Application Note AN-T-203

挥发性溶剂和化学中间体中的酸度

Objective and reliable determination according to ASTM D1613

The presence of acidic components in volatile solvents is due to contamination of the solvents, or through their decomposition during storage, distribution, or manufacture. Increased acid content in solvents could lead to a variety of problems like shorter storage stability or chemical corrosion, which can damage costly company assets and lead to shutdowns.
Using the Optrode for indication, the acidity is determined according to **ASTM D1613** by photometric titration with sodium hydroxide as titrant and phenolphthalein as indicator. The Optrode enables an objective recognition of the end point enhancing the accuracy of the results.

SAMPLE AND SAMPLE PREPARATION

The analysis is demonstrated on 4-methyl-2-pentanone (MIBK), 2-ethoxyethyl acetate, and 2-ethoxyethanol.

EXPERIMENTAL

The analysis is carried out fully automatically on an OMNIS system consisting of an OMNIS Sample Robot, an OMNIS Advanced Titrator, and an OMNIS Dosing Module. The Optrode is used for indication of the end point.

For water-soluble samples (e.g., 2-ethoxyethanol), an appropriate amount of sample is pipetted into the sample beaker. While stirring, deionized water is dosed and phenolphthalein indicator solution is pipetted into the sample beaker. The solution is then titrated with standardized sodium hydroxide until after the end point is reached. After each titration, the titrated solution is aspirated and the buret tips as well as the sensor are rinsed with deionized water in the sample beaker.

For samples that are not completely miscible in water (e.g., MIBK or 2-ethoxyethyl acetate), the same procedure is used as for water-soluble samples with the exception that carbon-dioxide free ethanol is used instead of deionized water.

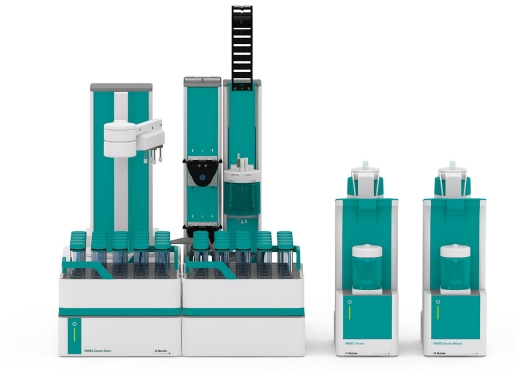

Figure 1. OMNIS system consisting of an OMNIS Sample Robot, an OMNIS Advanced Titrator, and an OMNIS Dosing Module.

RESULTS

Color changes are produced which are detected objectively with the Optrode and reliably evaluated by the OMNIS Software.

Table 1. Overview of the results obtained for 4-methyl-2-pentanone (MIBK), 2-ethoxyethyl acetate, and 2-ethoxyethanol (n = 6).

Substance	Mean value / %	SD(abs) / %	SD(rel) / %
4-methyl-2-pentanone	0.0151	$6.0\ 10^{-5}$	0.4
2-ethoxyethyl acetate	0.012	$8.0\ 10^{-5}$	0.7
2-ethoxyethanol	0.0003	$1.6\ 10^{-5}$	5.2

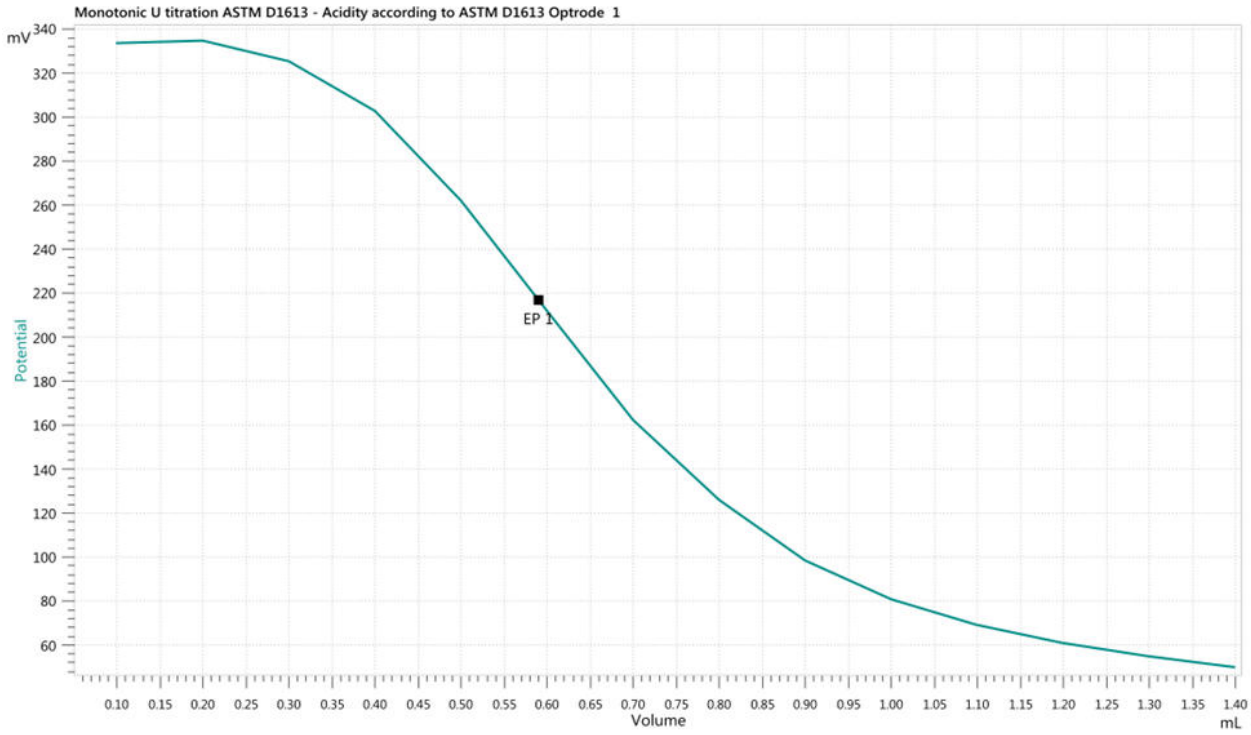

Figure 2. Titration curve showing the determination of acidity in volatile solvents according ASTM D1613.

CONCLUSION

This method shows the possibility to determine very low acidity according to ASTM D1613. The standard deviation is acceptable even at very low acidities due to the objective and reproducible detection of the color change by the Optrode.

With the possibility to easily change between 8 different wavelengths, the Optrode can be used for other applications. The fully automated OMNIS system allows to enhance the throughput of the analyses and enabling the best possible results.

Internal reference: AW TI CH1-1254-112018

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CONFIGURATION

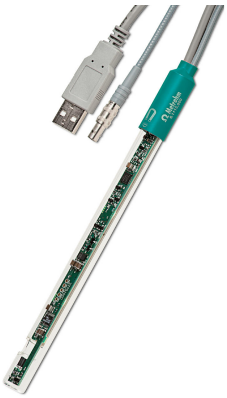
OMNIS Advanced Titrator

创新型、模块式电位分析 OMNIS Titrator 滴定仪,适于独立运行或作为 OMNIS 滴定系统的核心元件运行,用于使用 OMNIS Sample Robot 进行终点和等当点滴定(单一/动态)。由于采用 3S 试剂瓶适配器技术,处理化学品很安全。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螺旋搅拌器。在需要时可以通过相应的软件功能许可证为平行滴定升级 OMNIS Advanced Titrator。

- 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四个滴定模块或加液模块
- 螺旋搅拌器的连接方式
- 可提供不同大小的计量管:5、10、20 或 50 mL
- 采用 3S 技术的试剂瓶适配器:安全处理化学品,自动传输生产商的原始试剂数据

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),包括平行滴定:“Professional” 功能许可证



Optrode

有 8 种可用波长的光度滴定用光学传感器。可以通过软件控制(tiamo 2.5 及以上版本)或通过磁铁来进行波长切换。该玻璃鞘对溶剂耐受,并且易于清洁。这种节省空间的传感器适用于,例如:

- 按照 USP 或 EP 的非水溶性滴定
- 羧基端基的测定
- TAN/TBN 根据 ASTM D974
- 硫酸测定
- 混凝土中的 Fe、Al、Ca
- 水硬度
- 根据 USP 的硫酸软骨素

传感器不适合通过测量颜色强度(比色法)来测定浓度。