Determination of caffeine by iodometric back titration

Fast and accurate measurement of caffeine content in aqueous samples

Caffeine belongs to a group of alkaloids that stimulates the central nervous system, affecting breathing and the cardiovascular system. Due to its popular performance-enhancing effects, caffeine is considered the world's most consumed pharmacologically active substance.

Iodometric back titration is a simple and accurate method for the determination of caffeine in aqueous solutions or water-soluble samples. In acidic solution, caffeine reacts with iodine to form an insoluble, brown-red complex. Excess iodine is then back titrated with sodium thiosulfate. This method is suitable for food and substances from which caffeine can be extracted with water (e.g., coffee).

In this Application Note, the caffeine content in aqueous samples is accurately and reliably analyzed by iodometric back titration using the OMNIS Titrator equipped with a dPt Titrode.

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SAMPLES AND SAMPLE PREPARATION

This application is demonstrated on caffeine standard, guarana extract, guarana extract concentrate, ground coffee, and an energy drink.

An appropriate amount of sample is weighed into an amber glass beaker. Deionized water, iodine solution, and sulfuric acid are added, and the caffeine-iodine complex is formed. Afterwards, the solution is filtered.

EXPERIMENTAL

An aliquot of the filtrate is titrated until after the first equivalence point with standardized sodium thiosulfate solution (**Figure 1**). The determination is carried out with an OMNIS Titrator equipped with a dPt Titrode (**Figure 2**).
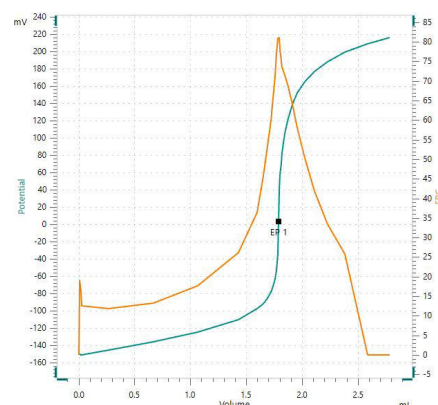

Figure 1. Exemplary titration curve of caffeine in an energy drink (Table 1) with sodium thiosulfate as titrant.

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EXPERIMENTAL

Figure 2. OMNIS Titrator equipped with a dPt Titrode electrode for the determination of caffeine content in aqueous samples.

RESULTS

This method offers very accurate results, as displayed in Table 1.

Table 1. Results of caffeine determination in different aqueous samples.

Sample (n = 6)	Caffeine in %	SD(rel) in %
Caffeine standard	100.1	0.9
Guarana extract	4.2	2.0
Guarana extract concentrate	40.7	2.1
Ground coffee (roasted)	1.3	2.9
Energy drink	0.07	2.4

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CONCLUSION

The iodometric back titration is a precise method used to accurately measure the caffeine content in various aqueous samples. Reliable determinations are made easy using the OMNIS Titrator equipped with a dPt Titrode. This system offers flexible analyses combined with high-end software. The dPt Titrode is maintenance-free and suitable for redox titrations like iodometry when the pH value remains constant.

Internal reference: AW TI CH-1330-112022

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CONFIGURATION

OMNIS Professional Titrator with magnetic stirrer
Innovative, modular potentiometric OMNIS Titrator for stand-alone operation or as the core of an OMNIS titration system for endpoint titration and equivalence point titration (monotonic/dynamic). Thanks to 3S Liquid Adapter technology, handling chemicals is more secure than ever before. The titrator can be freely configured with measuring modules and cylinder units and can have a rod stirrer added as needed. Including "Professional" function license for parallel titration with additional titration or dosing modules.

- Actuation via PC or local network
- Connection option for up to four additional titration or dosing modules for additional applications or auxiliary solutions
- Connection option for one rod stirrer
- Various cylinder sizes available: 5, 10, 20 or 50 mL
- Liquid Adapter with 3S technology: Safe handling of chemicals, automatic transfer of the original reagent data from the manufacturer

Measuring modes and software options:
- Endpoint titration: "Basic" function license
- Endpoint and equivalence point titration (monotonic/dynamic): "Advanced" function license
- Endpoint and equivalence point titration (monotonic/dynamic) with 5-way parallel titration: "Professional" function license

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