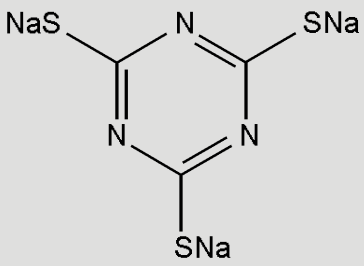
TMT 15® - Determination of the TMT concentration in aqueous solutions and effluents

Analytical procedure / Info 5 E



1. Principle

TMT 15® is used for precipitating heavy metals out of effluent in the form of virtually insoluble metal-TMT compounds. Since surplus precipitant is used in the process, it is desirable in certain cases to have a method for determining the concentration of free TMT 15®, which is not in combination with heavy metals.

TMT is a 15% aqueous solution of the sodium salt of trimercapto-s-triazine:

Empirical formula: $C_3N_3S_3Na_3$

Molar mass: 243.22 g/Mole

Density of TMT 15®: approx. 1.12 g/cm³

Content of $C_3N_3S_3Na_3$: min. 15 % by weight = 168 g/l

Even traces of TMT 15®, that is to say the active constituent $C_3N_3S_3Na_3$, can be determined not only in pure aqueous solutions but also in flue gas scrubbing water or industrial effluent containing salts. This is done by UV-spectroscopy. The $C_3N_3S_3Na_3$ must be present in the form of an alkaline solution (1 mole/l sodium hydroxide).

Concentrations as low as approx. 2 mg $C_3N_3S_3Na_3$ per litre of effluent are still detectable. At a concentration of approx. 168 g $C_3N_3S_3Na_3$ per litre of TMT 15®, the concentration of free TMT 15® per litre of effluent still has a measurable threshold value of only a few ml TMT 15® per m³.

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2. Determination

Below is a guide for recording the calibration curve and analysis of the effluent sample containing TMT 15®.

Equipment: Zeiss PD 2M spectral photometer with UV measuring device
Cuvette: 1 cm quartz cuvette
Wavelength: 285 nm

2.1 Recording the calibration curve

Standard solutions* are made up containing 1 - 15 mg $C_3N_3S_3Na_3$ per litre. To make up the solutions, water with approximately the same constitution as the original effluent sample is used. The salt content of this water should correspond to that of the original effluent sample. It must be neutral (approx. pH 7) and contain no heavy metals or TMT 15®. For 100 ml of standard solution*, 50 ml sodium hydroxide solution (concentration: 2 moles/l) are poured into a 100 ml volumetric flask. An appropriate amount of diluted TMT 15® solution then is added, corresponding to the required concentration of $C_3N_3S_3Na_3$, and the mixture is topped up to 100 ml with the above-mentioned water. In the standard solutions, the $C_3N_3S_3Na_3$ is now present at the sodium hydroxide concentration (1 mole/l) required for conducting measurements.

The standard solutions are mixed well. Their extinction's are measured in a 1 cm quartz cuvette against a control solution at a wavelength of 285 nm. The control solution is made up by mixing 50 ml of the water sample resembling the original effluent sample with sodium hydroxide solution (2 moles/l) in a volumetric flask and topping the mixture up to 100 ml. The extinction values are then plotted in a graph against the concentration of $C_3N_3S_3Na_3$ in each case to produce a calibration curve.

* To make up the standard solution, the TMT 15® can be diluted to the following intermediate concentrations:

A.	1	ml TMT 15® (conc.: 168 g $C_3N_3S_3Na_3$/l) diluted to 1000 ml
	1	ml of the diluted solution contains 0.168 mg $C_3N_3S_3Na_3$.
	0.595	ml of the diluted solution correspond to 100 ml standard solution with a concentration of 1 mg $C_3N_3S_3Na_3$ / l.
B.	5	ml TMT 15® (conc.: 168 g $C_3N_3S_3Na_3$ / l) diluted to 1000 ml
	1	ml of the diluted solution contains 0.840 mg $C_3N_3S_3Na_3$.
	1.786	of the diluted solution correspond to 100 ml standard solution with a concentration of 15 mg $C_3N_3S_3Na_3$ / l.

2.2 Analysis of the effluent sample

The same procedure applies here as described in 2.1. 50 ml neutral effluent are mixed with sodium hydroxide solution (2 moles/l) in a volumetric flask, after which the mixture is topped up to 100 ml. This sample is then measured in a 1 cm quartz cuvette against the control solution at 285 nm. From the extinction value obtained, the corresponding concentration of $C_3N_3S_3Na_3$ can be read off at the calibration curve. Since the original effluent sample was diluted to twice its starting volume with sodium hydroxide solution, the result must be multiplied by a factor of 2 in order to obtain the original concentration of $C_3N_3S_3Na_3$.

3. Notes

If the effluent sample is of high salinity, this can lower its extinction value and lead to erroneous measurements. In order to eliminate this side-effect of the matrix, it is important to make up the standard and control solutions using water which has a salinity corresponding to that of the original effluent sample. This water must also be neutral (approx. pH7) and contain no heavy metals or TMT 15®.

The samples to be measured must be clear. Solids must be filtered off beforehand. If the flue gas scrub water has a high calcium content, this can lead to the precipitation of calcium hydroxide as the sample is made alkaline. Calcium hydroxide does not adsorb any TMT 15®, so that even samples of this sort can be conditioned by simple filtration.

The concentration of $C_3N_3S_3Na_3$ in the TMT 15® can be determined by potentiometric titration against sulphuric acid. This set of analysis instructions for TMT 15® is available to all interested parties.