heavy rubber tube to a Bunsen suction pump and suction turned on, the surface of the filter is put in contact and kept in contact with the surface of the liquid in the beaker (Fig. 2) until all the liquid is drawn into the filter-tube, when the latter is withdrawn and reversed (Fig. 3) to permit the liquid to be sucked out. The suction is then discontinued, the fiber washed with 50 to 75 cc. of boiling water back into the beaker, stirred thoroughly, allowed, if need be, to subside a few moments, and the liquid sucked out through the filter, as above described. Three or four such washings will free the fiber of acid or alkali. When freed of acid, the fiber is washed back into the beaker with the requisite quantity of 1.25 per cent. alkali and digested. Finally, when filtered and washed (as above described) free of alkali, the fiber is washed with distilled water into a 50 cc. tared evaporation dish (royal Berlin porcelain in absence of platinum), the water evaporated on steam bath, the fiber dried, weighed, incinerated, the ash weighed, the fiber obtained by difference in the usual way. A small flexible steel spatula is often useful in removing the fiber from the linen and in bringing it into the dish. It will not infrequently happen that the dish will get full of water before all the fiber is washed into it; in such case, the excess of water is removed by suction through the small filter above referred to.

Experiments with filtering materials other than linen—hardened filter paper, flannel, chamois skin, unglazed porcelain discs (Fig. 8) were made—but the first and last are out of the question because of slowness, the two others are not suitable with hot alkali.

Results Obtained by the Method.—Duplicates on peanut hulls, corn bran, rice chaff, corn cobs, and peanut middlings gave results differing by 0.01 per cent., 0.14 per cent., 0.32 per cent., 0.20 per cent., 0.12 per cent., respectively. In the case of six samples of cocoas, duplicates were obtained differing by 0.02 per cent., 0.33 per cent., 0.03 per cent., 0.02 per cent., 0.06 per cent., 0.13 per cent. in each case. The fiber of cocoa, a fine powder, is one of the most difficult to filter and wash—next to impossible with a Gooch filter, but comparatively easy and quick by this method.

The filtrates and wash-water from the second or alkaline digestion were, after having passed through the linen filter, put through gooches (asbestos). It was found in six fiber determinations (wheat bran, shorts, cornmeal, "ship stuff") that the gooches retained 0.21 per cent. to 0.37 per cent. of solids, reckoned as fiber, that had passed through the linen; in the case of seven cocoas, filter paper (the hardened variety) retained 0.02 per cent., 0.02 per cent. 0.02

per cent., 0.26 per cent., 0.27 per cent., 0.35 per cent., 0.46 per cent., respectively, of solids which had passed through the linen.

The filtering (from the second or alkaline digestion), washing and transferring of the fiber to the evaporating dishes in the case of six cocoas consumed sixty-six minutes, an average of eleven minutes to the determination; the filtrates from these determinations were then put through hardened filter paper under suction, the shortest time of filtering in any of the six cases was one hour, the longest seven hours.

The above tests of the method were made several years ago. The author has hoped from year to year to make more extended and searching experiments, but has not been able to do so.

The official method of the U. S. Dept. of Agr. (Bur. of Chem., *Bull.* 107, p. 56, revised) for crude fiber says: "Filter through linen, asbestos or glasswool"—nothing as to particular grade of linen. One naturally selects, if linen, that of finest, closest mesh obtainable. Old filters of the author, still on hand, are 32 x 32 per square cm., that is, contain about thirty-two threads each way to the cm.; the threads are apparently about 0.2 to 0.3 mm. in diameter. Undoubtedly *standard*, or *better standardized* linen—if that material be finally found to be best—should be adopted.

There is a kind of felt, composed mainly of cotton, comparatively thin, uniform in texture; it would probably make an excellent filter. It is to be had at tailoring establishments.

RALEIGH, N. C., Jan. 10, 1910.

THE DETERMINATION OF CRUDE FIBER.

By G. M. MACNIDER.

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In carrying out the method of the A. O. A. C. for the determination of crude fiber in cattle feeds¹ we have tried a good many modifications of the apparatus to shorten the time required and to make the determination less laborious. As the apparatus in use in this laboratory at present has proved satisfactory, some of the modifications in use may be of interest to chemists engaged in agricultural work.

The digestions with 1.25 per cent. sulphuric acid and 1.25 per cent. sodium hydroxide are done in 600 cc. Jena glass beakers, 16.5 cm. in height and with a uniform diameter of 8 cm. During the digestion a clock glass is placed over the beaker to condense the steam. This is fairly satisfactory, but considerable trouble is frequently caused by the frothing of the liquid, thereby necessitating the washing of the

¹ Bureau of Chemistry. *Bull.* No. 107 (revised).

substance from the sides of the beaker, and by the loss by evaporation which varies the strength of the solution. A number of different forms of condensers were tried to overcome both of these difficulties, but with only a fair amount of success. The author has found the following apparatus very satisfactory: this consists of an upright Liebig condenser, the lower end of which passes through a large rubber stopper which fits into the beaker described above. The stopper also carries a small tube for the introduction of a stream of air. (The use of a stream of air is suggested in the official method.) When the digestions were done in this apparatus it was found that with the use of only a weak stream of air the frothing even on the most difficult samples, *i. e.*, low-grade cottonseed-meals, could be so controlled that no error was introduced from this source. By using the Liebig condenser in place of a glass tube condenser or a flask containing water placed on the beaker there was so little loss of steam that the volume of the liquid remained practically constant throughout the digestions.

A great many different forms of filters have been tried for filtering the fiber. The most satisfactory form is by reverse filtration, using linen as a filtering medium. (The official method directs to use either linen, asbestos or glass wool.) Any good grade of linen is satisfactory. The first form of this filter was made as follows: A circular piece of linen was fastened over the mouth of a glass funnel by means of a draw string around the edge. The stem of the funnel is connected with the suction apparatus and the linen immersed in the liquid and the liquid sucked up into a waste bottle. The fiber adhering to the linen is washed off with a jet of water.¹ This form of funnel was used for a while, but was found unsatisfactory and was discarded on account of the fact that the linen was not supported and consequently sucked back into the funnel, causing trouble in washing. It also required a considerable amount of time to prepare the lined filters and attach them to the funnel. To overcome this difficulty Mr. Harris (formerly of this laboratory) designed a funnel made of sheet copper, the mouth of which is closed by a perforated copper plate, which supports the linen. The linen filter is fastened over this by a copper band about 1 cm. in width which fits closely around the edge of the funnel. This keeps the linen stretched taut over the copper plate and makes it very easy to wash off any adhering fiber. By this arrangement the linen filters can be changed very rapidly. The funnel, as used at present in this laboratory, is made 7 cm. in diameter, or so as to just fit into the fiber beaker, thus giving as large a filtering surface as possible. When the fiber is finally washed free from

alkali it is transferred from the beaker to a crucible or porcelain dish and evaporated to dryness in the usual manner. The only objection to the funnel just described is that in transferring the fiber it requires a large volume of water, usually 120 to 150 cc., to wash the fiber from the filter and wash out the beaker. To overcome this the author has designed a small copper funnel 3.5 cm. in diameter, made very similar to the large one. This can be used for the entire filtration, but the work can be done faster if the acid washings and the first few washings of the alkali are done with the large copper funnel, then on the last washing of the alkali use the small funnel. As the liquid is sucked up the funnel is carefully moved around in the beaker and the greater portion of the fiber can be collected on the linen attached to the funnel and can then be washed into a small crucible with a very small amount of water. It requires only a small amount of water to wash out the fiber remaining in the beaker. It was found that by using this method the fiber could be transferred to a small crucible with from 25 to 40 cc. of water as compared to 120 to 150 cc. with the large funnel. Where a large number of determinations are to be made this is a great saving in time as well as in the amount of gas burned in evaporating the fiber to dryness.

A number of tests were made, using Gooch crucibles for both the entire filtration or for the alkali filtration. The nature of the material causes the filters to clog so readily that it requires too much time to filter in this way when a more rapid method is available. Very satisfactory results were, however, obtained when all the washings except the last washing of the alkali were done with the copper funnel and the fiber then washed into the gooch. With wheat products and similar materials this procedure is fairly rapid, but with cottonseed-meal and similar products the filters clog very readily and it requires a considerable amount of time to complete the filtration.

Tests were also made with the Monroe crucible, prepared according to the directions given by Snelling.¹ When the filtrations were divided as described for the Gooch crucible, the Monroe crucible gave very satisfactory results. The time of filtration is, of course, longer than with the copper funnel, but this will be more than compensated for by the time saved on the evaporations. Snelling suggests the use of a platinum wire gauze in the platinum felt to prevent cracking and curling of the felt when heated. This would probably be of considerable advantage in this class of work. After the fiber has been ignited and weighed the ash can be very easily removed with a camel's hair brush without injuring the platinum felt.

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¹ *J. Am. Chem. Soc.*, **31**, 4, 1909.

¹ This form of filter was first gotten up by J. M. Pickel of this division. See preceding paper, *THIS JOURNAL*.