Application Note AN-T-245

Photometric determination of ionic surfactants by two-phase titration

Accurate and reliable results with the Optrode according to the classical Epton titration

The two-phase Epton titration method is the standard approach for titrating ionic surfactants. It is used whenever the delivery conditions specify this method or when potentiometric surfactant titration cannot be carried out, such as with powdered washing agents. This Application Note presents the two-phase titration of ionic surfactants according to Epton. The titration is optimized for the OMNIS Software. Specifically, the concentration of sodium dodecyl sulfate (SDS) in shower gel is determined by titration with hyamine using an Optrode M2.

INTRODUCTION

Two-phase titrations were first developed in the early 1930s and still continue to be improved upon. In 1947, chemist S. R. Epton developed the Epton titration method. In this first iteration, an anionic sample is mixed with methylene blue in diluted sulfuric acid (as an indicator), and chloroform. Vigorous shaking forms an ionic salt that is extracted into the chloroform, turning the phase a blue color. Titrating with the classical cationic surfactant cetylpyridine bromide causes the color to slowly migrate into the upper aqueous phase. The endpoint is reached when both phases turn blue.

In the 1960s, Holness and Stone developed the method into its current form. They replaced methylene blue with a mixed indicator, dimidium bromide-disulfine blue, and used Hyamine® 1622 as cationic titrant. This new indicator clearly improved endpoint recognition because the color change is only observed in the chloroform phase. The chloroform phase is red in the presence of excess anionic surfactant ions and blue in the presence of excess cationic surfactant ions.

Depending on the titrant (e.g., hyamine or SDS), either anionic or cationic surfactants can be titrated. Despite this, the term «Epton titration» has stuck and is still used today.

SAMPLE AND SAMPLE PREPARATION

This application is demonstrated on shower gel. Sample preparation is not required.

EXPERIMENTAL

The determination is carried out on an OMNIS Professional Titrator equipped with OMNIS Dosing Modules (Figure 1) as well as an Optrode M2.

An appropriate amount of sample is weighed into the titration beaker, then deionized water, mixed indicator (dimidium bromide-disulfine blue), and chloroform are added. The solution is titrated with standardized hyamine until after the first equivalence point is achieved using a wavelength of 520 nm.

Figure 1. The OMNIS Professional Titrator with OMNIS Dosing Modules (shown here) are ideal for two-phase titrations of ionic surfactants when equipped with the Optrode M2.

RESULTS

The Epton titration method offers very accurate results, as displayed in Table 1. An exemplary titration curve of SDS in shower gel with hyamine is given in Figure 2, showing the sharp jump in voltage at the equivalence point.

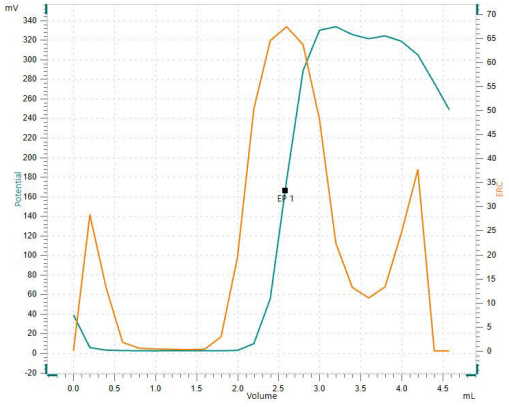

Figure 2. Epton titration curve at 520 nm with one equivalence point.

Table 1. Results of the Epton titration of SDS in shower gel (n = 6).

Sample (n = 6)	SDS (%)	SDS (mmol/100g)
Mean value	5.09	17.65
SD(abs)	0.05	0.19
SD(rel) in %	1.1	1.1

CONCLUSION

Precise and reliable determination of ionic surfactants according to the Epton two-phase titration can be achieved using an OMNIS system. The Optrode M2, with its glass shaft, is fully resistant against solvents and is therefore ideal for surfactant titrations with chloroform.

CONTACT

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CONFIGURATION

OMNIS Professional Titrator with magnetic stirrer

Innovative, modular potentiometric OMNIS Titrator for stand-alone operation or as the core of an OMNIS titration system for endpoint titration and equivalence point titration (monotonic/dynamic). Thanks to 3S Liquid Adapter technology, handling chemicals is more secure than ever before. The titrator can be freely configured with measuring modules and cylinder units and can have a rod stirrer added as needed. Including "Professional" function license for parallel titration with additional titration or dosing modules.

- Actuation via PC or local network
- Connection option for up to four additional titration or dosing modules for additional applications or auxiliary solutions
- Connection option for one rod stirrer
- Various cylinder sizes available: 5, 10, 20 or 50 mL
- Liquid Adapter with 3S technology: Safe handling of chemicals, automatic transfer of the original reagent data from the manufacturer

Measuring modes and software options:

- Endpoint titration: "Basic" function license
- Endpoint and equivalence point titration (monotonic/dynamic): "Advanced" function license
- Endpoint and equivalence point titration (monotonic/dynamic) with 5-way parallel titration: "Professional" function license

OMNIS Dosing Module without stirrer

Dosing module for connection to an OMNIS Titrator for extending the system to include an additional buret for titration/dosing. Can be supplemented with one magnetic stirrer or rod stirrer for use as separate titration stand. Freely selectable cylinder unit with 5, 10, 20 or 50 mL.

Optrode M2

Optical sensor for photometric titrations offering 8 different wavelengths. The wavelength can be switched using the software (tiamo 2.5 or higher) or with a magnet. The glass shaft is completely solvent-resistant and easy to clean. For example, this space-saving sensor is suitable for:

- Non-aqueous titrations in accordance with USP or EP
- Determinations of carboxyl end groups
- TAN/TBN in accordance with ASTM D974
- Sulfate determination
- Fe, Al, Ca in cement
- Water hardness
- Chondroitin sulfate in accordance with USP

The sensor is not suitable for determinations of concentrations via measurement of color intensity (colorimetry).

50 – 150 mL titration vessel

50 – 150 mL titration vessel with screw top for use with 6014050X0 titration vessel lids.