

Application Note AN-T-203

Acidity in volatile solvents and chemical intermediates

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.

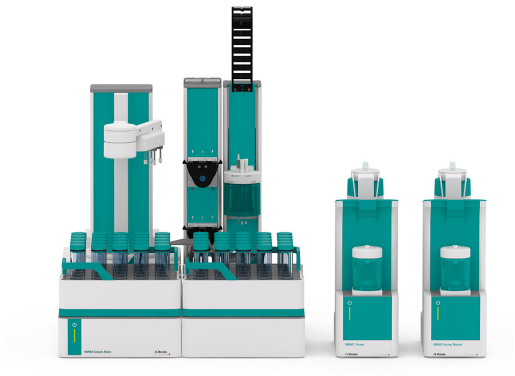


Figure 1. OMNIS system consisting of an OMNIS Sample Robot, an OMNIS Advanced Titrator, and an OMNIS Dosing Module.

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	$6.0 \cdot 10^{-5}$	0.4
2-ethoxyethyl acetate	0.012	$8.0 \cdot 10^{-5}$	0.7
2-ethoxyethanol	0.0003	$1.6 \cdot 10^{-5}$	5.2

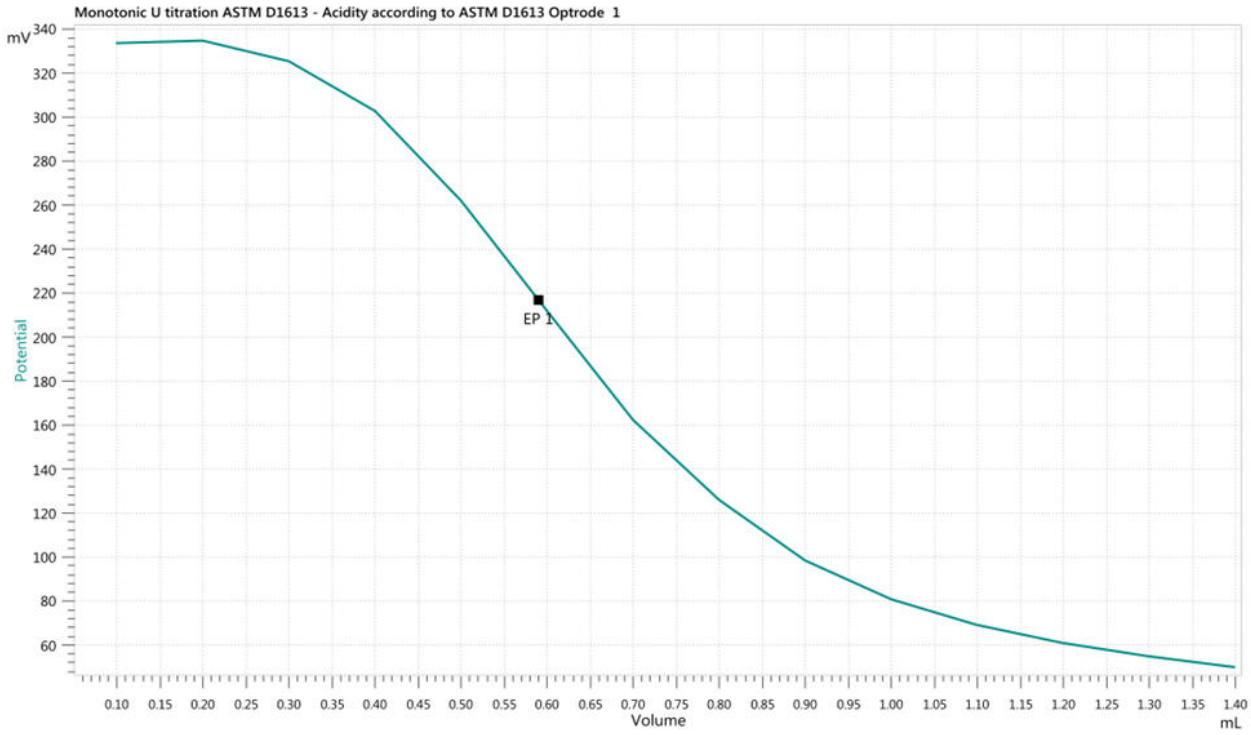


Figure 2. Titration curve showing the determination of acidity in volatile solvents according ASTM D1613.

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

CONTACT

Metrohm SG
31 Toh Guan Road East, LW
Technocentre, #06-08
608608 Singapore
info@metrohm.com.sg

CONFIGURATION



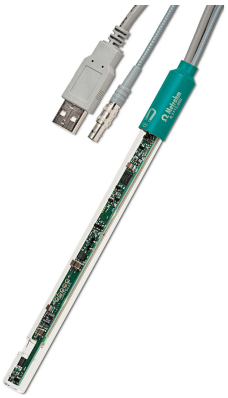
OMNIS Advanced Titrator with magnetic stirrer

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. If required, the OMNIS Advanced Titrator can be equipped for parallel titration via a corresponding software function license.

- Control 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: Secure 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 parallel titration: "Professional" function license



Optrode

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).