

least 24 hours. In the preserves tested the pigment concentration in the fruit was the same as in the surrounding jelly.

Some examples of the application of the method are shown in Table II. The total pigment contents of the four varieties of strawberries (measured in frozen samples stored at -18°C . for 6 months) were rather similar. Strawberry preserve sample 1 was unusually high in pigment content, the color intensity being about what one would expect from the results on frozen fruit, after allowing for the effects of dilution by other components of the preserve and for some color loss during manufacture. Preserve 3 was obtained from the same manufacturer as No. 1, and showed only about 27% as much red anthocyanin as No. 1.

The color difference was obvious upon visual inspection of the preserves. Sample 2 was purchased in March on the open market. Preserve 3 when stored at 15° and 38°C . showed rapid loss of pigment, especially at the higher temperature.

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Determination of Safrole in the Oil of *Ocotea cymbarum*

A Cryoscopic Method

ANTHONY J. SHUKIS AND HERMAN WACHS
Dodge & Olcott, Inc., Bayonne, N. J.

A cryoscopic method employing congealing temperatures has been developed for the determination of safrole in the oil of *Ocotea cymbarum* of commerce. A graph is included, by means of which the safrole content of the oil as determined by the mercuric chloride method, is correlated with the congealing temperature.

SAFROLE is of considerable technical importance as a flavoring material and as a raw material for the preparation of piperonal, and recently it has come into use as a starting material for the synthesis of insecticidally active materials (7). Prior to the recent war, Japanese camphor oil was the only available material from which safrole could be obtained in commercial quantities. It was a fortunate coincidence that a new source was made available in Brazil by the steam distillation of the oil of *Ocotea cymbarum* (2).

Since safrole is the only valuable constituent of the oil of *Ocotea cymbarum*, a simple and quick method for its determination was desired. A cryoscopic method appeared to be most suitable. However, for this purpose it was necessary to obtain correlation of physical constants, congealing temperature, and concentration. To determine the concentration of safrole in samples of the oil of *Ocotea cymbarum* the addition compound of safrole and mercuric chloride (1, 5) yielding hydroxychloromercuri dihydrosafrole was used. The methods described in the literature (3, 4) were modified as described below, to minimize the errors due to transfer and solubility of the hydroxychloromercuri dihydrosafrole in water, and thereby give more readily reproducible results. The method outlined by Huzita and Nakahara (3) was modified as follows:

The reaction is carried out in one vessel to avoid a troublesome transfer. The reaction is allowed to proceed in a homogeneous solution for 30 minutes, using mercuric acetate solution. Under the conditions of homogeneity the reaction readily proceeds to completion. Conversion to the chloride is then effected. Allowance is made for the solubility of hydroxychloromercuri dihydrosafrole in water at 0°C . by determining the solubility of the material at that temperature and applying a correction factor. By these means the error in general is reduced

from ± 1.5 to $\pm 0.7\%$ and readily reproducible results are obtained.

Safrole "drainings" were prepared by freezing a sample of the oil of *Ocotea cymbarum*. The congealed fraction was used as a source of safrole, which was subjected to further purification. The noncongealed fraction was taken as safrole drainings. Samples of safrole drainings, oil of *Ocotea cymbarum*, and safrole, the latter purified by freezing, were doubly distilled and center sections were taken as standards for analysis. Congealing points on each were taken, and repeated if necessary until checks within 0.1°C . were obtained. Each sample was then analyzed by the mercuric chloride method. Summarized in Table I are these data, with additional data taken to round out the information.

Table I. Properties of Analytical Standards

Sample	Congealing Point, $^{\circ}\text{C}$.	Safrole Content Weight %, Mercury Analysis	n_D^{20}	Specific Gravity, 25°C .
Safrole drainings	2.4	69.1 { 69.2 69.0	1.5299	1.0541
Oil of <i>Ocotea cymbarum</i>	8.8	91.9 { 91.7 92.1	1.5352	1.0843
Safrole	11.0	99.5 { 99.4 99.7	1.5382	1.0987

A plot of weight per cent of safrole versus the congealing points was prepared; the points in Table I fell on a straight line. To ensure conformity and check the linearity of the plot, samples differing in safrole content by approximately 1 weight %, in the range 85 to 95%, were prepared from mixtures of the safrole drainings and safrole mentioned above and their congealing points were determined. When plotted, these points fell on the straight line plotted using the standards. All the values fell within