



Application Note AN-T-239

弱的高酸非水滴定

Fast and accurate determination of the amine value with perchloric acid by nonaqueous titration

Amines are weak bases and are used in industrial chemical processes as well as in pharmaceuticals. An important parameter and quality indicator is the amine value (expressed in mg KOH/g sample). Weak bases such as amines can be titrated in aprotic solvents with good accuracy if a very strong acid is used as the titrant. The strongest acid in nonaqueous media is perchloric acid in glacial acetic acid.

SAMPLE AND SAMPLE PREPARATION

This application is demonstrated on triethanolamine. Sample preparation is not

The solvent used also plays an important role during the analysis. Acetic acid with <1% water content is an excellent solvent for weak bases. This Application Note presents the nonaqueous perchloric acid titration of amines. Specifically, the concentration of triethanolamine (TEOA) is determined by titration with perchloric acid in glacial acetic acid using a Solvotrode easyClean and lithium chloride in ethanol as electrolyte.

required.

EXPERIMENTAL

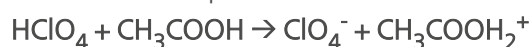
The determinations are carried out on an Eco Titrator equipped with a Solvotrode easyClean

with integrated Pt1000 temperature sensor (Figure 1).

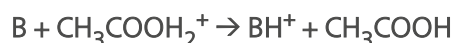


Figure 1. Eco Titrator equipped with a Solvotrode easyClean with integrated Pt1000 temperature sensor.

Perchloric acid (HClO_4) reacts with acetic acid (CH_3COOH) to form a strong acidium ion, as shown in this equation:



Then the weak base (B) reacts via the following neutralization reaction mechanism:



The strong acidium ion guarantees a clear

voltage jump and equivalence point even when titrating weak bases.

An appropriate amount of sample is weighed into the titration beaker, and then glacial acetic acid is added. Afterwards, the solution is titrated until after the first equivalence point with standardized perchloric acid in glacial acetic acid.

RESULTS

This method offers very accurate results, as displayed in **Table 1**. An exemplary titration curve of TEOA with HClO_4 is given in **Figure 2**,

showing the sharp jump in voltage at the equivalence point.

Table 1. Results of the potentiometric titration of triethanolamine (n=6).

Sample (n=6)	Amine value (mg KOH/g)	Recovery (%)
Mean value	375.42	99.83
SD(abs)	2.79	0.74
SD(rel) in %	0.7	0.7

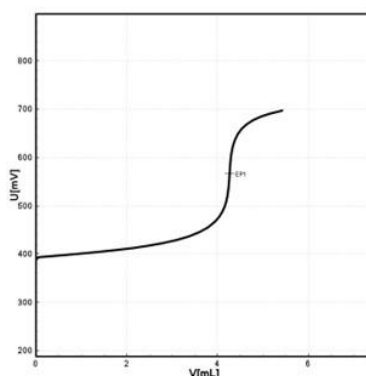


Figure 2. Perchloric acid titration curve with one equivalence point.

CONCLUSION

It is not always possible to titrate weak acids or bases accurately in aqueous solvents. The samples are often insoluble in water, or the potential of the titration is too low for a clear evaluation. In such cases, nonaqueous titrations are recommended. One example of this is perchloric acid titration in glacial acetic acid for weakly basic substances.

The compact Eco Titrator with integrated

magnetic stirrer and touch-sensitive user interface is ideal for nonaqueous titration of weak bases with perchloric acid. The system offers user-friendly handling at an attractive price point. The Eco Titrator is compact and takes up minimum lab bench space. Pre-installed methods make it easy for users without laboratory experience to quickly get precise, reliable, GLP-compliant results.

CONTACT

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CONFIGURATION



Eco Titrator Acid/Base

型 Eco Titrator 具有内置磁力拌器和触摸感式用界面,可以足日常分析需求。其始提供符合 GLP 准的果。

Eco Titrator Acid/Base 提供了用于在水性溶液中酸滴定的完整套件。套件包括了滴定、一个 20 mL 量管元以及一个合式 pH Ecotrode plus。



Solvotrode easyClean 1.2 m

活的 easyClean 隔膜 的合 pH 和固定 (1.2 m) 用于所有非水性酸/滴定。

玻璃膜性差的溶液行了化,并且由于易于清的 easyClean 隔膜,也用于非常的品(例如:旧油)。

可与非水参比解(化或四乙基化)一起使用。存在相的参比解中。

物品号 6.0229.020 的 Solvotrode easyClean 同有 2.0 m 固定度的版本可供。