THE DETERMINATION OF PHENOLPHTHALEIN IN MINERAL OIL EMULSIONS.*

BY C. F. BICKFORD AND R. E. SCHOETZOW.[1]

An accurate and practical method for the determination of moderately large quantities of phenolphthalein in mineral oil and agar emulsions involves two steps:

1. Separation of the phenolphthalein from the oil and the agar.
2. Determination of the phenolphthalein.

The separation of the agar is very simply carried out by treating the whole emulsion with a mixture of alcohol and ether. When the proportions of alcohol and ether are suitably chosen, this mixed solvent precipitates the agar and dissolves the oil and water. The most satisfactory mixture is 25 cc. absolute alcohol and 75 cc. ether; increasing the proportion of alcohol makes the subsequent isolation of the phenolphthalein more difficult. The precipitated agar is easily separated by filtration, but it carries down small quantities of phenolphthalein. These are recovered by dissolving the agar in a small amount of water and again precipitating with a fresh portion of the solvent mixture. After this reprecipitation the agar is tested for retained phenolphthalein by dissolving in water and adding a drop of dilute alkali; if no phenolphthalein is found it is discarded.

The solvent mixture containing the phenolphthalein is extracted with dilute alkali; the phenolphthalein passes into the aqueous layer to give the familiar colored solution. It is at this point that the proportion of alcohol and ether shows its effect; raising the alcohol above 25% renders the complete extraction of the phenolphthalein more difficult. The phenolphthalein is precipitated from the combined alkaline extracts by adding dilute sulphuric acid. This precipitate is not, in its present form, suitable for weighing. The mixture is extracted with ether and the resulting ether solution is extracted with alkali. Upon acidification this alkaline solution yields the phenolphthalein in a form which becomes coarsely crystalline after standing over night. The large crystals permit rapid collection on the Gooch filter.

The solubility of phenolphthalein in water is not negligible; 100 cc. of water at 21° C. dissolves about 3 mg. To the weight obtained must therefore be added a correction for the amount which remains in solution. The correction is determined for every assay by a colorimetric method. An exact description of the procedure follows:

A sufficient quantity of the emulsion to yield about 0.05–0.10 Gm. phenolphthalein is accurately weighed into a glass-stoppered Erlenmeyer flask. Seventy-five cubic centimeters of ether and 25 cc. absolute alcohol are added. The flask and contents are vigorously shaken and then allowed to stand for thirty minutes. The mixture is filtered into a separatory funnel and the precipitate washed with several small quantities of the solvent mixture. The agar is redissolved in about 5 cc. of water in the original Erlenmeyer flask and precipitated with fresh solvent mixture as previously described. The filtrate and washings from the reprecipitated agar are combined with the first filtrate in the separatory funnel. The agar is dissolved in water and tested for phenolphthalein by adding a drop of dilute sodium hydroxide, discarding if no phenolphthalein is present.

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The solvent mixture is repeatedly extracted with 10-cc. portions of 0.04 normal sodium hydroxide until absence of color shows complete removal of phenolphthalein. The solvent mixture is then discarded. The phenolphthalein is precipitated from the combined alkaline extracts by slow addition of 0.1 normal sulphuric acid in slight excess. The precipitated phenolphthalein is completely extracted from this mixture with ether. The ether solution is again extracted with 0.04 normal sodium hydroxide, discarding the ether after complete removal of the phenolphthalein. The alkaline solution is acidified with 0.1 normal sulphuric acid, being careful to use only one or two cc. excess acid. The resulting precipitate is allowed to stand over night to permit coarse crystals to form. The crystals are collected on a tared sintered glass Gooch crucible, washed with four (5 cc.) portions of cold water, dried at 100° C. and weighed. The filtrate and washings are transferred to a 250-cc. volumetric flask. The beaker which contained the crystals is washed with 0.04 normal sodium hydroxide until colorless washings are obtained. These beaker washings are added to the 250-cc. flask containing filtrate and the solution is made up to 250 cc.

A standard phenolphthalein solution is prepared by dissolving exactly 0.03 Gm. of pure phenolphthalein in 250 cc. of 0.04 normal sodium hydroxide solution. The quantity of phenolphthalein in the filtrate and washings is now determined by matching the color of that solution against suitable dilutions of the standard solution. This quantity is added to the weight previously found.

Determinations by the described method are consistent and agree with theory. An analysis in duplicate of an emulsion prepared to contain 0.321% phenolphthalein indicated an averaged phenolphthalein content of 0.311%, the individual values being 0.319% and 0.302%. Similarly, another emulsion, the phenolphthalein content of which was less exactly known, contained an averaged phenolphthalein content of 0.289%, the individual values being 0.280% and 0.297%.

Occasionally emulsions of this type show some separation after aging. The nearly clear aqueous layer at the bottom of such a separated emulsion contained less than 0.01% phenolphthalein. The following table gives the amounts of phenolphthalein found in the top and middle portions for three samples of mineral oil and agar emulsions contained in bottles.

	Per Cent Phenolphthalein.	
Sample.	Top Portion.	Middle Portion.
B	0.335	0.310
	0.333	0.332
E	0.315	0.320
F	0.377	0.373

These data show that, although the bottom aqueous layer of an emulsion which has undergone separation is low in phenolphthalein content, the remainder is quite uniform in respect to the quantity of phenolphthalein present. The whole sample will again have a uniform distribution of phenolphthalein after shaking to re-incorporate the separated water.

THE CLINIC PHARMACY.*

BY JOSEPHINE NICHOLS.

We speak of a Clinic as an organized group of doctors working together for the welfare of the patient. With such a group a pharmacist has a very definite place. It is here that pharmacy can be practiced in its most professional aspects and should therefore appeal to the pharmacist who is interested in the science itself and who need not be hampered by the economics of business management and

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