Application Note AN-T-042

柠檬酸和草酸混合物

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.

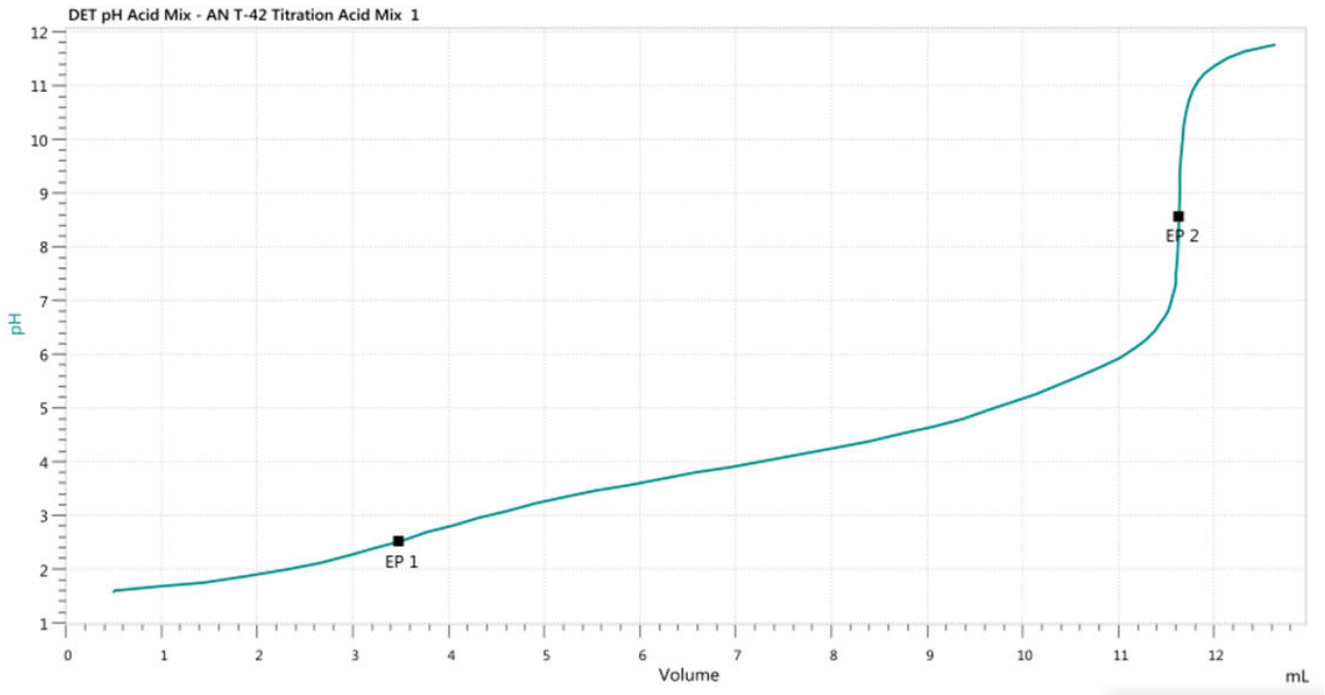

Figure 2. Titration curve of the determination of a mixture of citric and oxalic acid. The first equivalence point corresponds to oxalic acid, and the second to citric acid.

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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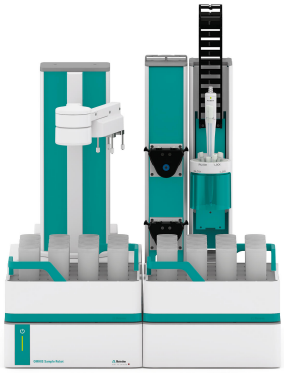
CONFIGURATION

OMNIS Titrator Food

OMNIS Titrator Food 提供了用于含水酸碱滴定的完整套件。套件包含:配有磁力搅拌器的 OMNIS Advanced Titrator、一个 20 mL 的计量管单元、一个用于含水酸碱滴定的 d-Ecotrode plus 电极以及 OMNIS 软件的独立许可证。



OMNIS Sample Robot S Pick and Place

OMNIS Sample Robot S 具有一个“蠕动”泵模块(2 通道)和一个 Pick&Place 模块以及大量附件,可直接进入全自动滴定。此系统具有两个样品盘位置,可用于 32 个 120 mL 的样品盘。此模块化系统供货时已完全安装完毕,因此可在极短时间内投入运行。

该系统也可根据需要扩展配备另外两台蠕动泵以及多加一个 Pick&Place 模块,由此使通量翻倍。如果需要更多工作台,则可将此 Sample Robot 扩展为 L 规格款型的 OMNIS Sample Robot,由此可使七个样品盘的样品在多达四个 Pick&Place 模块上并行处理,将样品通量扩大四倍。