

## Application Note 11

### Determination of Critical Micelle Concentration with DataPhysics DCAT Series

Determination of critical micelle concentration (CMC) of Triton X-100 in aqueous solutions and determination of the ethoxylation grade of the Triton X-100 mixture.

#### Problem

Determination of the critical micelle concentration (CMC) of Triton X-100 in aqueous solutions with the DataPhysics DCAT combined with the liquid dispense unit (LDU).

The literature values of the CMC of Triton X-100 differ in the range of 0.24 to 0.433 mmol/l. The reason for this variance is that the exact molecular structure of available Triton X100 differs, according to side chain configuration (due to the extent of ethoxylation). As an average the value of ethoxylation is described as 9.5. It has been suggested that the relationship between CMC and the extent of ethoxylation could be investigated. With the work of Li et al. it was demonstrated that it is possible to determine the extent of ethoxylation within a Triton X-100 mixture, by a study of CMC.

M. Li, et al. (2000): Small Variations in the Composition and properties of Triton X-100; *J. Coll. & Int. Sci.* (230); 135-139

S. Ledakowicz, et al. (1997): Critical micelle concentration of nonionic detergents; *Tenside Suf. Det.* 34; 190-194

#### Method

Surfactants, such as Triton X-100, are compounds with hydrophilic and hydrophobic constituents. In aqueous solutions the hydrophilic head of the surfactant interacts with the water molecules.

When surfactants are brought into an aqueous solution, a higher concentration of surfactants is found at the surface than in the bulk. This decreases the surface tension of the solution, as a function of the total surfactant concentration. Once the surface of the solution is saturated with surfactant molecules (see fig. 1), no more molecules can join the surface layer, irrespective of the concentration of surfactant reached. This means that no further decrease in surface tension is detected. Beyond this point agglomerates, called micelles, are formed within the bulk solution. This point is termed “the **Critical Micelle Concentration**”.

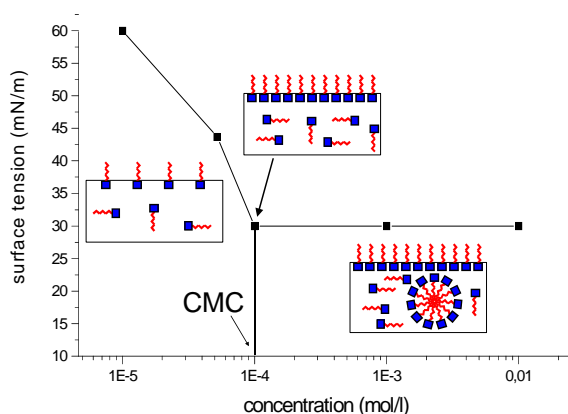


Fig. 1: Dependence of the surface tension on the surfactant concentration; the critical micelle concentration and the formation of micelles are indicated

In order to determine the CMC of a surfactant within an aqueous solution the Wilhelmy plate or Du Nouy ring method is used with the DCAT, in combination with the LDU. As the concentration of surfactant is increased, by software control of the LDU, it is dispersed via a “flea” driven by an electromagnetic stirrer. The temperature with the sample vessel is also controlled.

In practice and in contrast to the ideal case described in fig. 1., during *real* measurements the curve produced (surface tension vs. surfactant concentration) rarely consists of two straight lines which meet in a clearly defined point. During these *real* case studies it is likely that our surfactant contains contaminants or is, in fact, a mixture of surfactants. A typical curve of the dependence of surface tension on the concentration of a surfactant, with traces of contaminants, is displayed in Fig.2.

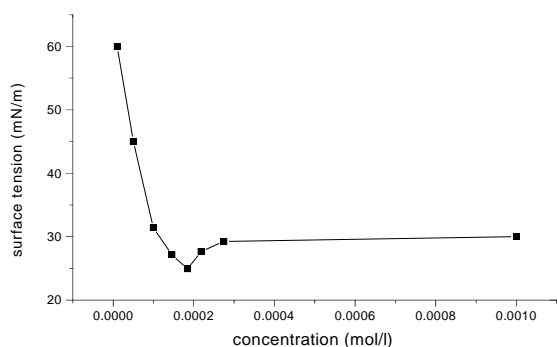


Fig. 2: Dependence of the surface tension concentration in the presence of contaminants

In most cases the contaminant is expected to be more surface active than the pure surfactant. This drives the surface tension (temporarily) below the value, observed in the curve typical for the pure surfactant, in the vicinity of the CMC. This is due to the contaminant occupying the surface in preference to the pure surfactant, at lower concentrations. As the experiment continues and more surfactant (mixture) is added the contaminant is absorbed in the forming micelles allowing the surface tension to stabilize at the value expected of the pure surfactant. When observed; this process, compensating for the dip below the value expected, results in an increase in surface tension, as the contaminant moves away from the surface to be replaced with pure surfactant molecules.

## Procedure

To determine the CMC of Triton X-100, with the DataPhysics DCAT and LDU, the surfactant was dispersed in water (1:100 v/v). All tubes and the syringe of the LDU were flushed, with water, to

ensure that there was no contamination during the dispensing process.

The LDU system has to be calibrated, be free of air bubbles and then charged with the Triton X-100 solution.

A test/sample vessel was filled with 100 ml of pure water and a magnetic “flea” added. Between measurements (surface tension determination with the Wilhelmy plate) a known volume of Triton X-100 solution was added and the mixture stirred for 40 s.

All elements of the method: dispense steps, stirring time, rest time and surface tension evaluation are controlled through the SCAT 33 software.

In order to determine the concentration *region* where the CMC will be found, the first project is carried out over a wide concentration range (1.0E-5 to 2.0E-2 mol/l).

A second, more precise, evaluation is then carried out (1.0E-4 to 1.0E-3 mol/l) once an estimate of the CMC has been determined.

In both procedures, a 20 step, logarithmic function, surfactant solution addition was carried out (see. fig 3).

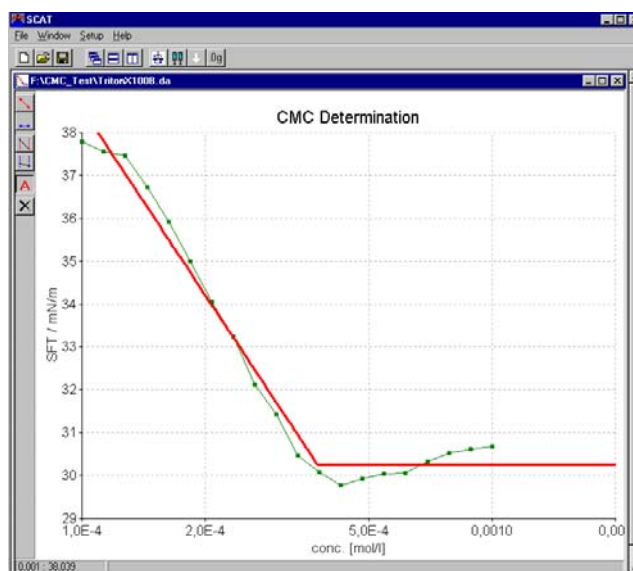


Fig. 3: CMC determination of Triton X-100. The 20 measurement steps were set as a logarithmic function of the concentration.

## Results

The result of the CMC measurements of Triton X-100 was 0.4 mmol/l.

The surface tension of the saturated solution was 30.6 mN/m.

This data tells us that the effective ethoxylation value of the Triton X-100 used here is near 10.0.

## Summary

The DCAT, in combination with the LDU, provides a convenient and reproducible way to determine the critical micelle concentration of Triton X-100.

Secondly, the measurements provide an easy and cheap way to determine the *effective* molecular structure of the Triton X-100 mixture, without expensive NMR-measurements.