



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 ($\beta(\text{citric acid}) = 20 \text{ g/L}$ and

$\beta(\text{oxalic acid}) = 20 \text{ g/L}$).

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**.

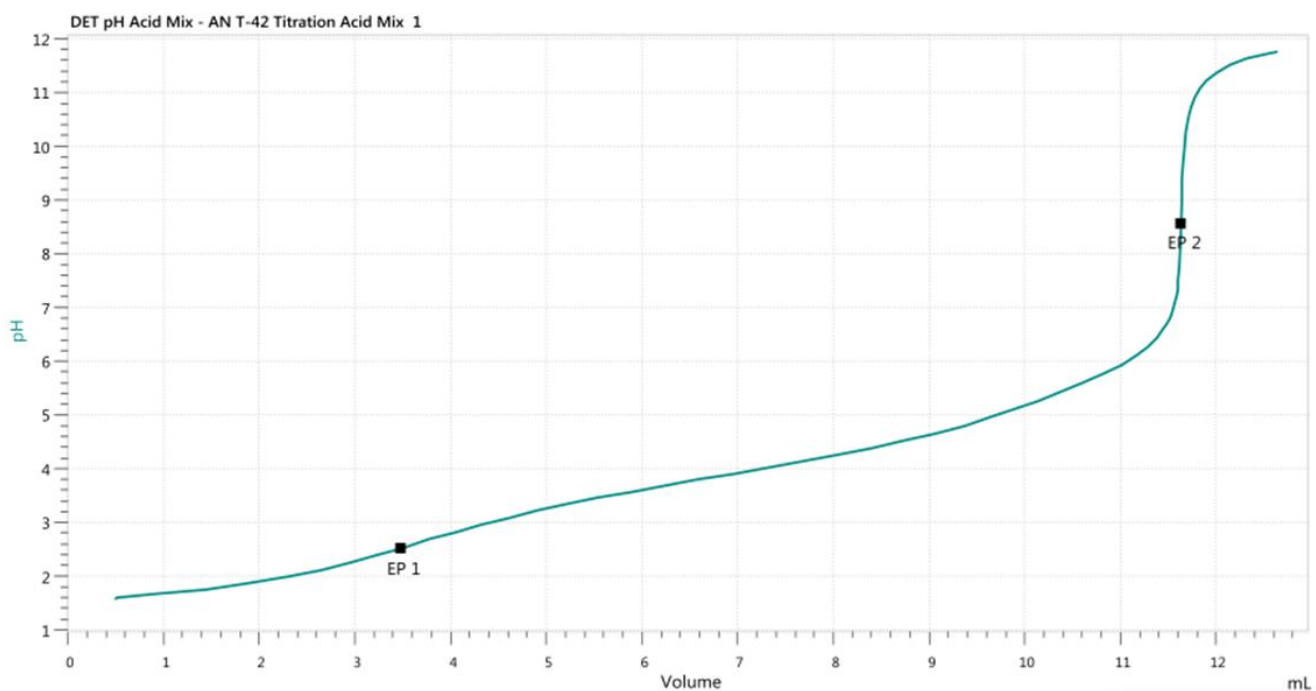


Figure 2. Titration curve of the determination of a mixture of citric and oxalic acid. The first equivalence point corresponds to oxalic acid, and the second to citric acid.

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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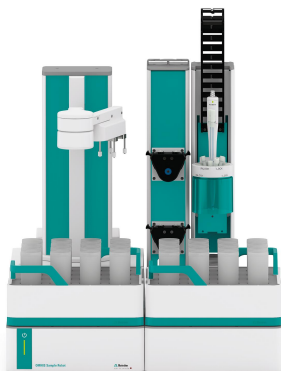
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CONFIGURATION



OMNIS Titrator Food

L'OMNIS Titrator Food vous offre un ensemble complet pour le titrage acide-base en milieu aqueux. Cet ensemble contient le titreur OMNIS Advanced Titrator avec un agitateur magnétique, une unité de cylindre de 20 mL, une d-Ecrotrode plus pour le titrage acide-base en milieu aqueux et le logiciel OMNIS Software avec une licence « standalone ».



OMNIS Sample Robot S Pick and Place

OMNIS Sample Robot S avec un module de pompe « péristaltique » (2 canaux) et un module Pick&Place et de nombreux accessoires pour un accès direct au titrage entièrement automatisé. Le système offre, dans ses deux racks d'échantillons, de la place pour 32 béchers d'échantillon de 120 mL. Ce système modulaire est livré entièrement monté et peut donc être mis en service dans un temps record.

Sur demande, il est encore possible d'ajouter au système deux pompes péristaltiques et un autre module Pick&Place, ce qui permet de doubler le débit. Si d'autres stations de travail sont nécessaires, ce Sample Robot peut évoluer jusqu'à la taille L de l'OMNIS Sample Robot. Les échantillons de sept racks peuvent ainsi être traités en parallèle sur quatre modules Pick&Place maximum, ce qui multiplie par quatre le débit d'échantillons.