Filter-Press Method of Extracting Water Samples For Chloride Analysis

GEOLOGICAL SURVEY WATER-SUPPLY PAPER 1544-A

Prepared in cooperation with the Nassau County Department of Public Works and the New York State Water Resources Commission

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GENERAL GROUND-WATER TECHNIQUES

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UNITED STATES GOVERNMENT PRINTING OFFICE, WASHINGTON : 1961

UNITED STATES DEPARTMENT OF THE INTERIOR

STEWART L. UDALL, *Secretary*

GEOLOGICAL SURVEY

Thomas B. Nolan, *Director*

For sale by the Superintendent of Documents, U.S. Government Printing Office
Washington 25, D.C.

CONTENTS

ILLUSTRATION

TABLE

603290—61

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ABSTRACT

Determinations of chloride concentrations of ground water in fresh-water, diffused-water, and salt-water bodies were made after extracting water by the filter-press method during the drilling of a test well at Atlantic Beach, Long Island, N.Y., in 1959. The filter-press method is an adaptation of techniques used in the petroleum field.

The filter-press method was found to be suitable not only for sand and silt but also for silty-clay and solid-clay core samples. It involves extracting interstitial water from core samples by means of a filter press and analyzing the filtrate for chloride content by titration. The method requires the identification and selection of the part of the core sample not invaded by the drilling mud during the coring operations. The invaded parts are identified by the distinctive green color of fluorescein dye added to the drilling mud. The results obtained by the filter-press method are consistent with those obtained by other methods.

INTRODUCTION

A detailed investigation of salt-water encroachment in southwestern Nassau County on Long Island, N.Y., was undertaken in 1958 by the U.S. Geological Survey in cooperation with the Nassau County Department of Public Works and the New York State Water Resources Commission. The investigation includes test drilling which is to provide basic data for the definition of the position of fresh-water, diffused-water, and salt-water bodies, and also for the definition of the salinities, densities, and hydraulic gradients of these bodies. A progress report on the first phase of the test-drilling program has been prepared (Lusczynski and Swarzenski, 1960).

The position and density of the fresh-water, diffused-water, and salt-water bodies are defined in part by the chloride content of the ground water brought up in core samples during the test drilling. Swarzenski (1959) described the centrifugal and dilution methods used during the first phase of the drilling program in 1958 to extract water from cores for determinations of chloride. The centrifugal method was found to be suitable for accurate determinations of the chloride content of water in samples of sand, clayey sand, and silt. It involves, first, the extraction of water by means of a mechanical centrifuge, and then titration for chloride content. The dilution method was devised for solid and silty clays which yield little or no water by centrifugal extraction. The laboratory procedure for this method is rather complex. It requires (a) a determination of moisture content from the loss in weight of a selected part of a core when it is dried in an oven at a temperature of 180°F for about 24 hours, (b) the restitution of original moisture content and further dilution with distilled water, (c) re-solution of the salts precipitated during drying, (d) filtration, and finally (e) titration for chloride content. The dilution method in its present stage of development yields only approximate results.

During the second phase of the drilling program in 1959, a filter-press method was used; it produced reliable results under controlled conditions, not only with sand and silt but also with solid-clay and silty-clay samples. The results obtained by the filter-press, centrifugal, and dilution methods at a test well at Atlantic Beach, Long Island, N.Y., are described and compared in this paper.

The filter-press method was used previously by C. W. Lauman & Co., Inc., well-drilling contractors of Bethpage, Long Island, N.Y., for defining chloride concentrations in sand zones during the construction of a test hole for a supply well. However, the results obtained by the contractor were only approximate, principally because no precautions were taken to select material not invaded by the drilling fluid.

FILTER PRESS

The filter press used during the study (fig. 1) is one of several types sold commercially for use in determining filtration properties of drilling muds (National Lead Co., undated). It is used also in mud logging to obtain a rough measure of salinity of formation water.

The filter-press unit consists of a chamber, a filtering medium, a graduated tube for catching and measuring the filtrate, and a pressure-source unit. A cell, base cap, screen, rubber gaskets, and top cap make up the chamber. The cell is 3½ inches high and has inside and outside diameters of 3 and 3½ inches, respectively. The filtering medium is a sheet of filter paper which fits on the screen over the base cap; it has a filtering area of about 7 square inches. The filter paper used is specially hardened to withstand the pressure in the chamber. A graduated cylinder is used to catch the filtrate.

The chamber is partly filled with selected material, assembled, placed in the frame, and made airtight by the T-screw (fig. 1). Pressure

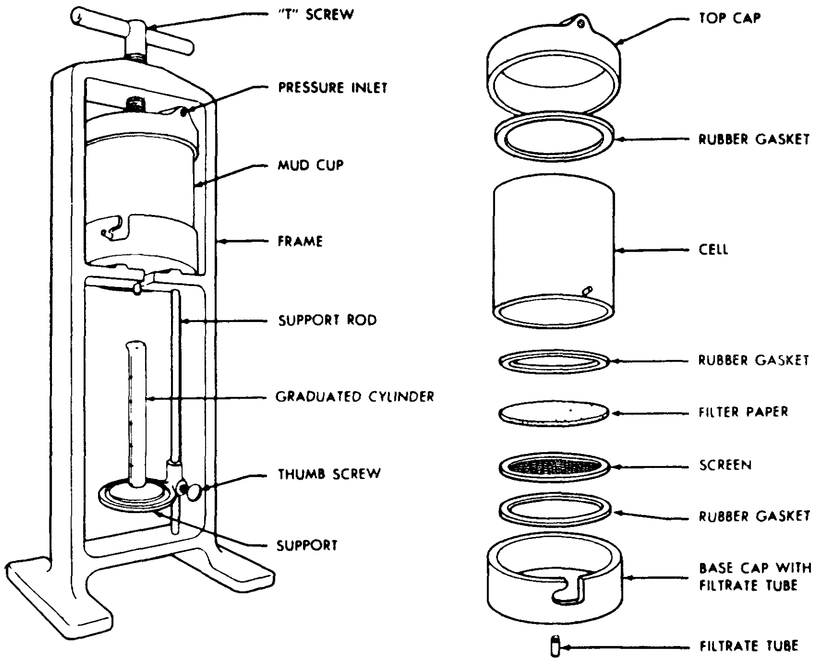

FIGURE 1.—Filter press and chamber assembly. Courtesy of Bariod Division, National Lead Co.

introduced through an opening in the upper cap of the chamber forces interstitial water out of the material and into the filtrate tube through a small opening in the base cap.

The pressure device used at test wells on Long Island includes a container for a carbon dioxide cartridge, a pressure gage, and a regulator. Pressures of as much as 120 psi (pounds per square inch) can be attained with the carbon dioxide cartridges. Other pressure sources, such as compressed nitrogen and carbon dioxide in cylinders, which are sold commercially can be used.

The filter press illustrated is a low-pressure unit. A unit for pressures of as much as 900 psi is also available.

FILTER-PRESS METHOD

The filter-press method involves filtering interstitial water from the core sample by means of the filter press, and analyzing the filtrate for chloride content by titration with a standard silver nitrate solution. The analysis is usually completed in the field within minutes after the core sample is brought to the surface. The filter-press method is an adaptation of techniques in use in the petroleum field (National Lead Co., undated).

As with the centrifugal method, the requisites for accurate determination of chloride by the filter-press method are (a) use of uninvaded parts of the core sample, (b) minimum loss of formational water in the core sample during coring and during transferral of the selected part of the sample from the core barrel to the filter-press chamber, and (c) sufficient filtrate, consisting only of formational water, for analysis.

The samples are obtained in a split-spoon core barrel, 2 inches in diameter and 18 or 24 inches long. Partial invasion of the core samples is unavoidable with the use of present coring methods and techniques. Invasion occurs when the drilling fluid is forced by hydrostatic pressure into the formation at or near the sampling point before the core is taken; it occurs also when the drilling fluid finds its way into the core barrel during coring, and as the core is raised to the surface. Loose sand and gravel samples are invaded to the greatest degree, and solid clay to the least. The amount of invasion increases with time. Therefore, the time from the attainment of the desired depth and coring at the depth until the core is brought to the surface should be kept to a minimum.

Addition of fluorescein dye to the drilling fluid gives it a distinctive green color and permits visual detection of the invaded parts of the core sample. A fluoroscope can be used also to assure the selection of uninvaded material and to check the filtrate for the presence of even minute amounts of the drilling fluid. Other dyes, such as methyl blue, may be used for assuring a contrast between the color of drilling mud and that of the formation.

Uninvaded parts consisting of loose material such as sand and gravel are transferred to the filter-press chamber by spatula or spoon. Usually it is not practical to remove much more than 25 to 50 percent of the uninvaded material from the core barrel by this method. Enough material is transferred to fill the chamber about a quarter to half full. It is then tamped lightly until an integrated unit is formed in the chamber and a film of water is formed on the surface of the material and along the cylindrical wall.

Usually less than 10 to 20 percent of a solid- or silty-clay sample is invaded by the drilling fluid. Plugs of the uninvaded material 1 to 2 inches long are placed in the chamber to fill it about a quarter full. Then they are molded and tamped into an integrated unit. Difficulty can be experienced in molding and tamping a relatively dry solid clay—one having a water content of less than 15 to 20 percent of the dry weight of the clay.

The total time between the opening of the split-spoon core barrel and the placing of the chamber in the frame is usually 1 to 2 minutes for the loose material and 2 to 5 minutes for the tight material. There is thus very little opportunity for evaporation.

After a sample is properly prepared for filtration, the chamber is fully assembled, placed in the frame, and made airtight by the **T**-screw (fig. 1). The gas pressure is then applied.

The pressure of the carbon dioxide gas in the chamber moves some of the interstitial water through the filter screen and filter tube into the graduated cylinder. Pressures of 5 to 30 psi suffice for the gravel, sand, and silt samples. Pressures of about 100 psi are usually sufficient for silty- and solid-clay samples. The carbon dioxide gas does not alter the chloride concentration of water forced from the material into the filtrate tube.

Chloride determinations of the filtrate are made in the field by the standard titration method using silver nitrate solution (1 ml $AgNO_3$= 1.0 mg Cl or 0.5 mg Cl). With these solutions, relatively large amounts of filtrate (25 to 50 ml) are needed when fresh water is to be tested, and relatively small amounts (1 to 10 ml) when salt water or diffused water is to be tested.

Usually enough filtrate can be obtained from the uncontaminated material of only one core if it is taken in a diffused-water or salt-water zone. However, more than one core may be necessary to obtain the required amount of filtrate if the core is taken in a fresh-water zone; this is particularly true for solid-clay samples, which yield only small amounts of interstitial water.

RESULTS AT ATLANTIC BEACH, LONG ISLAND, N.Y.

During the summer of 1959, an 850-foot test hole was drilled at Atlantic Beach, in the southern part of southwestern Nassau County, Long Island, N.Y. The hole penetrated unconsolidated material containing fresh water, diffused water, and salt water; core samples were taken at intervals of 5 to 20 feet. Chloride determinations were made by Swarzenski and the writer by means of the filter-press, centrifugal, and dilution methods. Determinations were made also of the chloride content of water pumped from three separate observation wells screened permanently at different depths in the test hole. (The hole was backfilled and relatively impermeable material was placed between the screens.) Results for sample tests for which more than one method was used, together with sample depths and descriptions, are listed in table 1.

Chloride concentrations determined by the filter-press method in the diffused-water zone at a depth of 150 feet and in the salt-water zone at a depth of 673 feet agree closely with those in related water samples from the individual wells screened at these depths. Chloride deter-

TABLE 1.—*Chloride content of ground water at selected depths at Atlantic Beach, Long Island, N.Y.*

Core sample	Depth, in feet, below land surface	Description	Moisture content, in percent of dry weight	Chloride content, in parts per million			
				Well sample	Filter-press method	Centrifugal method	Dilution method
1	41	Clayey silt			7,500		
2	42	Silt	33				9,000
3	52	Solid clay	56		1,500		1,500
4	91	do	41		490		430
5	101	do	37		1,600		1,360
6	111	do	35		2,550		2,280
7	150	Fine to medium sand		[1] 12,900	12,900	13,500	
8	160	Fine to medium clayey sand			13,100	13,200	
9	170	Solid clay	22				5,000
	170–171	Silt			6,000		
10	180–181	Solid clay, layers of silt			1,600		1,240
11	260–261	Clay and silt	24		50		60
12	282–283	Fine to medium clayey sand			18		
13	291	do		[2] 13	21		
14	311–312	Medium clayey sand			30		94
15	346–347	Fine to medium clayey sand			62		75
16	384–385	Fine clayey sand			204		
	386	Solid clay	18				210
17	445–446	Fine to medium clayey sand	19		1,850	1,910	1,570
18	454	do			3,150	3,200	
19	574	Clay and silt	19				14,000
	575	Very fine clayey sand			15,000		
20	673	Fine to coarse gravel in clay matrix		[3] 16,000	15,400		
21	693–694	Very fine clayey sand			15,300	14,800	
22	733	Solid clay, silt streaks	17				10,400
	733–734	Silt and laminated clay			12,000		
23	743–744	Very fine clayey sand			10,800	10,700	
24	763–764	Very fine to medium clayey sand			11,100		
	764	Solid clay, trace of silt	19		10,900		8,400
25	773	do	23				7,200
	774	Very fine clayey silt			4,400		

[1] Screen at 146–156 ft.
[2] Screen at 284–294 ft.
[3] Screen at 667–677 ft.

minations by the filter-press method in the fresh-water zone at depths of 282–283 and 291 feet are several parts per million higher than those of water pumped from the well screened at 284–294 feet. The agreement of the results by the filter-press method at each of these two depths, as well as at the other depths in the zone of fresh or nearly fresh water between 260 and 347 feet, is only fair because only one core was taken at each of these depths, and as a result not nearly enough filtrate was obtained for a good determination of chloride.

The results for water extracted from sand and silt core samples by the filter-press and centrifugal methods are in close agreement. Less overall handling and preparation was required and more water per given volume of sample was obtained in a much shorter time by the filter-press method than by the centrifugal method.

In the diffused-water and salt-water zones at Atlantic Beach, tests by the dilution method, in general, showed lower chloride content than those by the filter-press method. In the fresh-water zones, the tests by the dilution method showed higher chloride content than those by the filter-press method. Some small percentage of the difference in results by these two methods in the fresh-water, diffused-water, and salt-water zones may be due to actual differences in chloride content of water at points only a few inches to about a foot apart vertically at the site of the test hole. However, results from depths of 52, 91, 101, 111, and 764 feet in diffused water in solid clay are comparable because adjacent sections of the core sample were purposely selected for the determinations by the filter-press and dilution methods. At these depths, the dilution method produced results averaging, for the five samples, about 12 percent lower than those obtained by the filter-press method. At the other depths in sand, silt, and clay zones, results may not be comparable, because adjacent sections of the core sample may not have been used for analysis by each of the two methods.

Chloride determinations were made by all three methods on one core sample, that at the depth of 445–446 feet. Results obtained by the filter-press, centrifugal, and dilution methods were 1,850, 1,910, and 1,570 ppm (parts per million), respectively. The material was fine to medium sand. For the filter press, material between 445 and 446 feet was selected; for the centrifugal and dilution methods, material at 446 feet was used.

CONCLUSIONS

Experiments made during a program of test drilling for study of salt-water encroachment in southwestern Nassau County, Long Island, N.Y., indicated that accurate determinations of chloride concentrations of ground water in fresh-, diffused-, and salt-water bodies can be obtained from core samples by the filter-press method. This method can be used for sand, silt, and silty clay as well as solid clay. At the test well at Atlantic Beach, Long Island, N.Y., the results of chloride tests by the filter-press method, from gravel, sand, and silt samples, compare closely with those by the centrifugal method and those from analysis of water from finished wells. Results of chloride tests by the filter-press and dilution methods for fine sand, silt, and clay samples were generally in fair agreement.

The filter-press method is simple and direct. In this method, water is pressed out of the uninvaded part of a core sample and is analyzed for chloride content. The method is economical; basically it requires only a filter press, a chloride-testing kit, and a fluoroscope, all of which can be purchased for less than $200. It is rapid and convenient, in that each determination of chloride can be made in the field within minutes after the core is brought to the land surface.

Precautions must be taken to assure selection of material not invaded by the drilling fluid. These precautions include the use of fluorescein dye and a fluoroscope to identify the invaded parts of the core samples, and the use of a fluoroscope to check the filtrate for presence of any minute amount of drilling fluid.

It is planned to continue experimentation with the filter-press method during the third phase of the test-drilling program on Long Island. Experimentation will be directed specifically toward improving coring techniques in order to reduce the amount of invasion in loose material. Further experiments will be made in techniques of preparing relatively dry clay samples in the filter-press chamber and of extracting more interstitial water from relatively dry clay samples under pressures substantially higher than 100 psi.

Efforts will also be made to improve the dilution method.

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U. S. GOVERNMENT PRINTING OFFICE: 1961