

in phosphoric acid for rice, cotton and corn, but not for cowpeas. While cotton and cowpeas have the same solvent power, cotton needs more phosphoric acid in its growth.

(9) The sum of the phosphoric acid in the soil after growing the plants plus that in the plant, less the original content of the soil, is taken as the phosphoric acid made available by the plant. It varies from 24 to 136 per cent. of the original soil content in our eight experiments, thus varying widely according to the soil or the plant. No doubt, different soils are different in this respect.

(10) In experiments with 15 soils, there appeared to be a relation between the chemically available phosphoric acid as measured by N/5 nitric acid, and soil deficiencies as shown by pot tests.

(11) There is no doubt that the determination of chemically available plant food will give valuable indications as to the needs of soils in phosphoric acid, if due attention is paid to the factors which influence it, and to the other factors of availability.

[CONTRIBUTION FROM THE BUREAU OF CHEMISTRY, U. S. DEPARTMENT OF AGRICULTURE. PUBLISHED BY PERMISSION OF THE SECRETARY OF AGRICULTURE. SENT BY H. W. WILEY.]

HIGH VACUA IN THE SCHEIBLER TYPE OF DESICCATOR.

BY H. C. GORE.

Received April 28, 1906.

THE ether-sulphuric acid method for obtaining high vacua in desiccators, which was invented by Benedict and Manning,¹ is shown by them to be of great value in the determination of water in foods and physiological preparations. The method consists essentially in expelling the air by evaporating ether in the desiccator under diminished pressure and subsequently removing the ether vapors by means of sulphuric acid. Vacua of less than 1 mm. of mercury are readily attained by this means.

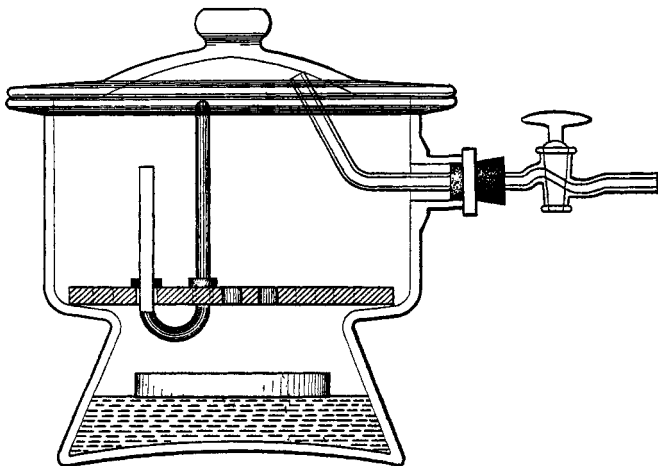
The Hempel desiccator, in which the sulphuric acid is located in the upper part of the apparatus, has been the type of desiccator mainly used by Benedict and Manning. The main objection to this form, in the opinion of the writer, and one which it is believed will prevent its general use, lies in the fact that the sulphuric acid is apt to spill, particularly at the time when the

¹ Am. Ch. J. 27, 340 (1902), and Am. J. Physiol. 13, 309 (1905).

upper compartment is removed.¹ In addition, it is a special form of apparatus not generally found in laboratories.

The Scheibler type of desiccator, in which, however, the sulphuric acid was contained in an acid dish in the upper part of the desiccator, was also used with success by the originators of the method. It is found by the writer that the Scheibler type works as well as the Hempel form and much more conveniently, when the sulphuric acid is contained in the *bottom* of the desiccator. Following are the details of the application of the ether-sulphuric acid method to the Scheibler desiccator in which the acid is contained in the bottom, together with results obtained in a comparison of the two types of desiccators.

A Scheibler desiccator of 20 cm. internal diameter is fitted with a one-holed new rubber stopper, through which passes a delivery tube reaching to near the top of the inside of the desiccator and terminating outside of the desiccator in a well-fitting stop-cock (best a stop-cock with a slant-bored hole). The rubber stopper before use is first well cleaned by being rubbed with a cloth moistened with chloroform, and then well greased with a mixture of vaseline and beeswax in which a little "pure gum" rubber tubing has been melted. The stop-cock is also lubricated



with this mixture. For the ground joint between the desiccator and its cover a vaseline and beeswax mixture is used. The

¹ It is found that the upper compartment of the Hempel desiccator is best loosened after the vacuum is destroyed, by driving the thin blade of a spatula between the ground surfaces by means of gentle tapping.

desiccator is provided with a simple U-shaped manometer, and charged with about 200 cc. of concentrated sulphuric acid on which is floated a Petri dish about 10 cm. in diameter (see drawing). Connection is made with the exhaust, best through two bottles (not shown in the figure), the first of which is arranged to act as a safety-bottle in case of back suction, and the second contains water in order that the outflow of gas may be observed.

When all is ready for the exhaustion, from 20 to 25 cc. of ether are added to the Petri dish, most conveniently through a long-stemmed funnel, the desiccator closed and suction applied. After the first rush of gas, a rapid, steady flow goes on as the ether evaporates from the Petri dish. The flow of vapors ceases as soon as the ether is volatilized and the stop-cock is now closed. The desiccator may now either be left to itself, when a gradual fall of the mercury in the manometer will be observed, reaching a minimum of about 1 mm. in a short time, or the apparatus may be gently moved about in such a manner that fresh surfaces of sulphuric acid will be exposed to the ether vapors. By the latter procedure a high vacuum is very rapidly attained. In case, however, materials are being dried which contain considerable quantities of water, loss by spattering may occur where the pressure is lowered so suddenly, and the former method is preferred. Such mechanical loss has been observed, for example, in connection with experiments in the drying of potato starch. When the pressure was very suddenly reduced from 50 mm. to 10 mm. or less, the water vaporized so suddenly that it carried the starch grains to all parts of the desiccator with almost explosive violence. The starch used contained only about 11 per cent. of water.

A set of experiments was carried out in which a Hempel desiccator, used according to the directions of Benedict and Manning,¹ and a Scheibler desiccator used according to the procedure given above, were run side by side. The material employed was a very pure sample of potato starch. Two-gram samples of the starch were weighed into small aluminum dishes, fitted with covers, of the kind recommended by Benedict and Manning.² The dishes with their contents were placed in the desiccators, their covers removed, and the process for obtaining the high

¹ Loc. cit.

² Am. J. Physiol. 13, 314 (1905).

vacuum applied to each desiccator. The desiccators were opened at the intervals indicated in the table. In opening, the air was admitted through a calcium chloride tube and the dishes were immediately capped with their respective covers and weighed. The percentages of water removed at the different intervals in the two desiccators are shown in the table below. The determinations were made in triplicate and the separate determinations in each set agreed closely throughout. The averages only are given in the table.

TABLE SHOWING THE DRYING OF STARCH IN HIGH VACUA IN HEMPEL AND SCHEIBLER DESICCATORS.

Desiccator.	Time. Hours.	Per cent. of water.
Hempel	30	10.20
“ ..	74	10.67
“	146	10.69
Scheibler	24	10.58
“	68	10.74
“	140	10.76

The starch contained 10.90 per cent. of water, this determination being made by drying for two hours in a partial vacuum of about 60 mm. at 102° C.

The results show that the Scheibler form with the sulphuric acid in the bottom of the desiccator, equals the Hempel form as an apparatus for the rapid drying of materials in the high vacua obtained by the ether-sulphuric acid process.

A criticism of the ether-sulphuric acid process must be noted. There seems to be some slight tendency for the formation of acid fumes after the apparatus has remained evacuated for some days and a faint odor can be detected in the desiccator on opening. The dried starch samples from both desiccators were notably darker in color than the original sample, or the samples dried at 102°.

The method, therefore, cannot be recommended unqualifiedly, but it is the nearest approach yet made to an ideal method. The modification suggested in this paper is made in the hope that the method will be more widely adopted by chemists.