

Portable and Smart Ion Analyser for Automated Monitoring of Alkali Metals in Battery Recycling Industries towards Circular Economy

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Summary:

A portable, accessible, and smart ion analyser is presented herein for automated monitoring of Li⁺ and Na⁺ in battery recycling processes, integrating potentiometric sensing, automated fluid handling, embedded data processing, and remote dashboard access in a compact modular platform. The system enables quasi-parallel multi-electrode measurements, autonomous recalibration, and near-real-time operation with minimal human intervention. The prototype shows wide linear ranges, low detection limits, and strong agreement with ICP-MS/MS reference measurements. Deployed in a battery recycling facility, it demonstrated reliable on-site monitoring of process streams over a 4-week trial. Overall, the system supports sustainable recycling, operational decision-making, and scalable industrial metal recovery.

Keywords: Alkali-metals, Potentiometry, Smart Ion Analyser, Battery recycling, Circular economy.

Background, Motivation and Objective

With the increasing demand for lithium-ion batteries and the growing need for efficient recycling strategies, accurate, fast, and accessible monitoring solutions for strategic metals are crucial to support sustainable recycling, raw material recovery, and quality control processes [1]. While conventional instrumental procedures are effective for benchtop analysis, the high cost, results delay, and laborious sample pre-treatment are still challenges to address. In context, we present a portable and smart ion analyser for automated and real-time monitoring of alkali metals (i.e. Li⁺ and Na⁺) in battery recycling processes.

Description of the New Method or System

The ion analyser (Fig. 1) combines potentiometric sensing, embedded signal processing, fluidic monitoring, and networked data communication into a compact digital-physical platform (50 cm × 35 cm × 30 cm). The system integrates eighteen ion-selective electrodes (nine Na-ISEs and nine Li-ISEs) and six solid-contact Ag/AgCl reference electrodes in a screen-printed configuration, assembled in a modular 3D-printed multi-flow cell, to enable multi-point statistical acquisition and multiple analyses over time. A syringe pump, two rotary selection valves, three

pillow bags with standard solutions (recalibration Li/Na solutions and ionic strength adjuster), and a custom-made 3D-printed module for sample dilution are integrated for sample and carrier delivery, enabling automated sensor recalibration and sample processing. A single EmStat-4M potentiostat was extended by a custom-made multiplexer hardware adaptor to quasi-parallel multi-electrode operation (1s interval). Beyond sensing, the system is embedded with a flowmeter for real-time flow characterisation and a derivative flat-region detection algorithm for data analysis. In addition, the ion analyser incorporates software with a dashboard interface, accessible via an integrated display or browser connection, supporting standalone, scheduled, or fully automated operation modes.

Results

Under optimal conditions, the analytical figures of merit of the proposed system include a sub-Nernstian sensitivity (≈ 55 mV dec⁻¹) and a wide linear response range from 0.1 to 100 mM for both metals. The limit of detection was 0.05 and 0.04 mM for Li⁺ and Na⁺, respectively. Impressive slope reproducibility for the same electrode or between electrode units was demonstrated by relative standard deviation (RSD) values lower than 7.7%, while small discrepancies were

observed for the intercept values ($RSD < 20\%$), which are circumvented by sensor recalibration before sample analysis. The system supports up to 45 consecutive measurements without consumable replacement or maintenance, with a sample throughput every 1 hour and 30 minutes. Before deployment, pregnant leaching solutions of black mass (PLS) from Lithium Nickel Manganese Cobalt Oxide batteries and battery discharge waters (BDW) samples were analysed using the ion analyser and compared to analysis performed by ICP/MS-MS at an external laboratory as the reference method. A strong agreement was observed between the two approaches, specifically for Na measurement at the BDW samples ($\text{diff} \leq 16\%$) and Li measurement at the PLS samples ($\text{diff} \leq 27\%$).

Tab. 1: Analytical performance of the Ion Analyser.

	Li ⁺	Na ⁺
Slope / mV dec^{-1}	56.0	56.1
R ²	0.999	0.999
LRR / mg L^{-1}	0.7 – 700	2.3 – 2300
LOD / mg L^{-1}	0.4	0.8
pH range	2 – 10	2 – 10

As a case study, the ion analyser was deployed at a local battery recycling facility (B.again, Braga, Portugal) to monitor the levels of Li⁺ and Na⁺ in PLS and BDW samples. During the trial period (4 weeks), the system performed intermittent analysis with a total of 35 measurements, and the samples were sent to an external laboratory to be analysed by ICP/MS-MS (results still pending).

This innovative portable ion analyser represents a step forward to resilient smart sensing solutions and circular economy [2], offering a cost-effective and modular smart system, on-site or near-line operation, automated analysis, and industrial connectivity. Ultimately, this system contributes to more efficient decision-making and sustainable metal recoveries in battery recycling industries [3].

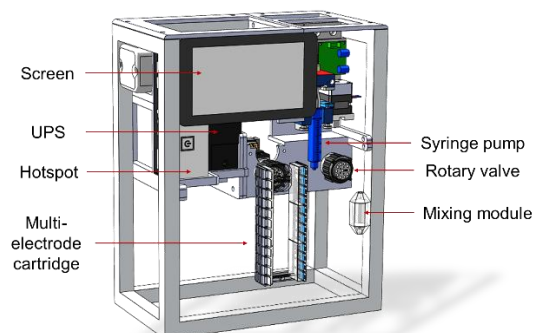


Fig. 1. Ion Analyser visualisation and description.

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