

## MISCELLANEOUS TECHNIQUES AND IMPURITIES

Record the density after about 5 minutes when the equilibrium conditions should be attained.

In extremes of ambient temperature it may be necessary to use a water bath to maintain the suspension at 20 °C.

### *Calculation*

$$\text{Density of suspension concentrate} = \frac{a}{2 - a} \text{ g/ml}$$

Where:

$a$  is the density of the 50% m/m dilution and the density of water is taken to be 1.0.

### 3.3.2 Density bottle method

#### OUTLINE OF METHOD

A portion of the sample is weighed into a density bottle and diluted with water containing an anti-foam agent, using vacuum, if necessary to remove occluded air. The volume of sample taken is then found by making up to capacity with the anti-foam solution and weighing.

#### REAGENT

*Anti-foam solution* 1% m/v aqueous solution of a silicone anti-foaming agent.

A very dilute solution of "Silcolapse 5000" has been found to be satisfactory at a dilution of 4 drops in 2.5 l of water.

#### PROCEDURE

Measure about 20 ml of well-mixed sample, previously adjusted to  $20 \pm 0.5$  °C by standing in a water bath, into a suitable 100 ml density bottle (Note 8), previously weighed and calibrated with water at 20 °C, and weigh. Add about 20 ml of anti-foam solution, previously brought to  $20 \pm 0.5$  °C, and mix by swirling. Fill the bottle to just below the mark with anti-foam solution, without mixing, and place in a water bath held at  $20 \pm 0.5$  °C for 10 minutes. Ensure that no air bubbles remain (Note 9) then, without delay (to avoid temperature changes), adjust the volume to the 100 ml mark with anti-foam solution. Dry the outside of the bottle and weigh. To obtain the density of the anti-foam solution, determine the mass of the density bottle filled with anti-foam solution at  $20 \pm 0.5$  °C.

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$$\text{Then density at } 20^{\circ}\text{C in g/ml} = \frac{\text{mass of sample in g}}{\text{volume of sample in ml}} = \frac{X - W}{100 - \frac{Y - X}{D}}$$

Where:

- $W$  = mass of bottle only (g)  
 $X$  = mass of bottle with sample (g)  
 $Y$  = mass of bottle with sample and anti-foam solution to capacity (g)  
 $Z$  = mass of bottle with anti-foam solution only to capacity (g)  
 $D$  = density at  $20^{\circ}\text{C}$  of anti - foam solution in g/ml =  $\frac{Z - W}{100}$

*Note 1* This method is based on method BS 718:1979 or IP 160/64 but does not include all the precautions necessary for precision determinations since it has been adapted for use with methods of analysis for pesticides. For more accurate determinations method BS 718 or IP 160 should be followed.

*Note 2* See method BS 718 or IP 160 for methods and tables of correction to be applied for more accurate determinations.

*Note 3* Unless otherwise directed for routine work,  $\pm 0.5^{\circ}\text{C}$  can be used.

*Note 4* The 'Warden' form (Figure 6) is recommended for all except viscous or solid products and should always be used for volatile products.

*Note 5* It is recommended that new pyknometers should be re-calibrated after one year, and thereafter at intervals depending upon the magnitude of any changes found.

*Note 6* Clean the pyknometer and stopper with chromic acid cleaning solution, rinse well with distilled water, and dry. Ensure that all traces of water are removed, using a current of filtered air if necessary. Cleaning should be carried out in this manner whenever the pyknometer is to be calibrated, or whenever the liquid fails to drain cleanly from the internal walls of the pyknometer or the capillary of the stopper. Normally the pyknometer may be cleaned between determinations by washing with light petroleum spirit, followed by vacuum drying.

*Note 7* Specific gravities (relative densities) as calculated in the text are apparent values in air.

Hence

$$\text{Apparent density} = \text{apparent SG} \times 0.99700 \text{ g} \times \text{cm}^{-3}$$

$$\text{True density} = (\text{apparent SG} \times 0.99700) + 0.0012 \text{ g} \times \text{cm}^{-3}$$

$$\text{True specific gravity} = \frac{\text{true density}}{0.99820}$$

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*Note 8* A suitable density bottle consists of a 100 ml volumetric flask, with the neck cut down to approximately 12 mm above the graduation mark.

*Note 9* If bubbles persist, place the bottle in a vacuum desiccator, evacuate cautiously, leave under vacuum for 5 min., then replace the bottle in the 20 °C water bath.

### MT 5 MATERIAL SOLUBLE IN ACETONE

#### SCOPE

These methods are intended for materials which are freely soluble in acetone.

#### 5.1 Hot solution

#### OUTLINE OF METHOD

The sample is refluxed with acetone, the acetone is evaporated, and the residue is dried and weighed.

#### REAGENT

*Acetone* ( $\text{CH}_3\text{COCH}_3$ ) anhydrous; RE 34

#### APPARATUS

*Soxhlet extraction apparatus or straight through extractor*

*Round bottom flasks* 250 ml with ground joint to fit the extractor; containing 2 glass beads and tared

*Distillation set* to fit the flask

*Soxhlet thimble* previously extracted with acetone then dried

*Flameproof oven* at 60 to 70 °C

#### PROCEDURE

Weigh (to the nearest mg) the specified amount ( $w$  g) of sample, transfer to the thimble, and extract completely with acetone for about 4 h into the tared flask ( $x$  g). Remove the flask from the extraction apparatus, distil off the acetone and dry the flask and residue in the oven to constant weight at 60 to 70 °C. Cool and reweigh ( $y$  g).

$$\text{Content of material soluble in acetone} = \frac{100(y - x)}{w} \% \text{ m/m}$$