Application Note AN-T-221

SET titration of HPLC mobile phases

Automated timesaving pH adjustment of semi-aqueous media

High pressure liquid chromatography (HPLC) requires the use of a mobile phase – mostly consisting of semi-aqueous media. These kinds of media are challenging to titrate as the electrodes behave differently compared to when working with aqueous media. Laboratory analysts often remark that manual pH adjustment using a pH electrode is very time-consuming, resulting in long waiting times between additions until a stable pH is reached.

This Application Note presents the automatic pH adjustment of a mixture of acetonitrile, water, and triethylamine using a Metrohm titrator. The time required to adjust the mobile phase pH has decreased from hours to approximately 10 minutes with the described setup. Additionally, the pH value at the end of the adjustment as well as the volume of titrant used can be automatically documented and traced for auditing purposes.

For accurate endpoint indication, the EtOH-Trode was used. This electrode has been specially designed to measure pH in nonaqueous solutions owing to its double junction system and special membrane glass.

SAMPLE AND SAMPLE PREPARATION

This application is demonstrated on a solvent mixture composed of 1600 mL acetonitrile, 400 mL deionized water, and 10 mL triethylamine.

EXPERIMENTAL

The analyses were carried out on an Eco Titrator in combination with the EtOH-Trode (Figure 1). The determinations were performed on 200 mL aliquots of the solvent mixture.

The adjustment of the pH value was achieved within a matter of minutes, whereas manual pH adjustment took hours to complete. The obtained pH at the end of the titration was stable and reproducible.

Table 1. Volume needed for the adjustment of the pH value of 200 mL solvent mixture.

	pH 10	pH 7
Titrant volume	0.15 mL	0.95 mL

CONCLUSION

This application example shows how easily manual pH adjustments can be automated by choosing the right device and electrode. Moreover, automation offers significant benefits for laboratories including time savings, increased precision, economical analysis, and traceability.

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CONFIGURATION

Eco Titrator

The compact Eco Titrator with integrated magnetic stirrer and touch-sensitive User Interface is ideal for routine analysis. It provides GLP-compliant results with minimum space requirements at all times (approx. DIN A4).

Universally compatible with almost all potentiometric titrations, such as, for example, for

- Food products: Acid content, chloride, Vitamin C, iodine and peroxide number in fats
- Water analysis: Carbonate and Ca/Mg hardness, chloride, sulfate, permanganate index
- Petrochemistry: Acid/base number, sulfide & mercaptans, chloride, bromine number
- Electroplating: Total acid, metal content, chloride
- Surfactant analysis: Anionic, cationic and non-ionic surfactants
- Photometry with the Optrode: p and m value, metals, water hardness

EtOH-Trode

Combined pH electrode with double-junction system for pH measurements in nonaqueous media (e.g., for pHe in ethanol).
The electrode is equipped with a fixed ground-joint diaphragm which is insensitive to contamination, and the bridge electrolyte can be freely chosen (aqueous or nonaqueous).
When c(KCl) = 3 mol/L is used as bridge electrolyte, storage in storage solution is recommended. When a different bridge electrolyte is used, storage in the respectively used bridge electrolyte solution is recommended.
The two chambers for reference electrolyte ("INNER FILLING") and bridge electrolyte ("OUTER FILLING") are each filled with c(KCl) = 3 mol/l at the time of delivery.