



Application Note AN-T-246

阳离子表面活性剂的银滴定标准法

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**).

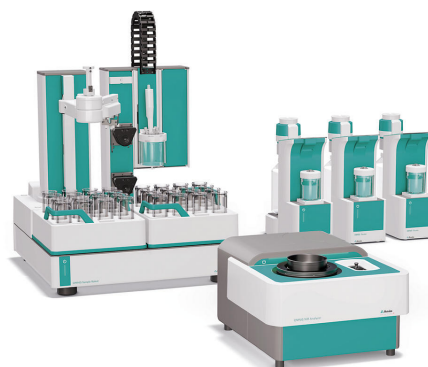


Figure 1. OMNIS Sample Robot S – WSM with OMNIS Professional Titrator and OMNIS Dosing Modules equipped with a dAg Titrode as well as an OMNIS NIR Analyzer Solid.

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.

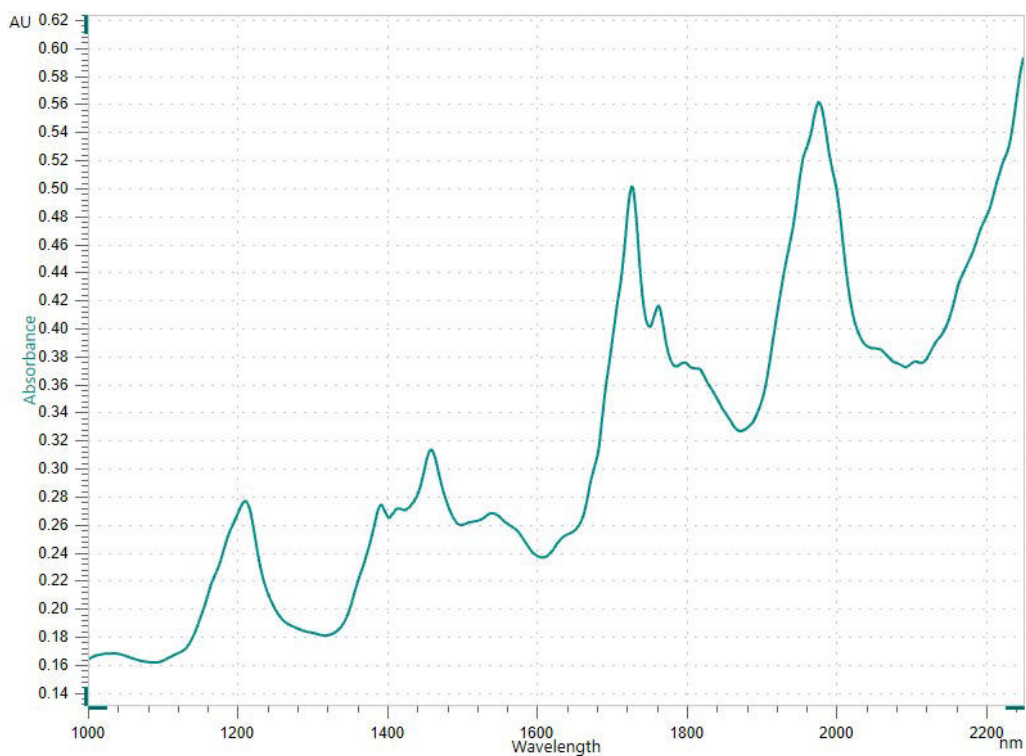


Figure 3. NIR spectrum of TEGOtrant between the wavelengths of 1000 nm and 2250 nm.

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

Ag₂S-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

瑞士万通中国
北京市海淀区上地东路1号
院1号楼7层702
100085 北京

marketing@metrohm.com.cn

CONFIGURATION



OMNIS Professional Titrator

创新型、模块式电位分析 OMNIS Titrator,用于进行终点和等当点滴定(单一/动态)。由于采用 3S OMNIS Liquid Adapter 技术,处理化学品从未像现在一样安全。可以使用测量模块和计量管单元自由配置滴定仪,并在需要时扩展一台搅拌器。包括用于使用其他滴定或加液模块平行滴定的“Professional”功能许可证。

- 通过计算机或本地网络控制
- 可以为其他应用或辅助溶液另外连接最多四个滴定模块或加液模块
- 可以扩展磁力搅拌器和/或棒式搅拌器
- 可提供不同大小的计量管:5、10、20 或 50 mL
- 采用 3S 技术的 OMNIS Liquid Adapter:安全处理化学品,自动传输生产厂家的原始试剂数据

测量模式和软件选项:

- 终点设定滴定:“Basic” 功能许可证
- 终点和等当点滴定(单一/动态):“Advanced” 功能许可证
- 终点和等当点滴定(单一/动态),包括 5 路平行滴定:“Professional” 功能许可证



OMNIS Dosing Module

用于与 OMNIS Titrator 滴定仪相连的加液模块,以扩展额外用于滴定/加液的滴定管。可以扩展磁力搅拌器和/或螺旋搅拌器,以作为单独的滴定台使用。可自由选择 5、10、20 或 50 mL 计量管单元。



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OMNIS Sample Robot S – WSM (1T/2P)

OMNIS Sample Robot S – WSM 配备 1 个 OMNIS Workstation Module,包括 2 台用于清洁和抽吸传感器及样品容器的泵、1 个工作站、棒式搅拌器以及丰富的全自动滴定直接入门附件。该系统的 2 个样品架提供了容纳 32 只样品杯,每只 120 mL 的位置。这套模块化系统在完整安装状态下提供,因此可以在最短时间内启动。根据要求还可以为系统升级另外 2 台蠕动泵以及另一个工作站,以使吞吐量加倍。如果需要其它工作站,Sample Robot S 可升级为 L 尺寸的 OMNIS Sample Robot,这样可以在最多 4 个工作站上同时加工 7 个样品盘的样品,达到四倍样品吞吐量。



dAg Titrode Ag2S

带 pH 玻璃膜的 OMNIS 用数字组合式银环形电极,用作参比电极。银环具有硫化物 (Ag_2S) 涂层,其适用于较高的灵敏度和更好的指示极限。

该免维护电极适用于 pH 值恒定的沉积滴定(硝酸银滴定剂),例如:

- 氯化物、溴化物、碘化物
- 硫化物
- 硫化氢
- 硫醇
- 氰化物

该电极存放在蒸馏水中。

dTodes 可在 OMNIS Titratoren 上使用。



OMNIS NIR Analyzer Solid

适合固体和粘性样品的近红外光谱仪。

OMNIS NIR Analyzer 是一种按照瑞士质量标准开发和生产的近红外光谱 (NIRS) 解决方案,用于整个生产链的常规分析。使用新技术和嵌入先进 OMNIS Software 反应在该 NIR 光谱仪的速度、可操作性和灵活使用上。

OMNIS NIR Analyzer Solid 的优点概览:

- 在 10 秒以内测量固体和粘性样品
- 自动化多位置测量,即使在样品不均匀时,也能够获得可重现的结果
- 方便地嵌入自动系统,或者与其它分析技术(滴定)关联
- 支持大量样品容器