A NEW FORM OF EXTRACTION APPARATUS.¹

BY CHARLES WILSON GREENE.

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A new form of extraction apparatus involving the principle of the Soxhlet extractor has been in use by me for several years, and I am led to publish this delayed description because of a number of requests for the description by those who wish to apply it in researches on animal tissues.

The apparatus was devised for the quantitative analysis of meat samples in order to secure an extraction in hot solvents. In the ordinary Soxhlet extractor the hot vapors pass from the flask through a side tube which re-enters above the sample. The extraction is accomplished in a cold, or at best only a lukewarm medium. In the apparatus here described the hot vapors pass up around the cup containing the sample, and the condensate is readily held at or just below the boiling point at all times. The extraction, therefore, proceeds at the boiling temperature. The device has all the advantages of the Soxhlet apparatus in that all parts of the sample are subjected to the actual contact of the hot fluid and this fluid is periodically drained off to be renewed from the condensation of the pure vapors.

Description of Apparatus. The form of the apparatus is illustrated in the accompanying diagram. The apparatus consists of five pieces, which are described as follows:

Number 1 is an ordinary short-necked, narrow-mouthed, flat-bottomed flask which must be pear-shaped for convenience in the transferring of extracting fluids. Its neck is ground to receive the stopper of piece number 3. A most convenient size for this flask is 250 cc. This flask should be made of glass of high resistance as in practice ordinary glass rather has proven to be too breakable. The reflux flask should be made in triplicate.

Number 2 is an ordinary Hopkins condenser, 300 mm. high and 35 mm. in diameter. It has a ground glass stopper at its base. This stopper,

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which is ground to fit in piece number 3, is 46 mm. in diameter. The stopper should have a relatively narrow ground surface and be dome shaped as shown in the diagram, as it is found that this form of stopper is more readily manipulated in the transfers that occur in the use of the apparatus. The end of the condenser tube within the stopper is punctured for the reception of a platinum wire where it is desired to suspend a Gooch crucible, paper extraction thimble or other sample container.

Piece number 3, which in practice I have usually called the middle piece, is as its form shows, somewhat like a filtering flask, but with its mouth ground to fit the condenser stopper and its funnel tube ground to fit the reflux flask. This piece must be large enough to receive the siphon cup described below. For the small Gooch a free diameter of 46 mm. is sufficient, and the depth of the bowl should be 100 mm.

Piece number 4 is a shallow cup with siphon tube turned as shown in the figure. This cup should be about 10 to 15 mm. deeper than the height of the Gooch used in extraction—that is about 50 mm. for the 25 cc. Gooch. The siphon should be turned at about the height of 40 mm. from above the bottom of the cup. It is better to have the siphon elbow at the level of the top of the Gooch. In practice one may adjust the height of the Gooch to the different types of material that are to be extracted by inserting a glass rod in the siphon cup on which the Gooch rests. This siphon must be turned snugly against the cup, otherwise it will

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1 The figures are given for a size of extractor adapted to the use of a 25 cc. porcelain Gooch crucible. For the larger Gooch crucible, 40 cc., see the figures given in the table. Special sizes for particular objects are not suggested here.
be broken in placing the cup in and out of the middle piece. The siphon should be constructed of tubing 2 mm. in inside diameter and its end cut so that it extends 1 cm. below the bottom of the stopper of the middle piece when it is set in place. Two small glass rests should be drawn on the floor of the cup, opposite the siphon, to hold the cup off from the wall of the middle piece in order to give free passage to the hot extraction vapors.

Number 5 is an ordinary porcelain Gooch, of the slightly oval form, in which material to be extracted is to be placed. The sizes given above are for a 25 cc. Gooch which conveniently holds a 15 gram sample of meat.

In changing flasks and solvents there is need of some care where, as in alternate alcohol and ether extraction, the boiling points of the solvents vary. Readjustment of the rapidity of heating is called for. There is a certain amount of danger of superheating a solvent of lower boiling point but this can readily be avoided by allowing the apparatus to cool somewhat and the first solvent to be washed out by one or two changes of the second solvent. In alternate alcohol and ether extraction if the change to ether is made while the alcohol in the extraction mass is too hot there is greater danger of an explosion.

This apparatus lends itself to ready adjustment in size and form to meet different purposes for which such apparatus is ordinarily used. The 40 cc. Gooch crucible will hold a meat sample of about 22 grams. For that size of sample, which in certain organs may be preferable to the smaller sample, it is only necessary to vary the size of the middle piece and of the siphon cup to correspond to the increased size of the Gooch crucible. This variation is met by increasing the inside diameter of the middle piece to 50 mm. and the siphon cup to a diameter of 42 mm. The heights in this case remain the same.

For certain types of fractional extraction, one may use a paper extraction thimble instead of the Gooch crucible. A simple variation in sizes to accommodate to the weight of the sample would make the apparatus available for this type of extraction. In practice, the triplicate flasks enable one to shift from one solvent to another by merely exchanging flasks.

I have used this apparatus in sets of five or seven, heating the set over a circular water-bath about 45 cm. in diameter, 12 cm. deep, and supplied with a constant water level 5 cm. from the bottom. The water bath is supplied with sets of porce-
lain rings, also with a wire gauze false bottom located 5 cm. below the top of the bath. When alcohol extraction is taking place, I settle the reflux flask number 1 into the bath some 4 or 5 cm. below the surface of the rings supporting it, letting the flask rest on the wire false bottom. A clean cloth around the set of apparatus tends to hold the steam up around the set. This allows the extraction to proceed at a most effective rate.

In ether extraction, on the other hand, the proper temperature is obtained by raising the apparatus out of the bath and supporting it with one or at most two rings out of the set.

The apparatus has proven very effective in certain extractions for histo-chemical studies. In this case it was desired that the preparation should always be immersed in the solvent and should at no time be allowed to dry. A shallow porcelain shell was set in the siphon cup and the Gooch then set in this shell. The solvent filtered through the Gooch and into the shell overflowing and siphoning off as usual. But the Gooch did not, of course, empty below the level of the top of the shell.

The apparatus has decided advantages by virtue of its dissectible parts. These are more or less interchangeable. When parts are broken they can be renewed with less expense and inconvenience. The cleansing of the apparatus is also facilitated.

**Dimensions of the Extractor for 25 cc. and 40 cc. Porcelain Gooch Crucibles.**

<table>
<thead>
<tr>
<th>Piece No.</th>
<th>Description</th>
<th>mm.</th>
<th>mm.</th>
<th>mm.</th>
<th>mm.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>The reflux flask, 250 cc. capacity, pear shape.</td>
<td>46</td>
<td>90</td>
<td>15-20</td>
<td>15-20</td>
</tr>
<tr>
<td>2</td>
<td>The Hopkins condenser, 300 mm. long, 35 mm. diameter.</td>
<td>50</td>
<td>90</td>
<td>15-20</td>
<td>15-20</td>
</tr>
</tbody>
</table>
Piece No. 4. Siphon cup:

<table>
<thead>
<tr>
<th>FOR GOOCH NO.</th>
<th>INSIDE DIAMETER AT TOP</th>
<th>HEIGHT CLEAR OF TURN OF SIPHON</th>
<th>HEIGHT OF SIPHON</th>
<th>LENGTH OF SIPHON FROM TOP OF TURN TO TIP</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>mm.</td>
<td>mm.</td>
<td>mm.</td>
<td>mm.</td>
</tr>
<tr>
<td>1</td>
<td>38</td>
<td>50</td>
<td>35-40</td>
<td>100-110</td>
</tr>
<tr>
<td>2</td>
<td>42</td>
<td>55</td>
<td>40-45</td>
<td>110-120</td>
</tr>
</tbody>
</table>

Piece No. 5. The Gooch crucible sizes:

<table>
<thead>
<tr>
<th>GOOCH NO.</th>
<th>CAPACITY</th>
<th>OUTSIDE DIAMETER</th>
<th>HEIGHT</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>cc.</td>
<td>TOP mm.</td>
<td>BOTTOM mm.</td>
</tr>
<tr>
<td>1</td>
<td>25</td>
<td>36</td>
<td>15</td>
</tr>
<tr>
<td>2</td>
<td>40</td>
<td>40</td>
<td>25</td>
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