



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

メトロームジャパン株式会社
143-0006 東京都大田区平
和島6-1-1
null 東京流通センター アネ
ックス9階

metrohm.jp@metrohm.jp

CONFIGURATION



Eco Titrator

ヒルトインのマクネチックスターラおよびタッチセンサーユーザーインターフェース付きのコンパクトな Eco Titrator は、ルーチン分析に理想的です。これは、いかなるときもGLPに適合した結果を、最小限のスペース要件 (およそ DIN A4) にて提供します。

ほぼすべての電位差滴定にて汎用的に使用可能。たとえば

- 食品: 酸性度、塩化物、ヒタミンC、油脂のヨウ素価および過酸化値
- 水質分析: 炭酸塩硬度およびCa/Mg硬度、塩化物、硫酸、過マンガン酸塩指数
- 石油化学: 酸/塩基価、硫酸塩およびメルカフタン、塩化物、臭素価
- 電気めっき: 総酸価、金属含有量、塩化物
- 界面活性剤分析: 陰イオン、陽イオン、および非イオン界面活性剤
- Optrodeを用いた測光: p値およびm値、金属、水硬度



EtOH-Trode

非水媒質における pH 測定 (例えばエタノール中の pH e など) のためのタフルジャンクションシステムを備えた複合 pH 電極。

電極は、汚れに耐性のある固定クラントポイントタイアフラムを装備しており、塩橋は自由に選択することかてきます (水性または非水性)。

塩橋として $c(\text{KCl}) = 3 \text{ mol/L}$ を使用する場合、保存液中での保存が推奨されます。その他の塩橋を使用する場合、使用するそれぞれの電解液中での保存が推奨されます。

出荷時、参照内部液 ("INNER FILLING") と塩橋 ("OUTER FILLING") 用の両方のチャンハには、 $c(\text{KCl}) = 3 \text{ mol/l}$ が充填されています。