



Application Note AN-V-239

Iron speciation in LiFePO_4 batteries

Simultaneous determination of Fe(II) and Fe(III) in lithium iron phosphate with the Multi-Mode Electrode pro

Lithium iron phosphate (also known as LiFePO_4 or lfp) batteries last for over 2000 charges and are safer because of their lower risk of overheating. LiFePO_4 batteries have a slightly lower energy density compared to lithium-ion batteries. However, high discharge rates of lfp batteries make them ideal for electric vehicles, renewable energy storage, and backup power systems. Lithium iron(II) phosphate is used as a cathode material in lithium iron phosphate batteries. Characterization of lfp and monitoring the oxidation state of iron in lfp batteries is relevant to battery performance in terms of durability, capacity,

and safety. Additionally, analyzing chemical composition can be useful for battery research and can aid eco-friendly recycling practices. This is essential for driving battery technology forward and promoting clean energy solutions.

Polarographic speciation of Fe(II) and Fe(III) can be used to evaluate the purity of LiFePO_4 and its usability as a cathode material in lithium iron phosphate batteries. It can further be used to study the concentrations of Fe(II) and Fe(III) in the cathode material after several charging and discharging cycles to evaluate the aging behavior.

SAMPLE

Pure LiFePO_4

EXPERIMENTAL

The lfp sample is weighed, mixed with degassed diluted sulfuric acid, heated at 85 °C for 15 minutes, and then cooled. Afterward, the digested sample solution is added to the measuring vessel that contains 20 mL degassed electrolyte. Quantification is done using two standard additions with separate Fe(II) and Fe(III) solutions.



Figure 1. 884 Professional VA manual for MME.

Table 1. Parameters

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

ELECTRODES

- Multi-Mode Electrode pro

RESULTS



Figure 2. Determination of Fe(II) and Fe(III) in digested lithium iron phosphate with the Multi-Mode Electrode pro.

The viva software delivers unmatched versatility and flexibility by automating data conversion and displaying data in different formats to save time and reduce the risk of errors. **Table 2** shows how viva effortlessly translates concentrations from g/L to mg/g of tested material, enhancing understanding of the results for inexperienced users.

Table 2. Result

Sample	Fe(II) (g/L)	Fe(III) (g/L)
Digested LiFePO ₄	2.8	0.09

Table 2. Result

Sample	Fe(II) (g/L)	Fe(III) (g/L)
Digested LiFePO ₄	2.8	0.09
Sample	Fe(II) (mg/g)	Fe(III) (mg/g)
LiFePO ₄	350	11

Internal reference: AW VA CH4-0543-112015

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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.