

## RESEARCH PAPER RP1067

Part of *Journal of Research of the National Bureau of Standards*, Volume 20,  
January 1938

# A LABORATORY EXTRACTION APPARATUS AND ITS USE IN SEPARATING A LUBRICATING-OIL FRACTION WITH ACETIC ACID<sup>1</sup>

By Sylvester T. Schick Tanz<sup>2</sup>

## ABSTRACT

There is described a glass laboratory extraction apparatus, operating with reflux, for use with solvents heavier than the liquids to be extracted. Data are given on the extraction, with acetic acid, of a narrow-boiling fraction of "water-white" lubricating oil.

## CONTENTS

	Page
I. Introduction.....	83
II. Apparatus and experimental procedure.....	85
III. Results.....	85

## I. INTRODUCTION

Solvent extraction is often useful for the separation of complex liquid mixtures when other methods of fractionation are not effective. Saal and van Dyck<sup>3</sup> pointed out the similarity between solvent extraction and distillation, the solvent phase in extraction being analogous to the vapor phase in distillation, and emphasized the importance of the reflux ratio in extraction. Similar calculations on the theory of solvent extraction with reflux were made by Fenske and his coworkers,<sup>4,5,6</sup> who applied the principles in actual practice and effectively separated narrow-boiling cuts of lubricating oils by this method.

The present paper describes a glass laboratory extracting column, operating with reflux, for use with solvents denser than the liquid being extracted. This is a modification of the larger columns developed by Fenske and his coworkers.<sup>7</sup> Data are also given on the use of this column in extracting a narrow-boiling fraction of "water-white" lubricating oil with acetic acid.<sup>8</sup>

<sup>1</sup> Financial assistance has been received from the research fund of the American Petroleum Institute. This work is part of Project 6, The Separation, Identification, and Determination of the Constituents of Petroleum.

<sup>2</sup> Formerly research associate at the National Bureau of Standards, representing the American Petroleum Institute, now with the Alcohol Tax Unit, Bureau of Internal Revenue, United States Treasury Department.

<sup>3</sup> Saal and van Dyck. *Proc. World Petroleum Congr. (London)* **2**, 352 (1933).

<sup>4</sup> M. R. Cannon. Thesis, Pennsylvania State College (1935).

<sup>5</sup> M. R. Cannon and M. R. Fenske. *Ind. Eng. Chem.* **28**, 1935 (1936).

<sup>6</sup> M. R. Fenske and W. B. McCluer. U. S. Patents 2037318 and 2037319

<sup>7</sup> See footnote 5.

<sup>8</sup> For the description of a 14-m column for use with solvents less dense than the material to be extracted, see B. J. Mair and S. T. Schick Tanz, *J. Research NBS* **17**, 909 (1936) RP953.

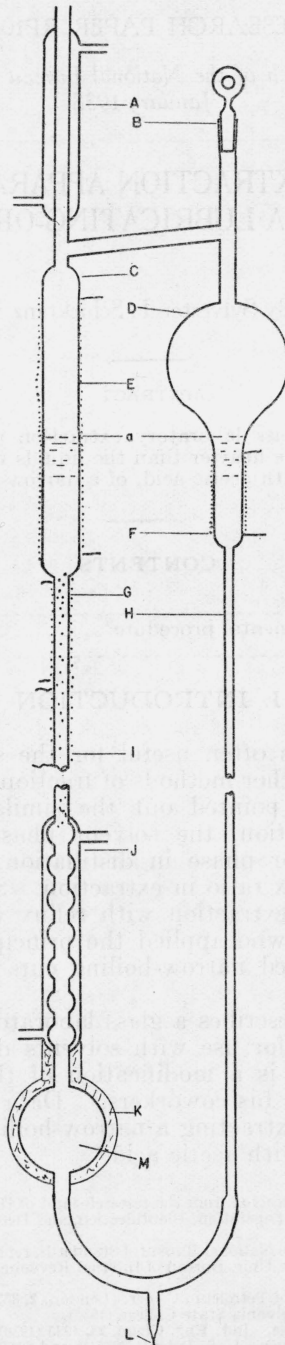


FIGURE 1.—Diagram of glass extraction apparatus for use with solvents heavier than the liquid to be extracted.

[The description is given in the text.]

## II. APPARATUS AND EXPERIMENTAL PROCEDURE

The apparatus is shown schematically in figure 1. The solvent is first placed in the apparatus to the height, *a*. The material to be extracted is then introduced through the condenser, *A*, into the section *C*, where it floats on the surface of the solvent. By means of the heater, *F*, the solvent in the evaporator, *D*, is distilled over into section *C*, the vapor being liquefied in the condenser, *A*. The evaporator, *D*, is an inverted 500-ml Kjeldahl flask, the lower section of which has fine particles of carborundum fused on the inner surface to insure uniform boiling. *B* is a ground joint. Section *C* is warmed by means of the heater, *E*, in order to increase the solubility of the material being extracted as the solvent passes down through it. For some distance below the section *C*, the temperature gradient of the tube, *G*, which has an outside diameter of about 8 mm, is controlled by means of the electrical heating unit, *I*. The saturated solution, gradually cooling, passes down the tube, *G*, to the cooled section, *J*, where most of the dissolved material is precipitated and returns up the tube in the form of small globules. The 200-ml flask, *M*, below the section *J*, serves to allow the finer particles, formed in *J* and carried down by the flow of the solution, to coalesce, because of the decreased velocity of flow, and rise up the tube, *G*, rather than be carried across to the tube, *H*, which returns the solution to the evaporator, *D*, to be recirculated. The flask, *M*, is covered with a 0.5-in. layer of magnesia, *K*. By means of a suction tube inserted through the opening at *B*, there is withdrawn from the evaporator, *D*, at appropriate intervals, such an amount of solution as will contain the quantity of material desired for one fraction. An amount of solvent equivalent to that in the solution withdrawn at *D* is added to the system through the condenser, *A*. The column is drained by breaking the lower tip, *L*. The tubes, *G* and *H*, are made as long as practicable, as the effectiveness of separation depends to a great extent on the length of column. The optimum dimensions of the tubes, *G* and *H*; the boiler, *D*; the sections *C* and *J*; and the flask, *M*, are a function of the quantity of material being extracted and of the solubility of the various constituents in the solvent. The rate of evaporation of solvent from the boiler, *D*, which determines the rate of circulation of solvent through the system (counterclockwise, as sketched), is adjusted to a value below that at which the circulation will be so rapid as to carry globules of oil across the lower end of the apparatus from the flask, *M*, to the tube, *H*.

## III. RESULTS

In table 1 are given the physical properties of the original fraction of "water-white" lubricating oil<sup>9</sup> and of the fractions obtained by solvent extraction with reflux in a column 14 m long, using acetic acid as the solvent. In this experiment, the temperature of the cooling section, *J*, was kept at about 17° C, in order to avoid solidification of acetic acid and to obtain the maximum separation possible. Fractions were withdrawn at intervals of about 12 hr.

<sup>9</sup> For a description of the "water-white" oil and its constituents, see B. J. Mair and S. T. Schicklanz, J. Research NBS 17, 909 (1936) RP953; B. J. Mair and C. B. Willingham, J. Research NBS 17, 943 (1936) RP954.

TABLE 1.—Physical properties of lubricating-oil fractions obtained by solvent extraction with acetic acid in a 14-m column

Number of fraction	Weight percentage of charge	Refractive index $n_D^{25}$	Kinematic viscosity at		Kinematic viscosity index <sup>1</sup>	Boiling point at 1-mm Hg pressure
			100° F	210° F		
Original.....	%	1.4703	<i>Stoke</i> 0.2563	<i>Stoke</i> 0.0447	102	°C 199
1.....	9.41	1.4839	.3931	.0539	70	-----
2.....	6.74	1.4811	.3517	.0511	76	-----
3.....	11.82	1.4775	.3288	.0497	82	-----
4.....	14.40	1.4775	.3126	.0485	89	-----
5.....	7.18	1.4721	.2834	.0466	94	-----
6.....	9.67	1.4687	.2557	.0442	99	-----
7.....	9.12	1.4663	.2333	.0431	113	-----
8.....	5.75	1.4640	-----	-----	-----	-----
9.....	11.37	1.4617	.1999	.0399	124	-----
10.....	7.17	1.4590	.1833	.0385	134	-----
Residue.....	7.06	1.4563	.1734	.0378	141	207

<sup>1</sup> These viscosity indices were computed with the aid of the tables of R. E. Hersh, E. K. Fisher, and M. R. Fenske, *Ind. Eng. Chem.* **27**, 1441-46 (1935).

The extent of the separation obtained by this method is clearly shown by a comparison of the physical properties of the fractions with those of the original oil. Coloration of the oil occurred during the extraction with acetic acid, the greater part of the color being concentrated in the first fractions. This coloration did not appear to affect very much the physical properties of the oil, as the recombination of the separated fractions gave a composite whose physical properties, except for the dark color, were nearly identical with those of the original oil.<sup>10</sup>

WASHINGTON, November 17, 1937.

<sup>10</sup> Note added in proof: Since this report was prepared, the author has noted a publication on an *Apparatus for the extraction of solutions with heavy solvents*, by S. Wehrli, *Helv. Chim. Acta.* **20**, 927 (1937).