Quality Control Testing of Pepper

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Pepper is probably the world's most important spice, both in terms of total production and value of production. The 1958 *production of* 65,000 *tons was valued at* $US70,000,000 *and current production is around* 100,000 *tons. World consumption of pepper is increasing, and the supply at present is only just meeting the demand. Thus the crop has good potential as a cash crop for Papua New Guinea, being comparatively easy to grow and process.*

Black pepper is produced by drying the green, unripe berries of *Piper nigrum*, a vine-like shrub of the *Piperaceae* family native to southern India. (see *Plate* I). White pepper is produced from the same plant but from riper orange-red berries. These berries have the cortex or skin removed before drying, leaving the white berry.

In commercial food processing such as meat canning, pie and sausage making, etc., whole pepper is rarely used. The manufacturer prefers to use an extract of the pepper known as oleoresin and/or the volatile oils of pepper.

Oil of pepper, obtained by steam distillation of the ground spice, is used to provide the aroma and some of the flavour of natural pepper. It has none of the pungency of pepper, however, since the pungent principles are not steam volatile. Oleoresin, on the other hand, contains the pungent principles (most important of which is piperine), the volatile oil and various resins. To prepare oleoresin, the ground pepper is extracted with a solvent such as alcohol, ether or acetone. Subsequent evaporation of the solvent leaves the viscous semi-solid pepper oleoresin. This can be standardized according to its pungency and flavour, and thus offers a uniform concentrated product that is more easily handled than the natural spice.

(Photo: D.I.E.S)

Plate I.—A pepper vine. The peppercorns grow close together on a stem about 6 in long

The need for quality testing

For a pepper to be accepted on the world markets, it has to meet certain analytical standards. It also has to measure up to other peppers in taste and aroma, which are not as easy to measure. Commercial processing companies evaluate pepper samples on the basis of yield of oleoresin, the amount of piperine in the oleoresin, the yield of volatile oil and its chemical composition. Naturally, the peppercorns also have to be of good colour and appearance and free from mould and insect damage.

The standards set by large processors such as Stafford Allen of England and the Essential Oil Association of America, are as follows:

Oleoresin content of pepper—8-11%

Piperine content of oleoresin—55-65%

Volatile oil content of pepper—2.3% min.

Volatile oil content of oleoresin—15-35%

Values recorded for Papua New Guinea peppers have ranged over the following limits:

Oleoresin—7-18%

Piperine—41-80%

Volatile oil—1.7-3.9%

Volatile oil in oleoresin—11-38%

It is obvious that there is a need for quality control of pepper, in order to produce the best possible product and so ensure that potential markets are not jeopardized by the supply of inferior grades of spice.

QUALITY CONTROL METHODS

Oleoresin: Finely ground pepper (about 10 g) is extracted with acetone in a Soxhlet extractor which permits continuous unattended extraction of the spice to be carried out. A description of the apparatus was given in *Harvest* Vol. 1, No. 4 of 1971 page 143. The solvent is then removed from the extract by drying at room temperature, usually under vacuum. Oleoresin remains behind as a viscous, greenish-black mass. A simple calculation then enables the oleoresin content of the pepper to be determined.

Piperine: There are several methods, of varying accuracy, that are used for estimating piperine.

One method employed measures the amount of nitrogen in the pepper oleoresin. As piperine contains 4.9 per cent nitrogen, multiplying the total nitrogen by a factor of 20.36 gives an estimate of the piperine content of the pepper. Most of the nitrogen in oleoresin comes from piperine but nitrogen in other compounds will also be measured so the piperine estimate may be slightly high.

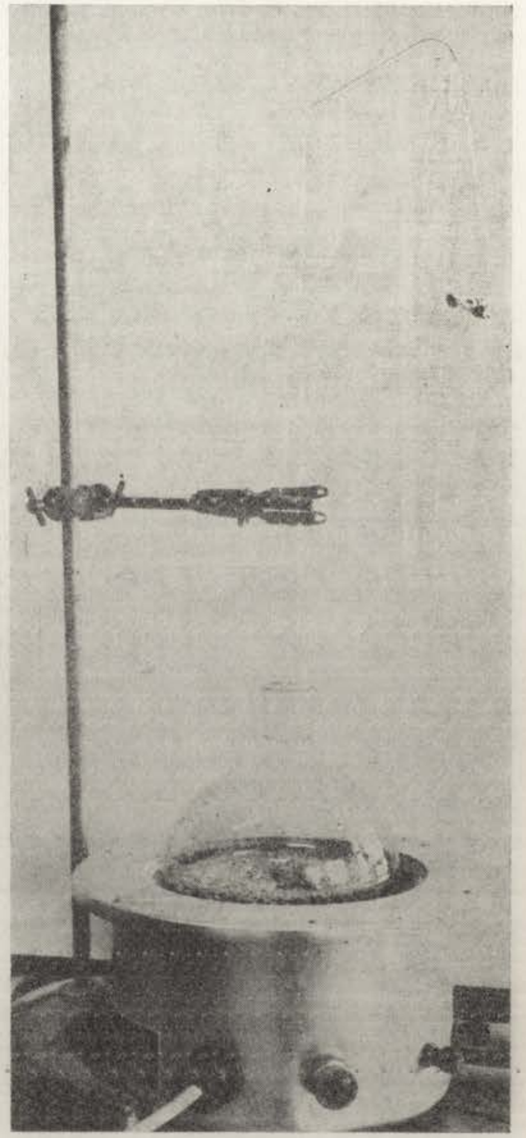
Another method employs sulphuric acid to break up the piperine molecule to produce formaldehyde and other by-products. The formaldehyde then reacts with chromotropic acid to produce a purple colour, which can be accurately measured by a spectrophotometer. Unfortunately, the chromotropic acid may also react with some other compounds present in pepper, so that the results obtained can be appreciably higher than they should be.

A third method which is very simple, and is the one used at our laboratory, is based on the fact that piperine absorbs ultraviolet (U.V.) light. The piperine in a sample of pepper is extracted with a suitable volume of alcohol and the amount of U.V. light absorption measured in a spectrophotometer. By also measuring the absorption of a series of solutions containing different but known amounts of piperine, we can calculate how much piperine there is in the pepper oleoresin.

Volatile oil: Ground pepper (about 50 g) is boiled with water in a flask fitted with an essential oil still (*Plate* II).

The pepper oil is volatile with steam and so passes up and over the condenser. Here the vapours are condensed and separated into oil and water. The oil floats on top of the water layer, while the water is continuously returned to the distilling flask. After the volume of the oil has been measured, it can be recovered and analysed.

Quality of Volatile Oil by TLC: Thin layer chromatography (TLC) is an analytical technique of great value to chemists. It utilizes



(Photo: C. A. Fowler)
Plate II.—An essential oil still

a very thin layer of finely ground powder such as alumina (aluminium oxide) coated on the surface of a flat glass plate. A small drop of the substance to be examined (in this case pepper oil) is placed near the bottom of the plate and the plate is then stood in a shallow layer of solvent (in this case toluene) in a glass tank. Capillary action draws the toluene up the thin layer of alumina, through the spot of pepper oil and then further along the layer. Each compound in the pepper oil tends to flow with the moving toluene, but is held back more or less strongly by the alumina. Thus the individual compounds are spread out up the plate. By drying the plate and spraying it with a suitable reagent to make the compounds coloured, we can produce a pattern characteristic of the pepper oil. Several pepper oils can be analysed on the one plate simultaneously so comparisons of Papua New Guinea pepper oil with commercially acceptable oils from overseas can be readily made (*Plate* III).

Composition of Volatile Oil by Gas Chromatography: Gas chromatography (GC) is an even more sensitive analytical method than TLC. The gas chromatograph (*Plate* IV) operates with a slow stream of nitrogen gas flowing through the coiled tubular column,

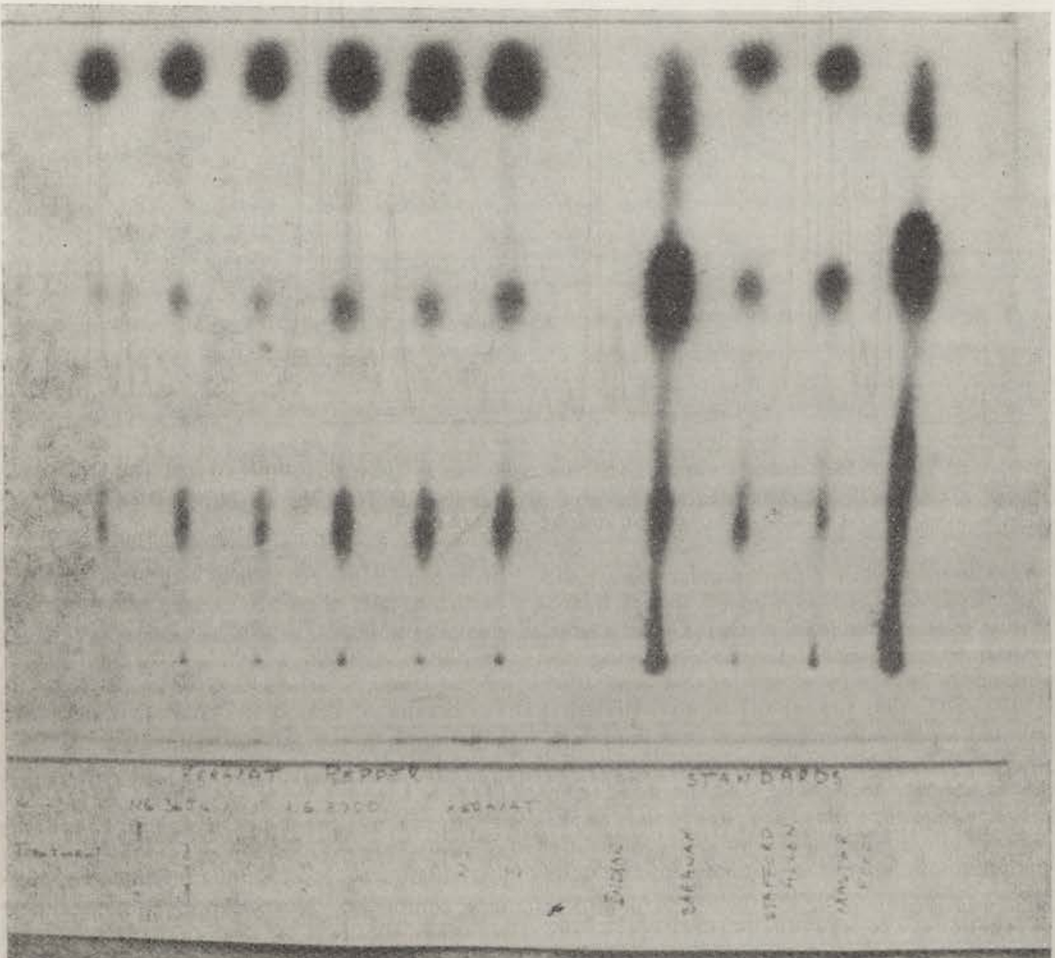


Plate III.—A thin layer chromatography plate. The different components in the pepper oil spread out on the plate, making it possible to compare the relative amounts of components in different samples of oil

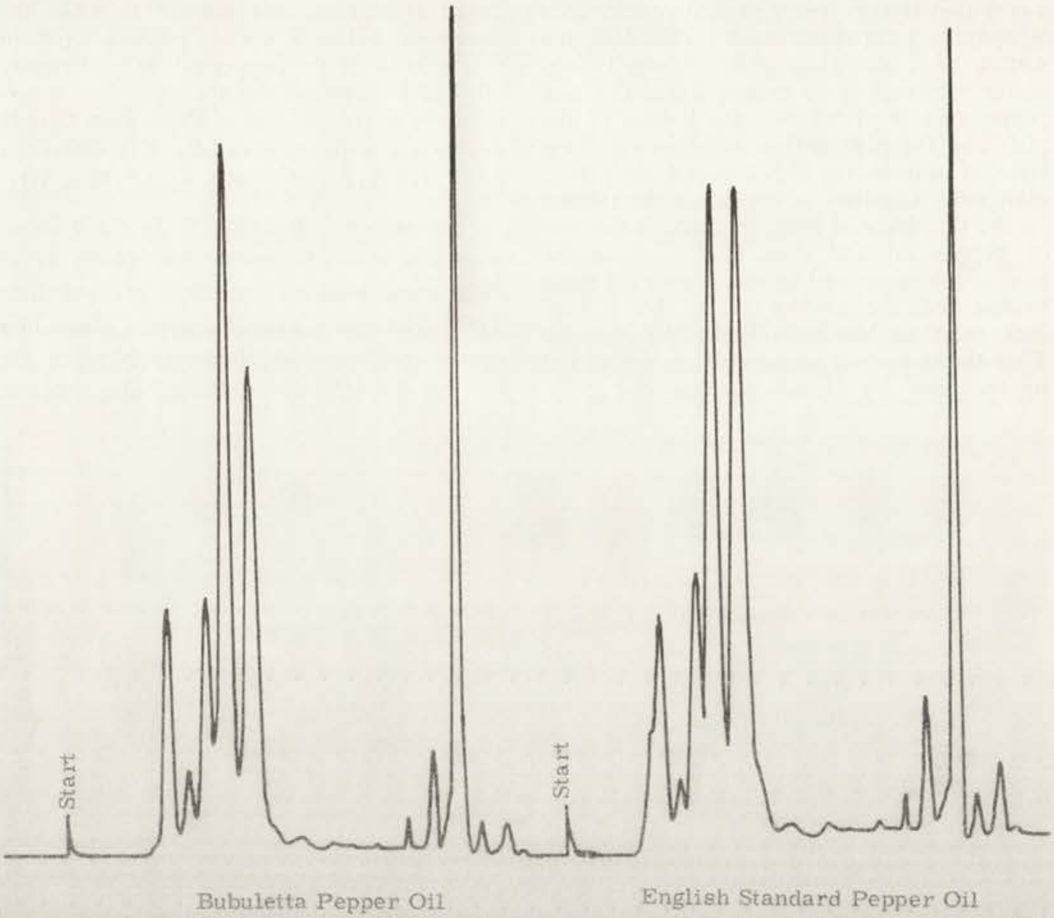


Figure 1.—A gas-liquid chromatograph trace of two pepper oils. Note the similarity between the PNG sample and the overseas standard

which is filled with a fine granular silica packing coated with a thin film of special hydrocarbon grease (stationary phase). Under normal operating conditions the oven containing the column is heated to something between 100° and 200°C. The pepper oil to be analysed is injected by hypodermic syringe onto the beginning of the column and immediately vaporises. These vapours are carried past the packing by the flowing nitrogen. Each compound in the pepper oil vapour tends to dissolve in the stationary phase, but also tends to evaporate and move with the flowing nitrogen. These two opposing factors separate the individual components, with the more volatile compounds emerging from the column first.

The end of the column is connected to a small jet of burning hydrogen. Normally a hydrogen flame contains very few ionized particles, but when an organic compound is present and burnt, it produces ions which make the flame conducting. The electrical conductance of the flame, which is directly proportional to the amount of compound present, is amplified and traced out by a recorder to give a chromatograph of the components in the pepper oil (*Figure* 1).

When testing first began, the TLC patterns of Papua New Guinea pepper oils bore little resemblance to those of oils provided by overseas companies. Now, however, they closely resemble the best of the overseas standards in both piperine and volatile oil content. Thus Papua New Guinea at present can produce pepper of a high standard, which should be readily saleable.

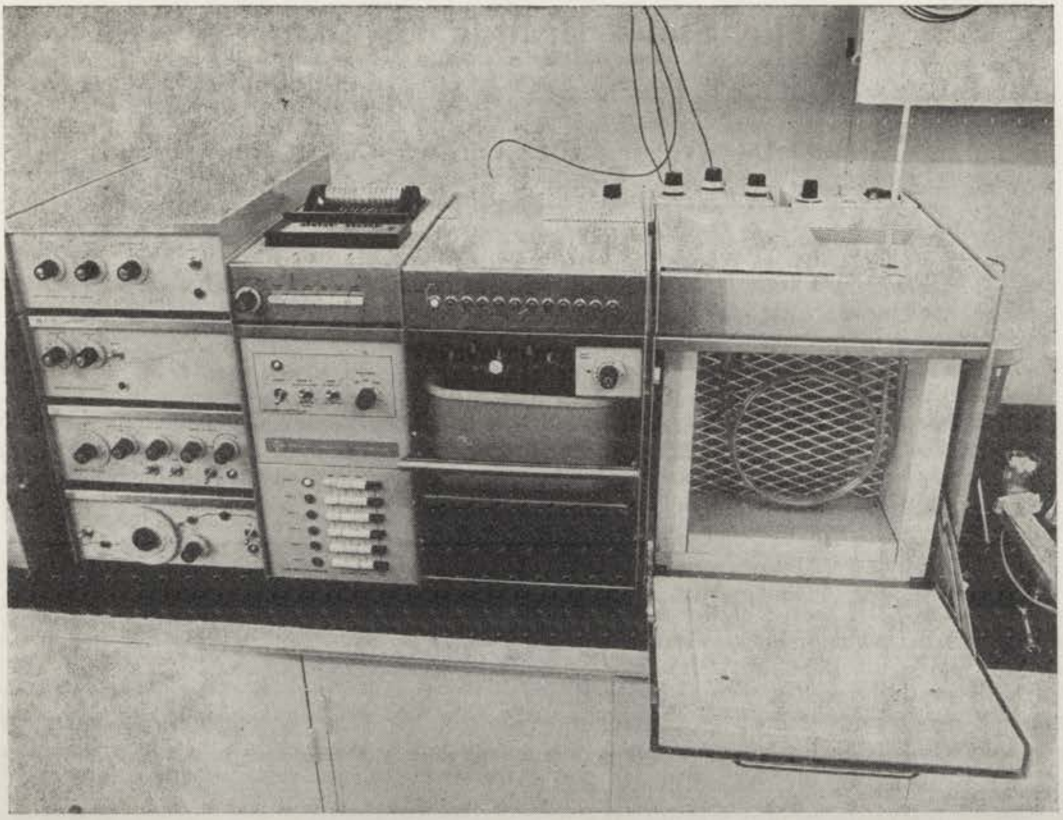


Plate IV.—The gas chromatograph (Photo: D.I.E.S)

The Fishes of New Guinea

BY IAN S. R. MUNRO

This book contains the most up-to-date information on local fishes yet published. It contains over 700 pages with 6 colour plates, 78 plates in black and white and 23 figures in text, and is published by D.A.S.F. Copies of *The Fishes of New Guinea* are available from the Government Printer, P.O. Box 3280, Port Moresby. The price is $14.50 retail plus 50 cents postage within Papua New Guinea, surface mail. Postage to Australia is 75 cents.