ANALYSIS OF EMULSIONS OF COD LIVER OIL AND MALT EXTRACT.

BY C. S. WAGGONER AND C. C. GLOVER.

The increasing popularity of cod liver oil and malt mixtures as tonics has lead to considerable rivalry as to the relative merits of various commercial brands. All reported attempts at an analysis of these mixtures have been thus far rather inaccurate and unreliable.

The main difficulties encountered are, the fact that the oil gains weight on heating hence offers difficulty in securing constant weight; the selection of a suitable solvent for extraction and lastly, the breaking of the emulsion. Experiments on a commercial product of cod liver oil, showed an increase of about 10 per cent by heating to constant weight. Among the solvents tried were ether, petroleum ether (30–60° boiling), carbon disulphide, chloroform and ethyl acetate. It was found that anhydrous ether and low boiling petroleum ether worked best for the method developed. Alcohol was found to be best for breaking emulsions.

The procedure first tried is as follows:—To a two or three gram sample, add 250 to 300 cc. of water and stir until a homogeneous mixture results. Place in a separatory funnel, add 75 cc. of ether, stopper tightly and shake for five minutes. Let stand until the ether separates from the water and forms a layer at the top. Draw off the aqueous layer into a clean beaker and the ethereal solution into another. Place aqueous portion back in separatory funnel and repeat the above procedure four or five times collecting the ethereal solutions in a common container. This solution is then evaporated on a steam-bath until no odor of ether remains. The residue is then extracted with ether to remove the oil from any water or other impurities that may be present. It may be necessary to extract more than once with ether with intervening evaporations in order to obtain the oil pure. The oil is then dried to constant weight in a desiccator over sulphuric acid, weighed and the per cent of oil calculated.

By this method it was only possible to recover within about two per cent of the correct amount in known samples containing from 13.04 per cent to 25 per cent of oil. This method was then discarded and a new one developed.

The procedure for the second method is as follows; use four to six grams of sample, add 15 cc. of water and stir until homogenous mixture results, add 50 cc. of ethyl alcohol, place in a 250-cc. separatory funnel and shake until emulsion breaks, add 50 cc. of low-boiling petroleum ether and shake for a few minutes, draw off the hydro-alcoholic solution in a clean beaker and the ether solution in another. Place the hydro-alcoholic solution back into the separatory funnel and repeat the above procedure four or five times, each time placing the ether solution in a common container. Evaporate slowly on a steam-bath until the odor of petroleum ether has disappeared. Dry the oil in a desiccator over sulphuric acid to constant weight. Weigh and calculate per cent of oil present.

Some of the results of the above method on known samples are as follows:

Known per cent.	Wt. of sample.	Wt. of oil recovered.	Per cent by wt. of oil recovered.
13.04	5.0906 Gm.	0.6536 Gm.	12.84
13.04	5.5921	0.7130	12.75
13.04	4.6700	0.6033	12.92
		Average	12.83%

20.00	6.1457	1.1944		19.43
20.00	4.2126	0.8197		19.45
20.00	5.2736	1.0372		19.66
			Average	19.51%
25.00	5.3531	1.3177		24.43
25.00	4.6211	1.1247		24.34
25.00	5.6343	1.3680		24.28
			Average	24.35%

This method was then tried upon three commercial samples with the following results:

Product.	Wt. of sample.	Wt. of oil recovered.	Per cent of oil by wt.
A.	5.3610	0.9674	18.04
A.	6.0795	1.1107	18.26
			Average 18.15%
B.	4.4574	0.9270	20.79
B.	5.0787	1.0352	20.38
			Average 20.58%
C.	5.7873	1.2967	22.40
C.	5.5490	1.2274	22.12
			Average 22.26%

Since it is common practice for manufacturers to express the percentage composition of cod liver oil and malt mixtures by volume rather than by weight, attempts were made to convert the above results into percentage by volume. Great difficulty was experienced in accurately determining the specific gravity of the original samples due to the viscosity of the samples, the variability of the malt extracts and the amounts of air bubbles entrapped in the mixtures. No concordant results of per cent by volume have therefore been obtainable.

In summarizing the work here presented one may see that great care must be taken in the amount of heat applied to the oil, the emulsion should first be broken with ethyl alcohol and that the best solvent for the oil is low-boiling petroleum ether. By inspection of the column of the percentage of oil recovered from known samples it may be seen that the error increases with the percentage of oil present in the sample. Commercial products on analysis show about twenty per cent by weight of oil. To the results obtained on the twenty per cent known sample, one might add about 0.5 per cent to the analysis and obtain a very close approximation to the amount of oil actually present.

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FOOD & DRUG LABORATORY,
UNIVERSITY OF MICHIGAN,
COLLEGE OF PHARMACY,
ANN ARBOR.