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ASSAY OF LINIMENT OF CAMPHOR.*

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Various methods have been suggested for the determining of camphor in Liniment of Camphor.

Mann (2) suggested counterbalancing two pairs of filter papers, pouring 0.4 to 0.5 Gm. of Liniment of Camphor on one pair and an identical weight of olive oil on the other pair. These two sets of papers were then to be exposed to the temperature of a hot air bath for about twenty minutes and again weighed to determine the amount of camphor present. Cook (3) found that heating at 100° C. for three hours drove off all but about one-half per cent of the camphor and, therefore, suggested adding about one-half per cent as a correction factor.

Cowie and Dickson (4) ascertained that 20 Gm. of camphor in 80 cc. of olive oil measured 100 cc. and that, therefore, the volume and weight of camphor in the finished liniment were identical. They proposed to take the specific gravity of the liniment at 15.56° C., place the weight equivalent to 10 cc. in a beaker, weigh 8 cc. of olive oil in a similar beaker and heat in a sand bath at 150° C. for thirty to forty minutes, making a correction for the loss in the oil.

Lothian (5) recommended placing the liniment in a shallow dish, such as the cover of a petri dish, to a depth of about one-half millimeter. The dish was then supported on a copper ring and leveled on the water-bath to obtain a uniform layer. This was heated one hour and weighed. He found that further heating would cause a gain in weight of the olive oil while the oil, itself, under these conditions did not gain. This indicated that the oil was affected by the camphor and, therefore, he placed no reliance on correction figures.

Wallace and Plummer (9) found that cottonseed oil oxidizes faster when heated with camphor than when heated alone as determined by the refractive index, the saponification value and the iodine value before and after heating. They found it was necessary to heat cottonseed oil at 120° C. for five hours to completely remove the camphor.

Kebler and collaborators (13) prepared a standard 20% solution of camphor in cottonseed oil and found the optical rotation to be plus 58.5 on the sugar scale in a 200 mm. tube. In a series of determinations heating to 150° C. to practically constant weight gave fairly concordant results with the polarimetric determinations. They stated that heating may give slightly high results, due to the presence of moisture.

Miller (14) says best results are to be obtained in a flat bottomed platinum dish at 110° C. for 90 minutes in a well ventilated oven.

Poe, Lipsey and Vaughn (11) made a study of the U. S. P. method for determination of camphor in Liniment of Camphor and found that the method gave consistently low results, due to the oxidation of the olein in cottonseed oil. Various kinds of dishes were used, but none proved satisfactory.

Dowzard (1) made a series of gravimetric assays on Liniment of Camphor and determined the optical rotation of the samples in angular degrees in a 100-mm. tube at 15° C. He then divided the rotation by the per cent of camphor found gravimetrically to obtain a factor whereby

* Section on Practical Pharmacy and Dispensing, A. P. H. A., Portland meeting, 1935.

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the observed rotation could be converted into per cent camphor. For the seven assays listed, the average factor was found to be 1.962.

Cook (3) pointed out that a polarimetric method becomes complicated by the fact that a solution of camphor in olive oil shows a different specific rotation (plus 50.63°) from that exhibited by an alcohol solution (plus 43.52°) and that a correction must, therefore, be made.

Hund (6) determined that cottonseed oil showed no optical rotation and on accurately prepared 10% and 20% solutions of camphor in cottonseed oil found that at 13° C. one per cent camphor showed an average rotation of 0.493° in a 100-mm. tube. Per cent by weight is, therefore, found by dividing the reading in a 100 mm. tube by 0.493. This gave fairly concordant results with the gravimetric method. Malosse (7) says that the specific rotatory power of camphor in olive oil increases with the dilution.

Von Friedrichs (8) studied the polarimetry of camphorated oil with reference to the oil used as a solvent. For liniment prepared with olive oil he derived the factor: Per cent camphor = $1.958 \times \frac{\text{Rotation observed}}{\text{Length of tube in decimeters}}$. Factors are also given for liniments prepared with other oils. He found that ordinary deviations of temperature were almost negligible.

David (10) suggested that 4.0 Gm. of camphorated oil be placed in a flask, together with 5 Gm. of calcium oxide and 100 to 120 Gm. of water, and the mixture distilled until 60 cc. of the distillate has been collected in a graduated cylinder. Twenty Gm. of sodium chloride were added to the distillate and the condenser washed with ether, collecting the ether in the same cylinder. Ether was added to about 50 or 60 cc. and the mixture thoroughly shaken. An aliquot of the ether was then poured into a tared flask, weighed and evaporated on the water-bath, and the residual camphor weighed. From this, the per cent of camphor in the original sample was calculated. Lajos (12) reports a method identical with this.

In connection with current work on U. S. P. revision, an assay method has been proposed (15) in which the liniment is dried in an oven maintained at 110° C. for two hours under a stream of carbon dioxide.

Work was undertaken to obtain comparative results by polarimetric and gravimetric methods. Doward's factor was used in the polarimetric work. The carbon dioxide method was followed as outlined in the "Bulletin of the Sub-Committee on Organic Chemicals." A third determination was made in an electric oven at 110° C. without carbon dioxide protection, controls being run on the oil of cottonseed used for the liniment.

Sample No.	Polarimetric.	Per Cent Camphor in Sample.	
		Under CO ₂ at 110° C.	Exposed to Air at 110° C. ¹
Sample No. 1	18.44%	18.63%	18.25%
Sample No. 2	19.19%	19.52%	19.02%
Sample No. 3	19.66%	19.68%*	19.41%

* The actual figure was 20.74 but correction of 1.06 was necessary due to loss when the oil, itself, was dried.

¹ The results given for the assays exposed to air at 110° C. are corrected for increase or decrease in weight of the oil used.

Twenty-eight samples of cottonseed oil were tested for volatile matter at 110° C. under carbon dioxide for two hours; only two samples showed any significant change. The results were as follows:

1 0.01 % increase	8 0.04 % increase	15 0.03 % increase	22 0.002% increase
2 0.01 % loss	9 0.04 % increase	16 0.004% increase	23 0.08 % increase
3 0.81 % loss	10 0.05 % increase	17 0.02 % increase	24 0.085% increase
4 1.79 % loss	11 0.005% increase	18 0.015% increase	25 0.039% increase
5 0.02 % increase	12 0.04 % increase	19 0.02 % increase	26 0.068% increase
6 0.01 % increase	13 0.07 % increase	20 0.06 % increase	27 0.066% increase
7 No loss or gain	14 0.007% increase	21 0.09 % loss	28 0.065% increase

A known solution of camphor in cottonseed oil was made up in a glass-stoppered, tared Erlenmeyer flask; 103.8016 Gm. of cottonseed oil were weighed into the flask and 25.9704 Gm. of camphor were added to make 129.8520 Gm. of exactly 20% Liniment of Camphor. The flask was tightly stoppered immediately after the addition of the camphor. The mixture was warmed gently (not over 40° C.) and shaken until the camphor was completely dissolved. The weight of the flask and contents was then checked to determine possible loss during solution; a loss of 0.0123 Gm. was found, but as this represented only 0.01% of camphor it was disregarded. Optical rotation of this liniment was then determined in a 50-mm. tube at 25° C. and found to be plus 5.07. The oil used in the preparation of the liniment had a rotation of minus 0.03 in a 50-mm. tube at 25° C. Using Dowzard's factor this gave 19.91% camphor based on the rotation of the liniment. When corrected for the rotation of the oil, the result was found to be 20.01% camphor. By the carbon dioxide method at 110° C. the prepared liniment showed 20.02% camphor, but a blank on the oil showed a loss of 0.13% making a net result of 19.89% camphor. Another portion heated in a glass evaporating dish at 110° C. till no odor of camphor was discernible showed 19.30% camphor, but a blank on the oil corrected this to 19.92% camphor.

	Polarimetric.	CO ₂ Method.	U. S. P. X Method.
Liniment	19.91% camphor	20.02% camphor	19.30% camphor
Correction for Oil	plus 0.10%	minus 0.13%	plus 0.62%
Net	20.01% camphor	19.89% camphor	19.92% camphor

It is apparently necessary to run a blank on the particular oil used in any given Liniment of Camphor if an accurate assay is to be obtained.

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"APOTHECARY SHOPS OF COLONIAL TIMES."*

A compilation of scattered historical data published in pharmaceutical literature and other sources of the past few years and describing apothecary shops, proprietors and distinguished customers of colonial days. The oldest American apothecary shop still in existence and doing business is the Rau Pharmacy in Bethlehem, Pa. The oldest record of an apothecary shop in America (1646) is that of Wm. Davies of Boston, Mass. This was probably the first store devoted exclusively to Pharmacy in America.

* Abstract of a paper before Section on Historical Pharmacy, A. Ph. A., Portland meeting 1935—by Millicent R. LaWall.