concentration broke the emulsion, presumably by "salting out" the soap film. This salting-out effect was a function of the salt concentration, not of the ratio of concentrations of salt to soap. A variation in anion appeared to have some effect in inversion by salting out, the minimum salting-out concentration in equivalents being as follows: Sodium sulfate, 0.24; sodium chloride, 0.22; sodium iodide, 0.18.

6—A study of the effect of the addition of certain sub-

stances present in commercial oils indicated a very weak stabilizing effect on oil-in-water emulsions of Nujol. Amylene, however, stabilized these emulsions somewhat in proportion to its concentration.

7—A brief study was made of the emulsifying tendencies of some crude oils with water and with sodium oleate solution together with the concentration of sodium chloride necessary to break these emulsions.

The Dehydration of Tar in the Laboratory¹

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The condensing systems which remove most of the tar from gas generally chill this gas well below its dew point; consequently raw tar usually contains considerable water. Subsequently the water content is usually reduced to a few per cent by gravity separation but this separation is never complete and in the case of very viscous tars, such as certain coal tars and tars having a gravity close to that of water, such as certain water-gas tars, the gravity separation is very unsatisfactory and some tars retain a very high percentage of water after standing indefinitely. For this reason, tars are generally held to be emulsions. Any tar containing more than a few tenths of a per cent water, if heated rapidly, may foam or bump badly. Consequently the elimination of the water is an extremely important problem for the tar distiller and the chemist engaged in the examination of tars.

For the dehydration of tar and other organic emulsions, a large number of procedures have been proposed.² In general the laboratory methods involve distillation following the addition of a solvent, or surface evaporation. The removal of water by centrifuging or by electrostatic attraction is not complete, and these methods are, therefore, not generally used in the laboratory. Grinding with carbon black and centrifuging with a solvent permit the rapid estimation of water,³ but present the disadvantage that the tar residue cannot conveniently be used for further study. Methods which involve the addition of a suitable solvent and distillation, such as for instance the excellent method of Dean and Stark,⁴ do not permit the determination of the light oil on the same portion of the sample and are generally not used when the chief object is the complete examination of the distillates and pitch.

For such studies, resort is had to surface evaporation processes which involve heating the tar from above; usually with a ring burner.^{5,6} These methods, however, present the disadvantage that the heat is generally not confined to the upper portions of the tar; consequently many tars froth and spatter disastrously in spite of the most exacting care on the part of the operator. A current of gas⁷ is sometimes passed through the tar to overcome this disadvantage; but this expedient is not ideal, because the gas current, unless chilled to low temperatures, will carry away uncondensed vapors of water and light oils. Moreover, the me-

chanical stirring action of the gas stream carries the heat to the lower portions of the still charge, thus assisting in superheating and the attendant dangers unless the entire operation is carefully watched.

Another expedient involves the slow addition of the tar to the still containing a small quantity of hot dehydrated tar. This is successful with very troublesome tars, but requires attention and special provisions for feeding.

To obviate these disadvantages, the writer has suggested a method now used in the laboratories of The Koppers Company. The tar is placed in the usual laboratory tar still, connected through the usual stillhead to the customary condenser and graduated receiver. Before closing the still, the position of the upper surface of the tar is marked. The lower portion of the still is then surrounded with a cylindrical receptacle open at the top—such as a pail—whose diameter is somewhat greater than the diameter of the still. This is so adjusted that the top of the receptacle is a little lower than the tar level. Water is added to the receptacle until it overflows. Heat is applied directly to the shallow layer of tar exposed above the water jacket, using the customary ring burner. This portion of the tar soon attains a temperature above the boiling point of water and the steam passes into the hot space above, where it cannot condense without passing out of the still. The water jacket absorbs heat which would otherwise be transmitted to the lower portions of the tar; consequently these are not heated above the steam point and no spattering or disastrous frothing can occur. The level of water falls gradually by evaporation, permitting the dehydrating zone to fall slowly. At any one time, the layer of *wet* tar heated sufficiently to cause steam formation is relatively shallow, consequently the steam has an opportunity to pass off quietly. When the water level has fallen below the bottom of the still, the tar is practically dehydrated. The receptacle for water is then removed, and the distillation is continued as usual.

The method is so simple that the writer finds it difficult to believe that it has not been used before, but if so, he is unaware of such use. It permits the approximate determination of light oil and water on the same portion of the sample, requires no new apparatus and practically no attention, handles efficiently very stiff tars and tars of high water content, and can be applied with advantage to the laboratory distillation of most tars.

In common with other surface evaporation methods which use direct flame heating, this may be subjected to the criticism that superheating of the vapor space may induce cracking. However, it presents the important advantage that the initial heating takes place at the tar level when the heat capacity of the tar assists in preventing the attainment of unduly high temperatures. The criticism can be eliminated by jacketing the upper portion of the still with material of high heat capacity. However, under the most unfavorable conditions the error introduced by such cracking is problematical, and it is generally disregarded.

¹ Presented before the Division of Industrial and Engineering Chemistry at the 62nd Meeting of the American Chemical Society, New York, N. Y., September 6 to 10, 1921.

² It is beyond the scope of this article to list these. Most of the important references have been listed by Dean and Stark, *THIS JOURNAL*, 12 (1920), 486.

³ Odell and Thiele, *Gas Record*, March 23, 1921, p. 30.

⁴ *Loc. cit.*

⁵ Senger, *J. Gasbel.*, 45 (1902), 841; also *J. Soc. Chem. Ind.*, 21 (1902), 1475.

⁶ Jayne, *J. Am. Chem. Soc.*, 25 (1903), 814.

⁷ If gas is used, it should be inert. The practice of blowing air through tar should of course be avoided, for tar at elevated temperatures may absorb oxygen, and with some tars, the absorption of oxygen is accompanied with a marked diminution in the quantity of recoverable oils.