

Application Note AN-T-246

Standardization of cationic surfactants by argentometric titration

Accurate assay determination of TEGO®trant with silver nitrate including near-infrared (NIR) quantification model

A well-known frequently used cationic surfactant (TEGOtrant, also known as 1,3-didecyl-2-methylimidazolium chloride) is standardized in this Application Note. The stoichiometric chloride is titrated argentometrically with standardized silver nitrate, enabling an extremely precise determination of the assay. Therefore, the result determined is equivalent to the total surfactant content, allowing the accurate calculation of the cationic surfactant concentration.

Furthermore, a quantification model was developed that uses NIR measurements in addition to titration. This enables users to quickly determine cationic surfactant content using only NIR spectroscopy.

INTRODUCTION

There are no primary or secondary standard methods for titrating or determining the active substance content of anionic and cationic surfactants. A cationic titrant is standardized using an anionic titrant, and vice versa. In short, the result of this titration is a sum parameter usually specified as «total surfactant content». This value is then used to determine the content in the actual sample.
The major weakness of this type of standardization is the significant margin of error one must accept. Because the exact concentration of the titrant is unknown, conclusions about the analyzed sample can only be made with limited accuracy.

SAMPLE AND SAMPLE PREPARATION

This application is demonstrated on 25 different batches of TEGOtrant A100 (1,3-didecyl-2-methylimidazolium chloride). The sample is ground into a fine powder before analysis.

EXPERIMENTAL

The determination is carried out using an OMNIS Sample Robot S – WSM, an OMNIS Professional Titrator equipped with OMNIS Dosing Modules, as well as a dAg Titrode along with an OMNIS NIR Analyzer Solid (Figure 1).

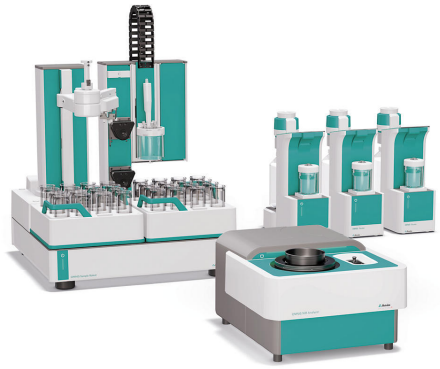


Figure 1. OMNIS Sample Robot S – WSM with OMNIS Professional Titrator and OMNIS Dosing Modules equipped with a dAg Titrode as well as an OMNIS NIR Analyzer Solid.

Titration

An appropriate amount of sample is weighed into the titration beaker, to which deionized water and a nitric acid solution are added. A titration is performed until after the first equivalence point with standardized silver nitrate.

NIR spectroscopy

A sufficient amount of sample is added to the small cup NIR accessory. This is placed in the corresponding holder which is already mounted on the OMNIS NIR Analyzer Solid.
Five determinations were carried out automatically in triplicate for each near-infrared measurement.

RESULTS

This method offers very accurate results, as displayed in Table 1. The comparative measurement of the control sample, summarized in Table 2, shows that the values from the titration and the NIR measurement differ by only 0.5%. Exemplary titration and NIR measurements of TEGOtrant are given in Figure 2 and Figure 3, respectively.

Table 1. Selected results of the potentiometric determination of TEGOtrant.

Sample (n = 3), batch number	Assay mean value in %	SD(rel) in %
20780065	96.16	0.3
11020053	94.11	0.1
11070002	95.65	0.3
00470397	95.19	0.1
00530513	93.71	0.3

Table 2. Summary of results for assay determination of the TEGOtrant «control sample» by titration and NIR.

Control sample, batch number	Assay mean value in %	SD(rel) in %
20650022 by titration (n = 6)	97.61	0.2
20650022 by NIR (n = 15)	98.13	0.3
Difference	0.52%	–

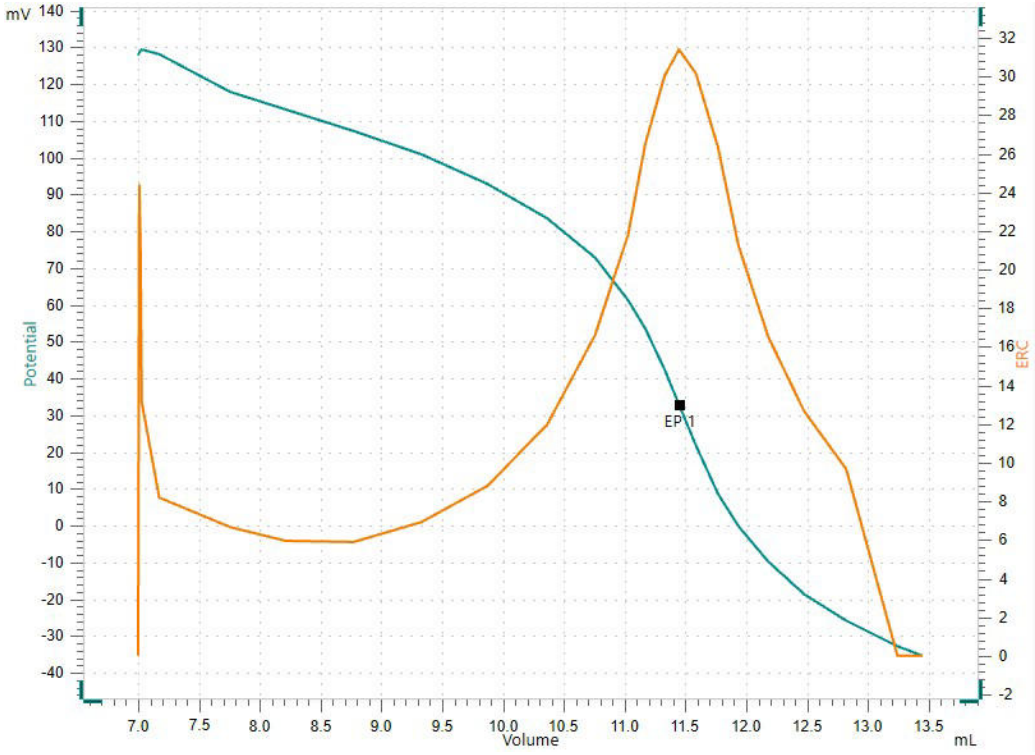


Figure 2. Titration curve of TEGOtrant with silver nitrate and the dAg Titrode.

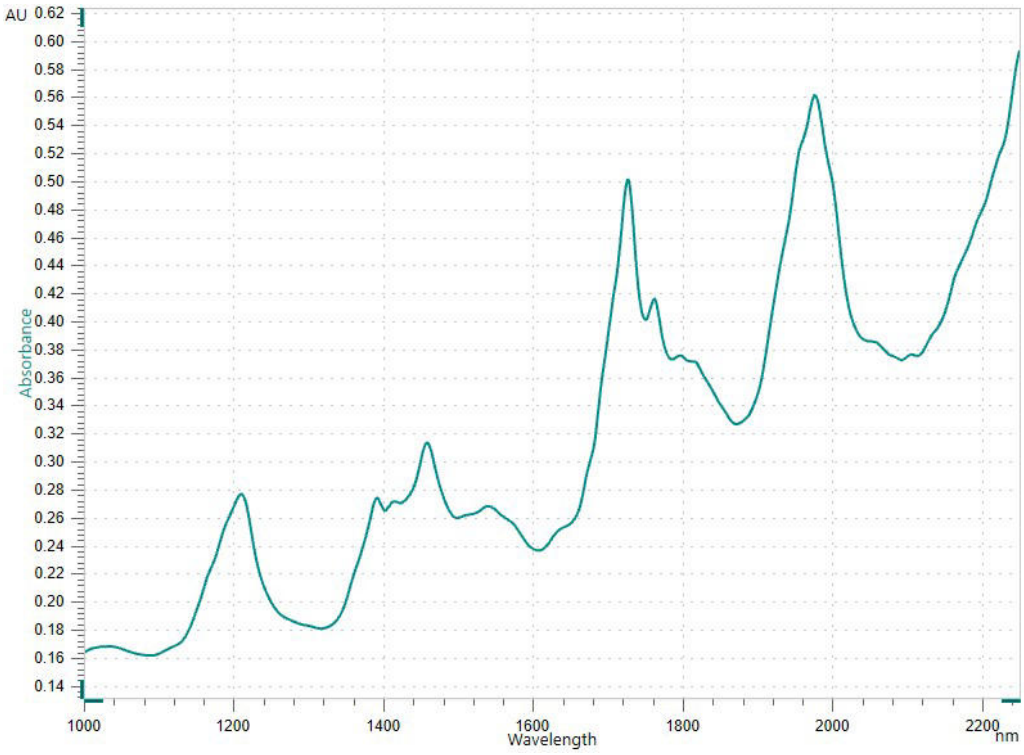


Figure 3. NIR spectrum of TEGOtrant between the wavelengths of 1000 nm and 2250 nm.

CONCLUSION

Potentiometric titration is an accurate and precise method that can be used to standardize the cationic surfactant TEGOtrant.

The OMNIS system used in this study is fully automated and enables fast and reliable cationic surfactant titration standardization. The argentometric determination with the digital Ag Titrode is highly accurate. Furthermore, the Ag_2S-coated silver ring increases sensitivity, delivering even better results.

When used with the corresponding OMNIS NIR Analyzer Solid, OMNIS Software can easily create a quantification model on a single platform, offering users real added value for the TEGOtrant standardization.

CONTACT

Metrohm Nordic AB
Box 11065
161 11 Bromma
mail@metrohm.se

CONFIGURATION



OMNIS Professional Titrator without stirrer

Innovative, modular potentiometric OMNIS Titrator for endpoint titration and equivalence point titration (monotonic/dynamic). Thanks to 3S Liquid Adapter technology, handling chemicals is safer than ever before. The titrator can be freely configured with measuring modules and cylinder units and can have a 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
- Can be supplemented with magnetic stirrer and/or 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.



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.



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.



OMNIS Sample Robot S – WSM (1T/2P)

OMNIS Sample Robot S – WSM, equipped with an OMNIS Workstation Module with 2 pumps for cleaning and extraction of the sensors and sample vessels, a workstation, rod stirrers, and extensive accessories for getting started directly with fully automated titration. The system provides space in two sample racks for 32 sample beakers of 120 mL each. This modular system is supplied completely installed and can thus be put into operation in a very short time. The system can also be extended upon request to include 2 additional peristaltic pumps and another workstation, thus doubling the throughput. If additional workstations are required, then the Sample Robot S can be extended to become an L-sized OMNIS Sample Robot, thus enabling samples from 7 racks to be processed in parallel on up to 4 workstations to quadruple the sample throughput.



dAg Titrode with Ag2S-coating

Digital, combined silver ring electrode for OMNIS with a pH glass membrane as reference electrode. The silver ring is coated with sulfide (Ag_2S) to increase the sensitivity and result in a better limit of determination.

The maintenance-free electrode is suitable for precipitation titration with a constant pH value (titrant: silver nitrate), e.g.

- Chloride, Bromide, Iodide
- Sulfide
- Dihydrogen sulfide
- Mercaptane
- Cyanide

This electrode is stored in deionized water.

dTrodes can only be used with OMNIS titrators and titration modules with digital measuring interface.



OMNIS NIR Analyzer Solid

Near-infrared spectrometer for solid and viscous samples.

Developed and produced in accordance with Swiss quality standards, the OMNIS NIR Analyzer is the near-infrared spectroscopy (NIRS) solution for routine analysis along the entire production chain. Its application of the latest technologies and its integration in the modern OMNIS Software are reflected in its speed, operability and flexible utilization of this NIR spectrometer.

Overview of the advantages of the OMNIS NIR Analyzer Solid:

- Measurements of solids and viscous samples in less than 10 seconds
- Automated multi-position measurements for reproducible results, even with nonhomogeneous samples
- Simple integration in an automation system or link with additional analysis technologies (titration)
- Supports numerous sample vessels