SCIENTIFIC SECTION

A METAL EXTRACTION APPARATUS FOR PHYTOCHEMICAL WORK.

BY. A. F. SIEVERS.*

In connection with studies on plants yielding essential oils, fixed oils and drugs, the Division of Drug and Related Plants of the U. S. Department of Agriculture required an apparatus that would permit the extraction of considerable quantitites of material with petroleum ether. No apparatus entirely meeting the requirements is available from dealers in laboratory supplies and scientific equipment, therefore it was necessary to design such and have it constructed in the Department Shops. A general idea of the type of apparatus desired was suggested by that described by Bryant[1] of the Ontario Agricultural Experiment Station. Certain modifications were necessary, however. A tubular condenser was preferred and a steam-jacketed receiver was considered essential. The apparatus finally constructed proved to be so generally satisfactory that its detailed description may be of interest to research workers who are required to extract relatively large quantitities of plant material.

Fig. 1.—Extraction apparatus for phytochemical work.

All the main parts of the apparatus are constructed of tin-lined copper weighing 32 ounces to the square foot. The illustration and detailed description here given should enable those interested to prepare working drawings by means of which the apparatus can be constructed by sheet metal workers.

Extraction Chamber.—This is 20 inches high and 10 inches in diameter with a total capacity of 6.8 gallons or about 6 gallons up to the line to which it may be filled. Around the top edge is a stout brass flange $2^1/_2$ inches wide and $^1/_8$ inch thick, to which is fitted the condenser which is provided with a similar flange at its base. A tight seal is obtained by means of a pressed cork gasket of the same shape and width as the flanges and $^1/_8$ inch thick. The condenser is secured by 12 threaded brass screws with wing nuts screwed upward through the lower flange which fit into corresponding

* Bureau of Plant Industry, U. S. Department of Agriculture.

[1] L. R. Bryant, *Industrial and Engineering Chemistry, Analytical Edition* (July 15, 1929), page 139.

but unthreaded holes in the upper flange. By this means removal of the condenser is quickly accomplished. The chamber is reduced at the base by means of an inverted cone to a diameter of 4 inches to which is attached a cylindrical extension $4\frac{1}{2}$ inches long, fitted at the base with a brass flange $1\frac{1}{4}$ inches wide and $\frac{1}{8}$ inch thick. To this is attached the receiver fitted at the neck with a similar flange, the seal being secured by means of a cork gasket and 6 brass screws. A perforated, tinned brass plate is fitted as a false bottom at the base of the chamber just above the cone and a solid plate at the base of the inverted cone forms the bottom of the chamber.

Extending upward from near the top of the cylindrical extension between the extraction chamber and the receiver to a point just below the condenser connection is a tin-lined copper tube, one inch in diameter, to carry the solvent vapors from the receiver to the upper part of the apparatus. This tube has no joints or elbows but is bent into the desired shape and has soldered connections, thus eliminating possible leaks. On the opposite side of the chamber is a glass gage of $\frac{5}{8}$ inch outside diameter. The siphon consists of a single piece of block tin tubing $\frac{3}{16}$ inch inside diameter with soldered connections. A draw-off cock (not shown) at the base of the chamber on the side opposite to the siphon provides for draining the chamber. A cotton bag slightly smaller than the extraction chamber was used in this case for holding the charge. Other receptacles may be used in accordance with the nature of the material to be extracted.

Condenser.—The condenser is a highly efficient tubular type. It consists of a vertical cylinder 18 inches high and 4 inches in diameter containing 33 evenly distributed, tin-lined, copper tubes $\frac{3}{8}$ inch in diameter. The condensed solvent flows freely from these vertical tubes without obstruction by the ascending vapors as is frequently the case when a worm condenser is used, especially with a low boiling solvent like petroleum ether. A tubular condenser such as described weighs less than the usual worm type when in use because it holds less water and is on the whole somewhat easier to handle.

Receiver.—The receiving vessel is constructed of the same material as the extraction chamber and is 10 inches in diameter and 15 inches high. It is reduced at the top to an opening of 4 inches by means of a cone to which is attached a short collar fitted with the brass flange, already referred to, by which the receiver is attached to the extraction chamber.

As this apparatus was primarily intended for extraction with low boiling, flammable solvents, the receiver is enclosed with a jacket extending around and under the vessel with a space of 1 inch between the walls and bottoms. To volatilize the solvent steam is introduced into this jacket near the top, there being an outlet at the bottom on the opposite side (not shown). The cock for drawing off the concentrated extract is shown at the base. It connects with the bottom of the receiver. In the cone top of the receiver is a 1-inch collared opening (not shown) through which the receiver may be filled without disconnecting it and in which a thermometer may be fitted.

The apparatus described should be exceedingly useful in general research work which requires the extraction of considerable quantities of material, especially with low-boiling, flammable solvents such as ether or petroleum ether. Experience has shown that it can be operated without leakage of solvent at any point but it is advisable to attach a tube to the top of the condenser to conduct to a convenient flue or window those traces of very low boiling fractions of the solvents mentioned that usually cannot be condensed. It is believed also that this apparatus would be most useful in phytochemical laboratories where the extraction by students of bulky plant material with flammable solvents involves considerable fire risk unless the escape of solvent from the apparatus is eliminated.

In addition to its use as an extractor the apparatus may also be conveniently used as a still for concentrating extracts and for fractioning solvents such as petroleum ether, the extraction chamber serving as the receiver in such cases. The immediate purpose for which the apparatus here described was intended was for the extraction of absolute essence of rose from roses. It was very satisfactory for this purpose but the rose "concrete" thus obtained, consisting of waxes and essential

oil, is present in small amounts, is readily soluble in petroleum ether and does not require long contact with the solvent nor frequent changes of fresh solvent. It was found, therefore, that much larger quantitities of roses could be handled in a given time by extracting these in five-gallon milk cans by maceration with several subsequent washings and using the apparatus for concentrating the extract. It is expected, however, that it will prove most useful in the usual way in the extraction of botanical drugs, oil seeds, oleoresins, etc., where continuous percolation with fresh solvent is desired.

AMMI VISNAGA.*

BY F. A. UPSHER SMITH, PH.C. (GT. BRIT.).

A PRELIMINARY REPORT.

The fruits of *Ammi visnaga* have been used in Egypt to relieve spasms of the ureter and for the removal of calculi. The author visited Cairo for the purpose of investigating this drug, and now submits a preliminary report with a complete bibliography showing what is known to date as to the pharmacognosy, pharmacology, therapy, pharmacy and chemistry of the drug, which is commonly known as *Khella.*

Most of the information that I have so far gathered is due to Dr. Karam Samaan, professor in the Department of Pharmacology in the University of Cairo. During the three weeks that I spent in Cairo, in the Spring of 1931, I had many interviews with Dr. Samaan, and he discussed with me what was then known as to the chemistry and pharmacology of Khella. As his results have been recorded in the literature there is no need for me to more than summarize them.

PHARMACOLOGY.

Khella appears to relax all smooth muscle, including that of the ureter. This explains the value claimed for the drug in allowing the passage of stone through the ureter.

The experiments leading to this conclusion were performed with a 10% tincture of the fruits of *Ammi visnaga* and 70% alcohol. The animals used included the pig, bull, cow, camel, sheep and dog. The relaxation of the ureter was greater in the case of the cow and bull, and poorest in the dog and camel. The drug lowers the tonicity of the ureter. Clinically, it relieves spasm of the ureter. The low toxicity of the drug suggests its usefulness in ureteral calculi and spasm of the ureter. Khella was found to possess a fairly strong diuretic action.

PHARMACOGNOSY.

I had the pleasure of meeting Dr. Ibrahim Ragab Fahmy, professor of Pharmacognosy in the University of Cairo. We made excursions into the Valley of the Nile, and he showed me Khella in its wild state, as well as another plant which was liable to be used in mistake for Khella, *viz., Ammi majus.*

Dr. Ragab Fahmy has contributed to the "Report of the Pharmaceutical Society of Egypt," Vol. III, 1931, illustrated descriptions not only of Khella, but

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* Scientific Section, A. PH. A., Toronto meeting, 1932.