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ALCOHOL-SOLUBLE EXTRACTIVE OF BENZOIN, MYRRH AND ASAFŒTIDA.

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There has been considerable complaint regarding the present U. S. P. method for determining the alcohol-soluble extractive of some drugs because of the loss of volatile constituents during the drying of the extract. This criticism applies particularly to Benzoin, myrrh and asafœtida. Of these, the one that apparently has been causing the most trouble is benzoin. It is suggested by Bickford and Bennett that the alcoholic extractive, including the water, be obtained by means of a Soxhlet extraction apparatus and that the water content be determined by the Xylol method. Other modifications of the U. S. P. method have been proposed but while they appear to give results which probably indicate a truer figure than the U. S. P. method, they are long and tedious and subject to other criticisms as well.

In this investigation there are, therefore, three questions involved, namely: *First*, is the proposed new method (Soxhlet and Xylol) for the determination of alcohol extractive reasonably speedy and satisfactory in that it gives concordant results? *Second*, is the proposed method suitable for other U. S. P. drugs such as, asafœtida and myrrh? *Third*, are these drugs as they occur in commerce at present, meeting the alcohol-soluble extractive requirements of the U. S. P.?

The method (No. 9) proposed by Bickford and Bennett is as follows:

"Weigh 2 Gm. of the sample into a dried and tared paper extraction thimble, using a glass stopper weighing bottle as a container. Extract in a continuous extraction apparatus with 95% alcohol containing about 0.5 Gm. NaOH for 5 hours. Dry and weigh thimble and calculate alcohol extractive matter plus water by difference. Deduct water as determined by xylol distillation method from the result and report as alcohol extract."

Below is a report of a brief study of this method as compared to the U. S. P. method.

Preparation of Samples.—Samples of Siam benzoin, sumatra benzoin and myrrh, consisting of about one pound each, were ground in a mortar and quartered. One quarter was further reduced to a No. 20 powder and used for analysis. In the case of asafœtida about one pound was broken up into small particles and quartered. One quarter was then used for analysis.

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Methods.—See U. S. P. X, page 466, for Siam benzoin, Sumatra benzoin and myrrh; U. S. P., page 67, for Asafœtida; and Bickford and Bennett method No. 9 as described in the foregoing.

TABULATION OF RESULTS.

Method.		Sumatra Benzoin.	Siam Benzoin.	Myrrh.	Asafœtida.
U. S. P. X		73.59%	89.80%	31.40%	55.44%
		73.20	90.90	31.10	54.74
	(Average)	73.39	90.35	31.25	55.09
No. 9		77.10	94.10	37.00	10.20
		78.50	94.30	36.80	13.30
	(Average)	77.80	94.20	36.90	11.75
Xylol Method (Moisture)		5.60%	1.90%	11.30%	7.60%

COMMENTS.

In checking the time required to make these determinations, it was found that with the U. S. P. method the process extended through an interval of from 26 to 30 hours, while for B. &. B. No. 9 from 7 to 9 hours were required, providing the moisture determinations were carried out concurrently with extraction, which is easily done. It is possible that this might be further reduced by limiting the extraction to 3 hours. In the case of the benzoin and myrrh the solvent appeared to be perfectly clear at the end of 2 hours, but this, of course, is a point to be determined by further experimentation.

While the U. S. P. method gives figures in the case of the Siam benzoin, myrrh and Asafœtida tested that meet the U. S. P. requirements, it is to be noted that the figures obtained by method No. 9 are consistently higher except for asafœtida. Inasmuch as this indirect method obviates the question of volatile matter, these figures are probably more nearly true, except for asafœtida, than those obtained by the U. S. P. method.

While the U. S. P. method for asafœtida is both tedious and cumbersome it appears, however, to give figures which meet the standard. Method No. 9 is not applicable with asafœtida in lumps as permitted by the U. S. P. method. Possibly if it were reduced to a powder by using purified sand, method No. 9 would give more consistent results. This, of course, would introduce another possibility for error but it appears that it is worth trying at least.

The limited observations covered by this report suggests that the method proposed by Bickford and Bennett is applicable to Siam benzoin, Sumatra benzoin and myrrh, and is to be commended for speed, accuracy and simplicity.

It is suggested that experimentation along this line be continued with enough commercial samples of these drugs to furnish sufficient data from which definite conclusions may be drawn.

REFERENCES.

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