STUDIES ON THE DETERMINATION OF CAMPHOR IN CAMPHOR LINIMENT. V. IN AN ATMOSPHERE OF NITROGEN.*

BY CHARLES F. POE.[1]

Several previous communications (1) from this laboratory have shown that the U. S. P. X method for the determination of camphor in camphor liniment was not satisfactory because of the oxidation of the cottonseed oil during the heating process. Two methods which largely overcome this defect have been suggested (2): The first in which antioxidants are added before the heating; the second in which the vacuum oven is used. Of the two the vacuum oven is the more satisfactory, but unfortunately this oven is not included in the equipment of smaller laboratories.

The present communication reports the investigation of a method which makes use of the ordinary oven in which the air is replaced by nitrogen.

An air-tight oven having an inlet and outlet tube was used. A large amount of nitrogen was run through the oven at first so as to displace all the air, and then a very small amount of nitrogen was allowed to pass through during the determination. Five grams of the liniment were weighed in tin dishes. Tin dishes were used because they are deeper than the porcelain dishes which were available, and no loss was suffered by the oil creeping up the sides.

The effect of different temperatures on the heating of a sample of camphor liniment was tried. Determinations were made at 100°, 110° and 120° C. As noted in the investigation with the vacuum-oven method (2), a temperature above 120° C. was found to decompose the oil. A temperature of 110° was satisfactory and was used throughout the experiments.

A series of determinations were made by heating 5-Gm. amounts of different samples of oil in the air oven at 110° C., both with and without an atmosphere of nitrogen. The results of these determinations are listed in Table I. In an atmosphere of nitrogen, the average change in weight at the end of six hours was 0.02 per cent as compared to an average gain of 1.00 per cent when corresponding samples were heated in an air oven.

TABLE I.—CHANGE IN WEIGHT OF OILS HEATED IN ATMOSPHERES OF AIR AND NITROGEN. TEMPERATURE 110° C.

No. of Oil.	In Air. 2 Hours.	4 Hours.	6 Hours.	In Nitrogen. 2 Hours.	4 Hours.	6 Hours.
1	+0.59[a]	+0.78	+1.07	+0.04	+0.05	+0.06
2	+0.41	+0.53	+0.94	+0.07	+0.08	+0.07
3	+0.44	+0.84	+1.22	+0.09	+0.11	+0.14
4	+0.31	+0.47	+1.02	+0.04	−0.04	−0.09
5	+0.37	+0.53	+1.08	+0.10	+0.11	+0.13
6	+0.41	+0.59	+0.82	−0.08	−0.10	−0.16
7	+0.52	+0.67	+0.89	−0.06	−0.09	−0.13
8	+0.46	+0.61	+0.96	+0.08	+0.08	+0.10
Average	+0.44	+0.63	+1.00	+0.04	+0.03	+0.02

[a] Percentage gain or loss in weight.

Samples of camphor liniment, made from different lots of oil and camphor, were analyzed separately in an atmosphere of air and nitrogen, and values obtained are listed in Table II. The results when nitrogen was used were very near the theoretical values and showed better agreement than the results obtained when samples of the same liniments were heated in the air oven. The average value obtained with 20 per cent liniments for the six-hour period in an atmosphere of nitrogen was 19.89 per cent; whereas the average result in the air-oven was 19.06 per cent.

* Presented before the Scientific Section, A. PH. A., Miami meeting, 1931.

[1] University of Colorado, Boulder, Colorado.

TABLE II.—COMPARISON OF THE DETERMINATION OF CAMPHOR IN ATMOSPHERES OF AIR AND NITROGEN. 20 PER CENT LINIMENT. TEMPERATURE 110° C.

No. of Oil.	In Air. 2 Hours.	4 Hours.	6 Hours.	In Nitrogen. 2 Hours.	4 Hours.	6 Hours.
1	19.18[b]	19.36	19.08	18.16	19.78	19.92
2	19.32	19.46	18.96	18.50	19.70	19.83
3	19.10	19.46	19.07	18.62	19.88	19.90
4	18.92	19.29	19.02	18.88	19.88	19.94
5	18.66	19.36	19.07	18.56	19.75	19.88
6	19.16	19.35	19.14	18.60	19.90	19.94
7	19.34	19.28	19.04	17.92	19.82	19.84
8	19.62	19.36	19.13	17.62	19.90	19.90
Average	19.16	19.37	19.06	18.36	19.83	19.89

[b] Percentage camphor found.

In order to confirm the work given above, new samples were prepared from different samples of oils and camphor. The results which were similar to those already given are set forth in Tables III and IV.

TABLE III.—CHANGE IN WEIGHT OF OILS HEATED IN ATMOSPHERES OF AIR AND NITROGEN. TEMPERATURE 110° C.

No. of Oil.	In Air. 2 Hours.	4 Hours.	6 Hours.	In Nitrogen. 2 Hours.	4 Hours.	6 Hours.
9	+0.29[a]	+0.71	+1.09	+0.06	+0.08	+0.08
10	+0.32	+0.60	+0.88	+0.04	+0.07	+0.09
11	+0.28	+0.40	+0.63	−0.01	−0.03	−0.01
12	+0.27	+0.48	+0.68	−0.01	−0.05	−0.04
13	+0.32	+0.80	+1.03	+0.05	+0.07	+0.09
Average	+0.30	+0.60	+0.86	+0.03	+0.03	+0.04

[a] Percentage gain or loss in weight.

TABLE IV.—COMPARISON OF THE DETERMINATION OF CAMPHOR IN ATMOSPHERES OF AIR AND NITROGEN. 20 PER CENT LINIMENT. TEMPERATURE 110° C.

No. of Oil.	In Air. 2 Hours.	4 Hours.	6 Hours.	In Nitrogen. 2 Hours.	4 Hours.	6 Hours.
9	19.06[b]	19.42	18.88	18.96	19.64	19.83
10	18.19	19.40	18.75	16.69	19.68	19.82
11	18.72	19.65	19.34	19.26	19.85	19.95
12	18.04	19.50	19.20	18.68	19.80	19.88
13	18.69	19.33	19.06	18.97	19.74	19.86
Average	18.54	19.46	19.05	18.51	19.74	19.87

[b] Percentage camphor found.

Because nitrogen is somewhat expensive, the author decided to run a similar set of experiments with carbon dioxide in lieu of nitrogen. The results are presented in Tables V and VI. The U. S. P. XI directs the use of carbon dioxide (3, 4).

TABLE V.—CHANGE IN WEIGHT OF OILS HEATED IN AN ATMOSPHERE OF CARBON DIOXIDE. TEMPERATURE 110° C.

No. of Oil.	2 Hours.	4 Hours.	6 Hours.
9	+0.06[a]	+0.07	+0.11
10	+0.04	+0.06	+0.09
11	−0.04	−0.02	+0.03
12	+0.06	+0.04	+0.07
13	+0.01	+0.13	+0.16
Average	+0.03	+0.06	+0.09

[a] Percentage gain or loss in weight.

TABLE VI.—DETERMINATION OF CAMPHOR IN ATMOSPHERE OF CARBON DIOXIDE. 20 PER CENT LINIMENT. TEMPERATURE 110° C.

No. of Oil.	2 Hours.	4 Hours.	6 Hours.
9	18.76[b]	19.59	19.72
10	19.48	19.72	19.82
11	19.09	19.65	19.89
12	19.17	19.53	19.86
13	19.43	19.63	19.83
Average	19.19	19.62	19.82

[b] Percentage camphor found.

The results obtained in an atmosphere of carbon dioxide are very similar to those obtained with nitrogen, except that the results with nitrogen are somewhat closer to the theoretical percentage of camphor. Also the average gain in weight for the oils alone is somewhat lower with the nitrogen than with the carbon dioxide.

SUMMARY.

1. Experimental results are presented which show that camphor in camphor liniment may be accurately determined by heating in an atmosphere of nitrogen.

2. The substitution of an atmosphere of carbon dioxide for nitrogen gives results which approximate the accuracy of those obtained with nitrogen.

3. The results obtained with an atmosphere of nitrogen or carbon dioxide are not quite as near the theoretical values as those obtained when the vacuum oven is used.

REFERENCES.

(1) Poe, Lipsey and Vaughan, JOUR. A. PH. A., 18, 580 (1929); 20, 1175 (1931).
(2) Poe, *Ibid.*, 21, 337 (1932); 25, 279 (1936).
(3) Grantham, R. I., "Proc. Amer. Drug. Mfrs. Assoc." (1932), page 221.
(4) Overbye, D. A., and Schoetzow, R. E., JOUR. A. PH. A., 24, 961 (1935).

The 85th Annual Meeting of the AMERICAN PHARMACEUTICAL ASSOCIATION will be held in New York City.