



Application Note AN-T-239

Nonaqueous titration of weak bases with perchloric acid

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

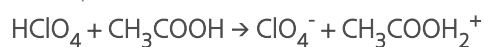
The determinations are carried out on an Eco Titrator equipped with a Solvotrode easyClean with

integrated Pt1000 temperature sensor (Figure 1).

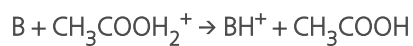


Figure 1. Eco Titrator equipped with a Solvotrode easyClean with integrated Pt1000 temperature sensor.

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

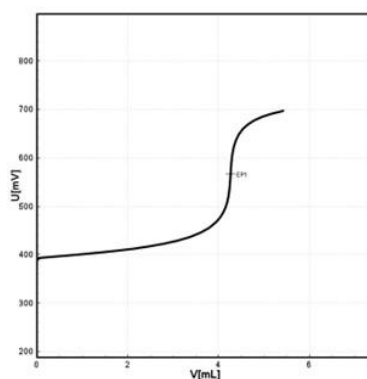


Figure 2. Perchloric acid titration curve with one equivalence point.

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

Metrohm AG
Ionenstrasse
9100 Herisau

info@metrohm.com

CONFIGURATION



Eco Titrator Acid/Base

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).

The Eco Titrator Acid/Base offers you the complete package for acid-base titration in aqueous solutions. Included in the package are titrators, a 20 mL cylinder unit, and a combined Ecotrode plus pH electrode.



Solvotrode easyClean (fixed cable 1.2 m)

Combined pH electrode with flexible easyClean diaphragm and fixed cable (1.2 m) for all non-aqueous acid/base titrations.

The glass membrane is optimized for poorly conducting solutions, and thanks to the easy-to-clean easyClean diaphragm, the electrode is also well suited for heavily contaminated samples, such as used oil.

This electrode can be used with non-aqueous reference electrolytes (lithium chloride or tetraethylammonium bromide). Storage in the respective reference electrolyte.

The Solvotrode easyClean is also available under the article number 6.0229.020 with a fixed cable length of 2.0 m.