

## SCIENTIFIC SECTION

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### STUDIES ON THE DETERMINATION OF CAMPHOR IN CAMPHOR LINIMENT. IV. THE USE OF ANTIOXIDANTS.\*

BY CHARLES F. POE.<sup>1</sup>

#### INTRODUCTION.

A number of methods have been studied and reported by the author with others (1, 2, 3) for the determination of camphor in camphor liniment. One of the objections to the method given in the U. S. P. X is that the cottonseed oil is oxidized during the heating process, and the increase in weight thereby causes considerable error in the determination. It was thought that this defect might be overcome by means of the use of certain antioxidants. The use of organic chemicals as antioxidants was first proposed by Moureu and Dufraisse (4) and has been studied and reported by these authors in a series of publications.

#### EXPERIMENTAL.

The determination was carried out according to the U. S. P. X method, except that different amounts of antioxidants were added to the weighed samples before heating. Tin dishes were used throughout the investigations.

In order to study the effect of these substances on the cottonseed oil alone, a number of samples of oils were selected. Five-gram samples were weighed out, and different antioxidants were added in varying amounts. The oils with added chemicals were heated in a constant temperature air oven for different periods of time. Similar determinations were made on samples of oil without the antioxidants. The results are given in Table I.

In comparing the gain in weight of the samples having the antioxidants added with those of the oils heated alone, it will be noted that the antioxidants were effective when added in certain amounts. With pyrogallol and *alpha* naphthol, 1 part in 500 or 1 part in 1000 seems to cause the oil to show practically no loss or gain in weight. With hydroquinone, 1 part in 1000 was more efficient, whereas with para-phenylenediamine the optimum amount was 1 part in 500.

The results in Table I indicate that very small amounts of the antioxidants are effective in preventing oxidation in cotton seed oil. Therefore, small amounts of several organic chemicals were used in a series of determinations of camphor in camphor liniment. The samples of liniment were made from different lots of oil. The results are recorded in Table II.

The results listed in Table II indicate that the use of antioxidants offers some promise for the determination of camphor in camphor liniment. Pyrogallol, *alpha* naphthol and hydroquinone seem to be effective in 1 part in 250, whereas para-phenylenediamine was not effective in the experimental amounts.

The procedure of the method may be summarized as follows: Place about 5 Gm. of camphor liniment in a tared dish having a diameter of at least 70 mm., weigh accurately, add either 20 mg. of pyrogallol, *alpha* naphthol or hydroquinone. The sample is heated for 4 hours at 110° C. in a constant temperature air oven, cooled in a desiccator, and weighed. The percentage of camphor is calculated from the loss in weight. Porcelain or tin dishes may be used, but aluminum dishes should not be used (1).

While the results were fairly satisfactory with some of the antioxidants, it seems to the author that the vacuum oven method (3) gives more satisfactory results.

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<sup>1</sup> University of Colorado, Boulder, Colo.

TABLE I.—CHANGE OF WEIGHT OF OILS WHEN HEATED WITH ANTIOXIDANTS OF 110° C.

Antioxidant Parts.	Sample Number of Oil Used.												Average. 4 Hrs.			
	7.		8.		9.		12.		6 Hrs.		2 Hrs.					
	2 Hrs.	4 Hrs.	6 Hrs.	2 Hrs.	4 Hrs.	6 Hrs.	2 Hrs.	4 Hrs.	6 Hrs.	2 Hrs.	4 Hrs.	6 Hrs.	2 Hrs.	4 Hrs.	6 Hrs.	
Pyrogallol.																
1:50	-0.30 <sup>a</sup>	-0.52	-0.58	-0.22	-0.34	-0.45	-0.38	-0.46	-0.50	-0.45	-0.59	-0.33	-0.34	-0.48	-0.46	
1:100	-0.22	-0.26	-0.26	-0.14	-0.14	-0.12	-0.08	-0.26	-0.20	-0.20	-0.26	-0.26	-0.16	-0.23	-0.21	
1:500	-0.10	-0.18	-0.10	-0.03	+0.01	+0.11	-0.02	-0.06	-0.04	-0.04	+0.10	+0.10	-0.05	-0.06	+0.02	
1:1000	0.00	-0.02	0.00	-0.02	+0.01	+0.02	+0.08	+0.10	+0.14	-0.03	+0.07	+0.21	+0.01	+0.04	+0.09	
Alpha Naphthol.																
1:50	-0.10	-0.32	-0.28	-0.27	-0.33	-0.39	-0.50	-0.62	-0.86	-0.52	-0.64	-0.71	-0.35	-0.48	-0.56	
1:100	-0.08	-0.22	-0.18	-0.09	-0.14	-0.14	+0.01	-0.10	-0.14	-0.30	-0.36	-0.40	-0.12	-0.21	-0.22	
1:500	0.00	-0.02	+0.02	+0.02	+0.08	+0.14	+0.08	+0.09	+0.14	-0.01	+0.04	+0.10	+0.02	+0.05	+0.10	
1:1000	0.00	0.00	+0.05	0.00	+0.04	+0.18	0.00	+0.02	+0.06	-0.04	-0.06	+0.10	-0.01	0.00	+0.10	
Hydroquinone.																
1:50	-0.51	-0.73	-1.19	-0.48	-0.94	-0.57	-0.76	-0.82	-1.28	-0.74	-0.92	-1.46	-0.62	-0.85	-1.38	
1:100	-0.35	-0.63	-0.43	-0.27	-0.48	-0.66	-0.26	-0.54	-0.84	-0.62	-0.83	-1.04	-0.38	-0.62	-0.74	
1:500	-0.02	-0.08	-0.05	-0.12	-0.14	-0.09	-0.16	-0.28	-0.32	-0.20	-0.16	-0.00	-0.13	-0.17	-0.12	
1:1000	-0.08	-0.14	-0.10	-0.03	-0.06	-0.10	-0.08	-0.08	-0.08	-0.08	+0.08	-0.04	-0.07	-0.09	-0.08	
Para-phenylene Diamine.																
1:50	-1.22	-1.28	-1.42	-1.00	-1.08	-1.26	-1.36	-1.48	-1.55	-1.36	-1.36	-1.36	-1.24	-1.30	-1.40	
1:100	-0.50	-0.62	-0.70	-0.46	-0.50	-0.50	-0.74	-0.60	-0.50	-0.52	-0.54	-0.52	-0.56	-0.57	-0.56	
1:500	+0.04	+0.14	+0.12	0.00	+0.04	+0.10	-0.04	+0.08	+0.20	+0.02	+0.16	+0.26	+0.01	+0.11	+0.17	
1:1000	0.00	+0.05	+0.36	-0.02	+0.08	+0.24	+0.05	+0.30	+0.50	+0.10	+0.26	+0.36	+0.03	+0.17	+0.37	
Oil alone	+0.44	+0.60	+0.90	+0.16	+0.38	+0.80	+0.36	+0.47	+1.08	+0.52	+0.68	+1.00	+0.37	+0.53	+0.95	

<sup>a</sup> All results expressed in per cent.

TABLE II.—DETERMINATION OF CAMPHOR BY USE OF ANTIOXIDANTS.

Antioxidant Parts.	20 Per cent Liniments Used. 110° C.									Average.		
	Sample No. 7.			Sample No. 8.			Sample No. 12.			2 Hrs.	4 Hrs.	6 Hrs.
Pyrogallol.												
1:250	19.12 <sup>a</sup>	19.88	19.92	19.50	19.87	19.73	19.42	19.94	19.90	19.35	19.90	19.85
1:500	18.98	19.92	19.94	18.48	19.70	19.60	19.00	19.80	19.56	18.22	19.81	19.70
Alpha Naphthol.												
1:250	18.30	19.88	19.92	18.54	19.84	19.80	18.75	19.86	19.70	18.53	19.86	19.81
1:500	18.82	19.84	19.90	18.44	19.80	19.65	18.80	19.72	19.52	18.69	19.79	19.70
Hydroquinone.												
1:250	19.60	19.90	19.94	19.60	19.96	20.00	19.00	19.94	19.80	19.40	19.93	19.91
1:500	18.10	20.04	19.99	19.40	19.82	19.50	19.08	19.84	19.54	18.86	19.90	19.68
Para-phenylene Diamine.												
1:250	17.94	19.74	19.68	18.34	19.14	19.08	18.60	19.80	19.67	18.29	19.56	19.48
1:500	19.04	19.95	19.70	18.22	19.28	19.26	18.34	19.56	19.53	18.53	19.60	19.50
No antioxidant	19.50	19.40	19.18	18.60	19.26	19.14	19.62	19.36	19.13	19.24	19.34	19.15

<sup>a</sup> All results expressed in per cent.

#### CONCLUSION.

1. Certain antioxidants have been investigated for use in the determination of camphor in camphor liniment.
2. Several antioxidants seem to show promise as a means of preventing the oxidation of the cottonseed oil used as a base for camphor liniment.
3. The results obtained with the use of the antioxidants seem to be more satisfactory than the results obtained by means of the U. S. P. X method, but less satisfactory than with the vacuum oven method.

#### REFERENCES.

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### THE ASSAY OF ORGANIC MEDICINAL PREPARATIONS CONTAINING ARSENIC.\*

BY EDWARD J. HUGHES.

During the past thirty years no less than a dozen distinctly different methods have been proposed for the estimation of arsenic in organic medicinals. Yet, up to the present time, no general method having the desirable qualities of accuracy, practical simplicity and rapidity has apparently been evolved.

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