Application Note AN-V-241

Fe(II)/Fe(III) speciation in iron citrate capsules by voltammetry

Fast iron speciation analysis with the Multi-Mode Electrode pro in complex citrate matrices

Iron citrate is used in food, pharmaceutical, and technical applications, where both total iron concentration and iron speciation (Fe(II) and Fe(III)) analysis are critical quality attributes. Citrate is an important ligand and efficient complexing agent in aqueous media, forming multiple Fe complexes rather than a single defined compound. Iron's oxidation state changes due to oxidation–reduction processes, light exposure, and oxygen ingress, directly affecting product stability and performance in biologically relevant formulations. Conventional methods (e.g., mass spectrometry, ICP) do not directly resolve Fe species or oxidation states without additional sample preparation. Voltammetry offers a direct and robust approach for iron citrate speciation, enabling selective determination of Fe(II) and Fe(III) in complex citrate matrices. By applying standard addition from defined stock solutions, matrix effects are efficiently compensated, allowing reliable quantification of individual iron forms.

SAMPLE

Commercially available iron citrate capsules were used, with 20 mg iron per capsule.

EXPERIMENTAL

The content of the iron citrate capsule is dissolved in acidic solution. The dissolved sample is added to a vessel (Figure 1) filled with purged electrolyte. Quantification is done using two standard additions with separate Fe(II) and Fe(III) solutions to both prevent oxidation and adapt to varying Fe(II) and Fe(III) concentrations.

- Electrode: Multi-Mode Electrode pro

Figure 1. 884 Professional VA manual for MME.

Table 1. Parameters for Fe(II) and Fe(III) analysis with the Multi-Mode Electrode pro.

Parameter	Setting
Mode	DME
Start potential	0.0 V
End potential	-1.5 V
Sweep rate	30 mV/s
Peak potential Fe(II)	-0.25 V
Peak potential Fe(III)	-0.8 V

RESULTS

The determined recovery rate for the total iron content in iron citrate capsule is 102%.

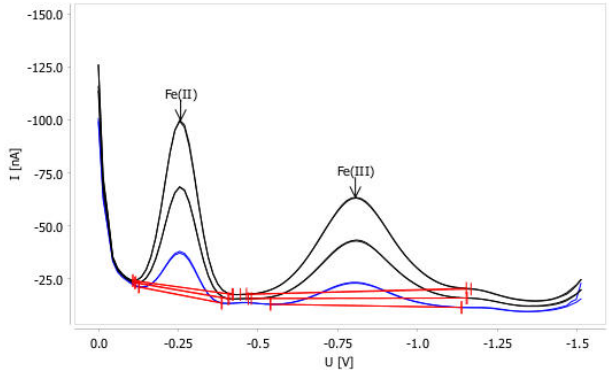

Figure 2. Determination of Fe(II) and Fe(III) in an iron citrate capsule (β(Fe(II)) = 16.77 mg/L and β(Fe(III)) = 15.69 mg/L). Expected total Fe content: 31.84 mg/L. Determined total Fe content: 32.46 mg/L.

Table 2. Results of the determination of Fe(II) and Fe(III) in iron citrate capsules.

Sample	Fe(II) (mg/L)	Fe(III) (mg/L)
Check standard solution 1 g/L	1003	928
Sample (mean value of three determinations)	16.03	16.51

Internal reference: AW VA CH-0646-032026

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CONFIGURATION

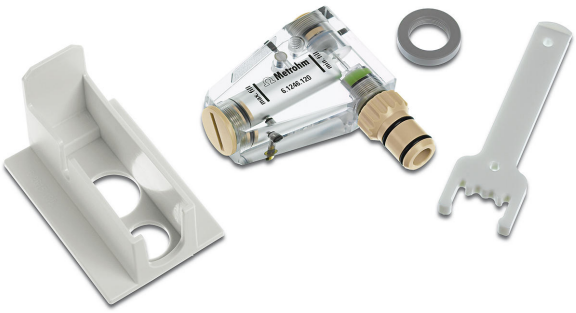
884 Professional VA manual for Multi-Mode Electrode (MME)

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.



Multi-Mode Electrode pro

Mercury electrode for voltammetry. Can be operated as DME, SMDE or HMDE.