



Determination of THPS using a Microtitration Test Kit

Procedure

A) Preparation of Iodine Titrant Solution

Using the dropper, transfer 1.5 cm³ of 0.1N Iodine solution to the 15 cm³ graduated tube and dilute to 15 cm³ with deionised water to produce 0.01N iodine solution titrant. Cap the tube and invert gently to mix. Transfer this titrant to the empty 50 cm³ amber glass bottle for storage. The iodine titrant solution will be stable for approximately 1 week if stored under cool conditions.

B) Sample Preparation (removal of hydrogen sulphide interference)

Hydrogen sulphide will interfere with the test for THPS and must be removed. If hydrogen sulphide is absent then this step can be ignored. If present, add 5 drops of Zinc Acetate Solution per 50ml of sample collected. Allow to stand for 1 minute then pass 20ml of this sample through the filter holder fitted with a GF/C filter paper in order to remove any precipitate that may have formed. Following filtration, continue as directed from step 2 of the analysis detailed below.

C) Analysis of Water Sample

1. Acquire the test sample and contain it in a plastic/glass bottle, filled to the top and capped to minimise the risk of oxidation by the air. Cool sample (see note 1) and filter if necessary using the 20 cm³ syringe connected to the 25mm filter holder (fitted with a GF/C filter paper).
2. Using a 10 cm³ syringe, transfer 10 cm³ of sample to the 50 cm³ glass titration jar and add approximately 10 cm³ deionised water.

3. Using a 2 cm³ syringe, add 2 cm³ DSP solution to the glass titration jar and swirl to mix.
4. Using a 1 cm³ pasteur pipette add 0.25 cm³ PSSA solution (see note 2) and swirl to mix.
5. Using the semi-micro spatula, add a small amount of starch indicator (see note 3) and mix well.
6. Titrate with 0.01N iodine titrant, using a 1 cm³ syringe plus needle, to a blue/black end point which does not disappear immediately on swirling (see note 4). Record the titre (S).
7. Repeat procedure (2-6) using a blank sample (system water not containing THPS). Record the titre (B).

Calculation: THPS Concentration (ppm) = (S-B) x 1.02

Notes

1. Under some circumstances, e.g. especially where a cationic surfactant is part of the THPS formulation dosed to the water, temperature may have an effect on the titration. For maximum accuracy, therefore, it is best to carry out the titration, consistently, between 20°-25°C.
2. PolyStyrene Sulphonic Acid (PSSA) is added to mask interference to the titration end point caused by the presence of cationic surfactants. If you are seeing a sharp blue black end point without the PSSA, this stage may safely be omitted.
3. Only a very small amount of starch is needed; large amounts will blur the end point. As a guide, half fill the indent in the plastic stirring rod with starch powder (i.e. approximately 50 - 100 mg) will be correct.

4. The end point is reached at the first appearance of a blue/black colour which does not fade within a few seconds. The end point is "fugitive" which means that the colour will fade on standing. It is strongly recommended that new users conduct a number of "practice titrations" to familiarise themselves with the end point before analysing real test samples. With practice, quite accurate analyses will be achieved.

Calibration

If calibration of the test kit is required, it may be accomplished by titration of a THPS solution of known concentration. However, standard solutions of THPS must always be made in a buffered medium to avoid oxidation of the THPS which would otherwise give a false result. A suitable method is as follows.

Weigh out 1.0 g of sodium bicarbonate and add to a 1 litre graduated flask. Add 800 ml of deionised water and swirl to mix. Weigh out, accurately, the quantity of THPS biocide which will contain exactly 1.00 g of THPS, active ingredient (e.g. 1.33 g of Tolcide PS75). Wash this into the sodium bicarbonate solution and swirl to mix. Top up the graduated flask to the 1 litre mark with deionised water and mix well. This standard solution will contain 1000 ppm of THPS (active ingredient).

For lower concentrations of THPS, dilute the 1000 ppm standard, as required, with deionised water (e.g. for a 100 ppm solution, take 10 ml of standard and dilute this, with deionised water, to 100 ml).

Note: For maximum accuracy, make up the THPS standard solutions fresh when needed and use them without delay.