



Application Note AN-V-070

Determination of iodide in glacial acetic acid

Iodide determination by cathodic stripping voltammetry (CSV) at the HMDE (hanging mercury drop electrode)

SUMMARY

Methyl iodide is a key ingredient to facilitate chemical reactions during production of glacial acetic acid. However, this raises the chance of iodide ending up in the final product. This is a problem because manufacturers commonly use acetic acid as a reagent to produce other chemicals, such as vinyl acetate. Even trace amounts of iodide can poison the catalysts used in these processes, increasing costs, and negatively affecting product quality. Regular determination of the iodide concentration ensures

the desired quality of acetic acid, safeguarding the integrity of various downstream industrial processes. Using ICP-MS to measure iodine in glacial acetic acid faces challenges due to memory effects that cause high background signals. Cathodic stripping voltammetry (CSV) at the hanging mercury drop electrode (HMDE) overcomes these limitations and provides a robust, cost-effective, and convenient alternative.

SAMPLE

Glacial acetic acid, 99.8%

EXPERIMENTAL

Add 10 mL acetic acid sample and then 2 mL ultrapure water into the measuring vessel. Carry out the determination of iodide using the 884 Professional VA (Figure 1) and the parameters specified in Table 1. The concentration is determined by two additions of iodide standard addition solution.



Figure 1. 884 Professional VA manual for MME.

Table 1. Parameters

Parameter	Setting
Mode	HMDE
Deposition potential	0.1 V
Deposition time	30 s
Start potential	-0.15 V
End potential	-0.5 V
Sweep rate	13 mV/s
Peak potential iodide	-0.32 V

ELECTRODES

- Multi-Mode Electrode pro

RESULTS

The method is suitable for the determination of iodide in acetic acid samples. The limit of detection of the method (for a deposition time of 30 s) is approximately 1 µg/L.

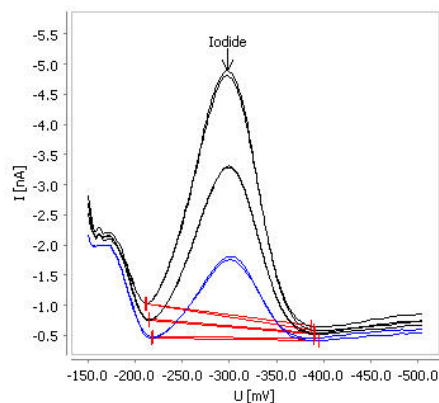


Figure 2. Determination of iodide in glacial acetic acid with CSV.

Table 2. Result

Sample	Iodide (µg/L)
Acetic acid	4.85

Internal references: AW VA CH-0629-022024

CONTACT

Metrohm AG
Ionenstrasse
9100 Herisau

info@metrohm.com

CONFIGURATION



884 Professional VA manual for Multi-Mode Electrode (MME)

884 Professional VA manual for Multi-Mode Electrode (MME) is the entry-level instrument for high-end trace analysis with voltammetry and polarography with the Multi-Mode Electrode pro or the scTRACE Gold or the Bismuth drop electrode. The proven Metrohm electrode methods in combination with a high-performance potentiostat/galvanostat and the extremely flexible viva software open up new perspectives for the determination of heavy metals. The potentiostat with a certified calibrator readjusts itself automatically before each measurement, thus guaranteeing maximum precision.

Determinations with rotating disc electrodes can also be performed with the instrument, e.g. determinations of organic additives in electroplating baths with "Cyclic Voltammetric Stripping" (CVS), "Cyclic Pulse Voltammetric Stripping" (CPVS), and chronopotentiometry (CP). The replaceable measuring head enables rapid changes between the various applications with different electrodes.

The **viva** software is required for control, data collection, and evaluation.

The 884 Professional VA manual for MME is supplied with extensive accessories and a measuring head for the Multi-Mode Electrode pro. Electrode set and **viva** license need to be ordered separately.