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A PRESSURE-MEMBRANE EXTRACTION APPARATUS FOR SOIL SOLUTION[1]

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The composition of the soil solution has been given considerable attention in connection with soil fertility and soil salinity investigations, and a number of different methods have been used for sampling the soil solution. It is the purpose of this paper to review briefly these methods and to present preliminary results on a new experimental procedure which gives promise of being useful for studying physical as well as chemical properties of the soil solution over the plant growth range of moisture contents.

REVIEW OF EXTRACTION METHODS

Methods for obtaining samples of the soil solution may be briefly classed under the following headings:

1. Displacement. The soil solution may be removed and replaced by a fluid which is caused to move into the soil pore spaces. Liquids are commonly used, but the displacement may be produced by a gas if the soil is saturated. The displacement method was reviewed and improved by Parker (18) and has been extensively used (3,4, 5, 6, 7, 11, 20, 21, 25). Good evidence for the reliability of the method has been given by Burd and Martin (3), but the range of soil textures and moisture contents on which it can be used successfully is definitely limited. Only exceptional soils can be displaced at moisture contents near the wilting point, and in many instances soils which are readily puddled require excessive periods for the displacement process.

2. Compaction. Liquids may be removed from a porous medium if the pore space can be sufficiently reduced by compaction. This method is commonly used in commercial filter presses for extracting moisture from plant residues and ceramic clays, and has been used on soils by a number of workers (9, 15, 22). Pressures of thousands of pounds per square inch are required to reduce the moisture content of soils to near the wilting point; because of the effect of high pressures on solubility, the method has been subjected to rather serious criticism by Northrup (16).

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3. *Centrifugation.* Water may be caused to move through and out of soil by centrifugation, and this procedure is commonly used for drying soil to standard conditions (2, 17, 24). Attempts have been made by a number of workers to obtain samples of soil solution by centrifugation, but a satisfactory procedure has not yet been developed.

4. *Molecular adsorption.* Gardner, Whitney, and Kezer (10) effected the extraction of solutions from slick spot soils by contact with rolls of dry filter paper. The transfer of solution from the soil to the paper was hastened by compaction in a cylinder with movable pistons. Neither compaction nor centrifugation will remove water from moist soil until the pressure in the soil water is increased to atmospheric pressure at the place provided for outflow; that is, the soil must become substantially saturated at this place. The molecular adsorption method is suitable for supplementing compaction or centrifugation methods when soil moisture contents are too low to satisfy the atmospheric pressure outflow condition.

5. *Suction.* The removal of water from soil by suction is a common procedure, accomplished by connecting the liquid phase of water in soil with liquid water at lower pressure. The mechanism for maintaining this pressure difference between extracted water and water in the soil is usually a porous ceramic wall or other membrane which, when wet, is readily permeable to water but not to air. The suction method as suggested by Briggs and McCall (1) makes it possible to extract moisture from soil until the negative pressure in the soil water is about one atmosphere. This pressure limitation arises from the fact that without elaborate precautions it is not possible in ordinary apparatus to reduce the pressure in liquid water below the aqueous vapor pressure. Porous ceramic cups have been used for extracting solutions from soil samples for fertilizer investigations (13) and for sampling submerged soil solutions (12). Adsorption of ions by the ceramic cell or other membrane material must be considered whenever the suction method is used (19).

The pressure limitation in the suction method may be avoided by increasing the gas pressure in the soil air. This has been done in connection with soil-moisture sorption-curve work by S. J. Richards (23) and has also been done by Lauritzen (14) for increasing the exudate from decapitated potted plants.

PRESSURE-MEMBRANE EXTRACTION APPARATUS

The apparatus which has been developed at this laboratory for removing solutions from soils makes use of the gas pressure modification of the suction procedure. The soil from which moisture is to be removed is placed in a chamber in which the gas pressure is increased above atmospheric pressure. The side of the chamber which supports the soil consists of a Cellophane membrane supported on a brass screen and a brass plate in such a way that any solution passing through the membrane is conducted away at atmospheric pressure and trapped under oil. In this way the moisture content of the soil in contact with the membrane will be reduced by the amount that would be necessary under normal atmospheric conditions to make the pressure deficiency of the soil water equal to the excess gas pressure in the extraction chamber.

It is not difficult to see why excess gas pressure applied on top of a layer of saturated soil should cause the soil water to move through an underlying membrane which is subjected only to atmospheric pressure on the side opposite to the soil. It is not entirely obvious why such a membrane should continue to extract moisture from soil after a continuous gas phase has been established throughout the soil mass and the soil-water system touches the membrane only at comparatively isolated points. The fact that the excess gas pressure can be maintained in the chamber indicates that surface tension action inhibits gas leakage through the membrane pores and there is established in the gas-liquid interface at the upper surface of the membrane an equivalent curvature determined by the excess gas pressure. The comparatively free movement of water through the membrane maintains this same curvature in the water interface at the contact points between the membrane and the soil. There is thus set up a curvature gradient across the layer of soil in contact with the membrane. It is the pressure gradient corresponding to this curvature gradient that moves the water or solution through the unsaturated soil toward the membrane. When equilibrium is reached and passage of water through the membrane ceases, the interface curvature of the water throughout the soil will be the same as at the membrane.

The moisture extraction chambers consist essentially of cylindrical sections clamped between flat steel plates. Two sizes were used in the work reported in this paper. In the small chamber the cylinder was $2\frac{7}{8}$ inches outside diameter with a wall thickness of $\frac{3}{16}$ inch. The end plates were $3\frac{1}{2}$ inches square, $\frac{1}{2}$ inch thick, and were clamped by four g-inch bolts. The cylinder for the large chamber was 12 inches outside diameter with a wall thickness of $\frac{1}{4}$ inch. The circular end plates were 14 inches in diameter, $\frac{5}{8}$ inch thick, and were clamped by eight Q-inch bolts. Tripod legs were screwed into the lower plate.

Figure 1 shows the constructional details for the large chamber. To prevent displacement of the gaskets by gas pressure, the rectangular groove shown in the sectional view was cut in the face of the cylinder and the annular rubber gaskets were attached to the cylinder with rubber-to-metal bonding cement. The brass screen is held taut on the brass plate by the smooth annular ring of solder which also serves as the bearing surface for the gasket. A short length of 0.040-inch bore copper tubing is riveted and soldered into the center of the brass plate so that the upper end of the copper tube is flush with the upper surface of the brass plate. This tube, passing downward through the brass plate and the lower steel plate, serves as the outlet for fluids which pass through the cellophane membrane from the soil chamber. The three lugs brazed to the cylinder make it possible to screw the cylinder to the lower steel plate and thus hold the Cellophane and gasket in place while the chamber is being loaded with soil.

The tendency for soil to shrink out of contact with the membrane during dehydration may be prevented by inserting a soft rubber diaphragm between the cylinder and the upper plate. A constant compressive force may thus

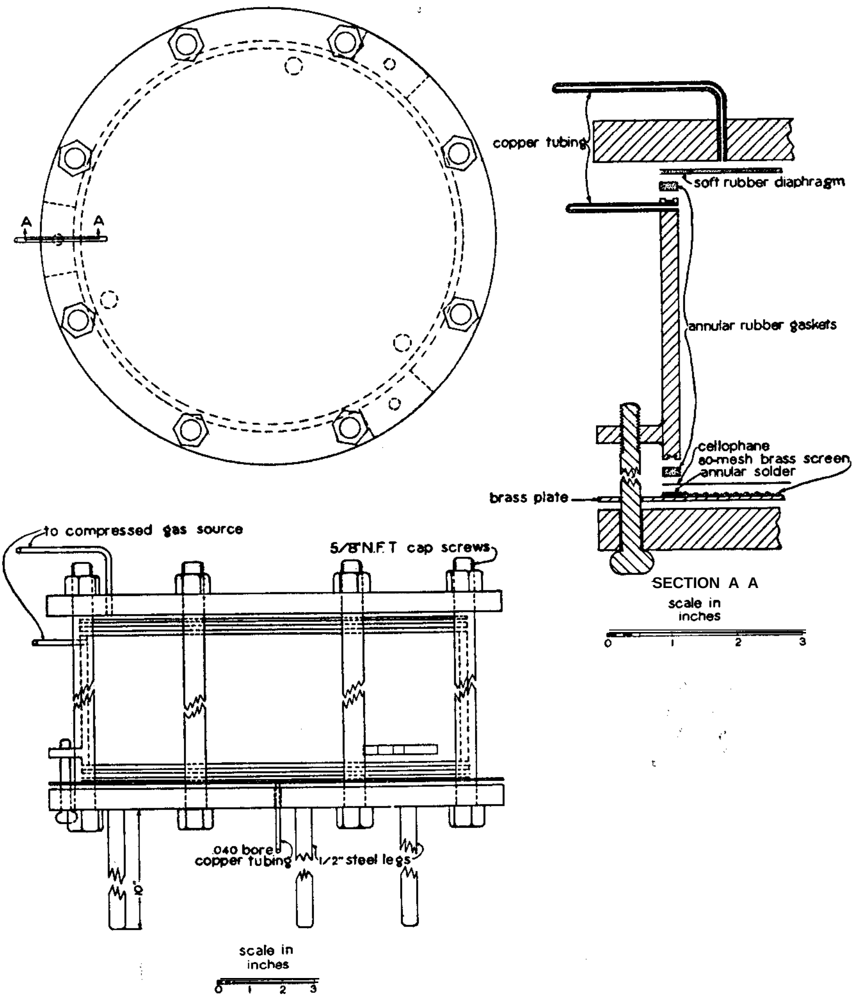

FIG. 1. PRESSURE-MEMBRANE EXTRACTION APPARATUS

be maintained on the soil by keeping the gas pressure above the diaphragm somewhat in excess of the extraction pressure.

The most suitable height for the cylindrical section of the extraction chamber will depend on the nature of the extraction work to be carried on. Cylinder heights of $\frac{1}{2}$, 2, and 4 inches have been found convenient for the work in progress at this laboratory.

MEMBRANE CHARACTERISTICS

Representative data on the permeability of plain transparent Cellophane are shown in table 1. Thickness values for the three gauge numbers commonly available are also given. It may be seen from the data that water transfer through Cellophane is not quite proportional to the pressure difference over the pressure ranges used. The permeability varies somewhat from one sheet to another, and tends to increase slightly during water transfer. The gas permeability measurements were made on the wet membrane immediately following the water-flow tests.

Only occasionally at the beginning of an extraction process have Cellophane membranes developed leaks, and these have been ascribed, for the most part,

TABLE 1

Permeability of plain transparent Cellophane to water, air, and nitrogen

Expressed as cm.^{3} cm.$^{-2}$ sec.$^{-1}$ atmos.$^{-1}$ X 10^6

GAUGE NUMBER	THICKNESS	TEMPERATURE	DRIVING PRESSURE IN ATMOSPHERES							
			Water				Air			
			2	4	6	8	2	4	6	8
	inches	°C.								
300	.00088	22.0	10.8	9.74	9.33	9.18	7.23	7.10	6.77	6.36
450	.0012	21.5	11.6	10.5	9.95	9.14	5.97	5.60	5.32	5.10
600	.0017	22.5	9.05	8.60	7.80	7.77	5.15	5.00	4.80	4.45

GAUGE NUMBER	THICKNESS	TEMPERATURE	DRIVING PRESSURE IN ATMOSPHERES					
			Water			Nitrogen		
			3.4	6.8	13.6	3.4	6.8	13.6
300*	.00088	27.5	17.5	19.6	17.0	150	208	348
450*	.0012	28.0	14.2	13.8	12.3		4.76	3.92
600*	.0017	26.2	7.88	7.85	6.80	1.91	3.48	2.82

* Cellophane obtained from a different supply house was used in these tests.

to faulty handling. The high permeability to nitrogen shown for the No. 300 sheet in table 1 is exceptional and in this case probably indicates a progressive failure of the membrane. Gas leaks through the membrane are rather likely to develop if the extraction process is continued for more than three or four days.

During preliminary tests it became apparent that solutions from certain saline soils markedly decreased the membrane permeability. It was found, for instance, that the permeability of a sheet of No. 600 Cellophane to 1 M Na_2CO_3 was exactly half the value obtained for distilled water.

Medium and tine grades of Zsigmondy Ultrafein filters were tested but were not found to be appreciably better than Cellophane and are much more expensive.

SOLUTION EXTRACTION TESTS ON SOILS

Preliminary tests indicate that the pressure-membrane apparatus may be used for extracting soil solutions over the whole plant growth range of soil moisture contents. Several soils were available for which the permanent wilting percentage had been determined by the sunflower method (8).

The soils were air-dried and had been passed through a Z-mm. sieve. A layer of dry soil approximately 1 cm. deep was spread on No. 600 Cellophane in the large chamber and wetted with distilled water. For these experiments the cylindrical section of the chamber used was ½ inch high. The soils were allowed to stand from 1 to 2 hours with an excess of water before the chamber was closed and a nitrogen pressure of 16 atmospheres (235 pounds per square inch) applied. This pressure was maintained constant by a regulator valve.

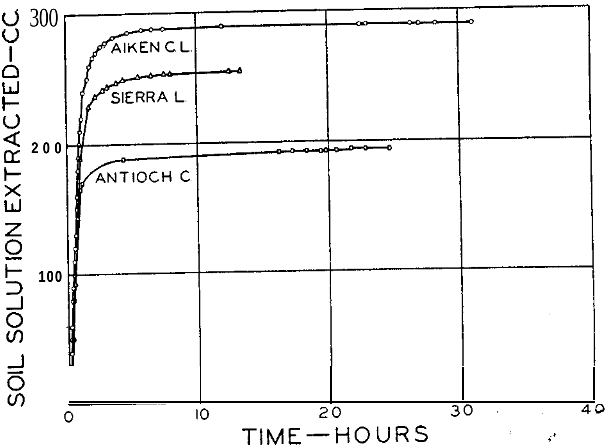

FIG. 2. SUMMATION OF THE SOIL SOLUTION EXTRACTED AS A FUNCTION OF TIME FOR A LAYER OF SATURATED SOIL 29.2 CM. IN DIAMETER WITH AN EXTRACTION PRESSURE OF 16 ATMOSPHERES. THE SOIL DEPTHS WERE: AIKEN, 1.0 CM.; SIERRA, 1.0 CM.; AND ANTIOCH, 0.5 CM.

From the curves in figure 2, it appears that the extraction rate is high and approximately constant until the water table in the soil layer reaches the membrane. The rate of delivery of water to the membrane then depends on flow through the unsaturated soil.

Antioch clay is an extremely impervious soil and shrinks considerably during dehydration. The curve for this soil shown in figure 2 was obtained for a 5-mm. layer on which a soft rubber diaphragm maintained a compressive stress of 10 pounds per square inch after the first half hour of extraction.

It is a matter of considerable interest that for the 5- to 10-mm. layers of soil that have been tried, moisture equilibrium with the 16-atmosphere membrane is attained within 24 to 36 hours, and as indicated in table 2, the equilibrium moisture contents have been below the wilting points for the respective soils. This seems to supply important new information on the possible rate of the movement of water to plant roots in dry soils.

The curves in figure 3 show extraction rates from 10-cm. layers of soil initially containing moisture at ½ atmosphere tension. Air pressure on the membrane was 6.8 atmospheres (100 pounds per square inch) and a compressive stress of 20 pounds per square inch was maintained by a pressure of 120 pounds

TABLE 2

Moisture content values for three soils under various standard conditions

SOIL	MOISTURE EQUIVALENT	WILTING COEFFICIENT	MOISTURE AT ½ ATMOS TENSION	MOISTURE AT 16 ATMOS. TENSION
Sierra loam.	8.6	3.7	6.0*	3.27*
Aiken clay loam..	36.2	22.0	29.5	21.24
Antioch clay..	21.0	12.0	20.6	11.2

* Although taken from the field at a later date, this sample was from the same depth and location as the sample on which the moisture equivalent and the wilting coefficient were determined.

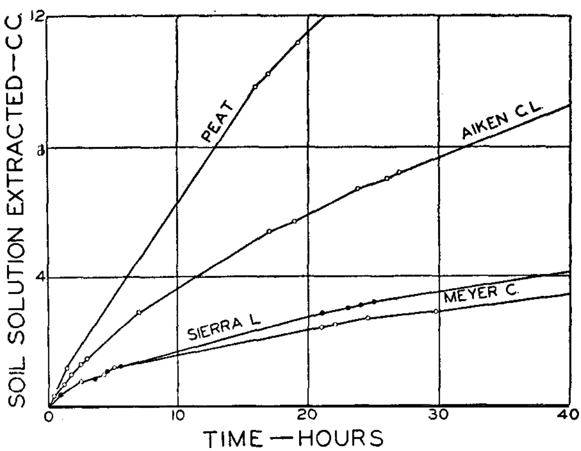

FIG. 3. SUMMATION OF THE SOIL SOLUTION EXTRACTED AS A FUNCTION OF TIME FOR A LAYER OF SOIL 6.3 CM. IN DIAMETER AND 10 CM. DEEP. INITIALLY THE SOIL CONTAINED MOISTURE AT ½ ATMOSPHERE TENSION AND THE EXTRACTION PRESSURE WAS 6.8 ATMOSPHERES

per square inch above a soft rubber diaphragm which served as the upper gasket for the cell.

It is likely that the soils were somewhat puddled by the handling process. The soils were wetted at zero soil moisture tension and dried to equilibrium at ½ atmosphere of tension in 6-inch double-walled irrigator pots. The cylinder of the extraction cell *was* then filled with this moist soil. All soils were handled as nearly as possible in the same way.

The curves illustrate in a general way the rate at which the pressure membrane apparatus will extract moisture from soils containing water at $\frac{1}{2}$ atmosphere tension. The small cell was used in these experiments; therefore, the extraction rate is only 5 per cent of what it would have been with the large cell.

SOIL SOLUTION STUDIES WITH THE PRESSURE-MEMBRANE APPARATUS

Because of several effects, the solution obtained from the pressure-membrane extraction apparatus may differ from the solution that would be delivered to an absorbing root in the same soil. Principal among these may be ion adsorption in the membrane. Since little information on this point is available for Cellophane, the matter will be carefully investigated. For a l-cm. layer of soil on No. 300 Cellophane, however, the soil-membrane thickness ratio is 450 to 1: therefore trouble is not anticipated in investigating the predominant ions in saline soils.

The 16 atmospheres of gas pressure necessary for low moisture content extractions may produce appreciable changes in the composition of the soil solution because of effects on solubility. The use of nitrogen should prevent large changes in carbonic acid concentration, but where detailed information is wanted on carbonate-bicarbonate relations, it may be necessary to use controlled mixtures of nitrogen and carbon dioxide.

For the experiments here described the liquid and the gas passing through the membrane were trapped in separate vessels by displacing white mineral oil. In some cases the water vapor contained in the gas passing through the membrane may appreciably concentrate the soil solution. This effect can be minimized by humidifying the gas before it enters the extraction chamber. During the 31 hours required for obtaining the Aiken curve in figure 2, about 255 mgm. of water vapor was carried off by the 11 liters of nitrogen that passed through the membrane. This corresponds to 0.03 per cent change in the moisture content of the soil.

In the large cell having a membrane area of 671 sq. cm., about 4 or 5 cc. of solution is required to wet the membrane and fill the spaces in the 80-mesh screen before liquid begins to emerge from the outflow tube.

OTHER USES OF THE APPARATUS

The pressure-membrane extraction apparatus seems to provide a means for considerably extending our knowledge of the flow of soil water at moisture tensions greater than 1 atmosphere. Steady-state moisture flow experiments in unsaturated soil can now be carried on over the whole plant growth moisture range and use can be made of well-known energy relations for expressing the results in standard units.

Sorption curves for the whole plant growth moisture range are readily obtainable with this apparatus and will provide useful information on the effect of such treatments as organic matter, fertilizers, salts, puddling, and freezing on the structure and pore size distribution of soils. Applications for the apparatus outside the field of soil science are also apparent.

SUMMARY

The methods that have been used for extracting solution from soils are briefly discussed under the headings: 1. Displacement, 2. Compaction, 3. Centrifugation, 4. Molecular adsorption, and 5. Suction.

A modified form of suction apparatus is described in which a carefully supported Cellophane membrane serves as the bottom of a gas pressure chamber in which soils are placed for solution extraction. In this way the moisture content of soil in contact with the membrane will be reduced by the amount that would be necessary under normal atmospheric conditions to make the pressure deficiency of the soil water equal to the excess gas pressure in the extraction chamber.

Data are presented for three soils showing that an extraction pressure of 16 atmospheres reduces the moisture content of 5- to 10-mm. layers of soil from saturation to below the wilting point in 24 to 36 hours.

The apparatus can be used for obtaining sorption curves and studying the permeability of unsaturated porous media over a considerably extended range of negative pressure.

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