A SOXHLET TYPE OF EXTRACTION APPARATUS FOR OPERATION AT LOW TEMPERATURES UNDER REDUCED PRESSURE

BY A. HAMBLETON

(From the Mara Laboratories at the Queen Alexandra Sanatorium, London, Canada)*

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The Soxhlet type of fat extraction apparatus is not well adapted to the extraction of thermolabile biological products, as the extracted matter is subjected to the boiling temperature of the solvent used. Ethyl ether, which has a conveniently low boiling point, is unsuited for many extraction purposes (1). In the course of a serological examination of serum lipids, the Soxhlet type of apparatus was found to give the most satisfactory extraction, but to permit the use of a wider range of solvents the apparatus was modified to operate under reduced pressure. This allowed the boiling point of the solvent, and hence the temperature during the extraction, to be adjusted as desired. The apparatus described herewith has been in use for 2 years, and has proved very satisfactory.

The first apparatus which we constructed for extraction under reduced pressure had tapered ground joints which were made airtight by a mercury seal. After holding the apparatus under a vacuum, these ungreased joints were difficult to separate. It is not feasible to grease the joints, since the organic solvents used would dissolve some of the grease and contaminate the extract. Nor can rubber stoppers be used at the joints without contamination of the extracts. This led us to use flat glass joints, which have met our requirements. The complete apparatus is shown in Fig. 1, and is constructed entirely of Pyrex glass. The bulb condenser is 30 inches long, as a large condensing surface is needed when working at lower temperatures under reduced pressure.

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The condenser is supported by rubber-padded clamps attached to a \( \frac{1}{2} \) inch iron rod, which runs from the top of the bench to the ceiling. There is only one joint in the apparatus, where the flask fits the condenser. This joint, as shown in Fig. 1, is made perfectly flat, finely ground, and then highly polished. The amount of air which leaks in through a well made joint of this type is very small. In operating this apparatus at absolute pressures of 100 to 200 mm. of Hg, the leakage amounts to 20 to 30 cc. of outside air per minute. For most work this is of no significance, although in extracting substances which are very subject to oxidation it is desirable to have the apparatus completely air-tight. This can be largely achieved by slightly changing the joint as follows: The joints are finely ground, but not polished, the flat surface of the flask being slightly larger than the corresponding flat surface.

Fig. 1. Complete extraction apparatus. A is a pipe leading to the vacuum line; B, a reflux condenser; C, a flat joint between flask and condenser; D, a wire which passes through small holes on opposite sides of the vapor pipe and supports the siphon extraction cup, E.
attached to the condenser (Fig. 1). The apparatus is connected with the flat joints clean and dry, and melted paraffin wax is then painted around the outside edge of the joint where it at once solidifies. When using this method, all our extractions were carried out at 50° or lower, so that no softening of the wax occurred. Although paraffin wax is soluble in many of the organic solvents employed, contamination of the extract with traces of paraffin was very rare. This method of sealing the joints is not ideal, and the dry polished joints are always to be preferred where the slight leakage of air into the apparatus will not impair the material which is being extracted. One great advantage of the flat polished joint over the tapered joint is that a number of flasks may be used interchangeably, and additional flasks can be made by the glass-blower without having the condenser available. The same type of joint is equally satisfactory for the preliminary concentration of the extracts, as indicated in Fig. 2.

To avoid the slight warping which may take place in glass soon after being worked in the flame, it is advisable that after making and rough grinding the flat joint, the apparatus should be laid aside for 2 or 3 weeks before the final grinding and polishing of the joint. The polished joints on our apparatus are ½ inch wide. Fig. 1 shows the 1 inch wide unpolished joint which was used when the edge of the joint was sealed by paraffin wax.

The neck of the extraction flask is 2 inches in diameter, which
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allows the use of extraction thimbles 1½ inches in diameter. It is not advisable to construct the apparatus for flasks greater than 2 inches in diameter at the neck, as it is difficult or impossible to grind and polish such large joints with the desired accuracy.

![Diagram of Manometer to maintain desired degree of vacuum. The top nut of the packing gland, which is threaded onto the brass rod, is loosened when the brass rod is being screwed up or down. The metal faces marked K are knurled to provide easier grip for the fingers. The manometer is mounted by means of putty into a suitable channel gouged out of a thick wooden board.](image)

We have found this apparatus easier to operate than the ordinary Soxhlet extractor. On holding the flask in position below the condenser and opening the tap to the vacuum line, the flask is held firmly in place by air pressure, and no clamp should be used.
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The electric heater should be covered by a piece of rather soft 1/4 inch asbestos board, the hole in the asbestos being of such size that the flask is heated only below the liquid level. The heater must always be removed before the vacuum is broken.

The vacuum may be held at any desired value by means of an automatic manometer such as is shown in Fig. 3. The long arm of the manometer tube is of 3 mm. bore, while the shorter limb is of 9 mm. bore. In the wall of this shorter limb is sealed a platinum wire to make a permanent contact with the mercury, while an adjustable platinum contact wire is attached to a threaded brass rod working in an air-tight packing gland. This contact can be adjusted to give any desired degree of vacuum, from air pressure to 1 cm. absolute pressure. The small current passing through the Pt-Hg contact in the manometer operates a relay mercury switch which starts and stops the motor for the vacuum pump. The pump is connected to a heavy 10 gallon glass bottle which acts as a vacuum reserve and stabilizer, and to this bottle also runs the vacuum line from the extraction apparatus. The pump should not be connected directly to the vacuum line running to the extraction apparatus, as this would cause larger fluctuations of pressure.

Most of our work with this apparatus has been carried out at temperatures of 35-45°, and extraction at these lower temperatures is satisfactory, provided that the sample is thoroughly dried upon the inert base. We have found finely ground Na₂SO₄ to be a suitable base, while CaSO₄·2H₂O has always been unsatisfactory, as Table I indicates. These figures are for an extraction of 40 minutes at 40° with absolute ethyl alcohol.

<table>
<thead>
<tr>
<th>Inert base</th>
<th>Solvent</th>
<th>Fatty acid extracted per 100 cc. serum</th>
<th>Efficiency of extraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na₂SO₄</td>
<td>C₂H₅OH</td>
<td>103.4 mg.</td>
<td>99.3 per cent</td>
</tr>
<tr>
<td>CaSO₄·2H₂O</td>
<td>&quot;</td>
<td>73.5 mg.</td>
<td>70.6 per cent</td>
</tr>
</tbody>
</table>

The sheep serum used contained 104.2 mg. of total fatty acid per 100 cc. by Bloor's method (2).

The vapor pressures of all the common solvents over a wide range of temperature are listed in the chemical handbooks (3).
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BIBLIOGRAPHY

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