



Application Note AN-T-042

Citric and oxalic acid in mixtures

Reliable potentiometric titration using a correction factor

Citric acid and oxalic acid are present in many products, such as foods or chemical solvents (e.g., decontamination solutions). Both acids are reducing agents, and citric acid is additionally a powerful antioxidant.

Both of these acids can be individually determined using titration. However, to determine their concentrations in mixtures, a content calculation is only possible with

correction factors for each acid due to their mutual impact (buffer effect).

A fast and accurate determination of these acids in various mixtures by potentiometric titration using the dEcotrode plus and sodium hydroxide as titrant can be realized. This Application Note explains more about this fast and easy analysis with reliable, automated titration instruments from Metrohm.

SAMPLE AND SAMPLE PREPARATION

The analysis is demonstrated on a mixture of citric acid and oxalic acid (β (citric acid) = 20 g/L

and β (oxalic acid) = 20 g/L).

No sample preparation is required.

EXPERIMENTAL

The analyses are carried out fully automatically on a OMNIS Sample Robot S in combination with an OMNIS Advanced Titrator and the dEcotrode plus for indication.

The sample solution is transferred into a sample beaker and deionized water is added. The solution is titrated with standardized sodium hydroxide until after the second equivalence point. After each titration, the solution is aspirated and the electrode is then rinsed with deionized water.



Figure 1. OMNIS System consisting of an OMNIS Sample Robot S and an OMNIS Advanced Titrator.

RESULTS

Reproducible titration curves (see **Figure 2**) are obtained for all analyses. The first equivalence point corresponds to oxalic acid and the second to citric acid. However, the two obtained equivalence points influence each other due to the close pKa values of the acids (oxalic acid = 1.25 and 4.14, citric acid = 3.13, 4.76, and 6.39).

Therefore, a correction factor is required for the titration. The correction factors used for this sample are 0.904 for citric acid, and 1.11 for oxalic acid.

The automated analysis leads to reproducible results with a RSD < 1.5% as shown in **Table 1**.

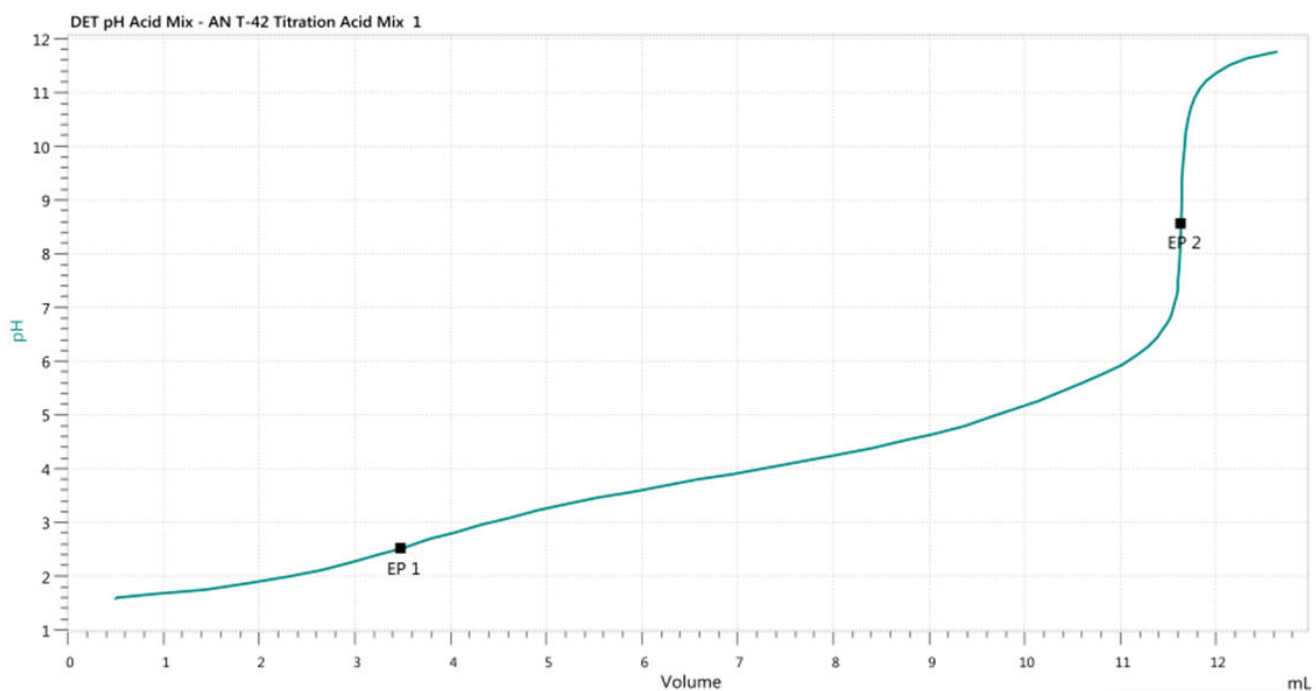


Figure 2. Titration curve of the determination of a mixture of citric and oxalic acid. The first equivalence point corresponds to oxalic acid, and the second to citric acid.

Table 1. Results of the determination of the mixture of β (citric acid) = 20 g/L and β (oxalic acid) = 20 g/L ($n = 5$).

Acid	Mean value / (g/L)	SD(abs) / (g/L)	SD(rel) / %
Citric acid	19.68	0.26	1.3
Oxalic acid	19.59	0.14	0.7

CONCLUSION

As a non-linear-correlation between the correction factors and the ratio of citric acid and oxalic acid exists, it is recommended to determine the correction factors at the expected ratio of citric and oxalic acid using standard solutions.

However, this method provides an easy and fast way to determine the citric acid and oxalic acid content in mixtures by potentiometric titration. The determination of the factors can be done automatically using the OMNIS system.

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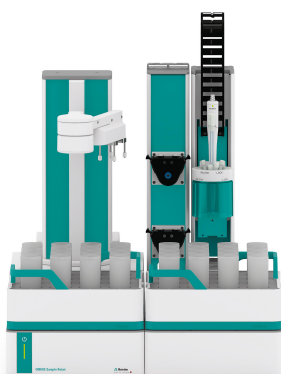
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CONFIGURATION



OMNIS Titrator Food

OMNIS Titrator Foodは、水性の酸/塩基滴定のための完璧なパッケージを提供します。パッケージには、マグネチックスター付きOMNIS Advanced Titrator、20 mLのシリンダーユニット、水性の酸/塩基滴定のためのd-Edotrode plus、およびOMNISソフトウェアのスタンドアローンライセンスが含まれています。



OMNIS Sample Robot S Pick and Place

ポンプモジュール「Peristaltik」(2 チャンネル)、Pick&Place モジュール、および全自動滴定を直に導入するための多種の付属品のついた OMNIS Sample Robot S。このシステムでは2つのサンプルラックにおいて、120 mL のサンプルビーカー32 個分のスペースが設けられており、モジュール式のシステムは、完全に組み立てられた状態で納品され、よって最短時間で稼働させることができます。

ご要望に応じて、システムには更に2つのペリスタリックポンプおよび追加の Pick&Place モジュールを装備することができ、スループットを倍増することができます。更なるワークステーションが必要な場合、この Sample Robot は L サイズまでの OMNIS Sample Robot に拡張することで最高4 つまでの Pick&Place モジュールにおいて7つのラックのサンプルを並行して処理し、スループットを4倍に増やすことができます。