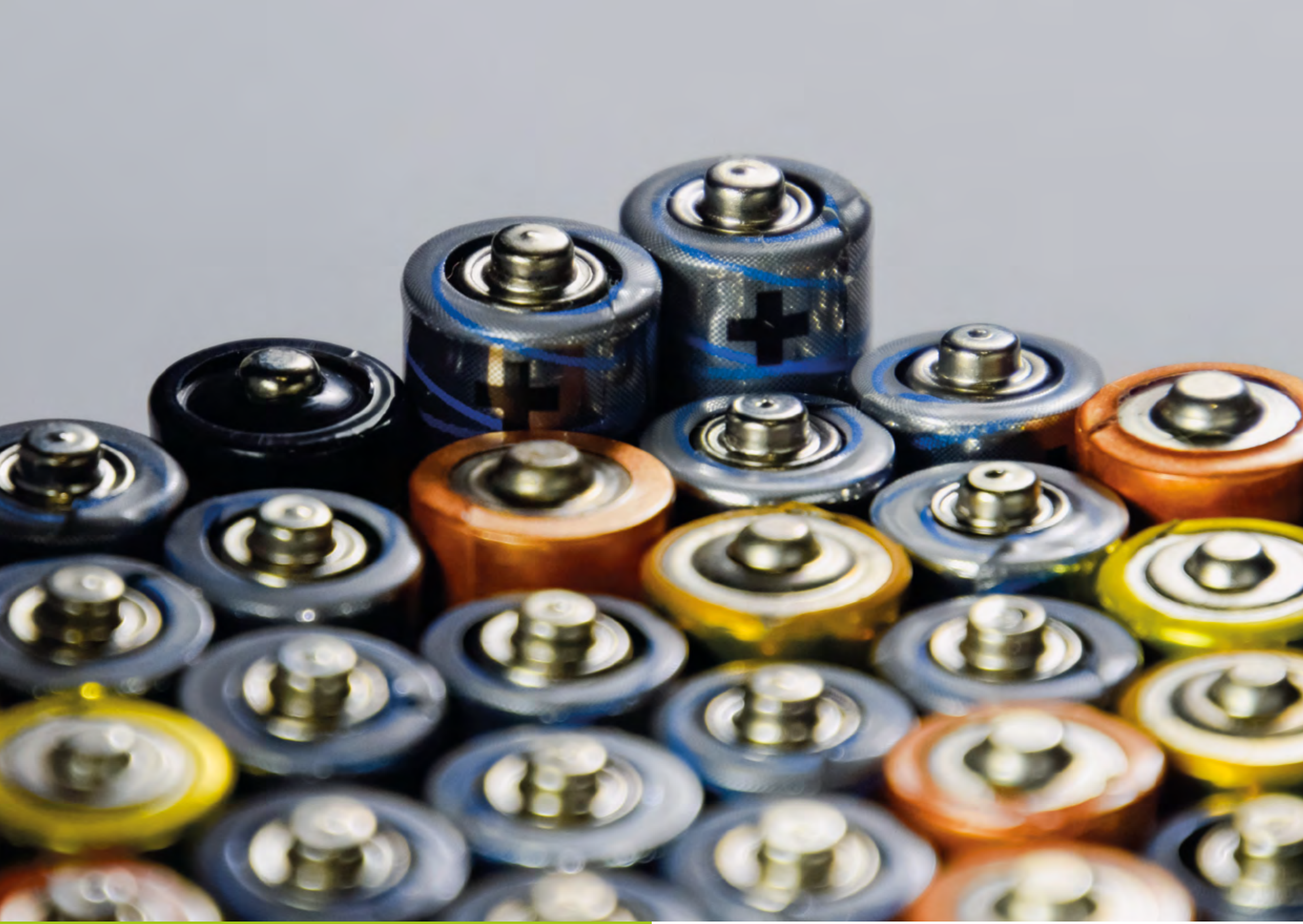

Battery research and production

Materials, parameters, and analytical methods

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A comprehensive portfolio

Battery research encompasses the development process from theory and concept validation of new materials to characterization and quality control of raw materials and finished batteries. Progress in the quest for higher energy, power density, and more efficient energy storage depends on sophisticated instrumentation for the characterization of materials and cells. Metrohm provides you with top-quality analytical instruments, know-how, and first-class, on-site service to enable and support your research.

Our White Paper WP-052 «A Guide to Li-ion Battery Research and Development» gives you a comprehensive overview of the electrochemical analysis of batteries and raw materials. The table below gives you an overview, which parameters of interests can be analyzed by which method using Metrohm instrumentation. Feel free to get more detailed information from the corresponding and linked Metrohm application documents. Missing your application? Contact your local Metrohm organization to discuss possible solutions.

Sample or process step	Parameter of interest	Standard	Analysis technique	Metrohm Application Documents
Anode (and raw materials)	Water content		Karl Fischer titration	AB-434, WP-084
	Identification/verification of carbon materials		Raman spectroscopy	410000059
	Oxygen content (Boehm titration)	ISO/TR 19733, GB/T 38114	Titration	AN-T-226
	Brightener & Suppressor in Cu deposition solution for copper foil production		CVS	
	Fluoride and chloride content in graphite		Combustion ion chromatography (CIC)	
	Intercalation and de-intercalation of lithium		Electrochemistry	WP-052
	Characterization of carbon materials in dependence of applied potential		Raman Spectroelectrochemistry	AN-RA-002, AN-RA-009
	Eluated anions and cations		Ion chromatography	
Cathode (and raw materials)	Lithium content in brine		Ion chromatography, Process analysis	AN-PAN-1058
	Assay of lithium salts (LiOH, Li_2CO_3, $LiNO_3$, LiCl)	GB/T 11064.1, GB/T 11064.2, GB/T 11064.3	Titration	AN-T-215, AN-T-216, AN-T-181
	pH value, LiOH, Li_2CO_3 and chloride content in lithium hydroxide refinement process		Titration, pH measurement, Process analysis	
	Ionic Impurities in lithium salts (e.g., sodium, ammonium , sulfate, fluoride, chloride)	GB/T 11064.15, YS/T 1569.4, YS/T 1569.5	Ion chromatography, Ion measurement	AN-C-189, AN-C-063, WP-084
	Ni, Co, Mn content in solution for cathode material production (single metal or mixture)		Titration, Voltammetry	AN-T-218, WP-084
	Total metal, nickel, manganese and cobalt content in NMC	YS/T 1006.1, YS/T 1472.1, SY/T 1472.2	Titration	WP-084
	Total iron content in LFP	YS/T 1028.1	Titration	WP-084
	Cobalt content in LCO	GB/T 23367.1	Titration	WP-084
	Manganese content in LMO		Titration	WP-084
	Cobalt content in NCA	YS/T 1263.2	Titration	WP-084
	Manganese content in LNMO	YS/T 1569.2	Titration	
	Fe(II) and Fe(III) in LFP		Voltammetry	AN-V-239
	pH value in carbon black, pCAM, CAM, and cathode slurry	ASTM D1512, ISO 787-9, GB/T 1717	pH measurement, Process analysis	AN-T-235
	Fluoride in cathode material (e.g., NMC)		Combustion ion chromatography (CIC)	
	Metal content in cathode slurry		Process analysis	

A comprehensive portfolio

Sample or process step	Parameter of interest	Standard	Analysis technique	Metrohm Application Documents
	Water content		Karl Fischer titration	AB-434, WP-084
	Eluated anions and cations		Ion chromatography	
	Through-plane tortuosity		Electrochemistry	AN-BAT-011
	Residual alkali, soluble base content (SBC)	GB/T 41704	Titration	WP-084
	Trace anions in recovered N-methylpyrrolidone (NMP)		Ion chromatography	
	Phosphate (phosphoric acid) as starting material for LFP		Titration	WP-084
Electrolytes, electrolyte additives, and electrolyte solvents	Water content	SJ/T 11723	Karl Fischer titration	AB-434
	Cations and anions in electrolyte salts (e.g., fluoride, chloride, sulfate in $LiPF_6$)		Ion chromatography	AN-CS-011
	Composition of lithium salts in battery electrolyte		Ion chromatography	AN-S-372, WP-084
	Electrolyte resistance		Electrochemistry	
	Solid electrolyte interface (SEI) investigation		Electrochemistry	AN-BAT-010
	Diffusion coefficient		Electrochemistry	AN-BAT-009
	Lithium ion transference number		Electrochemistry	AN-BAT-012
	HF content in electrolyte	SJ/T 11723	Titration	
	Ionic impurities	SJ/T 11723	Ion chromatography, Titration	WP-084
	Physicochemical changes occurring in electrolyte at different depths and electrode surfaces		Spectroelectrochemistry	AN-RS-042
Separators	Water content		Karl Fischer titration	AB-434, WP-084
	Eluated anions and cations		Ion chromatography	
	MacMullin number		Electrochemistry	AN-BAT-006
	Identification/verification of polymers		Raman spectroscopy	

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Sample or process step	Parameter of interest	Standard	Analysis technique	Metrohm Application Documents
Finished batteries	Voltage characteristics upon charge and discharge		Electrochemistry	AN-BAT-001, AN-BAT-002, AN-BAT-010, AN-BAT-007, AN-BAT-013, AN-EC-018
	Cycling performance and coulombic efficiency		Electrochemistry	WP-052
	Cycle and calendar life evaluation		Electrochemistry	WP-052
	Battery capacity and power		Electrochemistry	WP-052
	State of charge, depth of discharge, and state of health		Electrochemistry	WP-052
	Internal battery resistance		Electrochemistry	AN-EC-013, AN-BAT-006, AN-BAT-008, AN-BAT-011
	Charge transfer resistance		Electrochemistry	AN-BAT-013, AN-EIS-003, AN-EIS-001
	Diffusion coefficient (GITT and PITT)		Electrochemistry	AN-BAT-003, AN-BAT-004, AN-BAT-009, AN-BAT-012
	Eluated anions and cations		Ion chromatography	
Recycling	Fluoride and chloride content in black mass		Combustion ion chromatography (CIC)	
	Total metal, nickel, manganese and cobalt content in NMC	YS/T 1006.1, YS/T 1472.1, SY/T 1472.2	Titration	WP-084
	Cobalt content in LCO	GB/T 23367.1	Titration	WP-084
	Manganese content in LMO		Titration	WP-084
	Total iron content in LFP	YS/T 1028.1	Titration	WP-084
	Water content		Karl Fischer Titration	AB-434, WP-084
	Residual alkali, soluble base content (SBC)	GB/T 41704	Titration	WP-084

Analysis techniques

ELECTROCHEMISTRY – IDEAL FOR STUDYING THE PERFORMANCE OF BATTERIES AND BATTERY MATERIALS

Our specially designed potentiostats/galvanostats are used for the development and characterization of battery materials as well as finished batteries. Electrochemical measurements are based on a highly accurate control and measurements of voltage, current, electrical charge, or impedance.

Voltammetry is a highly sensitive method (LODs in the ppb range) for the analysis of electrochemically active substances, such as inorganic or organic ions. For example, voltammetry allows the simultaneous determination of nickel, cobalt, and manganese in cathode materials. This technique combines a wide range of applications, short analysis times, and high precision with comparatively low costs of the required instrumentation.

Potentiometric titration is ideally suited for determining the purity of lithium salts or metals used in cathode materials, such as cobalt, nickel, manganese, or iron. It is applied to analyze standard solutions used for the preparation of cathode materials or finished cathode materials. Unlike competing methods such as ICP-MS or AAS, titration does not require dilution of such samples. Hence, results obtained by titration are more reliable and accurate. Furthermore, running and maintenance costs are considerably lower compared with ICP-MS or AAS.

KARL FISCHER TITRATION – THE PREFERRED METHOD FOR WATER DETERMINATION

Lithium-ion batteries must be completely free of water (concentration of H_2O < 20 mg/kg), because water reacts with the conducting salt, e.g., $LiPF_6$, to form hydrofluoric acid. Sensitive coulometric Karl Fischer titration is the ideal method for determining water content at trace levels. Water determination for solids is carried out using the Karl Fischer oven method, where residual moisture in the sample is evaporated and transferred to the titration cell where it is subsequently titrated.

ION CHROMATOGRAPHY – HIGHLY EFFICIENT MULTI-PARAMETER ANALYSIS OF ELECTROLYTES AND METALS SALTS

Ion chromatography (IC) is an efficient and precise multi-parameter method to quantify impurities in lithium salts, decomposition products in electrolytes, as well as anions and cations in eluates of various battery raw materials.