

# Multiparameter Analysis of Battery Electrolytes

## LabX Workflow with Titration and UV/Vis

Battery manufacturers face increasing pressure to ensure safety, performance, and compliance while managing complex quality testing workflows. METTLER TOLEDO addresses this challenge with LabX® software, integrating titration and UV/Vis spectroscopy into one streamlined solution. Electrolyte quality directly determines battery reliability.

Parameters such as hydrofluoric acid (HF) concentration and APHA color values reveal impurities that can degrade materials, shorten lifespan, and compromise safety. Traditional workflows with disconnected systems and manual data handling create fragmented records, transcription errors, operator variability, and reporting delays, adding risk at critical decision points. LabX replaces these inefficiencies with centralized data capture, secure storage, and automated result transfer.

Operators receive standardized guidance directly on instrument interfaces, reducing training needs and ensuring reproducibility across teams and sites. Applied to electrolyte testing, HF titration and APHA color determination can now be seamlessly executed under one digital platform. The result is faster measurements, reliable documentation, assured compliance, and greater productivity—helping manufacturers protect quality while staying competitive in battery production.



Figure 1: Schematic of multiparameter setup with titration and UV/Vis spectroscopy.

## Introduction

This application note demonstrates a LabX®-driven multiparameter analysis for battery electrolytes, combining titration and UV/Vis spectroscopy into a single standardized workflow for improved quality control, accuracy, and compliance. In this application, electrolyte samples are first subjected to automated titration to quantify HF content using standardized protocols under LabX control. Color analysis is then performed using UV/Vis spectroscopy, with LabX capturing, storing, and linking the results automatically. Integrated operator guidance ensures correct handling steps, while secure digital transfer prevents errors and supports full data traceability.

## Material and Method

### Instruments and Accessories

- UV/VIS Excellence Spectrophotometer (e.g., UV7 30254726 or UV5 30254725)
- Titration Excellence (e.g., T5 30252672, T7 30252675 or T9 30252676) or Potentiometric Compact titrators (e.g., G10S 30267117 or G20S 30252669)
- Autosampler InMotion Flex 100 mL (30094120)
- Burette DV1010 10 mL (51107501)
- Compact Stirrer (51109150)
- DGi111-SC (51109500) or DGi115-SC (51109504) electrode
- Analytical balance (e.g., XPR205 30355411)
- Quartz cuvettes 50 mm (30258739)
- LabX software (30247984)

### Samples and Reagents

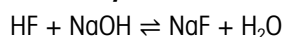
- Battery electrolyte (Lithium hexafluorophosphate solution in ethylene carbonate and dimethyl carbonate, 1.0 M LiPF<sub>6</sub> in EC/DMC=50/50 v/v, battery grade)
- Aq. sodium hydroxide, NaOH= 0.01 mol/L
- Potassium hydrogen phthalate, C<sub>8</sub>H<sub>5</sub>KO<sub>4</sub>
- Deionized water (DI water)

## Measurement

### Procedure (Titration measurement)

- Add approximately 40 mL of DI water into a 100 mL titration beaker and place the beaker on the balance.
- Tare the balance and add ca. 3 g of the electrolyte using a syringe.
- Then place the beaker on the InMotion autosampler rack and start method M813.
- Obtain the hydrofluoric acid content of the battery electrolyte.

### Chemistry



### Titrant

Aq. sodium hydroxide, NaOH, c(NaOH) = 0.01 mol/L.

### Analyte

Hydrofluoric acid, HF, M = 20.01 g/mol, z = 1.

Auxiliary Reagents: DI water

### Standard

Potassium hydrogen phthalate, C<sub>8</sub>H<sub>5</sub>KO<sub>4</sub>, M = 204.22 g/mol, z = 1

### Procedure (UV/Vis APHA Color Measurement)

- Electrolyte samples are measured as such, without any preparation, using a 50 mm quartz cuvette.
- Deionized water to be used as blank.

## Measurement Parameters

### 1. UV/Vis Spectrophotometer

Method	Scanning
Pathlength	5 cm
Measurement time	5 s
Observer	2°
Illuminant	C
Calculation	APHA

## Results

The color and HF content of the electrolyte are calculated fully automatically by using the LabX software [1]. The results are transferred automatically - avoiding manual data handling steps - stored into the central database and saved into a customized report.

Sample	APHA color	SD [%]	RSD [%]
Battery electrolyte	7.3	0.1	1.15

Table 1: Mean APHA color values from battery electrolyte samples obtained on a UV5 Excellence Spectrophotometer (n=6).

Sr.No	Sample size [g]	R1: Content [ppm]
1	2.8542	75.02
2	3.1214	76.78
3	2.8970	78.62
4	2.7795	75.61
5	2.8144	76.52
6	3.2041	76.97
Mean		76.59
s		1.24
srel		1.62%

Table 2: Mean HF content values from battery electrolyte samples obtained on a Excellence Titrator.

The color measurement and HF content of the electrolyte solution demonstrated excellent repeatability, with relative standard deviations of 1.2% and 1.6% respectively, on both the UV/Vis spectrophotometer and the titrator. Color measurements might be used to enhance the quality control of LIB electrolytes and solvents by monitoring discoloration, indicating contamination or deterioration that could affect battery performance, while HF content serves as a quality indicator and is listed in the specification of the electrolyte.

## Conclusion

In summary, our LabX-managed workflow seamlessly integrates titrator and UV measurements, eliminating manual data transfer and fragmented storage. It automates data handling, ensures consistent execution, and provides real-time result visibility, thereby enhancing throughput and reducing reporting bottlenecks. The solution guides operators step-by-step, minimizing training demands and procedural errors, while maintaining robust audit trails to meet regulatory requirements, eventually boosting accuracy, compliance, and analyst productivity.

## Remarks

Color measurement should be carried out as soon as possible since probable deterioration of electrolyte solution occurs with time, leading to an increase in APHA values.

The electrolyte solution should be kept and sealed properly since air and moisture can degrade it, leading to inconsistent color results.

LiPF<sub>6</sub> hydrolyzes slowly in aqueous solutions, while it is hydrolyzing faster in organic solutions with traces of water [2]. Thus, the titration is conveniently performed in water using aqueous NaOH as a titrant.

To prevent the further formation hydrofluoric acid by hydrolysis, the sample should be titrated readily.

This method was only tested on the sample presented. The compositions of Lithium-ion battery electrolytes vary, and some electrolytes may contain additives that hydrolyze to form of hydrofluoric acid very quickly. For such samples, it may be necessary to strictly work in non-aqueous conditions.

The titer of aq. 0.01 M sodium hydroxide solution is determined using potassium hydrogen phthalate (KHP) as the primary standard (M435). As the optimum amount of standard per measurement is very low (5 mg), a 5 mg/mL aq. KHP standard solution is prepared. After every sample, the electrode, stirrer and tubing are rinsed with deionized water. The titrations are performed in plastic beakers as HF can etch glass.

## Waste Disposal and Safety Measures

Although the hydrofluoric acid content of the electrolyte is quite low, every contact with skin or eyes should be prevented. Use gloves, safety goggles and a lab coat. Work in a fume hood. After the analysis, all solutions shall be neutralized and disposed of as aqueous waste.

## Reference

- [1] METTLER TOLEDO LabX battery electrolyte color and HF content methods.
- [2] Stich M., Göttlinger M., Kurniawan M., Schmidt U., Bund A., J. Phys. Chem., 2018, 122, 16, 8836–8842.

## Further Information

- ▶ [UV/Vis Spectrophotometry](#)
- ▶ [Titration Solutions](#)
- ▶ [LabX Software](#)

## Measured Values

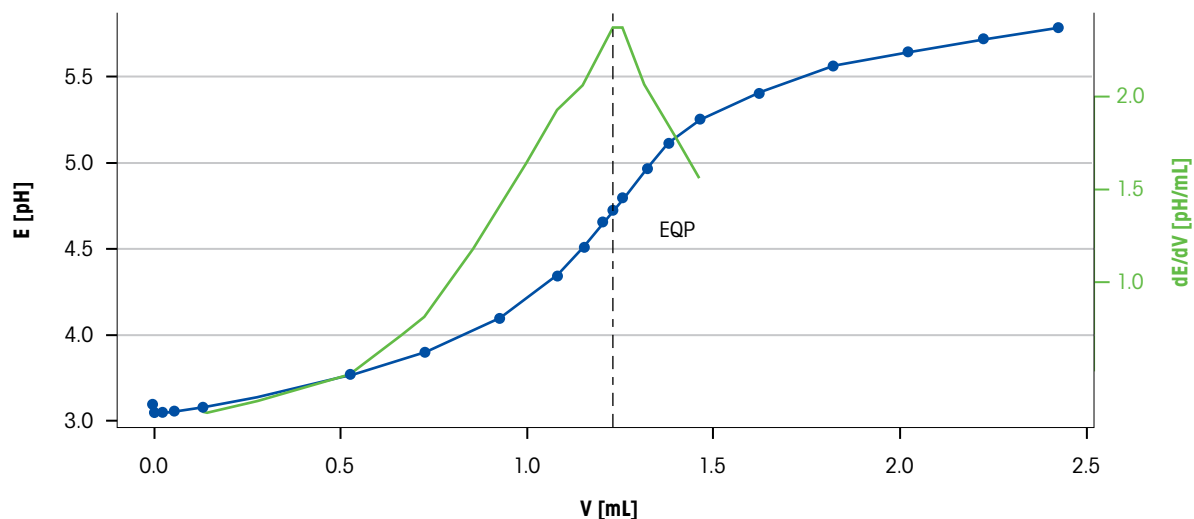


Figure 2: Titration curve (blue) and the first derivative of the titration curve (green) of sample 6/6.

t [s]	V [mL]	E [pH]	dE/dV [mV/mL]
0	0.0000	3.586	n/a
3	0.0050	3.553	n/a
6	0.0100	3.550	n/a
9	0.0225	3.549	n/a
12	0.0535	3.554	n/a
17	0.1310	3.580	0.30
23	0.3250	3.660	0.38
30	0.5250	3.766	0.51
37	0.7250	3.901	0.82
47	0.9250	4.097	1.38
60	1.0830	4.343	1.93
72	1.1530	4.505	2.07
86	1.2040	4.646	2.25
<b>EQP</b>	1.232515	4.726	2.37
100	1.2555	4.790	2.37
118	1.3180	4.964	2.05
138	1.3805	5.113	1.86
159	1.4645	5.250	1.56
181	1.6230	5.408	n/a
206	1.8230	5.555	n/a
222	2.0230	5.643	n/a
238	2.2230	5.723	n/a
251	2.4230	5.783	n/a

## Method

<b>001   Title</b>		
Type		General Titration
ID		M813
Title		Hydrofluoric Acid Content of Lithium-Ion Battery Electrolyte

<b>002   Sample</b>		
Sample Type		Sample
Number of IDs		1
ID 1		Electrolyte
Entry type		Weight
Lower limit		0.0
Higher limit		5 g
Density		1 g/mL
Correction factor		1.0
Temperature		25.0°C
Titration reader		None
Number of sample factors		0

<b>003   Titration stand</b>		
Type		InMotion T/Tower A
Titration stand		InMotion T/1A
Head position		Sample

<b>004   Stir</b>		
Speed		30%
Duration		5 s

<b>005   Titration (EQP) [1]</b>		
<b>Titration</b>		
Titration		NaOH
Concentration		0.01 mol/L
<b>Sensor</b>		
Type		pH
Sensor		DGi111-SC
Unit		mV
<b>Temperature acquisition</b>		
Temperature measurement		No
Stir		
Speed		30%
<b>Predispense</b>		
Mode		None
Wait time		0 s
<b>Control</b>		
Control		Cautious
Mode		Acid/base
Titration addition		Dynamic
dE (set value)		10 mV
dV (min)		0.005 mL
dV (max)		0.2 mL
Mode		Equilibrium controlled
dE		0.5 mV
dt		2 s

t (min)	3 s
t (max)	30 s

<b>Evaluation and recognition</b>		
Procedure		Standard
Threshold		1.5 pH/mL
Tendency		Positive
Ranges		0
Add. EQP criteria		No
<b>Termination</b>		
At Vmax		5 mL
At potential		No
At slope		No
After number of recognized EQPs		Yes
Number of EQPs		1
Combined termination criteria		No
Condition		No

<b>006   Calculation R1</b>		
Result		Content
Result unit		ppm
Formula		$R1 = Q \cdot C / m$
Constant C		$C = M \cdot 1000 / z$
M		$M[\text{Hydrofluoric Acid}]$
Decimal places		3
Result limits		No
Extra statistical functions		No
Send to buffer		No
Write to Smart Tag		None
Condition		No

<b>007   Rinse</b>		
Auxiliary reagent		Water
Rinse cycles		1
Vol. per cycle		10 mL
Position		Current position
Drain		No
Condition		No

<b>008   End of Sample</b>		
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<b>009   Park</b>		
Titration stand		InMotion T/1A
Position		Conditioning beaker
Condition		No

<b>010   Record</b>		
Report template		Titration report
Condition		No

<b>010   Record</b>		
Report template		Titration report