

METHODS IN USE AT THE LABORATORY OF THE BOURSE
DE COMMERCE, PARIS.

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I.—EXAMINATION OF WAX FOR ADULTERATIONS.

(1.) ESTIMATION OF STEARIC ACID.

INTRODUCE into a flask 3 or 4 grams. of the sample for analysis, and bring it to the boiling point with 60 c.c. of alcohol of 96° ; shake whilst cooling, and then titrate the alcoholic solution with half normal soda, employing phenol phthalein as an indicator. Wax being only slightly soluble in cold alcohol, there is no need to take notice of its acidity, and the amount of the mixture can be calculated as stearic acid from the number of c.c. normal soda used in the titration, knowing that 7.8 c.c. of half normal soda = 1 gram. of commercial stearic acid.

(2.) ESTIMATION OF PARAFFIN AND OF MYRISTIC ACID.

To the flask containing the neutralised alcoholic solution, add 3 to 4 c.c. of solution of soda of 50 per cent.; attach the flask to an upright condenser and heat for an hour to saponify. The saponification being complete, distil off the excess of alcohol, put the residue into a capsule, mix with dry sand and short asbestos, dry at 100° , pulverise and

extract it with warm chloroform (or petroleum ether), which dissolves the whole of the paraffin and the myristic acid, representing a part of the wax. To separate the paraffin, Horn has recommended acetilisation and solution of the produced ether by means of acetic acid, in which paraffin is insoluble. According to Horn, saponified wax should, under these conditions, yield 50 per cent. of matters soluble in glacial acetic acid. Following Horn's process, we have neither been able to properly separate the paraffin nor to obtain a constant factor for the part of the wax soluble in chloroform.

We effect this separation in the following manner :—

The chloroform holding in solution a part of the wax and all the paraffin is distilled off in a weighed flask, and the residue, having been dried at 100, is weighed.

Then weigh, in a small flask, a part of the residue left by the evaporation of the chloroform, and treat it under an upright condenser for an hour with 4 to 5 c.c. of anhydrous acetic acid. The acetilisation being complete, pour the resulting fluid into a glass tube graduated in 10 c.c. and divided into tenths; rinse the flask with boiling crystallisable acetic acid, and turn the whole into the graduated tube. The volume of the liquid should be about 9 c.c. Place the tube in a water-bath at 90°, then close it up with a cork and shake it forcibly so as to well emulsify the liquids, and replace in the water-bath.

When the acetic acid has become clear, the volume of insoluble matter which floats on the acid is read off. Renew the shaking and place in the water-bath until a constant volume of paraffin insoluble in acetic acid is obtained, of which calculate the weight, remembering that 1 gram. of paraffin = from 1.35 to 1.4 c.c. On deducting the weight of the paraffin from the weight of the residue furnished by the chloroform, we obtain by difference the weight of the portion of the saponified wax soluble in chloroform.

(3.) ESTIMATION OF STEARIN.

The saponified part insoluble in chloroform is formed by the soap of stearic acid and of stearin and by saponified cerotic acid. To estimate the stearin, dissolve in boiling water, filter to separate the sand and asbestos, and decompose the filtered liquor by a slight excess of nitric acid diluted so as to set free the fatty acids, filter and estimate the glycerine in the filtered liquid (after neutralisation and precipitation by plumbic acetate), by the potassium bichromate process. From the weight of the glycerine, calculate the stearin or suet, keeping in mind that 5 of anhydrous glycerine = 95 of stearin.

In cases where the proportion of stearin is small, it would be preferable to saponify 10 or 25 grams. of the substance and to estimate the glycerine by the bichromate process.

We therefore estimate by this method :—

1. Stearic acid by alkalimetry.
2. Paraffin by measuring the part insoluble in acetic acid.
3. A part of the wax (myristic acid) by deducting the paraffin residue from the weight of the residue soluble in chloroform.
4. Stearin by the estimation of glycerine.
5. The second part of the wax (cerotic acid) by difference.

II.—ANALYSIS OF WINE.

(1) ESTIMATION OF GLYCERINE.

Evaporate 250 c.c. of the wine to the volume of 100 c.c., then agitate this concentrated liquid with freshly precipitated plumbic oxide, and render it slightly alkaline with baryta water. Filter, wash, and neutralise the filtrate with dilute sulphuric acid. Concentrate in a flat porcelain capsule, and when the volume of the liquid has been reduced to 50 c.c. incorporate therein 5 grams. of plumbic oxide, 10 grams. of sand, and 20 grams. of barium sulphate, evaporate and dry at 100° C. During this drying the basin should be covered with a plate of glass to avoid spurting. The dried mass, having been powdered, is extracted with a mixture of equal parts of alcohol and ether, the extraction being continued until 60 c.c. of liquid has been obtained. Thirty cubic centimetres of this liquid is placed in a tared glass capsule, and 20 grams. of dried and powdered litharge having been added, the whole is evaporated in the water-bath, and then dried to a constant weight between 105 and 106° C. The other 30 c.c. is evaporated in a tared glass capsule 6 centimetres in diameter, and the residue is placed in the air bath between 160 and 170° C. until a constant weight is obtained.

The weight of residue No. 1, after deducting that of the litharge and capsule employed, *minus* the weight of residue No. 2, being first multiplied by 1.243, and then by 8, gives the weight of glycerine present in a litre of the wine.

(2) ESTIMATION OF ASTRINGENT ACIDS.

(a) *Estimation of Oenontannin.*

Concentrate 200 c.c. of wine down to 100 c.c., shake with an excess of freshly precipitated arsenious sulphide, filter and wash. Concentrate the filtrate to 50 c.c., add 10 grams. of silica, and 20 grams. of barium sulphate, and dry at 100° C. Powder the residue and extract it with warm ether; evaporate the ether and dissolve the residue in a little alcohol. Take 1 gram. of powdered hide which has been washed with alcohol, and dry at 100° C. Moisten it with a few drops of distilled water, and having added the alcoholic extract, allow the whole to macerate for half an hour. Filter through a square of cambric, previously dried and weighed, wash with alcohol, press out excess of liquid, and dry at 100° C. The increase in weight of the hide, multiplied by four, gives the oenontannin in one litre of the wine.

(b) *Estimation of Oenogallic acid.*

Dilute the alcoholic filtrate from the hide with distilled water to 100 c.c., and in 20 c.c. of this estimate the acid by means of a solution of iodine that has been previously standardised with gallic acid as follows:—

Prepare solution of iodine in potassium iodide containing 2 decigrams. of iodine per litre, and also a solution containing 0.125 gram. of gallic acid in 250 c.c. of distilled water. Mark a beaker at 50 c.c., place into it 10 c.c. of the gallic acid solution, and 3 c.c. of cold saturated solution of sodium bicarbonate. To this add the iodine drop by drop from a burette until a drop of the mixture, tested on thick filter paper dressed with powdered starch, leaves a stain surrounded by blue. Now add distilled water to the 50 c.c. mark and continue the addition of the iodine until a similar stain is again obtained. The amount of iodine thus used must be further corrected by making a blank

experiment on 50 c.c. of distilled water with 3 c.c. bicarbonate solution, and deducting the amount of iodine required to produce the stain. Having thus standardised the iodine, it is used in a similar manner on the 20 c.c. of the alcoholic liquid from the wine which has been previously neutralised with sodium bicarbonate.

(3) ESTIMATION OF COLOURING MATTER.

250 c.c. of the wine concentrated by evaporation (but without boiling) to 100 c.c. is rendered freely alkaline by ammonia, and then well shaken up precipitated arsenious sulphide. The whole is then filtered and washed with distilled water, and the filtrate having been rendered acid by acetic acid, to precipitate any sulphide of arsenic dissolved by the ammonia, is again filtered and washed. The two filters containing the sulphide of arsenic are digested on the water-bath in alcohol acidulated with acetic acid, and the whole having been again filtered, the residue is washed with hot alcohol until all the colouring matter is extracted. Finally, the alcoholic solution is evaporated in a tared capsule, desiccated at 105°C , and the residual colouring matter is weighed.

III.—PREPARATION OF BUTTER FOR THE REFRACTOMETER.

The butter is melted in a porcelain capsule, and then beaten up with two or three pinches of fused and pulverised calcium chloride, which takes up the water and the casein. The whole is then kept warm until it settles, and the clear butter-fat is decanted off and filtered through a plug of cotton wool; the clear butter-fat is heated to 60°C . and placed in the prism of the instrument, and the reading is taken at the moment when the inner thermometer marks 45°C .
