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.

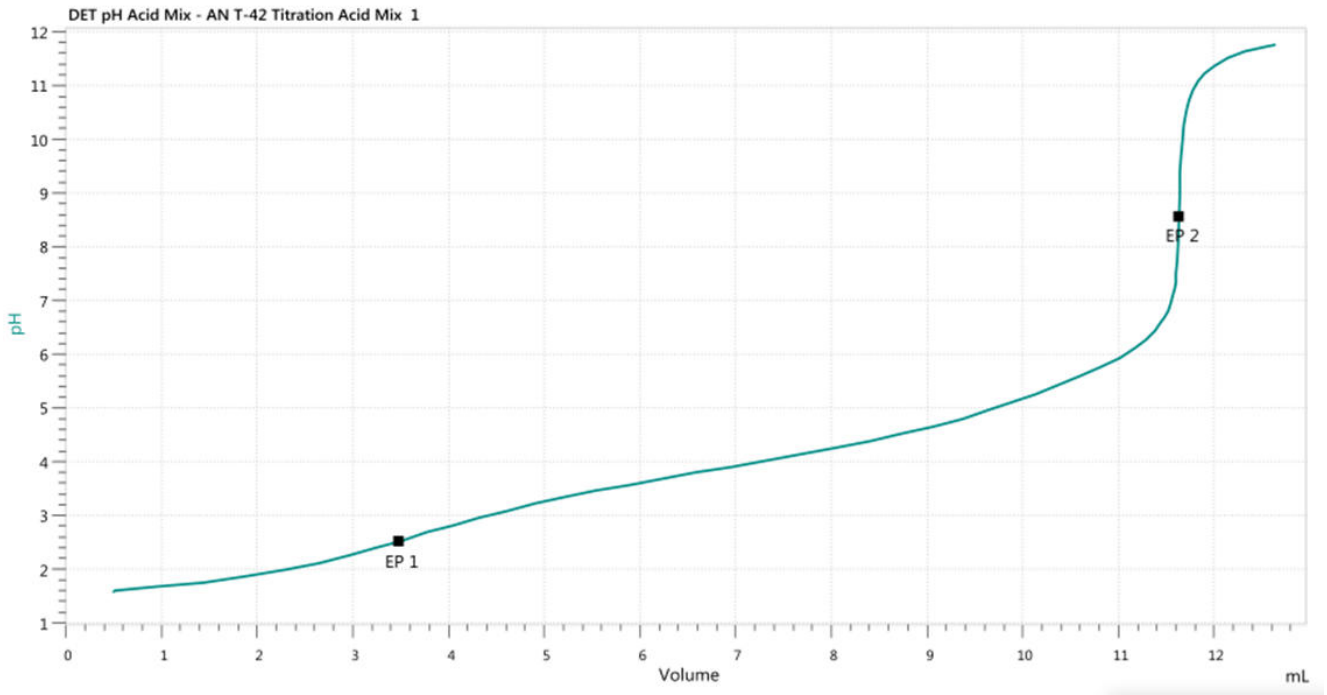

Figure 2. Titration curve of the determination of a mixture of citric and oxalic acid. The first equivalence point corresponds to oxalic acid, and the second to citric acid.

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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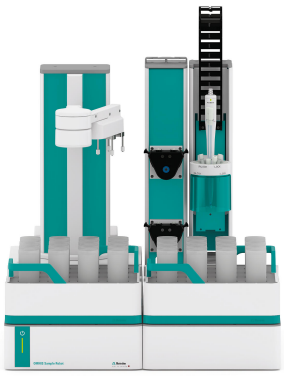
CONFIGURATION

OMNIS Titrator Food

The OMNIS Titrator Food offers you the complete package for aqueous acid-base titration. The package contains the OMNIS Advanced Titrator with magnetic stirrer, a 20 mL cylinder unit, a d-Ecotrode plus for aqueous acid-base titration and one stand-alone OMNIS Software license.



OMNIS Sample Robot S Pick and Place

OMNIS Sample Robot S with a "Peristaltic" (2-channel) pump module and a Pick&Place module in addition to extensive accessories for the direct transition to fully automatic titration. The system provides space in two sample racks for 32 sample beakers of 120 mL each. This modular system is supplied completely installed and can thus be put into operation in a very short time.

The system can also be extended upon request to include two additional peristaltic pumps and another Pick&Place module, thus doubling the throughput. If additional workstations are required, then this Sample Robot is already able to be expanded to become an L-sized OMNIS Sample Robot, thus enabling samples from seven racks to be processed in parallel on up to four Pick&Place modules and quadrupling the sample throughput.