



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.



Figure 1. Eco Titrator equipped with a EtOH-Trode for fast adjustment of the pH value.

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.

Internal reference: AW TI DE1-0810-032021

CONTACT

瑞士万通中国
北京市海淀区上地路1号院
1号楼7702
100085 北京

marketing@metrohm.com.cn

CONFIGURATION



Eco Titrator

型 Eco Titrator 具有内置磁力拌器和触摸感式用界面,是日常分析的理想。其始提供符合 GLP 准的果,并且具有最小的空需求(DIN A4)。

普遍用于几乎所有位分析式滴定,例如:

- 食品:脂肪的酸度、化物、生素 C、和化物
- 水分析:酸和 Ca/Mg 硬度、化物、硫酸、高酸指数
- 石化:酸/、硫化物和硫醇、化物、
- :酸度、金属含量、化物
- 表面活性分析:子、子和非子表面活性
- 光度与光度:p 和 m、金属、水硬度



EtOH-Trode

用于非水介(例如,用于乙醇中的 pHe)中 pH 量的双系的 pH。

配有防染的固定磨口隔膜,可以自由中解(水性或非水性)。

当使用 $c(\text{KCl}) = 3 \text{ mol / L}$ 作外参比液解,建在保存液中存。当使用其他外参比液解,建存放在相用的解液中。

。个用于内参比液解 ("INNER FILLING") 和外参比液解 ("OUTER FILLING") 的腔体在交付分填充有 $c(\text{KCl}) = 3 \text{ mol/l}$ 。