(12) Tschirch, "Handbuch der Pharmacog.," II, 2—page 1433.
(13) Formanek, *Zeitsch. Anal. Chem.*, 1897, 409.
(14) Leger, *Jour. Pharm.*, 1902, 335.
(15) Kremel, *Pharm. Post*, 1895, 422; *Pharm. Zentrlh.*, 37, 656.
(16) Tschirch, *Ber. d. d. pharm. Ges.*, 1898, 196.
(17) Oesterle, *Arch. Pharm.*, 1889, 81.
(18) Schunck, *Lieb. Ann.*, 1841, 1 and 65; 1848, 234.
(19) Stenhouse, *Ibid.*, 1851, 208.
(20) Liebermann and Giesel, *Ber. d. Chem. Ges.*, 1875, 1643; 1876, 329.
(21) Seel, *Ibid.*, 1900, 3212; *Südd. Apoth.-Zeit.*, 1906, 624.
(22) W. Scharf, "Oxydationsprod. d. Aloebestandth. mit Caroscher Säure," Diss. Zurich, 1909.
(23) Tschirch and Pedersen, *Arch. Pharm.*, 1898, 206.
(24) Rochleder and Czumpelik, *Chem. Zentralblatt*, 1863, 606; 1866, 29.
(25) Aweng, *Apoth. Zeit.*, 1902, 422.
(26) Tschirch and Hoffbauer, *Schweiz. Woch. Chem. Pharm.*, 1905, 43, 153.
(27) Tilden, *Jahresber. d. Fortschritte d. Pharm.*, 1871, 15.
(28) Plenge, *Pharm. J.*, 1884, 44, 330.
(29) Schäfer, *Pharm. Zeit.*, 1897, 42, 95.
(30) Leger, *J. pharm. chim.*, 6 (1902), 15, 519.
(31) v. Itallie, *Pharm. Weekblad.*, 1905, 42, 553.
(32) Kröber, *J. Landw.*, 1900, 379.

ACKNOWLEDGMENT.

The writer wishes to express his appreciation to Dr. Charles H. Rogers, Professor of Pharmaceutical Chemistry, whose encouragement and guidance made this work possible.

CHEMICAL AND PHYSICAL DETERMINATIONS ON THE GUM AND VOLATILE OIL OF ASAFŒTIDA.

BY JOSEPH F. CLEVENGER.*

It has been apparent for some time that the method of assay of asafœtida and the requirements for that product, as given in the U. S. P. X, are not entirely satisfactory. The New York Station has recently made a study of the subject and as a result has adopted a method of assay which gives figures that appear to be a truer measure of the quality of the drug.

To obtain information to serve as a basis for suggesting a suitable quality standard for asafœtida, each of 41 lots of the gum offered for entry at the port of New York during the past three years was analyzed. Samples were made uniform by passing them through a meat grinder and for the determinations of alcohol-soluble extractive, acid-soluble ash, moisture content and yield of volatile oil the following methods were adopted:

ALCOHOL-SOLUBLE EXTRACTIVE.

Weigh 10 Gm. of the sample in a tared 250-cc. Erlenmeyer flask. Add 100 cc. of alcohol. Attach a reflux condenser and boil for one hour or until the sample is completely disintegrated. Dry a filter paper in an oven at 110° C., cool and weigh. Fit the paper in a Buchner funnel of slightly smaller diameter so that the edges turn up approximately one-fourth of an inch. Filter the gum and alcohol hot, under suction, taking care not to fill the Buchner funnel above the edges of the filter paper. Wash with hot alcohol until no cloudiness is produced when the filtrate is

* Pharmacognosist at the New York Station of the Food and Drug Administration.

dropped into water. Remove the filter paper containing the residue, roll it and return it and contents to the tared flask, avoiding the loss of any of the residue. Dry at 110° C. to constant weight (over night drying is sufficient). The difference between the weight of the sample and the sum of the weights of the residue and the water, determined by the xylol or toluol method[1] is the weight of the alcohol-soluble extractive.

VOLATILE OIL.

Transfer 50 Gm. of the sample to a 500-cc. short-neck flask. Set up apparatus as illustrated in Fig. 1. The distillation is carried to complete exhaustion and should be conducted at a rate sufficiently slow to avoid the escape of vapors around the condensers and consequent loss of volatile oil.

The yield, specific gravity, optical rotation and refractive index of the volatile oil were determined as outlined by Clevenger.[2]

The sulphur content of the volatile oil was determined as follows:

SULPHUR.

Place approximately 0.5 Gm. of the volatile oil, weighed accurately, into a 150-cc. acetylization flask. Through the condensing tube add 5 cc. of water, then 5 cc. of concentrated nitric acid. Heat gently on the steam-bath until the reaction is started, then set aside in hood until the reaction has practically subsided. Add 3 Gm. of powdered potassium bromide through the condenser tube, then heat on the steam-bath for 20 minutes and cool. Add 10 cc. of 50 per cent sodium hydroxide solution through the condensing tube. Disconnect the condenser from the flask, transfer the contents to a nickel crucible, carefully evaporate to dryness and ignite. Dissolve the residue in water, remove nitrous and nitric acids by evaporation with hydrochloric acid and determine the sulphur as barium sulphate.

Of the 41 lots of asafœtida examined the following are typical:

TABLE I.—RESULTS OBTAINED BY PRECEDING METHODS.

Source.	Alcohol-Soluble Extract,[1] Per Cent.	A. I. A., Per Cent.	Moisture, Per Cent.	Volatile Oil. Cc. per 100 Gm.	Sp. gr. 20° C./20° C.	Op. rot. 20° C.	Ref. ind. 20° C.	Sulphur,[1] per cent.
Bombay	60.4	1.4	4.9	11.5	0.973	−9.0	1.518	22.4
Bombay	74.7	2.9	2.7	8.0	0.942	−4.5	1.510	28.0
Bombay	59.1	6.9	4.3	9.2	0.915	+6.3	1.495	18.9
Bombay	58.1	9.6	4.4	8.6	0.910	+9.3	1.495	18.2
Aden	60.3	1.7	4.4	9.0	0.928	+4.5	1.498	18.0
Bombay	54.5	4.6	2.8	7.5	0.927	+3.5	1.497	15.6
Bombay	57.0	4.9	4.6	7.8	0.920	+4.1	1.497	15.8
Bombay	61.0	3.7	3.8	8.0	0.908	+8.7	1.493	17.9
Bombay	58.6	6.1	8.0	9.5	0.922	+3.8	1.498	29.0
Bombay	63.9	6.0	4.3	10.0	0.921	+5.5	1.497	19.8
Bombay	70.2	3.6	3.6	9.0	0.935	+2.7	1.504	18.4
Bombay	58.5	7.2	3.8	11.0	0.906	+8.0	1.493	17.9
Bombay	63.0	12.9	4.0	12.0	0.929	+2.1	1.501	22.9
Bombay	59.6	4.4	4.3	10.0	0.960	−9.0	1.509	15.3
Bander Abbas	58.9	3.4	11.8	9.5	0.933	+7.3	1.497	19.6

[1] Determinations were made by O. C. Kenworthy; J. A. Batscha and J. A. Reilly, chemists in drug laboratory.

[1] Book of Methods of the A. O. A. C., 1930, page 227.

[2] JOUR. A. PH. A., 17 (1928), 345.

CONCLUSION.

The only assay so far recognized for gum asafœtida is alcohol-soluble extractive. The assay prescribed in U. S. P. IX was indirect and consisted in the determination of the alcohol-insoluble residue from which the alcohol-soluble extractive was calculated. The method was faulty in that the moisture content which was included could not be called properly alcohol-soluble extractive.

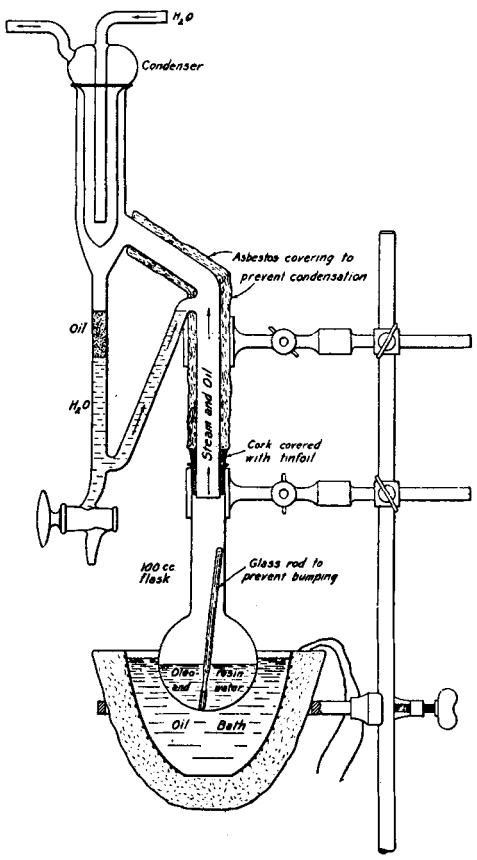

Fig. 1.

On the other hand, the assay in U. S. P. X is direct in that the alcohol-soluble extractive is dried to constant weight. The method is faulty in that an undetermined proportion of the volatile oil is lost and the resulting values for alcohol-soluble extractives are too low.

The method of assay adopted by the New York Station is similar to one used by Bennett and Bickford[1] in determining the alcohol-soluble extractive in gum benzoin and is essentially that given in U. S. P. IX with suitable corrections for moisture.

Since the volatile oil constitutes from 10 to 19 per cent of the alcohol-soluble extractive and since there is a probability of loss of volatile oil in certain manufacturing processes, it is recommended that in addition to the requirement for alcohol-soluble extractive a suitable volatile oil standard for asafœtida be adopted in the forthcoming U. S. P.

TURBIDIMETRIC MEASUREMENTS FOR PHARMACEUTICAL PREPARATIONS.*

BY SAMUEL CLAMAN, C. JELLEFF CARR AND JOHN C. KRANTZ, JR.

INTRODUCTION.

Clarity Standards for pharmaceutical preparations serve a twofold purpose. *First*, they insure that solutions intended for intravenous medication are free from insoluble particles. *Second*, they enable the establishment of a high degree of pharmaceutical elegance in such preparations as the elixirs and waters. In conformity with this view the new Netherlands Pharmacopœia makes the requirement:

[1] *Jour. A. O. A. C.*, 11 (1928), 386.

* Section on Practical Pharmacy and Dispensing, A. PH. A., Miami meeting, 1931.