complete review is beyond the limits of the project.

A review and evaluation of the most common methods as far as the beginning of 1950 have previously been made at SGI (Silfverberg, 1957).

Silfverberg came to the conclusion that dry combustion with determination of the evolved carbon dioxide was the most accurate method of determination.

However, not even this method is exact, as the total carbon content is measured and the conversion to organic content is somewhat disputed. At that time the method was complex and required an experienced chemist. A more simple method of wet combustion (Thorn & Shu, 1951) followed by titration was recommended for routine purposes. Even this method has been considered too advanced by most geotechnicians and therefore the method of loss on ignition has been used almost solely for geotechnical purposes in Sweden. Since then, the dry combustion method has been further developed and modern carbon analyzers have been introduced. However, these are very expensive. New and better wet combustion methods have been developed and the rapid wet combustion methods have been further simplified. Titration has been replaced by colorimetric determination. The latest method is to measure the reflection from the surface of a section of the soil using light rays of different wavelengths and a spectrophotometer and to relate this to the organic content of the soil. However, this method is in an early stage of development and its potential is unknown.

3.2 Loss on ignition

The most common method used in soil mechanics to determine organic content has been to measure the weight loss on ignition. The procedure is as follows: a dried, pulverized and weighed sample is heated for a certain time in a furnace. After cooling in a vacuum exsiccator, the sample is weighed again and the loss on ignition is calculated.

Different heating temperatures from 350°C to 900°C have been recommended depending on the sources of error found for the particular materials and the method of preparing the samples. In general, the lower the tem-

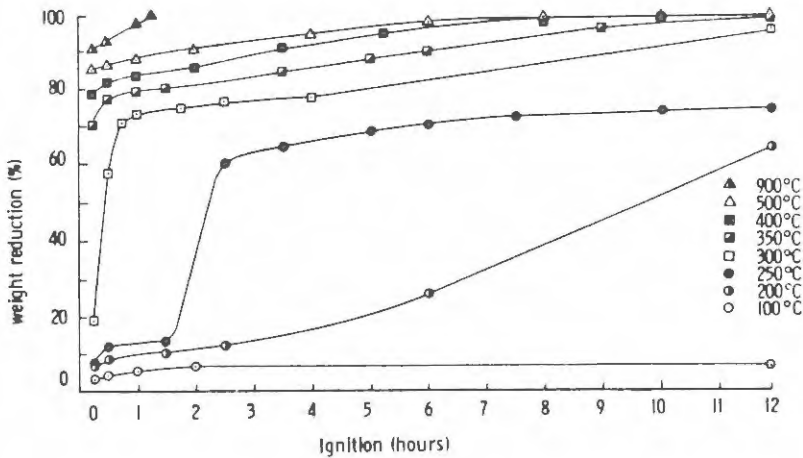


Fig. 1. Loss in weight for wood fibres as a function of time at heating to different temperatures. (From Al-Khafaji & Andersland, 1981).

perature used, the longer it takes for complete combustion of the organic material. Al-Khafaji & Andersland, 1981, found that complete combustion at 900°C was obtained after 1-1.5 hours while at least 12 hours was required at 350°C. Fig. 1.

During ignition, all organic material is assumed to be consumed and the loss in weight in per cent of the mass of the dried sample (loss on ignition) is taken as a measure of the organic content of the soil. Also other changes in weight occur at ignition depending on the composition of the soil. Losses in weight are primarily caused by the evaporation of OH-groups from the crystals in the mineral particles and from the decomposition of carbonates. Oxidation of certain minerals can cause an increase in weight. (Thalme & Almén, 1975).

Different minerals have different properties concerning change in weight at different heating temperatures. This fact is used, for example in determination of the occurrence of different minerals with differential thermal analysis. Typical curves for the change in their weight of different minerals at different heating temperatures are shown in Fig 2.

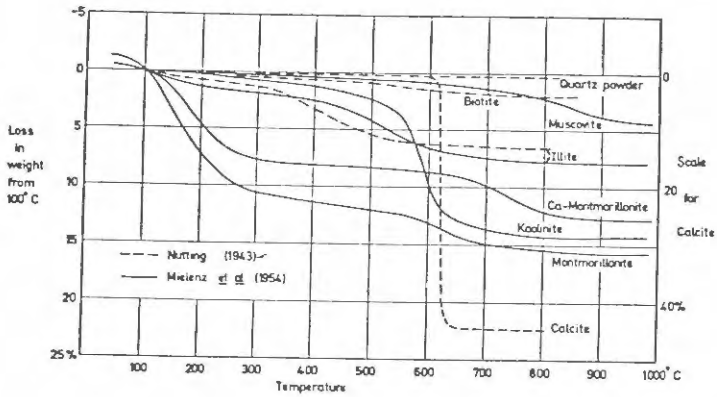


Fig. 2. Loss in weight of a mineral during heating. (From Skemp-ton and Petley, 1970). N.B. the scale for calcite.

These curves illustrate why different recommended temperatures for ignition have been chosen in different parts of the world. In regions where the soil has a certain composition, the loss in weight of the mineral phase can to a great extent be avoided by keeping the ignition temperature low, e.g. 440 °C (Arman, 1970).

In other regions, where many different minerals are included in the composition of the soil, the losses in weight change continuously and are not stabilized until a temperature of 800°C - 900°C is reached. In these regions, it is more common to use ignition temperatures in this order and adjust for the loss of weight in the mineral phase with an empirical correction. Such corrections are valid only for the type of soil from which they have been obtained and therefore the empirical corrections used in Sweden cannot be used for soils from other geological regions with other mineral compositions.

In Sweden, as a rule, a heating temperature of 800°C has been used and the time for heating has been at least one hour. The loss on ignition obtained has then been corrected with empirical values given by Ekström 1927. These empirical corrections are based on experience from tests on top soils in cultivated areas in Sweden. Table 1.

Table 1. Values based on experience for correction of the loss on ignition according to Ekström 1927.

Difference between loss on ignition and organic content for various mineral soils containing organic matter.

Soil classification	Loss on ignition subtracted by organic content	
	Variation	Mean value
Sandy soil	0,4-1,2	1
Sandy silty clay	1,4-2,2	2
Silty clay	2,0-2,9	2,5
Clay	2,9-4,4	3,5
Very fine clay	3,9-5,3	4,5

Loss on ignition for mineral soils free from organic matter and carbonates

	Loss on ignition
Sandy soil	0,4-1,3
Sandy silty clay	1,5-2,0
Silty clay	1,7-2,5
Clay	2,1-3,9
Very fine clay	3,9-6,0

The values from Ekström were processed by Pusch 1974 to get a better correlation with the geotechnical classification. Table 2.

Table 2. Loss on ignition in inorganic soil free from carbonates. (From Pusch, 1974).

Soil type	Clay content ¹⁾	Loss on ignition
Gravel	<5	1,0
Clayey sand, silt and moraine	5-15	1,5
Sandy, silty clay	15-25	2,0
Silty clay	25-40	2,5
Clay	40-60	3,5
Clay	>60	4,5

¹⁾Calculated for material <20 mm.

Pusch also made a compilation of Swedish investigations of the mineral composition of the clay fraction. Except for areas with cambrian or younger bedrock where carbonates and swelling clay mineral may be abundant, the clay fraction was mainly composed of illite, chlorite, vermiculite, caolinite, mica, amphibol, quartz, felspar and mixed minerals. As a general rule, when carbonate and montmorillonite are missing, the clay fraction is composed of 30-50% illite, mica and mixed minerals, 20-30% caolinite, chlorite and vermiculite and 20-50% quartz, felspar and rock forming minerals other than mica. As an approximation, caolinite, chlorite and vermiculite were assumed to be present in equal amounts.

From the mineralogical literature representative values for the loss on ignition were estimated for different minerals. **Table 3.**

Disregarding the relatively small content of clay minerals in coarser fractions and, except for mica, the relatively small loss on ignition in the rock forming minerals, Pusch used these values and the normal distribution of clay minerals to calculate a correction factor in the order of 0.05 times the clay content¹⁾. The correction assumes that the soil is free mainly from carbonates.

Table 3. *Loss on ignition for different clay minerals. (From Pusch, 1974).*

Mineral	Symbol	Weight loss in per cent at heating from 105°C to 800°C
Kaolinit	K	12 - 13
Illit	I	6 - 7
Montmorillonit	M _m	9 - 10
Vermikulit	V	≈ 7
Klorit	Kl	10 - 12
Muskovit	M	4,5 - 6

1) The clay content is calculated on material <20 mm.

Based on the values from Ekström, 1927, Thalme & Almén (1975) proposed a correction for carbonate free soils of 0.09 times the clay content if this is 15-25% and 0.06 times the clay content when this is more than 25%. The different corrections are compiled in Fig 3.

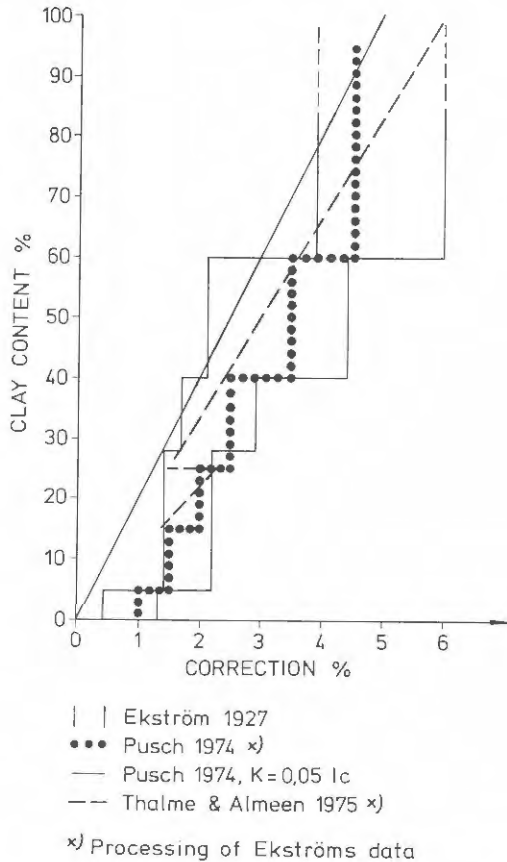


Fig 3. Different corrections for the loss on ignition in carbonate free soils.

The figure shows that the correction calculated by Pusch largely explains the empirical values found by Ekström. However, the correction values from Ekström are generally about 1% higher than the theoretically calculated values. As far as is known, the values from Ekström are the only experimentally proven values for Swedish soils. For soils containing carbonates large losses in weight occur when the carbonates de-

compose at heating into oxide and carbon monoxide. Complete decomposition of calcite (calcium carbonate) gives a loss in weight of 44% of the content of CaCO_3 . For dolomite (magnesium carbonate) and siderite (iron carbonate) the loss of mass is less, generally 10-30% according to Pusch.

To obtain a true correction of the loss on ignition for carbonates, their character as well as their part of the solid phase must be determined. Calcium carbonate, however, is the dominating type and generally a determination of the carbonate content is used as a measure of the content of CaCO_3 and the correction is made accordingly.

Calcium carbonate occurs in various amounts in most soils, except in the upper dry crust where it has disappeared due to decomposition and leaching. The content of calcium carbonate must therefore always be determined. Alternatively, the soil can first be examined regarding carbonate content by sprinkling hydrochloric acid on a sample of the wet soil (the so-called frizzle test). If no frizzle at all occurs, the soil can be assumed to be mainly free from carbonates. If there is some frizzle the carbonate content must be determined and the loss on ignition corrected accordingly.

Thalme & Almén (1975) recommend that the temperature at ignition be raised to more than 900° to ensure complete decomposition of the calcite.

In other methods, where the organic content is determined by measurement of the carbon content, the soil is sometimes treated with hydrochloric acid in order to remove the carbonates. This method is not suitable for determination of the loss on ignition, as the treatment can cause other changes in weight on heating. Shell remains are as far as possible removed before carbonate content and loss on ignition are determined. Occurrence of sulphur compounds, such as ferrous sulphides, also gives rise to changes in weight on heating. In soil with a high

content of ferrous sulphides or other sulphur compounds the organic content cannot therefore be directly determined from the loss on ignition.

Estimation of organic content by determination of the loss on ignition is consequently not as simple a method as it seems. To achieve an acceptable determination of the organic content in a soil also determination of the clay and carbonate contents with subsequent corrections, must be performed, as well as the heating procedure. The corrections normally used in Sweden are valid only for Swedish soils. The method is not suitable for determination of organic content in sulphide soils or soils with high contents of carbonates.

Furthermore, determination of clay content in organic soils can create special problems.

In determination of high organic contents, however, the determination of loss on ignition is one of the better methods to use, due to the relatively large mass of sample that can be used and because the sources of error in these cases can as a rule be neglected. Loss on ignition is almost always used for the determination of organic content in peat. In this connection, the remains after heating are generally denoted ash and the ash content of peat is often used instead of the organic content.

3.3 Treatment with hydroperoxide

Various methods for determining organic content by treatment with hydroperoxide have been proposed (e.g. Robinson 1930, Jackson 1958). In this connection hydroperoxide is poured upon the natural samples, with or without pre-treatment, until the reactions cease, whereupon the samples are washed, dried and weighed. Material washed away is evaporated, ignited and weighed, after which the loss in weight, due to the treatment with hydroperoxide is determined by comparison with dried, untreated samples. Samples with carbonate content must be specially

treated. From a practical view, the methods are complex and require timeconsuming attention as the reactions need to be speeded up by heating. At the same time, boiling-over must be prevented. A simpler method of treatment with hydroperoxide was proposed by Rosenqvist (1946). In this method, the results in losses on ignition were compared between an untreated sample and a sample treated with hydroperoxide.

The methods were evaluated by Silfverberg (1957). He found that in treatment with hydroperoxide, in general only part of the organic material is oxidized and that this part may vary within very wide limits. Treatment with hydroperoxide can furthermore cause other changes in the sample which affect the losses on ignition. Silfverberg thus found that the method could not be used to determine the organic content in soil and especially not with subsequent heating at low organic contents.

3.4 **Determination of organic content by determination of the content of organic carbon**

The method considered most accurate for determination of organic content in soil is the determination of the content of organic carbon. This utilizes dry or wet combustion. In this context the total carbon content is often measured and afterwards corrected for carbonate coal. The content of organic carbon is then converted to organic content. At this conversion of measured mass of organic carbon to organic content or humus content, the Wolff or van Bemmelen factor is normally used, which states that 58% of the organic material is composed of organic carbon. Ekström (1927) and Silfverberg (1957) found in their literature surveys that considerable variations in this value had been reported. The value of 58% is generally used for conversion of the content of organic carbon to organic content. However, it must be made clear that this is not an exact value but a standard value for guidance. Mohr (1979) states that for Danish soils the content of organic carbon in

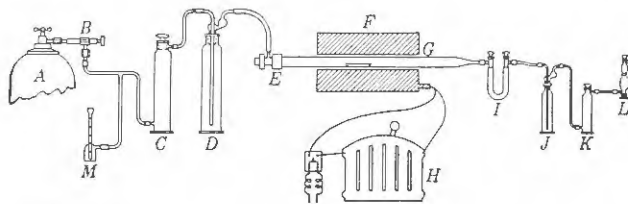
the organic material may be 10-20% lower in some organic materials with low degrees of decomposition, such as waste and some forms of peat.

However, in comparisons with other methods it must be observed that an error in the conversion factor gives a calculated error in the organic content itself and not, as with other sources of errors in various methods, an error in per cent of the total solid material.

3.5 Determination of organic carbon by dry combustion

The carbon content in a sample can be determined by combustion of the sample in a stream of oxygen gas or air free from carbon dioxide. The combustion gases are then purified from dust, water and sulphur compounds. Any carbon monoxide present should preferably be catalytically burnt to carbon dioxide and the evolved carbon dioxide then determined quantitatively. Combustion normally takes place at a temperature of 900°C. In this procedure, the total carbon content is determined. Carbon in the form of carbonates in the soil must first be destroyed or alternatively determined and corrected for when the organic content is to be determined. Elimination of carbonate coal can be achieved by pre-treatment of the sample with hydrochloric acid. The method is old and there are many variants. It is still considered to be one of the most accurate methods. In its older forms, the method requires comprehensive equipment and specially trained staff, and is very time-consuming. Fig. 4.

Today, use is made of more or less automatic equipment, so-called carbon analyzer, based on the principle of dry combustion. Combustion can take place at various temperatures, e.g. 550°C and 1000°C and in this way organic carbon and carbonate coal can be separated. In simpler models, the carbon dioxide is captured in ascarite bottles whose change in weight is measured. In the fully automatic carbon analyzers, the



- A. Oxygen tank
- B. Pressure regulator
- C. Bottle containing ascarite for cleaning from CO₂
- D. Bottle containing sulphuric acid for cleaning from water
- E. Removable connection
- F. Electrical furnace
- G. Combustion tube
- H. Rheostat for temperature regulation
- I. Wash bottle for dust
- J. Wash bottle for sulphur
- K. Wash bottle for water
- L. Bottle for absorption of carbon dioxide
- M. Manometer and safety valve

Fig. 4 Relatively simple equipment for determination of carbon content. From Kolthoff & Sandell 1950.

carbon dioxide is measured e.g. through the electrical conductivity of the combustion gas or with an infra-red detector, as in the latest models. Fig. 5. The carbon analyzers are, however, very expensive and the mass of the samples that can be analyzed is often very limited.

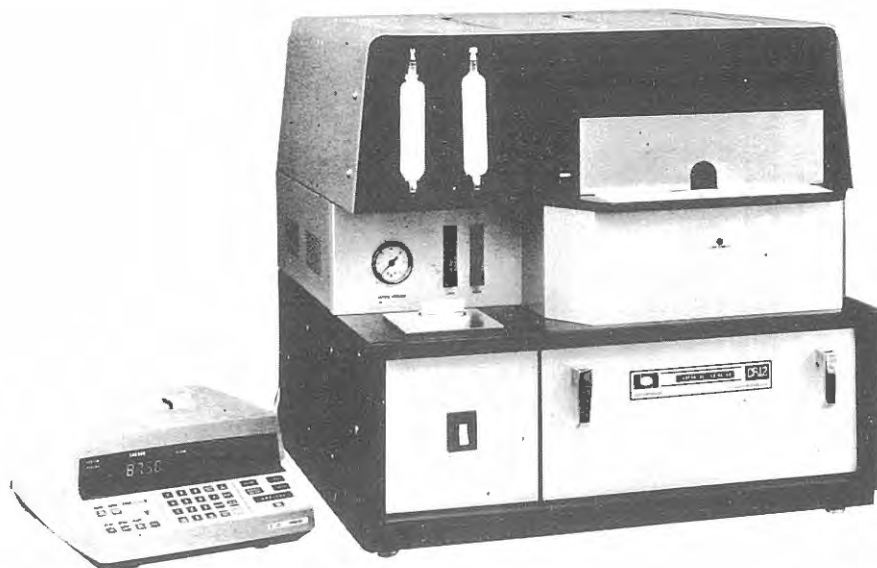
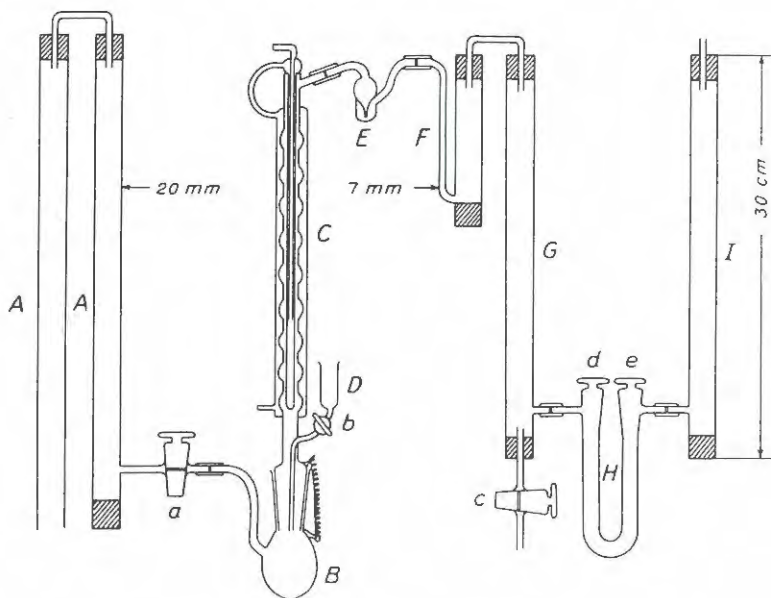


Fig. 5. Automatic carbon analyzer model Leco CR-12.

3.6 Determination of organic carbon by wet combustion

Wet combustion of carbon takes place by means of a strong oxidizing agent and an acid, usually potassium or sodium chromate and sulphuric acid. The soil sample is boiled in the oxidizing fluid and the carbon dioxide is cleaned and accumulated in the same way as at dry combustion. A number of variations regarding oxidation agents, acids, additives, catalytic combustion of carbon monoxide, together with collection and measuring of the carbon dioxide, have been proposed.

Combustion can take place at normal pressure while air cleaned from carbon dioxide is blown or sucked through the system. It can also take place under vacuum with subsequent flushing of the apparatus with air free from carbon dioxide. The degree of heating can also be varied. The more the heat used, the larger the proportion of the most resistant organic material that is oxidized. Every variant of wet combustion thus



- A. Tubes for absorption of CO_2 in the air
- B. Reaction bottle
- C. Condenser
- D. Buret for acid
- E. Bubble counter containing conc. H_2SO_4
- F. Tube containing granulated zinc
- G. Tube for absorption of water
- H. U-tube containing ascarite for absorption of CO_2
- I. Tube filled with granulated calcium chloride to prevent backward diffusion of moisture and air

Fig. 6 Equipment for wet combustion according to McCreedy & Hassid (1942).

has its own degree of recovery expressed in per cent of the total carbon content. In more accurate methods, the recovery degree is very high.

Besides measuring the weight of developed carbon dioxide at wet combustion methods of measuring the gas pressure (Slyke & Folch, 1940) and the volume of the carbon dioxide have been proposed (Hillebrand & Lundell, 1953). Moum, 1967, proposed a simple method of measuring the gas

pressure occurring when a soil sample in a closed system, previously evacuated to vacuum, is boiled in an oxidizing fluid consisting of sodium dichromate and sulphuric acid. The later methods encounter special problems as the temperature influences the measurements. Sources of error also occur if the soil contains for instance salts, as other gases than carbon dioxide can then be developed.

The total carbon content in wet combustion is measured in the same way as at dry combustion. When determining organic content correction for the carbonate coal is necessary.

3.7 "Rapid" titration methods

The first so-called rapid titration method was developed by Schollenberger in 1927. In this method a determination of the quantity of oxidizing agent used is performed by titration after wet combustion. In the Schollenberger method, the soil is mixed with a certain amount of potassium dichromate, whereupon sulphuric acid is added. The mixture is heated during stirring and the temperature is kept at 170-175° for 90-120 sec. The mixture is then cooled and diluted with water containing sodium fluoride. Thereafter it is titrated with ferrous ammonium sulphate using diphenylamine as indicator. Apart from its relative rapidity, the method has the advantage for determination of organic content that it is only the organic carbon that is oxidized and measured indirectly, so that no correction for carbonate coal is necessary.

A number of variants of the Schollenberger method have been suggested (e.g. Degtjareff 1930, Tiurin 1931 and 1934, Allison 1935). The simplest method was presented by Walkley & Black 1934.

The Walkley & Black method mainly differs from the Schollenberger method in that heating has been omitted. The potassium dichromate is dissolved before the addition of water and the heat release that takes

place when the sulphuric acid is diluted is sufficient for oxidation of the main part of the organic material.

Different procedures give different degrees of recovery of the organic carbon.

A survey by Crowther (1935), where samples were sent to different laboratories, showed that dry as well as wet combustion and different fast titration methods yielded primarily the same results in organic carbon, provided that the degree of recovery of the method was taken into consideration. Similar results have been obtained in other surveys (e.g. Smith & Weldon 1940). Large differences reported by some researchers like Allison (1960) are valid for deviations calculated as percentages of the amount of carbon at very low carbon contents and are hardly interesting from geotechnical aspects. The Walkley & Black method has been used widely and is a standard method in Australia, Great Britain, New Zealand and Hungary, among other countries.

The main sources of error in the rapid titration methods are possible deviations from the chemicals and the procedure by which the degree of recovery is determined. Walkley, 1947, studied different sources of error in the method. Occurrence of sodium chloride yields too high an organic content. The error in the carbon content is, however, no greater than one twelfth of the chloride content. This would give an error in the order of 0.2% in the organic content for a soil with a salt content of 3% in the pore water. In soils with very high chloride content, the error cannot be neglected, but in these soils the results must be corrected for the salt content. The washing away of chlorides is unsuitable, as part of the organic material may disappear during washing. Iron sulphide could give erroneous values when oxidized. Walkley as well as Vestervall (1963), however, came to the conclusion that the content of ferrous sulphide in an air-dried sample is too small to have any practical importance in this connection. Mohr (1979) has also found that pyrite hardly affects the results from the Walkley & Black method.

Several researchers point out that the presence of manganese dioxide could give too low an organic content since part of the oxidation could take place as a reaction with this. As far as is known, no investigator has shown this effect for natural soils. Walkley (1947) examined manganese-rich soils and found that many different forms of manganese oxides occur in soil. At determination of organic carbon only one, laboratory prepared manganese oxide, had any evident effect on the results.

Metson (1956) as well as Vestervall (1963) state that the degree of recovery of organic carbon in fast titration methods may vary somewhat depending on the organic content, but that the variation is relatively small.

A considerably modified form of wet combustion with accompanying titration was proposed by Nömmik 1971. The method requires special equipment. The sample is boiled in a solution with chromic oxide and phosphoric acid at 160°C for approx. 3 hours. The evolved carbon dioxide is captured in potassium hydroxide, the carbonates are precipitated with barium chloride and the excess potassium hydroxide is titrated with hydrochloric acid.

In soils with carbonate contents, the sample has to be pretreated and the same equipment can be used for pretreatment of the sample with metaphosphoric acid and determination of carbonate coal. The Nömmik method is to be considered as an advanced wet combustion method rather than a rapid titration method. It is judged to be very accurate. A similar but simpler and less accurate procedure was proposed by Thorn & Shu in 1951. This method was recommended by Silfverberg (1957).

3.8 The colorimeter method

Scollenburger (1945) was first to suggest a further simplification of the "rapid titration methods". At oxidation with dichromate the colour

of the oxidation fluid changes from orange to green when Cr^{6+} is transformed to Cr^{3+} . By measuring the intensity of the green colour with a colorimeter a faster and simpler measurement of the organic carbon content is obtained. The method was rapidly taken up by a number of researchers e.g. Graham (1948), Carolan (1948), Barkoff (1954) and Riehm & Ulrich (1954).

Graham (1948) found that the colorimeter method was insensitive to the user, while the titration could be difficult for untrained staff. He therefore suggested a method with fast wet combustion according to Walkley & Black, with accompanying colorimeter measurements using a light filter for wavelengths close to 620 μm . The method is primarily the same as is used today. With the Graham method, it is necessary to wait until the finer particles which are stirred up at combustion and the following dilution have settled, before the fluid can be decanted and the colour intensity determined. Carolan (1948) suggested filtration instead of waiting to accelerate the determination.

Riehm and Ulrich (1954) found that a heating procedure as in the "rapid titration methods" gave a higher degree of recovery. Barkoff (1954) used the colorimeter method with good results in Finnish soils with up to 85% organic content. Different procedures with different heating all yielded good results.

A larger investigation on Swedish soils with emphasis on colorimeter determination was made by Vestervall 1963. He found that the colorimetric measurement should be made at wavelengths over 590 m in order to avoid influence of unconsumed oxidizing agents and recommended measuring in the interval 600-640 m . During control measurements on inorganic material the colorimeter yielded a low reading. In a comparison between the colorimeter method (K) and determination of organic content with advanced wet combustion methods (O), the relation $O = K \cdot 1.0376 - 0.22$ obtained demonstrated a coefficient of correlation of 0.9993. If the advanced wet combustion methods are considered to give

true values, this means that the colorimeter-determined organic content should be reduced by 0.2%. In samples with common salt as an additive, errors of the same order as at titration were obtained, i.e. a salt content of 3% in the pore water yields an increase in the measured organic content in the soil amounting to about 0.2%. Sulphide contents which after air drying could yield a noteworthy effect can be considered as out of the question.

In the same way as in the titration methods, every method of wet combustion has its own degree of recovery. It is therefore important that exactly the same procedure be used in calibration as in the following determinations. The method with wet combustion and colorimeter measurement is a standard method in Finland and Hungary.

3.9 Measurement with reflections

New methods for determining the organic content by measuring the reflection of rays with different wavelengths with spectrophotometer are being tested at present. (Krishnan et al, 1980, Stoner & Baumgardner, 1980). The methods are, however, in an early stage of development and therefore their usefulness and accuracy have not been determined.

4. METHODS FOR DETERMINATION OF CARBONATE CONTENT

The carbonate content is usually determined by wet combustion with a non-oxidizing acid, frequently hydrochloric acid. All types of wet combustion methods with measurement of the developed carbon dioxide may be used, including the methods with titration of developed carbon dioxide (e.g. Nömmik). The carbonate content may also be measured by comparing the losses on ignition at heating to 900°C for untreated samples and for samples where the carbonates have been removed by treatment

with hydrochloric acid. An alternative method is to compare the losses on ignition with heating to 550°C and 900°C respectively. At these determinations it is usually assumed that the carbonates are calcium carbonates, which is the dominating carbonate in Swedish soils. When the amount of dolomite or siderite is great, the error becomes important. For determination of calcium carbonate only considerably simpler methods are available. The simplest method is the frizzle test where a wet sample is sprinkled with a few drops of diluted hydrochloric acid and the frizzle is studied. From a scale for the intensity of the frizzle, the carbonate content can be roughly estimated according to Table 4.

Table 4. *Rough estimation of the carbonate content in a frizzle test, (according to Thalme & Almèn, 1975).*

Frizzle	Estimated content CaCO_3
None	< 0,5%
Very weak	0,5 - 1%
Weak	1 - 3%
Strong but not prolonged	3 - 5%
Strong and steady	>5%

The method, however, has to be considered as very subjective and the main advantage with the frizzle test is that soils with carbonate content can be fairly well separated from soils free from carbonates. The simplest apparatus for quantitative determination of the calcium carbonate is the Passon apparatus. Fig. 7.

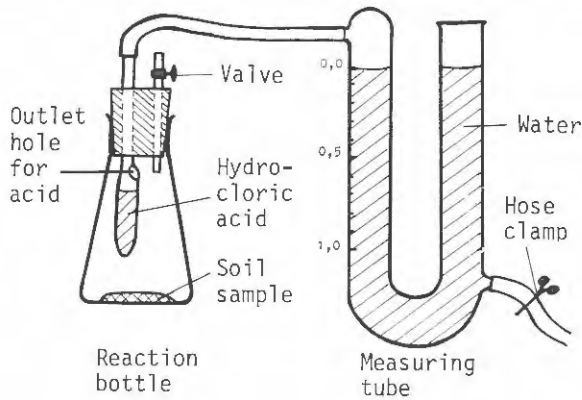


Fig 7. Apparatus for determination of the carbonate content (the Passon apparatus). From Thalme & Almèn, 1975.

In the test, the hydrochloric acid is poured onto the sample by tilting the bottle. After the evolution of gas the water is let out of the U-tube until it is equally high in both legs. 1 mol CaCO_3 yields 1 mol CO_2 and the carbonate content can be calculated from the evolved gas volume.

The Chittick apparatus (Fig. 8) is a further development of the Passon apparatus. The tilting reservoir has been replaced by a drop funnel with pressure equalization and the measurement system has been improved.

Moum proposed in 1967 an apparatus in which the reaction between acid and soil takes place in a closed system evacuated to vacuum. The amount of evolved CO_2 is then measured indirectly with a mercury manometer.

Fig. 9.

All the simple apparatuses are somewhat sensitive to temperature due to the relation between volume, pressure and temperature for gases. During the test itself, very little heat (max 2°) is evolved and no cooling or extra temperature measurement besides measurement of the

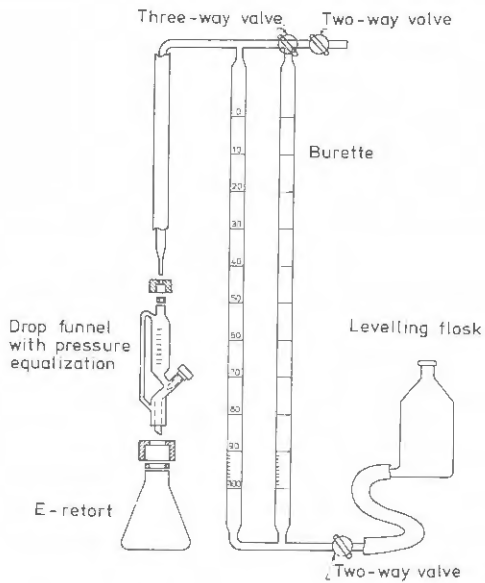


Fig. 8. The Chittick apparatus (from Fredriksson & Kjellin, 1973).

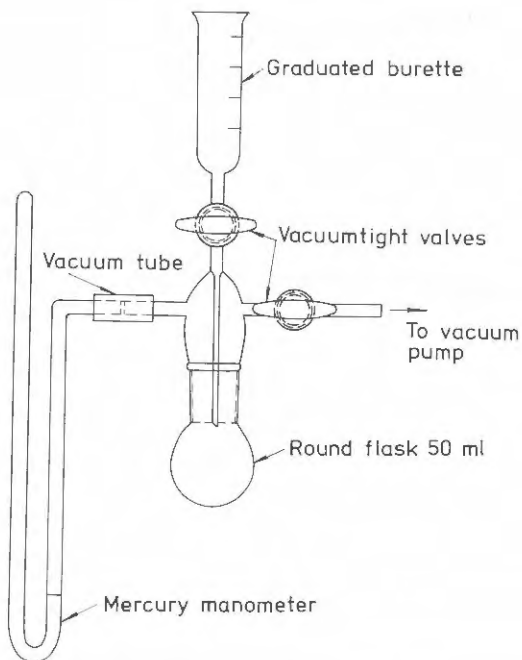


Fig. 9 Apparatus for determination of the carbonate content according to Moun 1967.

room temperature is needed. When using the Passon or Chittick apparatus, the current air pressure must be measured. Calibration of the apparatus is performed with known amounts of pure calcium carbonate. Sources of errors may be the appearance of manganese oxide or evolution of hydrogen sulphide. These errors can be eliminated by adding ferrous chloride and copper sulphate respectively to the hydrochloric acid.

A comparison between determinations of calcium carbonate with simple apparatus (Passon and Chittick) and more complicated wet combustion methods was made by Fredriksson & Kjellin 1973. This comparison showed that the simple methods yielded results almost identical to the more complicated methods. The spread in results for the simple methods was not greater than for the more complicated methods. The simple methods furthermore have the advantage that calcite (calcium carbonate) and dolomite (magnesium carbonate) can be separated from each other. Dreimanis (1962) has shown that in samples within the finer soil fractions, the total reaction between hydrochloric acid and calcite is concluded within 30 seconds. During this period only 4% of the dolomite has been dissolved. Dissolution continues for 15-45 minutes depending on whether the material is stirred or not. After this period, approx. 95% of the dolomite has been dissolved. For complete dissolution 6-24 hours is required.

In determination of calcite and dolomite a reading of the volume (or pressure) is therefore made 30 seconds after the start of the reaction, which continues for 15-45 minutes. The shorter time is valid when the sample is stirred with a magnetic agitator which is started after the first reading. When the evolution of gas has stopped, a new reading of the volume (or pressure) is made. **Fig. 10.**

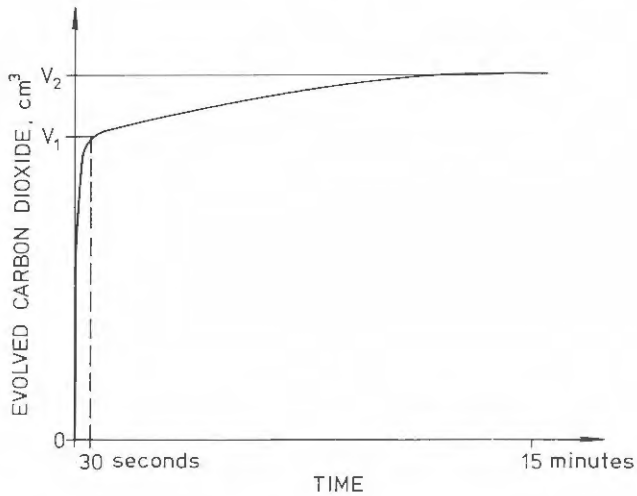


Fig. 10 Evaluation of gas versus time at treatment of calcareous soil with hydrochloric acid in the Passon- or Chittick apparatus. (From Dreimanis 1962). When using the Moun apparatus readings are taken of pressure instead of volume.

The content of calcium carbonate is calculated from the calibration curve of the apparatus and

$$V_{\text{calcite}} = V_1 - 0.04(V_2 - V_1)$$

As reading No 1 is equal to calcite + approx. 4% of the dolomite and reading No 2 is equal to calcite + approx. 95% of the dolomite the dolomite can be evaluated from

$$V_{\text{dolomite}} = (V_2 - V_1) / 0.91$$

A certain amount of carbon dioxide evolved by dolomite corresponds, however, to a dolomite content approx. 8% lower than the calcite content corresponding to the same amount of carbon dioxide. The dolomite content can therefore simply be calculated from the calibration curve for calcium carbonate by

$$V_{\text{dolomite}} = V_2 - V_1$$

5. METHODS FOR DETERMINATION OF SULPHIDE CONTENT

Sulphur occurs in many different forms in the soil. It may occur as free sulphur, sulphide, sulphite or sulphate. It may also occur as compounds difficult to dissolve such as pyrite and as part of the organic substances in the ground. The organic sulphur is, however, usually less than one per cent of the organic substance. In anaerobic conditions below the ground water level sulphur also occurs as easily oxidized sulphides and free sulphur. In the presence of free sulphur, the different kinds of ferrous sulphides are transformed to pyrite. Therefore the content of free sulphur in soil containing ferrous monosulphide may be expected to be low. With access to oxygen, e.g. at draining, the easily oxidized forms are oxidized to sulphuric acid and/or sulphates. Bacterial processes may also lead to oxidation of the sulphur.

The oxidation and reduction of the sulphur are reversible processes. Therefore, the state of the sulphur in the upper soil layers varies with season and ground water level. The total sulphur content is also affected by the sulphur fall-out from precipitation and winds as well as from leaching. (Rasmussen 1961, Wiklander, 1976).

Below the ground water level, the conditions are more stable. Investigations of different occurrences of sulphur in soil have mostly been effected by agricultural chemists and concern mainly the upper soil layers. However, some investigations of deeper profiles have been made by geologists and have mainly concerned "svartmocka" which is a black soil rich in ferrous sulphides (Georgala 1980, Nystrand 1980, Bergqvist 1983). Despite ferrous sulphide and its importance for the technical properties of soil being of great interest for several years, no direct investigations of contents for these purposes have been made until recently (Nystrand 1980, Bergqvist 1983).

For agricultural and forestry purposes, usually only the total sulphur content is determined. This is performed with dry combustion and can

today be carried out in automatic incinerators with sulphur detectors. According to e.g. Georgala (1980) there may be a connection between the content of ferrous sulphide and the content of total sulphur in the anaerobic zone below the ground water level. Determination of sulphide content in soil is usually performed by wet combustion followed by collection and quantitative determination of evolved sulphur.

The mass of sulphur determined is then converted to content of ferrous sulphide. Quantitative analyses of ferrous sulphide shown in the literature have yielded values equivalent to the total content of acid-soluble sulphides. All acid-soluble sulphide is then assumed to be ferrous sulphide and any other metals are assumed to occur only in very small quantities. (Georgala 1980).

The acid-soluble ferrous sulphide may occur as an amorphous iron monosulphide (FeS), magnetic pyrite ($\text{Fe}_{(1-x)}\text{S}$) and other forms such as $\text{Fe}_{(1+x)}\text{S}$ or Fe_3S_4 . Attempts have been made to separate and quantify different sorts of acid-soluble ferrous sulphides but this has failed (Georgala 1980). Normally the acid-soluble ferrous sulphide is assumed to consist of iron monosulphide (FeS). In soils from the anaerobic zone containing iron monosulphide, the difference between the total sulphur content and the content of iron monosulphide can be assumed to be a measure of the content of pyrite (Georgala 1980).

Soil samples that are to be analysed with respect to ferrous sulphide must not be oxidized before analysis. To avoid errors in analysis, the samples must be handled very carefully. In very accurate analyses the samples, which are usually taken with piston sampler, must be placed in plastic bags where the air is replaced with nitrogen and further sample preparation takes place in a glove box with a pure nitrogen atmosphere to prevent oxidation (Jacobsson 1984). Such high demands cannot normally be fulfilled, but it is important that the samples be sealed rapidly and tightly in the field and that the examination be made as soon as possible.

Different methods of analysis, which all imply wet combustion with collection of the sulphur evolved and accompanying titration for determination of the mass of sulphur, have been presented by e.g. Wiklander and Hallgren (1949), Berner (1963), Steinrath (1966) and Hesse (1971). The methods are identical in principle and it is mainly the chemicals used that vary. Other analysis methods than titration may be used. Georgala (1980) describes e.g. a method where an unoxidized sediment sample is freeze-dried in vacuum and then weighed and placed in a reaction bottle where hydrochloric acid is added. Evolved hydrogen sulphide is transmitted by a nitrogen stream to an absorption bottle with a cadmium chloride solution where the sulphur is precipitated as cadmium sulphide. Fig. 11. The precipitate is filtered, dried and weighed and then the mass of cadmium sulphide is converted to content of ferrous sulphide.

In control tests with pure sodium sulphide, the method appeared to have a very good degree of recovery. The technique with freeze drying for sample preparation should facilitate dissolution of the sample at extraction and no heating is necessary according to Georgala (1980).

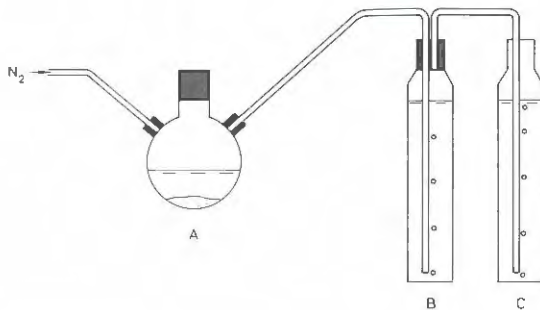


Fig. 11. Apparatus for determination of the sulphide content (Georgala 1980).

- A. Reaction bottle
- B. Absorption bottle
- C. Control bottle

As sample preparation and analysis at determination of the sulphide content are complex and time-consuming, a diploma project at the University of Linköping was initiated to select the most simple testing procedure and, if possible, improve it (Svensson 1983). In this work, among other things, the possibility of performing the quantitative determination of sulphur by spectrophotometer measurement was investigated. The relation between absorption of light and sulphur content in the cadmium sulphide solution proved, however, to be non-linear and therefore such an evaluation became complicated. The most suitable procedure according to Svensson (1983) is primarily based on the Steinrath method (1966). In the analysis, a natural, wet, unoxidized specimen is taken from a larger sample. The specimen is weighed and immediately placed in the reaction bottle and then the drop-funnel is put in place, see Fig. 12. This procedure must be executed rapidly to minimize oxidation. Two more specimens are taken from the larger sample in direct connection with the first and the water contents for these are determined. The mean value from these water contents is then used to calculate the dry weight of the analysed specimen.

Before and during the extraction, a slow stream of nitrogen is passed through the apparatus. As extraction agent 6 molar hydrochloric acid saturated with tin chloride is used. This extraction agent dissolves the iron monosulphide but not the pyrite. In the reaction bottle there should be some pellets of granulated zinc together with the sample. These pellets oxidize during the extraction process. In this way, the reducing effect of the solution is maximized and liberated sulphur is prevented from oxidizing to sulphate.

The extraction fluid is added in portions during a period of approx. 15 minutes. The solution is then boiled to dissolve the sample. The hydrogen sulphide evolved is transmitted to an absorption bottle by the stream of nitrogen. This continues for approx. 15 minutes after boiling has stopped. The transmission tube to the absorption bottle is cooled

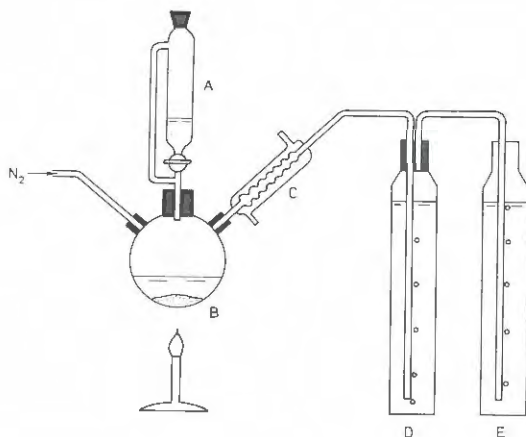


Fig 12. Apparatus for determination of the sulphide content according to Svensson 1983.

- A. Drop-funnel with pressure equalizer
- B. Reaction bottle
- C. Cooler
- D. Absorption bottle
- E. Control bottle

with a water cooler to separate gaseous hydrochloric acid. The solution for absorption consists of an acetate buffer containing cadmium. Percolating hydrogen sulphide causes cadmium sulphide to be evolved, whereby the solution for absorption is coloured yellow. After passing the absorption bottle, the stream of nitrogen passes one more absorption bottle which ensures that all sulphur has been absorbed in the absorption bottle. This is indicated by the fluid in the control bottle remaining uncoloured during extraction.

After extraction, a known amount of dissolved iodine is added to the absorption fluid and the sulphide is then oxidized. The amount of iodine added must be larger than that consumed and the fluid is then titrated to obtain the surplus of free iodine. In titration a solution of sodium thiosulphate is used, with a standard starch solution as indicator.

Svensson performed comparative analyses on synthesized ferrous sulphide and obtained a very good degree of recovery. In tests on natural clays, it was found that longer heating periods and stirring could be required for complete dissolution of the sample. With some reservation as to how representative the sample is for natural conditions, the method is considered to give satisfactory results. In spite of the simplifications it is not, however, a particularly simple method.

6. INVESTIGATIONS PERFORMED AT SGI

6.1 Comparative analysis with carbon analyzers and wet combustion at different institutions

Within the larger project "organic soil" a special investigation has been conducted as to the accuracy of measurement of organic and carbon contents which can be obtained in routine tests.

In this investigation, samples from different levels from a test site in south Gothenburg have been dried, pulverized and homogenized. "Identical" specimens have then been sent to various institutions having access to modern carbon analyzers and in one case wet combustion according to Nömmik has been performed. The carbon analyzers at the various institutions have been of different makes and models.

The methods for determination of carbonate coal have also varied between the different institutions. The Passon apparatus, pretreatment with hydrochloric acid, combustion at different temperatures and wet combustion according to Nömmik have been used. Furthermore, the carbonate contents for the same material have been determined with the Moum apparatus at the Norwegian Geotechnical Institute as well as at SGI.

Contents of total carbon and inorganic carbon have been determined in the analysis. The organic carbon has then been estimated by subtracting the inorganic carbon from the total carbon. The organic content has been estimated on the assumption that the organic material contains 58% organic carbon. The carbonate content has been estimated as the content of calcium carbonate which contains 12% inorganic carbon.

A compilation of the measured total carbon contents yields very similar results between the different institutions and methods. A typical spread in measured total carbon content is approx. 0.2% calculated for the total weight of sample. Fig. 13.

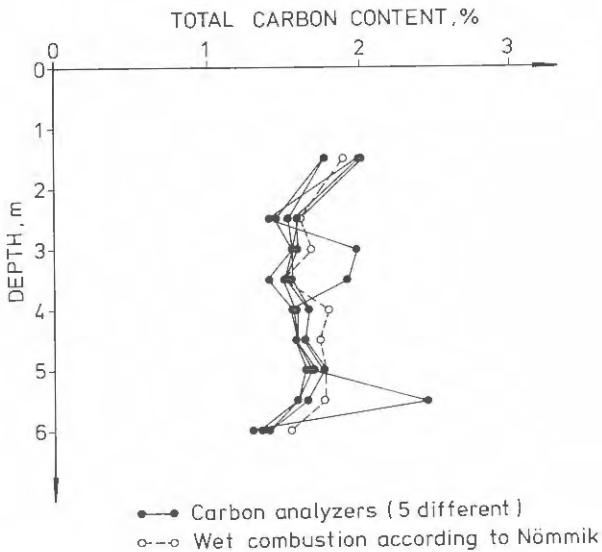


Fig. 13. Compilation of measured total carbon contents at comparison between different carbon analyzers and wet combustion according to Nömmik.

A compilation of the determinations of carbonate coal yields similar results for the different methods. Fig. 14. Also here, the spread is in the order of $\pm 0.15\%$ of the total weight of the sample. As the inorga-

nic carbon constitutes only 12% of the carbonates, the spread in carbonate content becomes more than $\pm 1\%$. It should be noted that the simple methods yield results which are just as reliable as the more complicated methods.

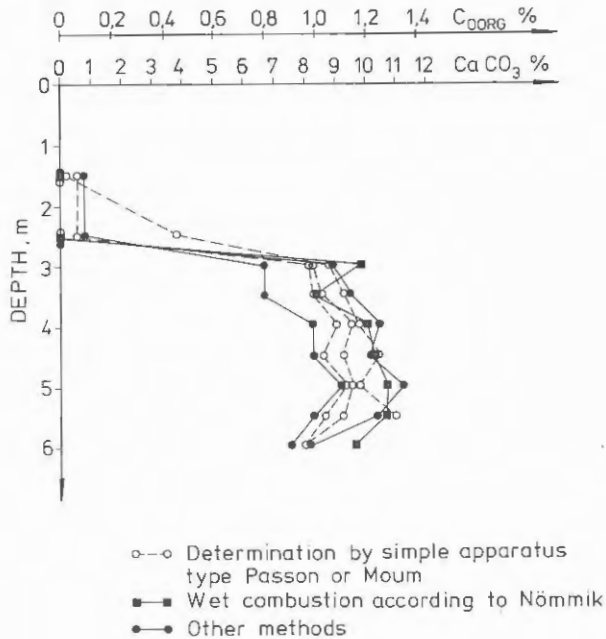


Fig. 14. Compilation of determinations of inorganic carbon and carbonate coal with different methods.

The content of organic carbon has then been calculated by reduction of the total carbon content with the relevant value of the content of inorganic carbon determined at the same institution. The results have been compiled in Fig. 15.

The compilation shows that all determinations yielded similar values but that occasional odd values may occur. In this type of soil with a low organic content, the typical spread was $\pm 0.4\%$ in organic content calculated on the total weight of the sample.

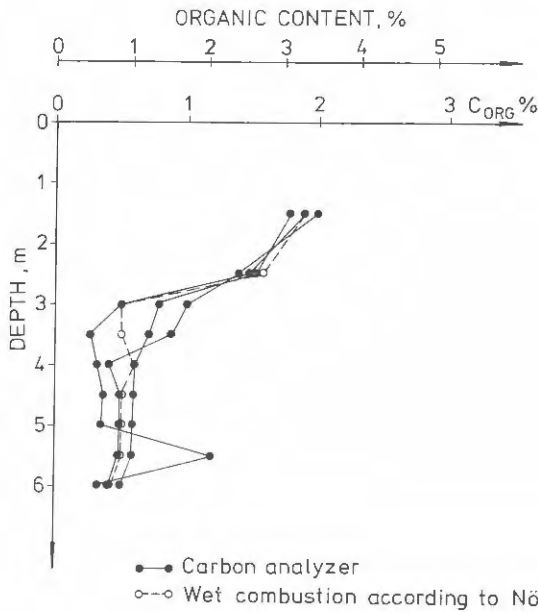


Fig. 15. Compilation of determinations of organic carbon and organic content with more accurate methods.

In connection with the SGI investigation, it was found that a similar investigation had been made at the Swedish Geological Survey (SGU) on sediments from Öresund (Fredrikson & Kjellén, 1973). The results from the two investigations are unanimous. Thus Fredrikson & Kjellén found a similar spread in the results at low organic contents. The spread increased with increasing organic content. The results from wet combustion and carbon analyzers yielded similar results regarding both magnitude and spread. In determination of carbonate contents the simple methods (the Passon and Chittick apparatus) yielded results similar to more advanced wet combustion methods. The spread increases in all methods with increasing carbonate content.

The spread in results is not a direct measurement of the accuracy of the methods. Instead, it shows the variation to be expected when natural samples are homogenized and small specimens are taken for analysis.

The higher the content to be determined, the smaller the specimens. The comparative investigation combined with the investigation at SGU shows that, for natural samples, the most accurate methods can routinely determine the organic content with a typical accuracy of about $\pm 0.5\%$ for low organic contents. The spread increases with increasing organic content. Fredriksson & Kjellén found a spread of about $\pm 1\%$ at an organic content of 6%.

In this connection, the determinations of carbonate content with simple apparatus (Passon, Chittick, Moum) yield results equally reliable to more complicated wet combustion methods. Also in this case, the spread increases with increasing contents. A typical spread for the compared soil specimens with a carbonate content of 9-10% was approx. $\pm 0.8\%$. Fredriksson & Kjellén obtained a spread varying from $\pm 0.3\%$ for low carbonate contents to $\pm 2\%$ at carbonate contents of about 40%.

6.2 Loss on ignition

In a number of different soils, including the material from Gothenburg used in the comparative investigation, various simpler determinations of organic content and carbonate content have been performed. These determinations have in turn been compared to determinations on "identical" specimens performed with carbon analyzer at the Swedish Laboratory for Agricultural Chemistry.

One of these "simpler" methods has been the determination of organic content by measurement of the loss on ignition.

In the determinations of the loss on ignition, the procedure recommended by Thalme & Almén has been used. In this method, the sample is heated to 900°C for at least one hour. Samples with very high contents of organic material are stirred with a platinum needle at least once

during heating. For all samples, the carbonate contents have been determined with the Moum apparatus as well as with carbon analyzer and the loss on ignition has been corrected with 44% of the carbonate content. No obvious contents of dolomite have been found, except at very high carbonate contents (>50%). The clay contents in the samples have been determined by sedimentation analysis after removal of humus. The loss on ignition corrected for carbonate content has then been compared to the organic content determined by carbon analyzer. The difference should be the correction for water evaporation of crystalline water which should be applied to the loss on ignition. The calculated correction is shown in Fig. 16. Materials with very high contents of organic substances or carbonates are not included in the comparison. As shown in the figure, the corrections are mainly within the range given by Ekström 1927. They also correspond fairly well to the correction obtained by Pusch (1974) by processing the values given by Ekström. Differences of some per cent may occur, however. At very high organic contents or carbonate contents, the differences may be even larger. They are, however, probably due to the inaccuracy of the measurements and have therefore not been included in this comparison. Furthermore, the clay content is difficult to determine at high organic contents.

As a correction in steps partly amplifies the differences, it is better to use a correction continuously changing with clay content. A correction of the loss on ignition for evaporation of crystalline water in the order of $(0.75 + 0.06 l_c) \%$ corresponds fairly well to the corrections obtained empirically. l_c is the clay content in per cent.

When comparing organic contents from loss on ignition and carbon analyzers it should be noted that the assumptions are not quite the same. In one case, it is assumed that all organic material is burnt and that the loss in weight is equal to the organic material. In the other case it is assumed that the organic material contains 58% carbon and thus can be calculated from the content of organic carbon. The latter method

is considered to be correct and is used for accurate analysis of organic content in all related sciences. At very high contents of organic material and especially if the soil is free from carbonates, the loss on ignition can, however, be at least as accurate due to the larger amount of sample and the fact that the errors become less important.

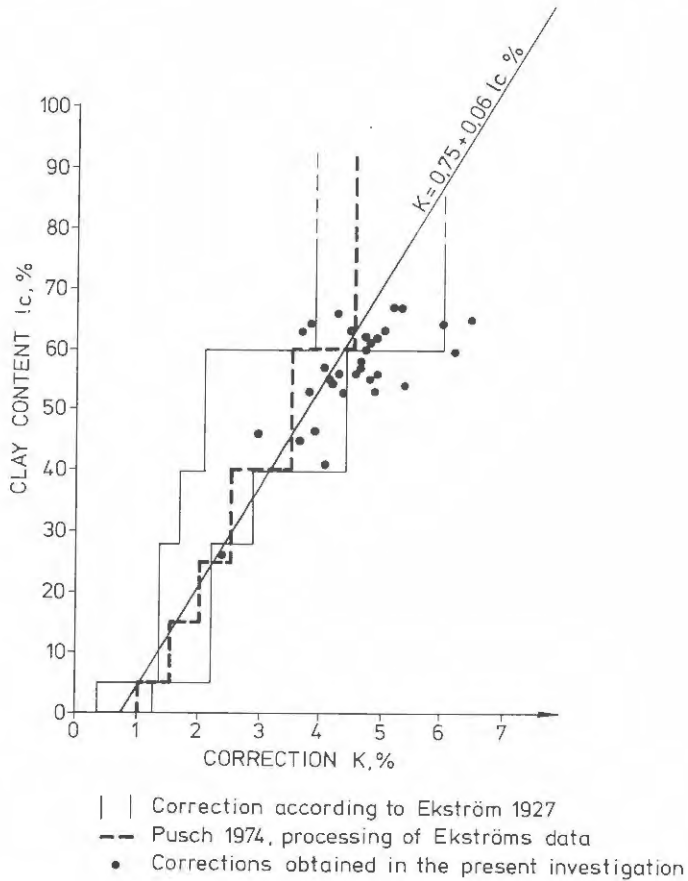


Fig. 16. Calculated corrections for evaporation of crystalline water at ignition.

Results from comparative determinations of organic contents using loss on ignition and carbon analyses are shown in Fig. 17.

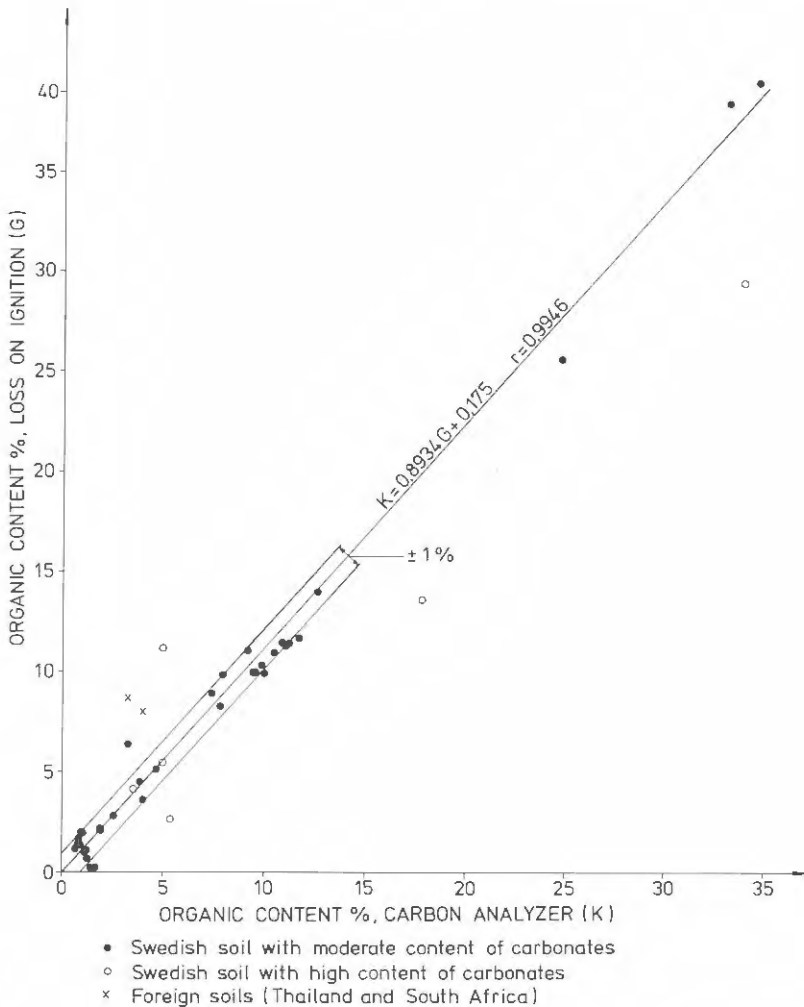


Fig. 17. Comparison between organic content determined at loss on ignition and determined with carbon analyser.

For Swedish soils with moderate lime contents, a clear correlation is obtained between organic content determined with a carbon analyzer (K) and corrected loss on ignition (G); $K = 0.8934G + 0.175$ with a coefficient of correlation $r = 0.9946$. The spread is normally within $\pm 1\%$. In a corresponding comparison between wet combustion (V) and corrected loss on ignition, Vestervall (1963) found the correlation $V = 0.8992G$

+ 0.65 with the same coefficient of correlation. The correlations indicate that the carbon content in the organic substance should be 52% rather than 58% for the investigated soils. If this factor is used, statistical agreement is obtained between corrected loss on ignition and organic content determined with the carbon analyser.

If the carbon factor 52% is used this entails a smaller displacement of the correction points in Fig 16. This displacement is small as no samples with high organic contents have been included and the result shows even better agreement with the empirical values given by Ekström. Ekström used a carbon factor of 54% in his comparisons.

However, the relatively good agreement obtained between organic content and loss on ignition is valid on the conditions that the clay content as well as the carbonate content is determined, that the carbonate content is moderate, that the sulphide content is small and that a continuous correction for evaporation of crystal water of $0.75 + 0.06 l_c\%$ is used. This is valid only for Swedish soils. In Fig. 17, some comparisons for marl and calciferous soils, together with some foreign soils, have been included and the differences are definitely not acceptable.

The importance of determination of the clay and carbonate contents at loss on ignition can be illustrated with the values measured in the profile used for comparison between the carbon analysers.

Losses on ignition in the order of 9.5% were measured in this material. Fig. 18. After correction for the evaporation of crystalline water, about 5.5% remains and after further correction for carbonates, only 1.5%. Despite a loss on ignition of about 9.5%, the greater part of this profile should not be classified as an organic soil as the Swedish lower limit for introducing them under the designation "organic" is at 2%. This final result is in accordance with the results from the carbon analysers.

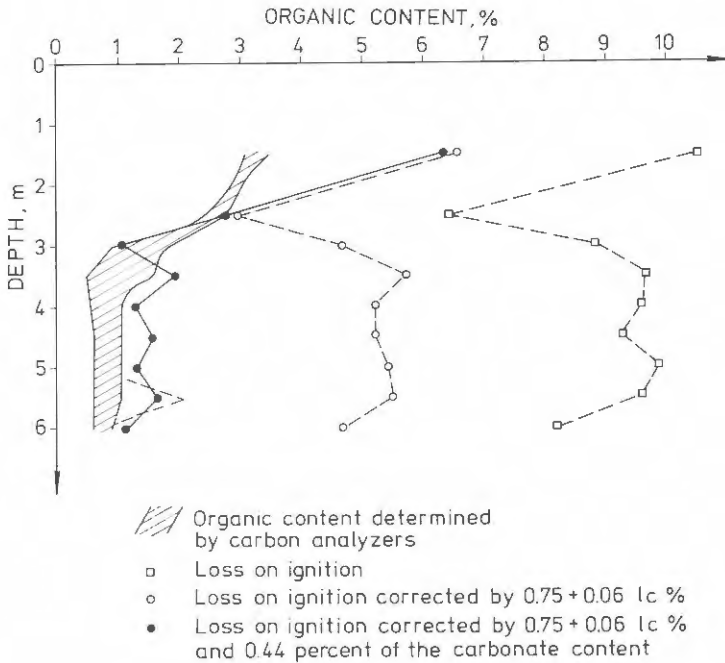


Fig. 18. Correction for loss on ignition in the profile used for comparison.

The carbonate content in the profile used for comparison was 9-10%. Carbonate contents in this range and higher occur in large areas in Sweden.

6.3 Wet oxidation with measurement of the gas pressure

In 1967 Mowm proposed a method for determining total carbon as well as inorganic carbon by measuring the gas pressure evolved at wet oxidation in a closed apparatus which had at first been evacuated to vacuum. The inorganic carbon is determined by wet combustion with nonoxidizing hydrochloric acid. (For details, see the method description in the Appendix). The total carbon is then determined in a separate test by combustion with an oxidizing acid; diluted sulphuric acid saturated with sodium dichromate. In this determination the sample is boiled for 15

minutes. During this procedure a cooling coil in the upper part of the apparatus, a so-called "coldfinger", is flushed with cold water to prevent gaseous acid coming into contact with the manometer. After boiling, the apparatus is cooled to room temperature and the pressure is read. Fig.19 .

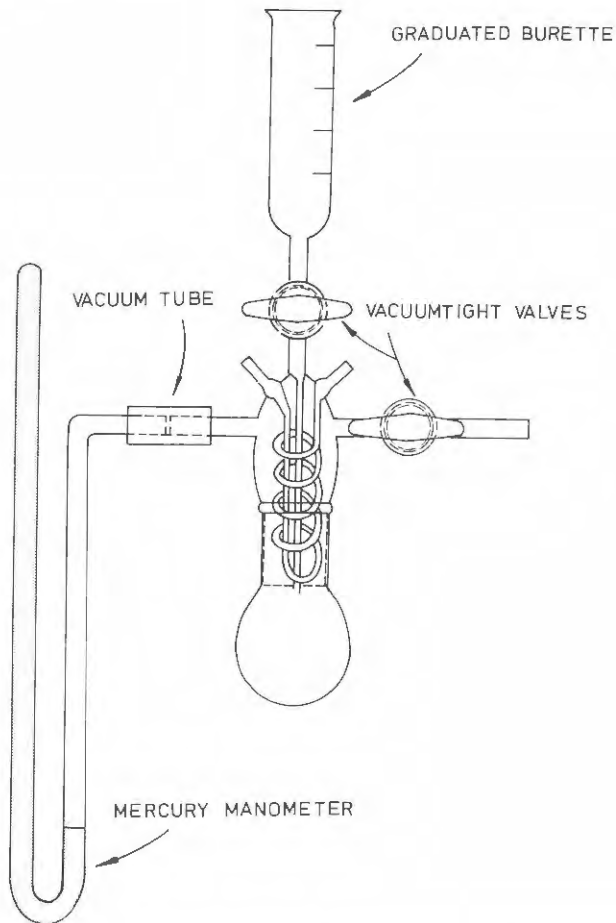


Fig. 19. The Mowm apparatus for determination of total and inorganic carbon.

Tests in the Moum apparatus have been made with a mercury manometer as well as with an electronic pressure transducer. In the attempts to determine total carbon, it proved very difficult to obtain repeatable results. The reasons may be incomplete and varying combustion, leakage in the apparatus during the relatively long testing period with heating and cooling, or problems with the temperature effects as the apparatus is sensitive to temperature.

After relatively comprehensive tests to solve the problems this method for determination of total carbon was abandoned as it did not correspond to the aim of finding a simple and reliable method for determination of the organic content. However, in the tests with the Moum apparatus for determination of the inorganic carbon and carbonate content, very good results were obtained.

Wet combustion of carbonate coal with hydrochloric acid is performed without any supply of heat and practically without heat release and thus all problems associated with changes in temperature can be avoided. Furthermore, the test is completed within 15 minutes and possible leakage can thus be minimized. In **Fig. 14**, results from two series of carbonate content determinations with the Moum apparatus on samples from the profile used for comparisons of more accurate methods are inserted. The tests have been performed at the Norwegian Geotechnical Institute as well as at SGI.

For the other soils in this investigation, determination of the carbonate content on "identical" specimens has been performed at the Swedish Laboratory for Agricultural Chemistry.

The Moum apparatus for determination of the carbonate content is largely equivalent to the Passon and Chittick apparatuses. In all three apparatuses diluted hydrochloric acid is added to the samples and the evolved carbon dioxide is measured. The only difference is in the measuring system.

The results from the investigations with the Moum apparatus have therefore been supplemented with the results obtained by Fredriksson & Kjellin (1973) for the Passon and Chittick apparatuses in the comparison between simple and more advanced methods. Fig. 20.

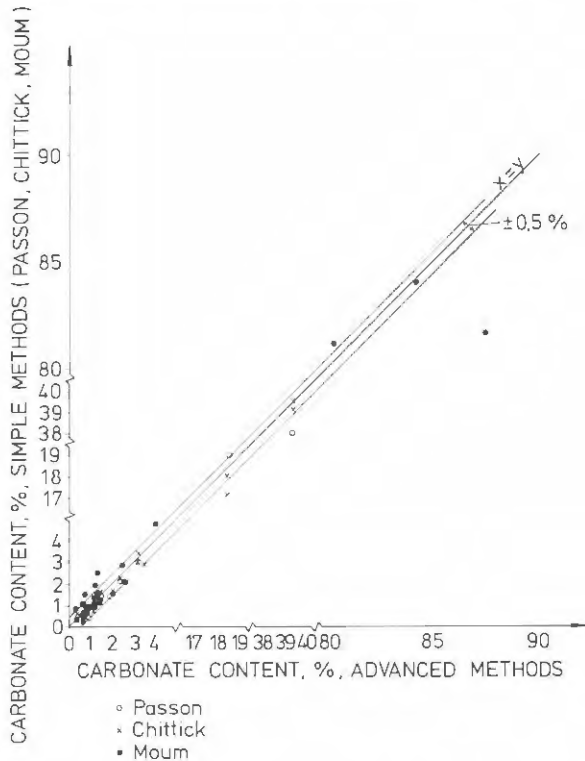


Fig. 20. Comparison between carbonate contents determined with simple and more advanced methods.

As shown in the figure, there is a very good agreement between carbonate contents determined with simple and more advanced methods. The spread is normally within 0.5% calculated on the total sample. Larger deviations may occur, especially at high carbonate contents when the analyses are performed on very small amounts of samples. From the investigation by Fredriksson & Kjellin, it appears that the spread is equally large for more advanced methods as for simple methods. The re-

sult from the investigation of the Moun apparatus was that it can be recommended for routine determination of the carbonate content. The apparatus and its handling are described in detail in the Appendix. Equally good results can be expected when using the Chittick apparatus and also the Passon apparatus normally gives acceptable values.

6.4 "Rapid" titration methods

After contact with colleagues at Földmero es Talavjizsgalo Vallalat in Budapest, a "rapid" wet combustion method followed by titration was tested. The method is almost identical to the Walkley & Black (1934) method. The only difference is that the samples are boiled for 10-15 minutes after adding sulphuric acid. The organic content was determined by this method for twenty samples and the results were compared with results from determinations with carbon analyzers. **Fig. 21.**

The comparison showed a fairly good agreement but the apparatus required a certain training of the staff before this result was obtained. This procedure includes several chemicals and mixtures which must have exact amounts and proportions. One of the mixtures also has to be fresh.

"Rapid" is furthermore a relative concept. In comparison with advanced wet combustion methods or older dry combustion methods, the tested method can probably be considered as rapid. Also these tests, however, are very time-consuming, especially for staff who do not usually perform chemical analysis. Together with the "rapid" titration method, different sorts of colorimetric determinations were tested. These methods were faster and simpler than the titration method and more suited for untrained staff, as stated by Graham (1948). Even the simplest of the colorimetric methods with colour comparison proved to yield results as reliable as the titration methods under these circumstances.

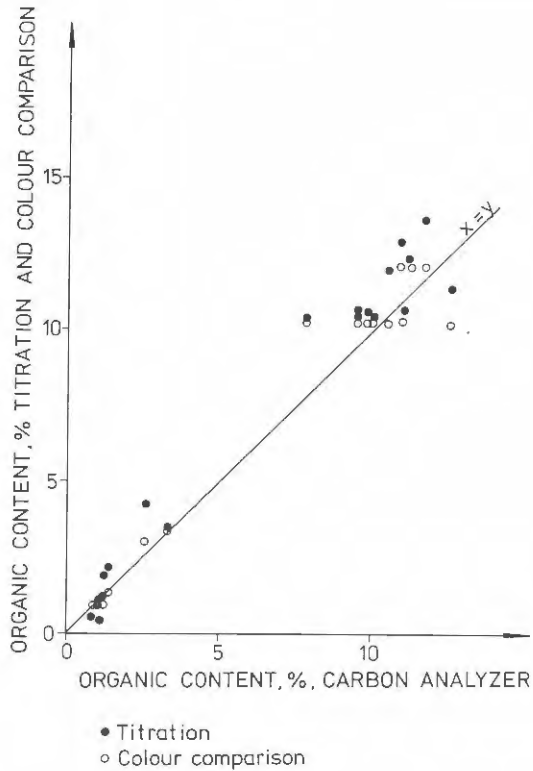


Fig. 21. Comparison between organic content determined with the "rapid" titration method and colour comparison and also organic content determined with carbon analyzer respectively.

The tests with the "rapid" titration methods were therefore concluded after this limited test series.

6.5 Colorimetric determination

The test methods with colorimetric determination were taken from Föld-mero es Talajvizsgalo Vallalat in Budapest and from the Technical Research Center in Helsinki.

The wet combustion technique is identical in the two cases. This is the simplest variant of combustion with potassium chromate and sulphuric acid but without extra heating, i.e. according to Walkley & Black (1934). After dilution with distilled water and clarifying, the colour reaction in combustion is measured.

In the Hungarian method, a number of reference solutions are produced where samples with known organic contents (organic carbon) are combusted according to the standard procedure. The diluted, clear liquid is then transferred to a test tube which is sealed. The steps in organic content between the reference solutions should be about 0.5%.

Determination of organic content is then performed with a simple ocular comparison between the colour for the particular test, where the liquid has been transferred to an identical test tube, and the reference solutions.

In the method from Finland the colour intensity is measured by a photo-electrical colorimeter. The colorimeter method with instrument reading is described in detail in the Appendix.

The simplest method with determination by comparison with a series of test tubes with known organic contents was tested first, see Fig. 22.

The results proved to be approximately equal to the results obtained at titration, Fig. 21. The ocular comparison was, however, rather difficult to perform. Very good ability in separating the colour shades was demanded from the staff and the results could vary somewhat with the room lighting. Furthermore, the reference solutions faded with time, although they were kept in a dark place. Therefore, new reference solutions had to be produced for every testing occasion.



Fig. 22. Reference solutions for determination of organic content.

However, the colorimeter, which is a relatively cheap instrument proved to be very reliable and to give repeatable results. Further investigations were therefore concentrated to instrument readings.

The organic contents determined with the colorimeter for the material used at comparison of various more accurate methods are shown in Fig. 23.

The results are largely equal to the results obtained with the more accurate methods. In this material, also the advantage of the method for determination of the organic content is illustrated, as it is insensitive to the carbonate content and the results do not have to be corrected.

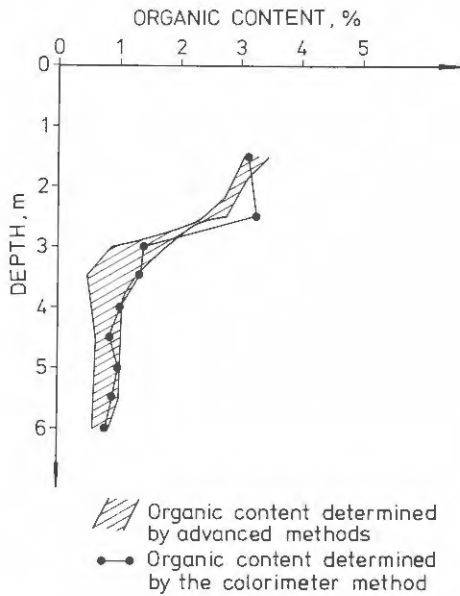


Fig. 23. Organic content determined with the colorimeter method in the profile used for comparison. (See Figs. 13-15). The colorimeter values should be reduced empirically by 0.2%.

Tests on pure calcium carbonate yielded a reading corresponding to an organic content of 0.2%. Vestervall (1963) obtained a corresponding reading in tests on soils freed from organic content by ignition.

In the same soils that have been investigated regarding loss on ignition the organic contents have also been determined by colorimeter measurements. The determinations have been compared with the results obtained in determination of organic content with the carbon analysers, Fig. 24.

All investigated soils have been included in the comparison, Swedish as well as foreign soils, soils with low contents of carbonates and calcareous soils. The results yield a very good level of agreement. The statistical correlation in this investigation between organic content

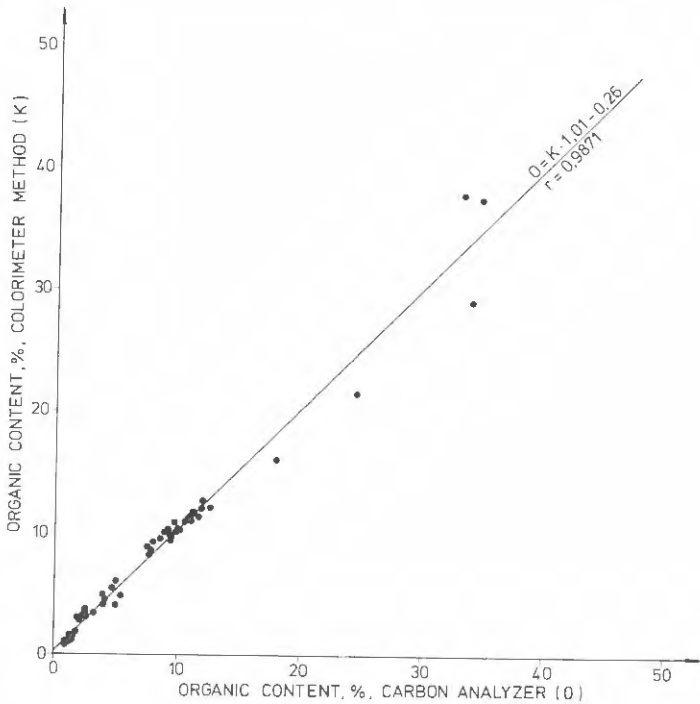


Fig. 24. Comparison between organic contents measured with colorimeter and carbon analysers respectively.

determined with the carbon analyzer (O) and the colorimeter determination (K) was $O = K \cdot 1.01 - 0.26$ with a correlation coefficient of 0.9871. Vestervall (1963) obtained the relation $O = K \cdot 1.04 - 0.22$ with the correlation coefficient 0.9993 in his investigation, where the colorimeter determination (K) was compared with an accurate wet combustion method (O).

The spread in the test results increases from about $\pm 0.5\%$ at low organic contents to $\pm 1\%$ at about 6% organic content. At higher organic contents the spread increases correspondingly. The spread in the results in comparison between the colorimeter and the carbon analyzer is thus approximately the same as the spread when "more accurate" methods are compared with each other.

Slunga (1983) performed a similar investigation on soils from Finland and found a relation $O = K \cdot 1.23 - 0.51$, where O was determined by a wet combustion method. The comparison made by Slunga differs somewhat from the comparisons by Vestervall and at SGI. In the comparisons by Vestervall and at SGI, two determined contents of organic carbon were compared to each other. In the investigation by Slunga, the colorimeter is calibrated for a soil with a known content of a certain organic substance. The colorimeter determination of organic content has been performed on soils with other organic substances and the comparison has then been made with a determination of organic carbon converted to organic content. A condition for complete agreement would be that the organic substance in the calibration material as well as in the soil has an organic carbon content equal to the content assumed at the conversion of the measured carbon content. The investigation by Slunga shows, however, that even if the carbon content can be determined relatively well, there is still the problem of converting it to organic content.

Together the investigations show that the organic content is largely equal to the value determined with the colorimeter reduced by 0.2% and with the reservation that the conversion factor of 58% may vary.

Colorimeter determinations have also been performed to measure the influence of chlorides on the determination of the organic content. For pure clays with a salinity of the pore water of 3% and a natural water content of 100%, the measured errors correspond to an organic content of 0.2%. At a lower natural water content the errors become correspondingly lower. At higher natural water content, the error becomes correspondingly larger, but in these cases there is also usually a considerable true organic content. Corresponding results were obtained by Vestervall (1963). For routine determinations of the organic content, the error is usually so small that it can be neglected. High chloride contents are revealed in wet combustion by evolution of red-yellow fumes when the sulphuric acid is added.

6.6 Determination of sulphide content

After the diploma work by Svensson (1983) the recommended equipment for wet combustion, absorption and titration was acquired. A large series of determinations of sulphide contents was performed on samples from a soil profile near Stockholm. Complementary determinations were made on samples of "svartmocka" with relatively high sulphide content supplied by courtesy of the Department of Geotechnology in Luleå. Furthermore, the total sulphur content was determined for all samples. This determination was performed in an incinerator with a sulphur detector by Micro Kemi AB in Uppsala.

In the investigations the samples from Stockholm were studied partly to determine the errors that could occur in the results of the analysis if the samples are allowed to oxidize beforehand and partly to find whether the nitrogen stream through the equipment could be replaced by an air stream, which would have simplified the equipment.

The investigation of errors due to oxidizing concerned maximum error. The sulphide contents were determined on oven-dried and ground samples and were then compared with the sulphide contents determined in unoxidized samples. In the determinations on dried samples, the nitrogen stream was replaced by an air stream. Comparative determinations of sulphide content were then performed on unoxidized samples with an air stream and a nitrogen stream respectively through the equipment during extraction.

Several tests were performed on the same levels and the results shown in Fig. 25 are mean values. However, the spread in the test results was moderate.

The results show that the sulphide contents in the profile are moderate. The highest contents are in the order of 0.6 per cent of weight of the dry substance. In the layers where these contents occur the clay

is black, however, and must be regarded as very rich in sulphide for conditions in the southern and central parts of Sweden. The measured contents of ferrous sulphide in the "svartmocka" from Luleå varied between 1.8% and 3.4%. Here, the spread in measured sulphide contents was considerably larger.

The results show that in tests with an air stream instead of a nitrogen stream through the equipment during extraction, consistently lower sulphide contents were obtained in the air stream tests than in the nitrogen tests. Consequently, nitrogen cannot be replaced by air in quantitative analyses.

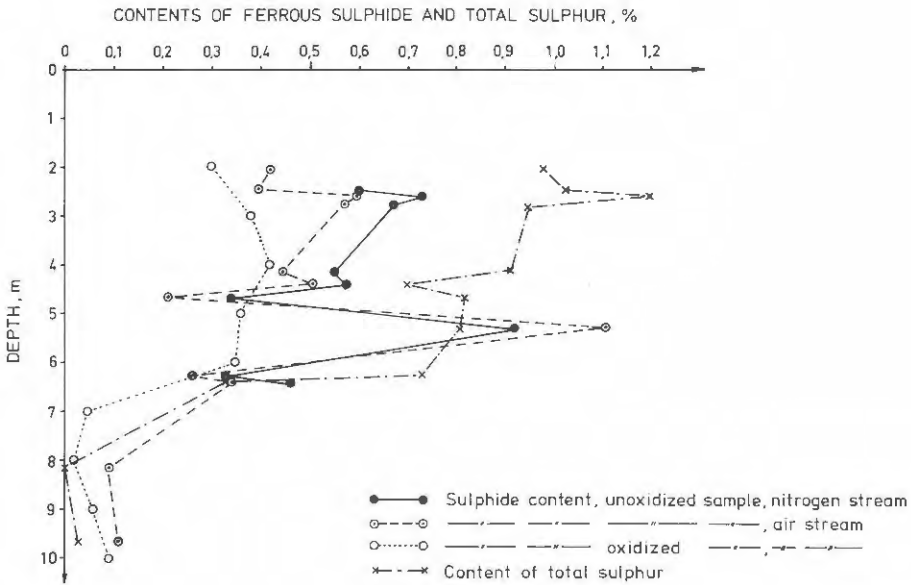


Fig 25. Contents of ferrous sulphide and total sulphur measured in a soil profile near Stockholm.

The tests with oxidized samples showed that a very large part of the sulphide content may be lost if the samples are oxidized before analysis. Prevention of oxidation during sampling, transport, storage and

preparation of the samples must therefore be considered to be of the utmost importance.

Georgala (1980) suggested that there could be a connection between the total sulphur content and the sulphide content in the soil below the ground water level. Fig. 25 shows that the sulphide content in the profile varies largely in the same way as the total sulphur content. In Fig. 26 measured contents of ferrous monosulphide are plotted versus the total sulphur content.

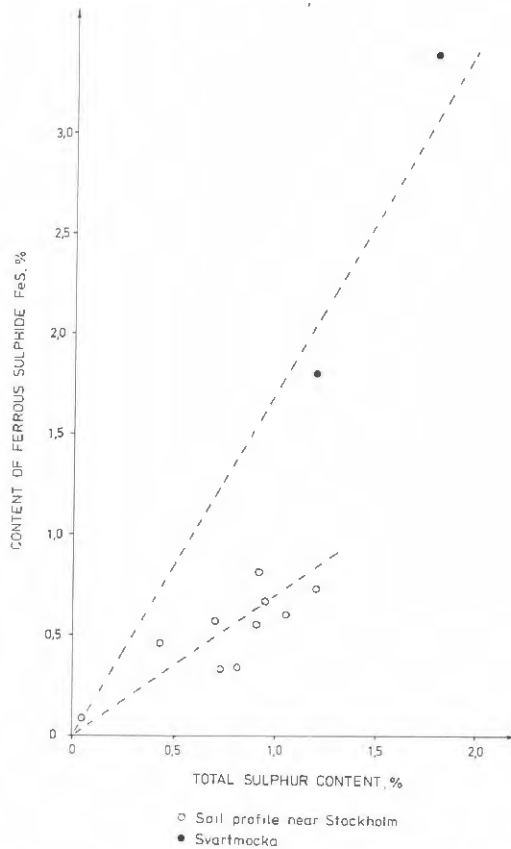


Fig. 26. Measured contents of ferrous monosulphide plotted versus the total sulphur content.

The results show that the sulphide sulphur in the Stockholm profile constitutes 25% of the total sulphur content. According to Georgala (1980) the remaining sulphur can primarily be considered as pyrite. This would mean that in terms of weight, there is approximately twice as much pyrite as ferrous sulphide in the Stockholm profile. A similar relation can be obtained from values measured by Georgala for a soil profile near Kalix. In "svartmocka" from Luleå the relation is different. Here, the sulphide sulphur amounts to about 65% of the total sulphur content, which means that the pyrite content is only one third of the content of ferrous sulphide.

There thus appear to be local correlations between total sulphur content and sulphide content in the soil profiles below the ground water level, although the relations vary from place to place.

How much of the spread in Fig. 26 that depends on the variations in sulphide content in the soil samples cannot be directly estimated. The spread in total sulphur content and sulphide content for dried samples, where the determinations were performed on homogenized specimens from a larger amount of sample is, however, much smaller than the spread in sulphide content determined for small natural samples, see Fig. 25.

Despite the fact that the spread in the determinations of sulphide contents performed on samples from the same levels in this material was moderate, it can still be considered justified to perform a number of tests on specimens from the same sample tube to obtain a representative mean value. The variations in "svartmocka" were, as mentioned above, much larger.

No more accurate reference method for determination of the sulphide content has been available. Control of the equipment used and the reliability of the method has therefore been performed with tests on synthesized ferrous sulphide. The synthesized ferrous sulphide available

on the market is not completely pure, but contains only 70-80% ferrous sulphide.

Tests with the current method yielded contents of ferrous sulphide between 70 and 83% ,which indicates an acceptable accuracy.

7. CONCLUSIONS

Determination of organic content in natural soil samples that have been dried and homogenized can be performed by routine test with an accuracy of about 0.5%. The accuracy is reduced to about $\pm 1\%$ at 6% organic content and decreases further with increasing organic content. This spread is mainly due to natural variations in the relatively small specimens that are analyzed. The amount of sample in the analysis is normally reduced with increasing organic content. Practically the same accuracy as for advanced methods can be obtained with the simple colorimeter method, as the differences in accuracy in the analysis are overshadowed by the variations in the specimens.

Organic contents determined by the colorimeter method should be empirically reduced by 0.2%.

For mineral soils and mineral organic soils, determination of organic content by measurement of the loss in weight at ignition can only be performed under the following special conditions:

- o the carbonate content of the soil is $\leq 20\%$
- o the carbonate content of the soil is determined and the loss on ignition is corrected accordingly
- o the clay content in the soil, I_c , is determined and the loss on ignition is reduced by $(0.75 + 0.06 I_c)\%$. (The reduction suggested by Pusch on the basis of empirical values from Ekström can be used but the accuracy is lower.)
- o the soil has a mineral composition which coincides fairly well with the average Swedish conditions.
- o the sulphide content in the soil is small.
- o the samples are ignited at 900°C for at least one hour.

Even under these conditions the spread is normally larger than for the colorimeter and more accurate methods.

For soils from other geological regions with different mineral compositions, the determination of organic content by loss on ignition can be performed with corresponding restrictions for local conditions and with corrections and testing procedures adjusted for these.

For highly organic soils and especially for peat the loss on ignition can be used to advantage for determination of the organic content. The only condition is that the carbonate content must not be too high. The advantage with the loss on ignition in these soils is partly based on the larger and more representative amount of sample in ignition, and partly on the uncertainty in the conversion from organic carbon to organic content in the other methods.

The result from this investigation indicates that the conversion factor for organic content to organic carbon should be 52% rather than 58%. However, the base is too limited for a recommendation to exchange the earlier adopted value.

Determination of carbonate contents in natural soils that have been dried and homogenized can be performed routinely with an accuracy of about $\pm 0.5\%$ at low carbonate contents ($< 5\%$). The spread in results increases when the carbonate content increases. The spread is primarily due to the variations in the carbonate contents in the relatively small specimens analyzed. The amounts of tested sample are normally reduced with increasing carbonate content.

The same accuracy as for advanced wet combustion methods can in practice be obtained with the relatively simple Chittick or Moun apparatus. The Passon apparatus also normally gives acceptable values. This is probably due to the fact that the differences in accuracy in the analyses are overshadowed by variations in the specimens. In the more simple methods calcite and dolomite can furthermore be easily separated.

As a result of this investigation it can therefore be recommended that routine determinations of organic content for geotechnical purposes be made by colorimetric determination and that corresponding determinations of carbonate contents be made with the Chittick or Moun apparatuses.

In determination of very high organic contents, e.g. determination of ash content in peat, measurement of the loss on ignition is the preferred method.

The method tested for determination of sulphide content with wet oxidizing, absorption of evolved sulphur and titration, yields values with sufficient accuracy for use in geotechnical investigations.

(The colorimetric determination of organic content and the determination of carbonate content with the Moun apparatus together with the tested method for determination of sulphide content are described in detail in the Appendix.)

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DETERMINATION OF ORGANIC CONTENT BY COLORIMETRIC MEASUREMENT

Accessories

- Colorimeter with filter for wavelengths of 620 μm (600-640)
- Balance with an accuracy of 0.01 g
- Erlenmeyer retorts 500 ml
- Voll pipette 20 ml
- Voll pipette 40 ml
- Pipette filler type Peléus ball
- Potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$)
- Concentrated sulphuric acid
- Water-free glucose (pro analysi)
- Distilled water
- Protective equipment (rubber apron, rubber gloves, eye protectors)
- Potassium dichromate solution
Produced by dissolving 86 g $\text{K}_2\text{Cr}_2\text{O}_7$ in distilled water so that the total volume becomes one litre. The solution should be produced in advance as the potassium dichromate requires a certain time to dissolve. The solution should be kept in an airtight glassbottle.
- Glucose solution with exact content.
Produced by dissolving e.g. 2.5 g glucose in 97.5 g distilled water. The solution will then contain 1% organic carbon by weight.

~ The following points should be observed in measurement and handling of the samples.

- Use protective equipment
- Contact with the skin and inhalation of potassium dichromate and sulphuric acid must be avoided.

- In transfer to kyvettes and other handling of liquids it should be remembered that also after reaction and dilution it still is an acid which is being handled.
- Use only reagents intended for analysis.
- The amounts of chemicals to be used must be carefully measured.
- All glassware used must be dry and clean.
- Frothing should be avoided when liquids are transferred from one container to another. Air bubbles that may occur in a sample are often a cause of poor accuracy.
- The test tubes or the kyvettes must be optically clean. Hold them only in the upper part so that no fingerprints occur on the part of the glass passed by the light beam. If any drops have been spilled, the outside of the tube must be wiped clean.

Procedure

The soil samples are dried.

Any larger roots or pieces of wood should be removed. The amount of this should be noted, as well as if the determination is performed on the whole sample or only on the fines.

The samples are ground in a mortar and weighed into the Erlenmeyer retorts, 0.2-3.0 g in each retort depending on estimated organic content in the samples. The amounts are chosen according to the following table

3 g	when the estimated organic content is	0- 3%
1 g	"-	3-10%
0.5 g	"-	10-20%
0.2 g	"-	20-60%

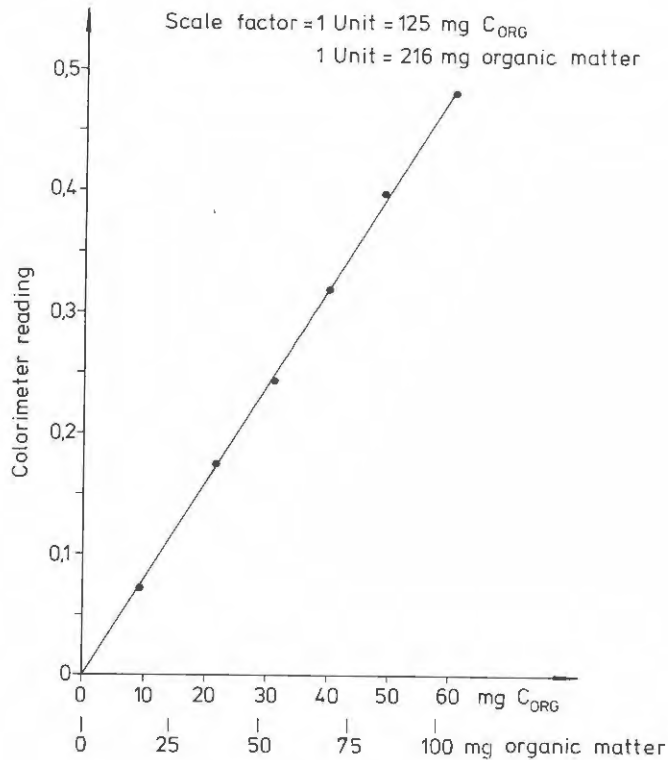
If in doubt, the smaller amount should be chosen or several determinations with different sample amounts performed. Doubletests are always recommended. 20 ml of potassium dichromate solution is added in each Erlenmeyer retort. 40 ml of concentrated sulphuric acid is then carefully added. When the sulphuric acid is added, considerable heat is produced and therefore the retorts should be placed on a suitable base. After the addition of sulphuric acid the retort is shaken. The solutions should stand for 15 minutes and then 200 ml of distilled water is added to each retort and the solutions well mixed. The solutions are then left standing overnight. At the same time, a "zero solution" is produced in an Erlenmeyer retort which does not include any soil but only the reagents.

After the solutions have stood overnight colorimeter measurement is performed. The instrument requires a certain time for warming up and should be switched on in advance. The clear fluid in the solutions is transferred to kyvettes fitting in the colorimeter. The instrument is zero calibrated with the "zero solution" and then the colour intensity in each solution is measured. Zero calibration is performed after each measurement. The reading should be made twice for each sample to check the reading. The instructions for the colorimeter instrument must also be taken into consideration. The amount of organic material in the solution is then obtained from the calibration curve and the organic content is calculated by dividing with the amount of material weighed into the Erlenmeyer retort.

Calibration

Calibration is performed with a series of colorimetric determinations where the soil samples have been replaced by different amounts of glucose solution. If the proposed glucose solution with 1% of organic carbon is used, a number of Erlenmeyer retorts with 0-6 g glucose solution

are prepared. In the same way as for soil samples, potassium dichromate solution, sulphuric acid and distilled water are added. Furthermore, a zero solution is produced. After colorimetric measurement, a calibration curve is drawn with the read colorimeter value on one axis and the amount of organic carbon, i.e. $0.01 \times$ weighed amount of glucose solution, on the other axis. The amount of organic carbon can then be converted to organic content assuming that 58% of the organic material is organic carbon.

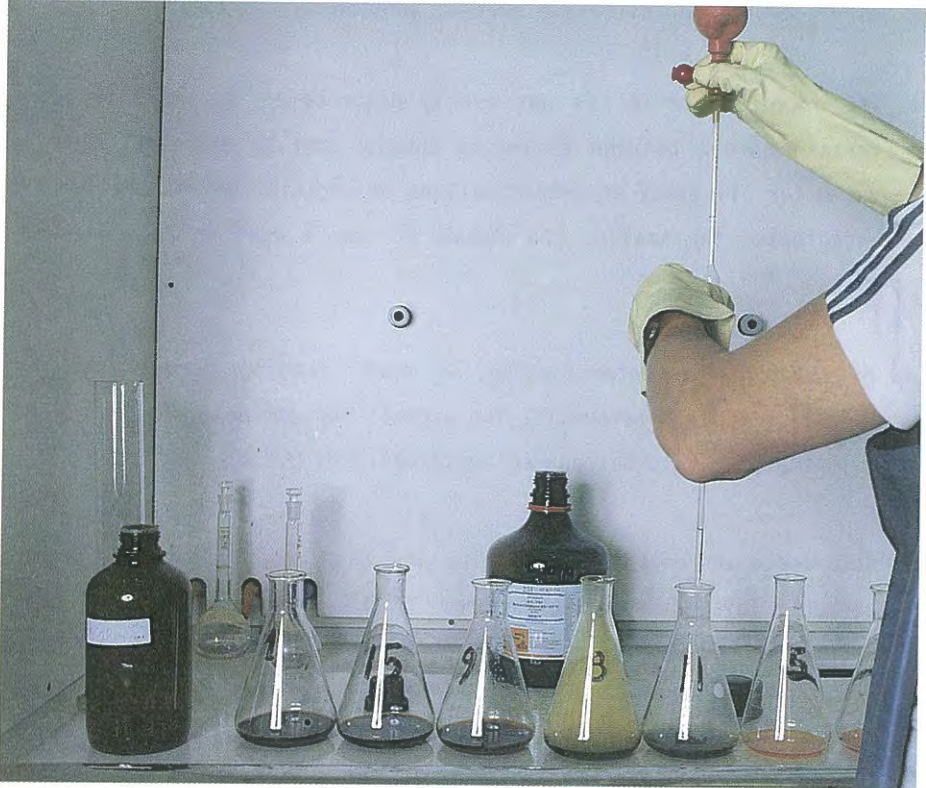


Example of calibration curve for colorimeter.

"The calibration curve" should be a straight line through origo and from this the scale factor is determined. The scale factor is only valid for the procedure followed during calibration and it is important that this procedure be followed exactly in the determination of organic content.

Sources of error

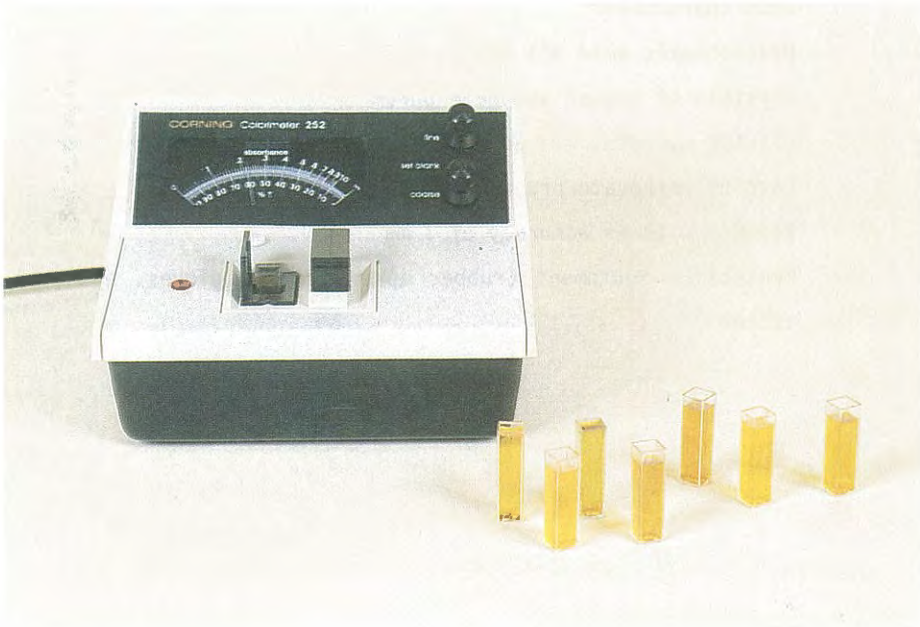
- o A special source of error in colorimetric measurement is deviation from the procedure at calibration in the determination of organic content.
- o The conversion factor between organic carbon and organic content may vary somewhat for different organic substances.
- o The organic material is not evenly distributed in the soil but a certain spread between different samples can be expected. This is valid for all types of determinations of organic content and is more accentuated the smaller the amount of sample used in the determination.
- o In colorimetric determination, a small reading occurs even for totally inorganic materials. The organic content determined with the colorimeter should be reduced empirically by 0.2%.
- o Occurrence of chlorides may give too high a value of the organic content. This error is in the order of 0.2% for normal Swedish clays deposited in salt water. High chloride contents are detected by evolution of red-yellow fumes when sulphuric acid is added. If the chloride content has been determined, the measured organic content must be reduced by one twelfth of the chloride content.



Adding sulphuric acid. The sample in retort No 3 is specially prepared with an additive of sodium chloride to illustrate the yellow-red fumes occurring at high chloride contents.



The samples after reaction, dilution and clarifying.



Measurement in a colorimeter.

DETERMINATION OF CARBONATE CONTENT BY MEASUREMENT OF EVOLVED GAS PRESSURE

(THE MOUM APPARATUS)

Accessories

- Reaction vessel (Fig 1) with graduated burette, valves and outlets to the manometer and a vacuum pump. The lower part has a conical connector fitting to the 50 ml round flasks. This equipment must be specially ordered from a glass workshop.

- Round flasks 50 ml
- Mercury manometer 0 - about 200 mm Hg (alt. vacuum meter)
- Vacuum pump
- Magnetic stirrer
- Room thermometer
- Hydrochloric acid HCl 37% purum
- Crystals of copper sulphate purum
- Diluted hydrochloric acid with an additive of copper sulphate
- Calcium carbonate pro analysi
- Balance with an accuracy of 1 mg
- Protective equipment (rubber apron, rubber gloves, eye protectors)

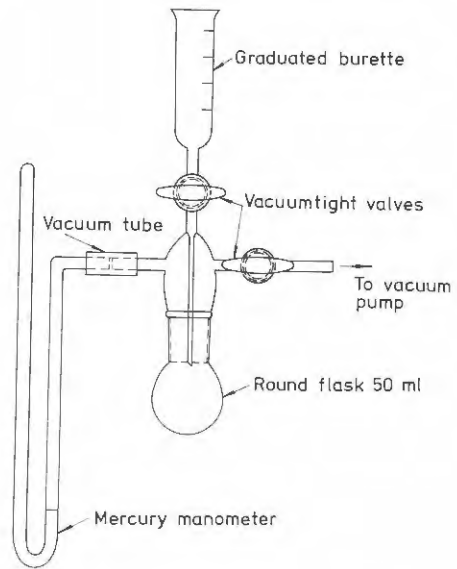
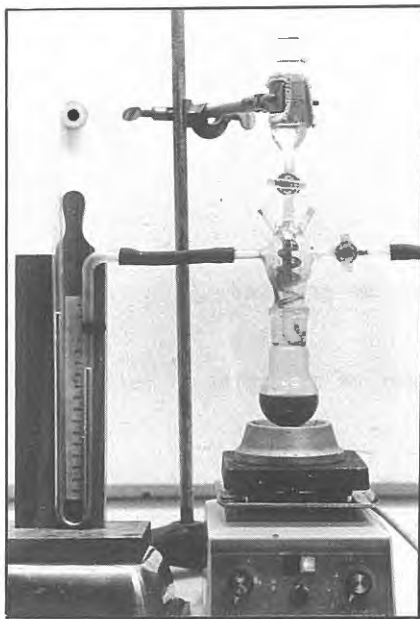


Fig. 1. The Moun apparatus for determination of carbonate content.

Preparations

The hydrochloric acid is diluted with distilled water in the proportion 1 part of hydrochloric acid and 2 parts of water. Crystals of copper sulphate are added until the solution is saturated.

The mercury manometer is controlled and any air in the closed branch is removed by tilting the manometer. Valves and connections are greased with airtight silicone grease. The vacuum pump is checked by test suction, after which the valves are closed and the tightness of the apparatus is checked. Defective rubber tubes are replaced.

Calibration

At reaction between calcium carbonate and hydrochloric acid carbon di-

oxide is evolved. The amount of carbon dioxide produced is measured by reading the pressure on the manometer and is calculated from the general gas law;

$$p \cdot V = n \cdot R \cdot T$$

where p = partial pressure in carbon dioxide produced

V = volume of the apparatus

n = number of carbon atoms = number of molecules of carbon dioxide

R = gas constant

T = absolute temperature °Kelvin

In calibration, the developed pressure is measured when a certain amount of hydrochloric acid reacts with a weighed amount of pure calcium carbonate. In this way the volume of the apparatus is determined. The volumetric determination must be performed several times to secure the value and it must be repeated every time the apparatus is changed by e.g. replacement of tubes. The volumetric determination is valid only for the amount of hydrochloric acid used hereby. Calibration can be performed as follows:

- o About 0.1 g of calcium carbonate is weighed into the round flask which is then fitted to the apparatus. The small magnet used for stirring must be inside the round flask.
- o Evacuate the apparatus with the vacuum pump and close the valve when vacuum has been obtained. Read the pressure p_0 . Ensure that the apparatus is airtight.
- o Add diluted hydrochloric acid in the graduated burette. Ensure that no air bubbles are left in the lower part of the burette. Open the valve and allow a certain amount of hydrochloric acid to be sucked

into the apparatus (normally 25 or 30 ml). As the reaction at the beginning is intense, the hydrochloric acid should at first be added in drops so that bubbling and frothing are limited.

- o Read the thermometer.
- o Read the pressure p and calculate the partial pressure of the produced carbon dioxide. $\Delta p = p - p_0$.
- o Estimate the effective volume of the apparatus with the gas law.

$$p(V_0 + \frac{\Delta p}{10} \pi r^2) = n \cdot R \cdot T$$

where p = partial pressure in produced carbon dioxide, mm Hg

V_0 = volume of the apparatus at pressure p_0 , cm^3

r = inner radius of the manometer tube, cm

n = number of moles of calcium carbonate used (weight in gr/100)

R = gas constant = 62358

T = absolute temperature °Kelvin ($^{\circ}\text{C} + 273$)

Determination of carbonate content

- o The sample is dried. Any non-representative particles such as pieces of wood, shells etc. are removed. This must be noted in the presentation of the results. The rest is crushed so that all material is within the fraction for fines (<0.06 mm).
- o Weigh the sample in a round flask. A suitable amount varies from 2 g in soils free from carbonates or only slightly calciferous soils to 0.1 g in pure calcareous soils.
- o Evacuate the apparatus and read the pressure p_0 .

- o Add the amount of hydrochloric acid for which the apparatus is calibrated. The first drops are added carefully to limit bubbling and frothing.
- o Read the manometer pressure, p_1 , approx. 30 secs. after starting to add acid.
- o Start the magnetic stirrer and let the process continue until all gas evolution has stopped (normally within 15 min.).
- o Read the manometer pressure p_2 .
- o Read the thermometer.
- o Calculate $\Delta p_{\text{calcite}}$ as $p_1 - 0.04(p_2 - p_1)$
- o Calculate " $\Delta p_{\text{dolomite}}$ " as $p_2 - p_1$
- o Calculate the number of moles of calcite (n_1) and dolomite (n_2) from

$$n = \frac{\Delta p \left(V_0 + \frac{\Delta p}{10} \pi r^2 \right)}{RT}$$

- o Calculate the content of calcite as

$$\% \text{ calcite} = \frac{n_1 \cdot 10^4}{\text{sample weight in grams}}$$

- o Calculate the dolomite content as

$$\% \text{ dolomite} = \frac{n_2 \cdot 10^4}{\text{sample weight in grams}}$$

- o The total carbonate content is the calcite content plus the dolomite content.

- o The content of inorganic carbon can be calculated as 12% of the total carbonate content.

Sources of error

- o The most common source of error is leakage in the apparatus when tubes and valves are dried and worn out. The problems can be rectified by replacement of tubes and greasing with silicone grease.
- o If hydrochloric acid is used without adding of copper sulphate there is a risk of evolution of hydrogen sulphide.
- o The calibration is valid only for a specific amount of hydrochloric acid and must be repeated if this or the apparatus is changed in any way.
- o In the reaction, a certain heat release occurs which affects the results. This is, however, normally very low (max 2°) and can be neglected. The room temperature must, however, be constant so that all pressure readings are taken at the same temperature.
- o If the sample contains manganese dioxide and organic carbon, a certain oxidation of organic carbon to carbon dioxide could occur. This risk can be eliminated by using a diluted hydrochloric acid added with 3 g of ferrous chloride ($\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$) per litre.

DETERMINATION OF FERROUS SULPHIDE CONTENT BY WET OXIDIZING AND TITRATION

Accessories

- Extraction equipment (Fig. 1) including:
 - Reaction vessel 100 ml with a tight fitting lid containing four conical tube connectors
 - Metallic clamp ring for the lid on the reaction vessel
 - Nitrogen connection tube with a conical connector
 - Cooler with conical ends
 - Drop-funnel 100 ml with tube for pressure compensation and conical fittings
 - Gas washing bottles 250 ml with filter plate
 - NS-plug with one tube connector and one conical end
- Balance with an accuracy of 0.01 g
- Connection to nitrogen tube with pressure regulator
- Magnetic stirrer with heating plate
- Stirring magneto
- Burette 25 ml with straight tap
- Measuring flasks 250 ml
- Graduated glass 500 ml
- Erlenmeyer retorts 100 ml
- Voll pipette 15 ml
- Voll pipette 50 ml
- Pipette filler type Peléus ball
- Concentrated hydrochloric acid 37% (HCl) pro analysi
- Tin(II)chloride (SnCl_2) pro analysi
- Granulated zinc (Zn) puriss
- Concentrated acetic acid 100% (CH_3COOH) pro analysi
- Sodium acetate, water free (CH_3COONa) pro analysi
- Cadmium acetate ($\text{Cd}(\text{CH}_3\text{COO})_2$) pro analysis
- Sodium thiosulphate solution 0.1 M ($\text{Na}_2\text{S}_2\text{O}_3$) ampoule

- 0.05 M iodine solution, ampoule
- Thyodene, indicator for iodine test
- Distilled water
- Protective equipment (protective coat, protective gloves and eye protectors).

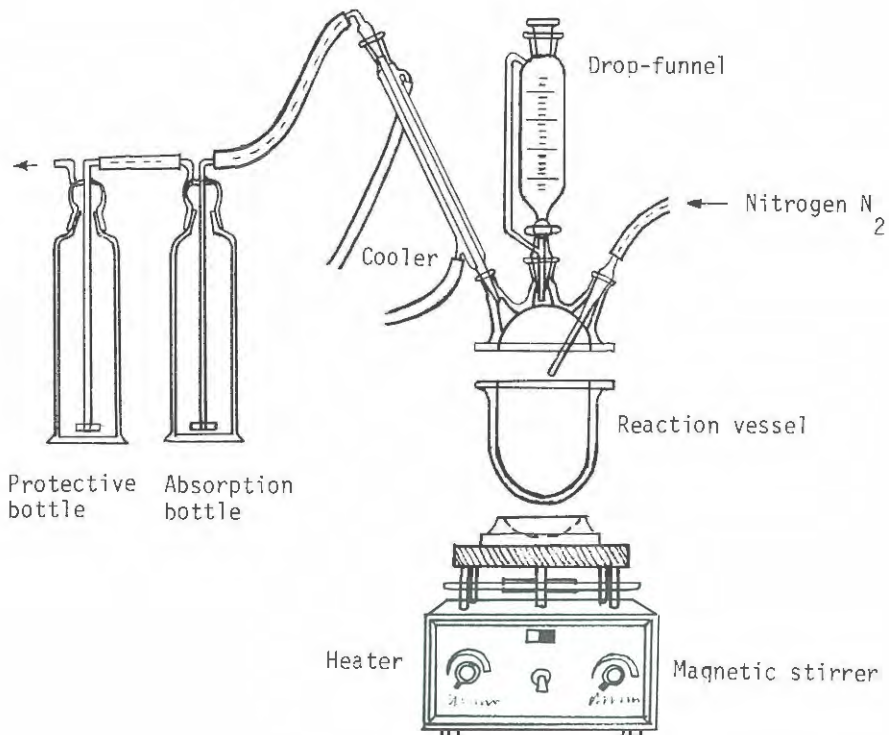


Fig. 1 Equipment for extraction

Preparation of solutions:

- Extraction fluid:

Produced by mixing 450 ml of concentrated hydrochloric acid in 550 ml of distilled water and adding 10 g of tin (II) chloride to the solution.

- Absorption fluid: Acetate buffer with cadmium.
Produced by mixing 200 ml of concentrated acetic acid in 750 ml of distilled water and adding 130 g of water free sodium acetate and 5 g cadmium acetate to the solution.
- 0.1 M Sodium thiosulphate solution (store solution)
Produced by mixing one ampoule with distilled water according to the instructions on the package or by dissolving 24.818 g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in 1.00 litre of distilled water.
- 0.02 M Sodium sulphate solution (user solution)
Produced by diluting 40 ml store solution with distilled water to 200 ml. This solution has a usable life of only one day.
- 0.05 M iodine solution
Produced by mixing one ampoule with distilled water according to the instructions on the package.

The following points must be observed in analysis and handling of samples:

- Contact with the skin and inhalation of hydrochloric acid, acetic acid and their solutions must be avoided. When mixing acid solutions the acid is always added to the water.
- Use only reagents with purity as specified in the list.
- All glassware used must be dry and clean.
- The amounts of chemicals to be used must be carefully measured.
- Only new, unoxidized and undisturbed samples should be used in the analysis.
- It is important that the extraction apparatus is airtight so that no hydrogen sulphide leaks out.
- Use protective equipment.

Procedure

The extraction equipment except for the reaction vessel, is assembled. The nitrogen is connected and the cooler is connected to a cold water tap. 100 ml of extraction fluid is added in the drop-funnel and 200 ml acetate buffer with cadmium is added in both the absorption bottle and the protective bottle. The reaction vessel is placed on the balance and this is tared. A natural wet unoxidized sample, usually taken by piston sampler, is cut into pieces. The outer parts are usually oxidized and have a lighter colour compared to the unoxidized material, which is dark blue or black. 5-10 g of unoxidized material is taken from the inner parts of the sample. This is placed in the reaction vessel and weighed. Immediately after the weighing two pellets of granulated zinc and the stirring magneto are put into the reaction vessel, which is then connected to the other extraction equipment. From the remaining sample, specimens are taken close to the specimen removed for analysis. These are weighed and dried for determination of the water content.

After connection of the reaction vessel with soil sample, zinc pellets and stirring magneto to the equipment, a quiet nitrogen stream is passed through the equipment for some minutes before extraction starts. The nitrogen stream must then continue during the whole extraction procedure. Water cooling is started to prevent gaseous hydrochloric acid being transferred to the absorption solution. The extraction fluid in the drop-funnel is then added in smaller portions during a period of approx. 15 minutes. Stirring is started when the extraction fluid is added. The solution is then heated to just boiling, whereby the sample is expected to completely disintegrate. If this does not happen during the first heating period the procedure is repeated until complete disintegration has been achieved. The nitrogen stream is then passed through for a further 15 minutes so that all H_2S is evacuated. During the extraction procedure the acetate buffer with cadmium in the absorption bottle is coloured yellow when cadmium sulphide is evolved, Fig. 2. If the fluid in the protective bottle is coloured yellow, extraction

should be repeated, either with a smaller amount of sample or with a quieter nitrogen stream. Both may have to be adjusted. After extraction, the nitrogen, stirrer and water cooling are shut off and the equipment is dismantled.

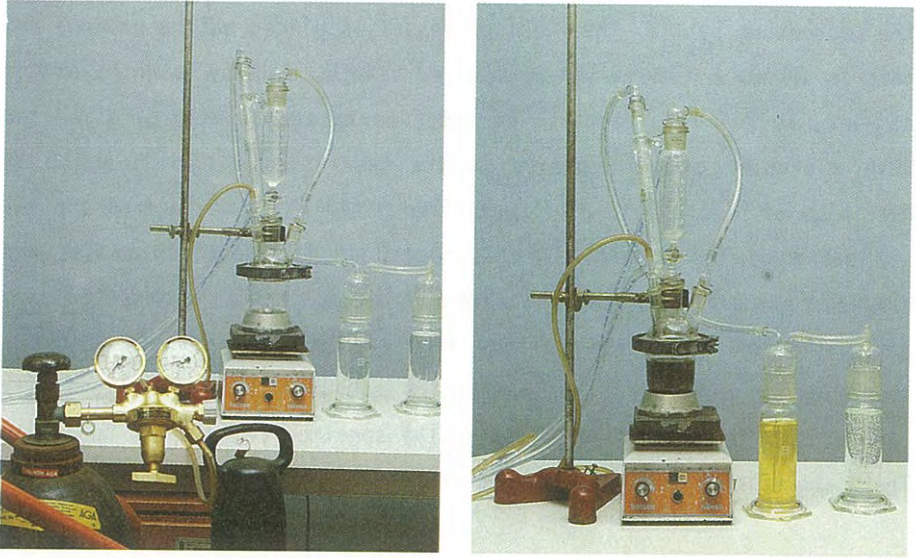


Fig 2. Extraction of sulphide from a soil sample. The absorption fluid is clearly yellow coloured from cadmium sulphide. The fluid in the protective bottle must be completely clear.

15 ml of the iodine solution is added in the absorption bottle with a voll pipette. Great care should be taken when using the pipette and the tip of the pipette should be held against the wall of the bottle for 10-15 seconds so that all the iodine solution is emptied. The solution is then transferred to a 250 ml measuring flask. The absorption bottle is cleaned with distilled water, which is also poured into the measuring flask until all absorption solution is transferred. The solution is then diluted with distilled water to the mark on the measuring flask. The measuring flask is shaken until the contents are uniformly mixed. The colour is now orange.

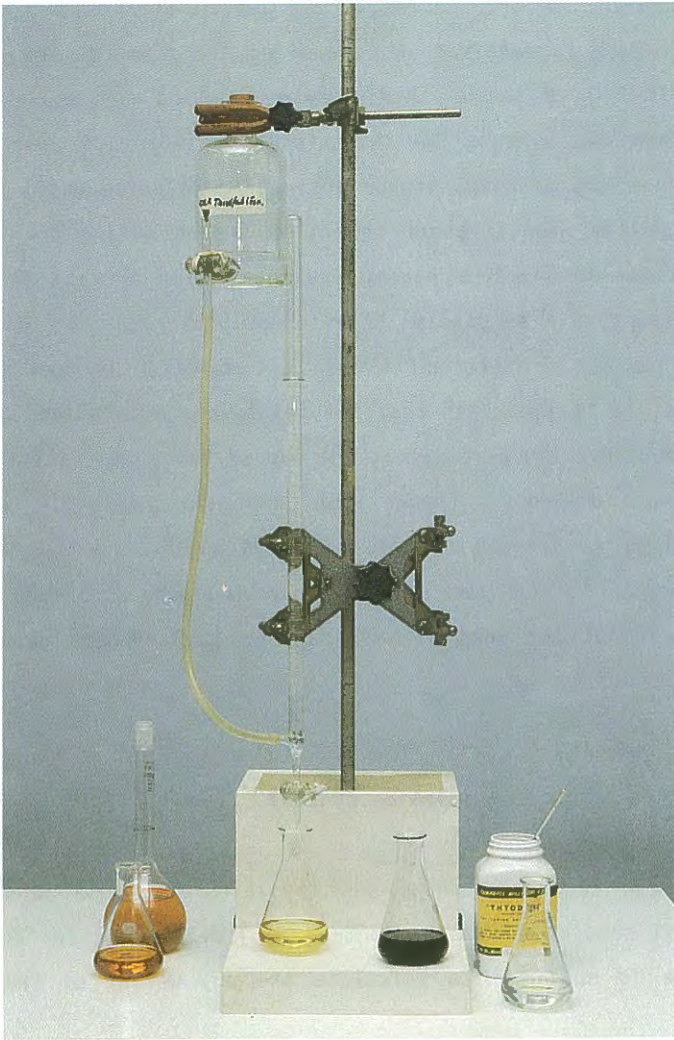


Fig. 3 Titration. The E-retort to the left with the orange liquid contains absorption solution with iodine. The same solution is in the retort under the burette but the liquid here is light yellow after titration with thiosulphate. Thyodene powder is added to the light yellow solution so that the colour changes to brown-purple or purple (the second E-retort from the right). After further titration by drops, the solution becomes completely colourless (the E-retort to the right). The titration is then finished and the burette is read.

50 ml specimens are transferred to 2-3 100 ml Erlenmeyer retorts with a pipette. Care is required when handling the pipette. The burette is filled with 0.02 M sodium thiosulphate solution and is set to zero, Fig. 3. From the burette the thiosulphate solution is added in drops into the E-retort with the orange absorption solution until the colour is light yellow. The E-retort should be shaken during the titration. A few pinches of Thyodene powder are then added, so that the solution becomes brownpurple to purple. More thiosulphate solution is carefully dropped into the Eretort until the test solution becomes completely colourless. It is important that the thiosulphate solution be added in single drops into the E-retort at the end of the titration, as only one drop can make the colour change from purple to completely colourless. The titration is stopped at the change in colour and the fluid level in the burette is read and noted. One or two more specimens should be titrated so that the mean value of sodium thiosulphate solution used can be calculated.

Determination of ferrous sulphide content

The content of ferrous sulphide, FeS, is calculated from the dry weight of the soil sample. The water content (w_n) determined for the natural wet sample is now used to calculate the dry weight of the analysed sample.

The content of ferrous sulphide can then be calculated as follows:

$$\text{FeS in percentage of weight} = \frac{0.4395 \times (15-V)}{m_D} = \%$$

where V = read value on the burette for used 0.02 M natrium thiosulphate at titration in ml.

m_D = the dry mass of the analysed sample calculated according to:

$$m_D = \frac{\text{mass of natural wet analysed sample}}{1 + w_N}$$

Sources of error

- o Deviations in the procedure may imply that the given formula for calculation is not valid. This is primarily the case in the titration of concentrations other than the proposed. It is therefore important that great care be taken when mixing solutions, handling the pipette and at titration.
- o The ferrous sulphide is not evenly distributed in the soil but varies considerably, both within the same sample and between different samples. It is therefore important not to use too small specimens in the analyses. Several analyses may be required for the same sample.
- o Another source of error is the approximation of the content of ferrous sulphide as FeS. Acid-soluble ferrous sulphide occurs in several different forms. Some free sulphur and organic sulphur may also occur. The amorphous ferrous sulphide FeS is, however, considered to be dominant in strata below the ground water level where reducing conditions prevail. Other metal sulphides such as copper sulphide CuS may occur, although in contents that can probably be neglected.
- o Samples taken for sulphide analysis must be treated in such a way that they do not oxidize. At contact with the oxygen in the air the ferrous sulphide oxidizes, for example, to ferrous sulphate, which is not acid soluble. The oxidation of the sample material may give analysed contents considerably lower than the real contents. In very accurate analyses, the samples should be kept and handled under conditions free from oxygen, i.e. in a nitrogen atmosphere.



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