4.5 Determination of unsaponifiable matter

The term "unsaponifiable matter" refers to those substances present in oils or fats that are not saponified by alkali hydroxides and are extractable into ether.

Recommended procedure

Place a quantity of the test substance, accurately weighed, as specified in the monograph, in a flask provided with a reflux condenser and boil in a water-bath for 1 hour with 25 mL of potassium hydroxide/ethanol (0.5 mol/l) VS, with frequent swirling of contents. Wash the contents of the flask into a separator by means of 50 mL of water and, while the liquid is still slightly warm, extract by shaking vigorously with 3 successive quantities, each of 50 mL, of ether R, washing out the flask with the first quantity of ether R. (CAUTION: Ether should be free of peroxides.) Take care to release frequently and carefully the pressure that may build up inside the separator. Combine the ethereal solutions in another separator containing 20 mL of water. (If the ethereal solutions contain solid suspended matter, filter them into the separator through a fat-free filter-paper and wash the filter-paper with ether R.) Gently rotate the separator for a few minutes without violent shaking, allow the liquids to separate, and run off the aqueous layer. Wash the ethereal solution by shaking vigorously with 2 successive quantities, each of 20 mL, of water; then treat 3 times with 20 mL of potassium hydroxide (0.5 mol/l) VS (NOTE: aqueous reagent), shaking vigorously on each occasion and washing with 20 mL of water after each treatment. Finally wash with successive quantities, each of 20 mL, of water until the aqueous layer is no longer alkaline to phenolphthalein/ethanol TS. Transfer the ethereal extract to a weighed flask, washing out the separator with ether R; distil off the ether with the necessary precautions and add 3 mL of acetone R.

By the aid of a gentle current of air remove the solvent completely from the flask, which is preferably held obliquely and rotated, almost entirely immersed, in a water-bath at about 60 °C. Dry to constant weight at a temperature not above 80 °C and dissolve the contents of the flask in 10 mL of ethanol (~750 g/l) TS, previously neutralized to phenolphthalein/ethanol TS. Titrate with carbonate-free sodium hydroxide (0.1 mol/l) VS, using phenolphthalein/ethanol TS as indicator. If the amount of carbonate-free sodium hydroxide (0.1 mol/l) required does not exceed 0.2 mL, the amount weighed is to be taken as the unsaponifiable matter. Calculate the unsaponifiable matter as a percentage of the oil or fat. If the amount of carbonate-free sodium hydroxide (0.1 mol/l) VS required exceeds 0.2 mL, the amount weighed cannot be taken as the unsaponifiable matter and the test must be repeated.