

failed to reveal any definite data on its solubility except a statement by Muller¹ which gives it as from 0.01 to 0.03 per cent at 25° C. We have determined its solubility at different temperatures by agitating an excess of benzidine sulfate with pure water in a constant temperature thermostat.

The benzidine sulfate was prepared by adding an excess of dilute sulfuric acid to an aqueous solution of Kahlbaum's benzidine. The precipitated sulfate was filtered and washed with distilled water till free of acid. From 3 to 5 g. were placed in a 500 cc. Pyrex flask containing pure water and the flask immersed in a thermostat which was regulated to 0.5° C. The contents of the flask were continually agitated by a revolving glass stirrer. After a specified number of days the solution was removed and immediately filtered, the first 20 cc. of filtrate being discarded. Solubility determinations were made at 0°, 25°, 50°, and 80° C.

Two methods for determining the solubility of the benzidine sulfate were used. One method consisted in evaporating a measured volume of 100 or 200 cc. to dryness in a platinum dish on the steam bath. The residue was dried at 100° C., cooled in a desiccator, and weighed. The other method consisted in titrating a measured volume of the solution with 0.05 *N* potassium permanganate after the addition of 5 per cent of sulfuric acid. The latter method is described in detail elsewhere.²

¹ *Ber.*, **35** (1902), 1587.

² A. W. Christie and C. S. Bisson, *THIS JOURNAL*, **12** (1920), 171.

The solubility of benzidine sulfate in water as determined by the above methods is given in the accompanying table.

SOLUBILITY OF BENZIDINE SULFATE IN WATER AT VARIOUS TEMPERATURES

Temperature ° C.	Determination	
	By Weight G. per Liter	By Titration G. per Liter
0	0.049	0.048
25	0.098	0.096
50	0.141	0.149
80	0.290	0.252

The two methods gave concordant results at all the temperatures except 80° C. The solutions for the determination of solubility at 0° and 25° C. were stirred in the thermostat for four days. Those at 50° and 80° C. remained in the thermostat only 24 hrs., since it was found that after several days the solutions became badly discolored, indicating that the benzidine sulfate had undergone decomposition. The solution obtained at 80° C. was slightly discolored even after 24 hrs., as was also the residue obtained on evaporation. This is probably due to the partial oxidation of the benzidine at the higher temperature, as evidenced by the low figure for solubility obtained by titration. The residue obtained from the 50° solution showed only a very slight discoloration and the results may be considered to give the approximate solubility at this temperature.

From a consideration of these results it is evident that in washing the benzidine sulfate in a quantitative determination of sulfate, the minimum amount of cold water should be used.

LABORATORY AND PLANT

A CONVENIENT METHOD FOR THE DETERMINATION OF WATER IN PETROLEUM AND OTHER ORGANIC EMULSIONS¹

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INTRODUCTION

One of the problems frequently confronting the petroleum chemist is the determination of emulsified water. Numerous methods have been devised, several of which have been described in a previous publication² issued by the Bureau of Mines. Allen and Jacobs discuss in that paper the advantages and limitations of various types of procedure and recommend as most desirable the scheme of distilling in a special electrically heated still. This type of still involves heating the entire outer surface of a 250 cc. distilling flask and its use permits the "breaking" of troublesome froth that is usually formed when a viscous petroleum emulsion is heated. Certain difficulties in the construction and operation of this still have led to discontinuing its use in favor of the more common method of diluting the emulsion before distillation with a solvent immiscible with water. The present

paper reports the work of the Bureau in modifying this latter method to obviate certain of its admitted disadvantages.

DISTILLATION IN THE PRESENCE OF AN IMMISCIBLE SOLVENT

There are various conditions under which it is desirable to determine water in petroleum emulsions and no one method is best in all cases. The method of gravity separation¹ is a convenient one and is satisfactorily reliable for emulsions that are not too viscous or that contain water in not too fine a state of subdivision. For the estimation of extremely small percentages of water, in such products as transformer oils, special methods are necessary which are too delicate for ordinary needs. The distillation method is without doubt most generally applicable, highly reliable, and sufficiently accurate for all usual requirements. The use of a solvent not miscible with water offers several advantages, the most important of which is prevention of frothing. Its principle disadvantages concern details of operation, and the modified procedure described in the present paper has been found to obviate most of these difficulties.

This method of determining water has been used in a

¹ Published by the permission of the Director of the U. S. Bureau of Mines.

² See Bibliography, p. 490.

¹ A common procedure is to place a measured volume of oil in a suitable graduated tube, dilute with naphtha, and spin in a centrifuge. The water is driven to the bottom of the graduated tube and its volume can be read directly.

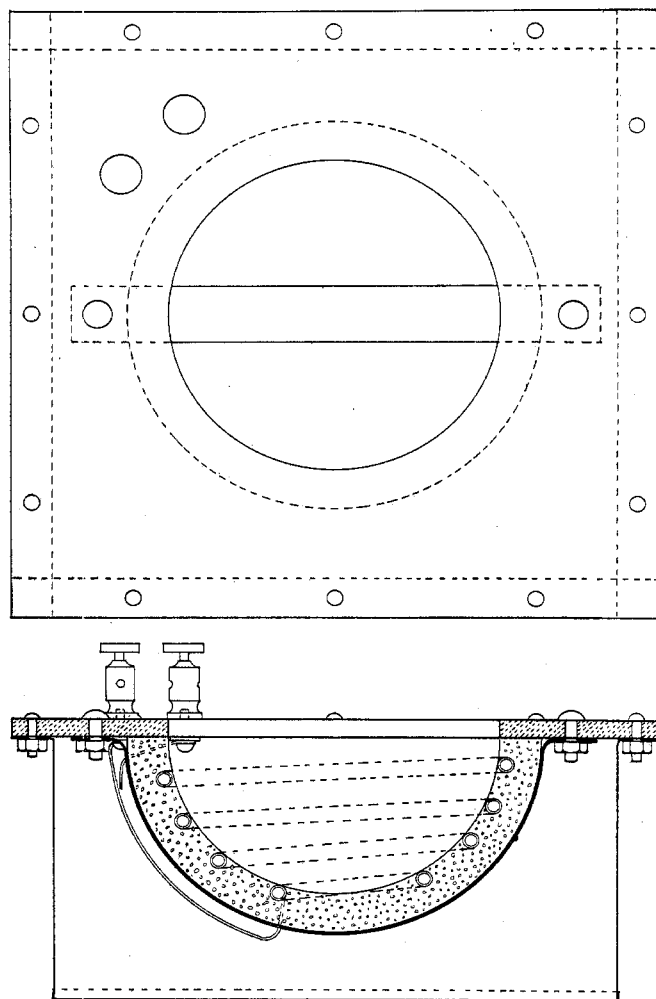


FIG. 1

variety of modifications and discussed in a number of published articles.¹ The following description covers the procedure commonly employed in petroleum laboratories.

A measured volume of the oil to be tested is placed in a glass or metal distilling flask and a suitable volume of solvent, generally petroleum naphtha of proper distilling range or a mixture of xylene and benzene, is added. The flask is connected with a condenser which delivers into some sort of a graduated receiver, heat is applied, and the distillation continued until the volume of water no longer increases. This volume is then read and the percentages of water in the original emulsion calculated. The principal objections and difficulties are:

1—Drops of water sometimes stick in the cool end of the condenser and are not easy to remove quantitatively.

2—The distillation requires a certain amount of attention and the distillate must be examined from time to time to determine when all the water has been driven over.

3—When emulsions of high-water content are tested it is sometimes necessary to add several successive portions of solvent to the contents of the distilling flask before all the water is driven over.

¹ See Bibliography, p. 490.

4—If a solvent such as xylene-benzene mixture is used there is an error due to the fact that an appreciable amount of water is dissolved in the upper layer of the distillate. Results must be corrected for this error or, preferably, the solvent must be saturated with water before adding it to the emulsion.

BUREAU OF MINES METHOD

The experimental work incident to the development of the method described in the present paper brought out a number of facts, which, though interesting, do not seem of sufficient general importance to warrant a detailed discussion. The results of the work are, therefore, reported in the form of a definite recommendation concerning apparatus and procedure. The specific recommendations are discussed in a subsequent portion of the paper. The following description covers the method as employed by the Bureau for all ordinary types of petroleum emulsions.

APPARATUS—The apparatus consists of a source of heat, a 500 cc. short-necked distilling flask, a special device combining the functions of distilling tube and receiver (hereafter referred to as "distilling tube receiver") and a condenser.

The source of heat may be an alcohol or gas burner, but the authors prefer a special type of electric heater, similar to that described by one of us in a previous article.¹ The form of the heater is, however, slightly different, as indicated in Fig. 1. The resistance elements now in service (with 110 volt alternating current) are composed of 23 ft. of No. 27 gauge nickel-chromium wire, having an approximate resistance of 72 ohms and an approximate capacity of 165 watts. These heaters are used without regulating rheostats, the capacity indicated having been found just sufficient to maintain the desired rate of distillation.

The 500 cc. round-bottomed distilling flask is a readily obtainable stock article. The authors have found Pyrex flasks particularly satisfactory.

The "distilling tube receiver" (Fig. 2) is usually made² by attaching a proper side tube to a stock type of tapered graduated test-tube. It is the one essential part of the apparatus that must be specially constructed and the authors have been assured that the work is simple and that the article ought not to be expensive when put on the market.

The condenser, readily obtainable from chemical

¹ E. W. Dean, "A Convenient Electric Heater for Use in the Analytical Distillation of Gasoline," *THIS JOURNAL*, 10 (1918), 823.

² The authors hereby acknowledge their indebtedness for aid in the design of this device to F. E. Donath, glass blower in the Pittsburgh Experiment Station of the Bureau of Mines.

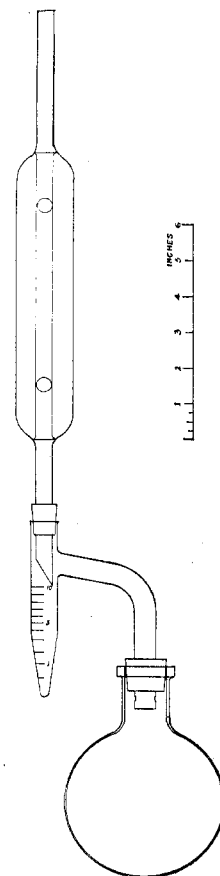


FIG. 2

supply houses, is about 15 in. long over all, and has an 8-in. sealed-on water jacket. The outside diameter of the inner tube is about $1\frac{15}{32}$ inch. Before being put into service the tip should be ground diagonally, as indicated in the figure.

The general method of assembling the glass parts of the apparatus is indicated diagrammatically in Fig. 2. Cork stoppers are used for the two junctions and if a good grade of cork is employed it is unnecessary to lute these joints.

For its own work the Bureau of Mines has developed a 4-unit electrically heated installation which has proven exceedingly satisfactory. A photograph of this installation is shown in Fig. 3.¹

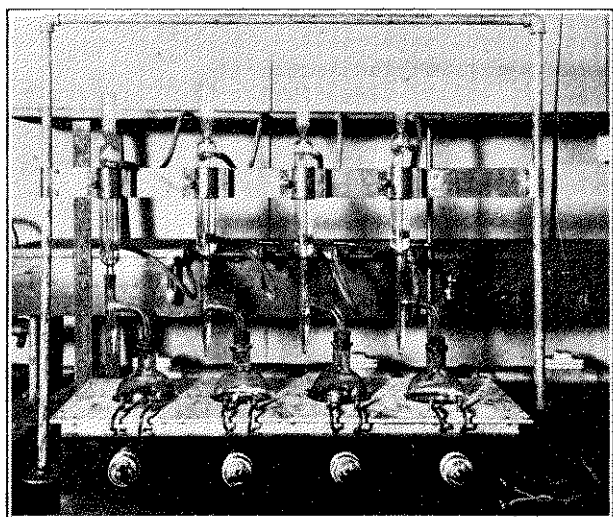


FIG. 3

IMMISCIBLE SOLVENT—The authors have found the most satisfactory solvent to be a so-called cleaner's naphtha derived from petroleum. The following specification covers the distillation range of this product, as determined by the standard method recommended by the Bureau:²

5 per cent mark not below 90° C. (194° F.)
nor above 100° C. (212° F.)
90 per cent mark not above 195° C. (383° F.)

A mixture of 80 per cent of commercial xylene and 20 per cent of commercial (so-called 90 per cent) benzene has also been found satisfactory. This mixture should not be saturated with water before using.³

¹ The authors acknowledge their indebtedness to W. A. Jacobs, Assistant Chemical Engineer, and Harry Buss, Chief Instrument Maker, of the Pittsburgh Experiment Station of the Bureau, for the design and construction of this highly satisfactory installation.

² E. W. Dean, "Motor Gasoline, Properties, Laboratory Methods of Testing, and Practical Specifications," Bureau of Mines, *Technical Paper 214* (1919), 18.

³ The commercial hydrocarbon products derived from the distillation of coal appear to dissolve water in slight but appreciable degree. With the original distillation method the full amount of solvent was distilled and collected in the receiver in contact with the water layer. A convenient means of compensating for the amount of water necessarily dissolved was to saturate the solvent before using. With the modified method herein described the volume of solvent in contact with water in the receiving graduate is of the order of 10 cc. or less and the error introduced through solution of water by this quantity is negligible. If the solvent is saturated before using, the amount of water collected in the receiver is always in excess of that actually contained by the oil tested.

PROCEDURE—Place 100 cc. of the emulsion to be tested in a graduate and measure out 100 cc. of solvent. Pour the emulsion into the 500 cc. flask, letting drain for a minute or two. Then wash all the emulsion out of the graduated cylinder, using successive portions of the measured 100 cc. of solvent. Drop a "boiling stone" (a bit of unglazed porcelain) into the flask. Connect with the distilling tube receiver and the condenser, making sure that the ground tip of the latter is placed so that drops of the condensate must fall into the receiving graduate and not into the side tube, and making sure that the position of the diagonally ground tip is approximately as indicated in Fig. 2.¹

Apply heat until the distillation is proceeding at the rate of 2 to 4 drops per second, observing the rate of fall from the ground tip of the condenser, and establish the degree of heat necessary to maintain this rate. Allow the distillation to continue until all droplets of water in the flask, the distillation tube, and the condenser have disappeared. This usually takes less than an hour and in routine work it is convenient to start the distillation, let it run for an hour, then come back and make the final readings. In case a ring of droplets of water is observed in the condenser tube the rate of distillation should be increased for a few minutes so that the vapor level on the condenser tube is raised slightly, thereby sweeping off the water. The rate of distillation can be increased by applying more heat, or by placing an insulating shield of asbestos paper over the flask. The formation of a ring of water generally does not occur if the condenser tube is clean.

The heat is then turned off and the distilling flask disconnected. (It is not necessary to wait until it cools provided care is taken not to let the escaping vapor ignite.) A drop of water sometimes hangs on the ground tip of the condenser; this, however, practically always falls off when the graduated receiver is detached from the condenser.

The volume of water collected in the graduated receiver is then read, taking care that the tube is held in a vertical position. The receiver is emptied and dried, and is ready for another determination. The flask is usually set aside to cool and a previously cleaned and dried one used for the next determination.

DISCUSSION OF DETAILS OF THE ABOVE PROCEDURE

SOURCE OF HEAT—The authors have tried electric heaters of the design described, and gas burners.² The former are by far the more satisfactory but are not essential to the accuracy of the method. Perfectly

¹ When determinations are made in an atmosphere of high humidity it is advisable to take certain precautions designed to prevent any extraneous water from entering the receiving graduate. A protecting shield such as a circle of blotting paper may be slipped over the condenser tube and held in place between the cooling jacket and the cork stopper connecting the condenser and distilling tube receiver. This obviates any possibility of water dripping from the condenser and passing through a cork that is not absolutely tight. Another more essential precaution for operations in an atmosphere of high humidity is to place a loose plug of absorbent cotton in the upper (open) end of the condenser tube, thereby preventing free access of air which might be deprived of moisture with a resulting increase in the volume of water apparently separated from the oil. A properly trapped calcium chloride tube can, of course, be used instead of a plug of cotton, but the latter is regarded as adequate protection for all cases except those requiring extreme accuracy.

² If a flame heater is employed it seems advisable to have the flask bottom protected by a piece of wire gauze.

satisfactory results were obtained with gas burners but a little more attention was required. It is possible that the easily obtainable electric hot plate would serve, although in this case it probably would be desirable to employ a flat-bottomed flask.

FLASK—The authors generally employ a short-neck, round-bottom, 500 cc. Pyrex glass flask. None of these particular details are, however, essential. The size of the flask, in particular, may be varied in case the total volume of the charge of emulsion and solvent is notably more or less than 200 cc. Metal flasks can, if desired, be substituted for glass, and the scheme of using flat-bottomed distilling vessels has already received mention.

DISTILLING TUBE RECEIVER—Numerous designs for this device have been tried and the type recommended has proved most satisfactory. For certain types of work it can undoubtedly be modified to advantage and occasion has already arisen in the Bureau laboratories to substitute a plain tube with a glass stopcock. This modification permitted the handling of emulsions containing large percentages of water, the water being drawn off through the cock into a measuring cylinder from time to time. It is entirely possible that other modifications may be desirable under conditions that may be encountered and the present device is recommended as simply the most convenient type that has been used with the types of emulsions that are generally tested in the Bureau laboratories.

CONDENSER—The type of condenser described was selected more or less by accident but has been found entirely satisfactory.

SOLVENT—A considerable variety of solvents have been tried and the two recommended above were found most satisfactory. The desirable properties are those which insure "smoothness" in distillation and a vapor temperature slightly above the boiling point of water. The solvent must have a certain percentage of constituents boiling below 100° C., but this percentage must not be too great. For instance, ordinary motor gasoline, with about 20 per cent boiling below 100° C., was notably less satisfactory than the special naphtha that is described in a previous connection. The final boiling point of the solvent apparently should not be too high as was evidenced by the fact that unsatisfactory results were obtained by the use of a solvent made by blending kerosene with ordinary motor gasoline in sufficient proportion to reduce the portion distilling below 100° C. to between 5 per cent and 10 per cent.

The solvent must be immiscible with water but should, under ordinary conditions at least, be miscible with the oil or other non-aqueous constituent of the emulsion. The application of this latter requirement is illustrated later when the results of tests on coal tar are discussed. The degree of solubility of water in the solvent is of some importance, though such errors are minimized by the fact that the quantity of non-aqueous distillate in the receiving tube is never more than 10 or 12 cc. Water is almost perfectly insoluble in the petroleum naphtha. It is probable, however, that many

laboratories will find it more convenient to prepare the xylene-benzene mixture than to obtain a special grade of petroleum naphtha. Water is slightly soluble in coal-tar distillates and in addition there is sometimes a tendency for the distillate to be cloudy, due to the holding back of minute particles of water. Permitting this cloud to settle has never, however, added as much as 0.01 cc. to the volume of the water layer.

PROCEDURE—The use of a charge of 100 cc. of emulsion and 100 cc. of solvent has proved satisfactory for oils containing between 0.5 and 10.0 per cent of water. If an accurate determination of water content of less than 0.5 per cent is desired it is necessary to use 200 cc. or more of both emulsion and solvent, employing, of course, a correspondingly larger distilling flask. If the emulsion to be tested contains more than 10 per cent of water the quantity taken should be some suitable fraction of 100 cc. According to the authors' experience it has not seemed necessary to change either the quantity of solvent or the size of the distilling flask when this method of handling emulsions of high water content is used.

The procedure recommended for measuring out the emulsion is a convenient one but not essential to the method. In some cases a pipette can probably be used to advantage. The use of a boiling stone has proved necessary and a number of determinations have been spoiled through failure to remember this detail. If it is omitted boiling sometimes begins explosively and oil is blown out through the top of the condenser.

RELIABILITY AND ACCURACY OF METHOD

The method has been tried with a number of emulsions prepared by mixing known amounts of water with anhydrous oils. Results have almost invariably checked the theoretical water content within ± 0.1 cc., which apparently represents the normal magnitude of error. The applicability of the method for the separation of water from refractory emulsions has been questioned and tests have been made to settle this point. Water was determined according to the procedure described. The flasks were allowed to cool somewhat, then connected with an ordinary distilling tube and condenser and the distillation continued until a vapor temperature of 250° C. was reached. In no case was any additional water separated or observed, thereby indicating that distillation at the temperature existing in the presence of the solvent was adequate to "break" the emulsion.

APPLICABILITY TO SUBSTANCES OTHER THAN PETROLEUM

The method was tried with several samples of grease and yielded results that were considered highly satisfactory by the operator who had had considerable experience with the conventional methods for determining the water content of greases. It was also tried with several samples of coal tar. The naphtha solvent proved useless for this type of emulsion, the tars in question being quantitatively insoluble in petroleum distillate. The xylene-benzene solvent, however, was satisfactory and the results of the determinations were

apparently as reliable as when petroleum emulsions were tested.¹

The authors believe that the method herein described is applicable to a wide variety of organic emulsions, possibly solid as well as liquid, and recommend its trial in all laboratories not fully satisfied with the methods at present in use. While it is not believed that results may necessarily be more accurate than those now obtainable, an increase in convenience and rapidity is anticipated.

PREVIOUS WORK ALONG SIMILAR LINES

On account of the simplicity of the modified method the authors have found it difficult to believe that their work could be new. The literature has been searched diligently, however, and it has been found that practically all the distillation methods for water determination have involved the use of apparatus of the conventional type and arrangement. The methods of Besson and Gray approach most closely the procedure recommended in the present paper. The basic idea of the authors' method was conceived after reading a description of the apparatus of Besson. Neither his method nor that of Gray is, however, believed to be as practical as that of the authors. The apparatus is not as simple and convenient and no attempt is apparently made to make the distillation continuous by refluxing the solvent into the flask.

SUMMARY

General experience has indicated that the method of distilling in the presence of an immiscible solvent is the most reliable and most generally satisfactory scheme of estimating the water content of organic emulsions, particularly petroleum emulsions.

The Pittsburgh Petroleum Laboratory of the Bureau of Mines has developed modifications of this method that add considerably to its convenience.

A description of the apparatus and procedure employed by the Bureau appears in the present paper.

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¹ Coal-tar emulsions are commonly believed to be more difficult to break up than petroleum emulsions and with the samples of tar tested particular care was taken to prove that no water remained after distillation in the presence of xylene-benzene mixture. The scheme of subsequently distilling the residue was employed and no water was discovered when the vapor temperature was carried as high as 250° C.

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HEAT BALANCE OF A DISTILLATION PLANT FOR THE RECOVERY OF FATTY ACIDS FROM COTTONSEED FOOTS

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Some years ago the writer was engaged in development work along lines of fatty acid distillation. Before this could be undertaken it was necessary to know the value of the heat of vaporization of fatty acids under operating conditions, *i. e.*, at the temperature and reduced pressure usually employed in distillation. A search of the literature of this subject failed to yield the desired information and the determination of these values was, therefore, undertaken by the writer. The results are given here in the hope that they may be of value to the profession.

The heat of vaporization can be calculated from the vapor pressure curve of the fatty acids, and the latter can be determined in the laboratory. As a commercial fatty acid distillation plant was available, however, advantage was taken of this fact to make a large-scale determination. This was preferred, as it was thought that it would not be possible to reproduce working conditions on a laboratory scale. One of the most important of these factors is the effect of the pitch in the still bottom. As it accumulates it becomes necessary to raise the distillation temperature sometimes