



Application Note AN-T-239

弱碱的高氯酸非水滴定

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.

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.

SAMPLE AND SAMPLE PREPARATION

This application is demonstrated on triethanolamine. Sample preparation is not

required.

EXPERIMENTAL

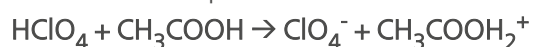
The determinations are carried out on an Eco Titrator equipped with a Solvotrode easyClean

with integrated Pt1000 temperature sensor (Figure 1).

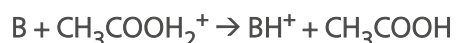


Figure 1. Eco Titrator equipped with a Solvotrode easyClean with integrated Pt1000 temperature sensor.

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

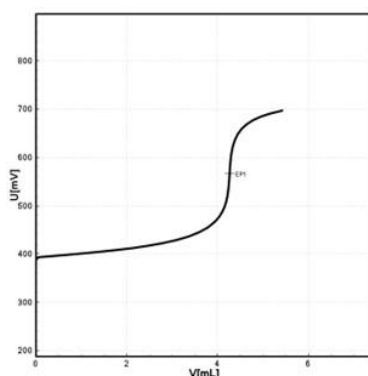


Figure 2. Perchloric acid titration curve with one equivalence point.

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

紧凑型 Eco Titrator 具有内置磁力搅拌器和触摸感应式用户界面,可以满足日常分析需求。其始终提供符合 GLP 标准的结果。

Eco Titrator Acid/Base 提供了用于在水性溶液中酸碱滴定的完整套件。该套件包括了滴定仪、一个 20 mL 计量管单元以及一个组合式 pH 电极 Ecotrode plus。



Solvotrode easyClean 1.2 m

带灵活的 easyClean 隔膜的组合 pH 电极和固定电缆 (1.2 m) 用于所有非水性酸/碱滴定。

玻璃膜针对导电性差的溶液进行了优化,并且由于易于清洁的 easyClean 隔膜,该电极也适用于非常脏的样品(例如:旧油)。

电极可与非水参比电解质(氯化锂或四乙基溴化铵)一起使用。储存在相应的参比电解质中。

物品编号为 6.0229.020 的 Solvotrode easyClean 同样有 2.0 m 固定电缆长度的版本可供选购。