

A ROBUST METHOD DEVELOPED FOR SPECIES ANALYSIS DURING A TYPICAL HYDROMETALLURGICAL TEST WORK PROGRAMME FOR PRODUCTION OF BATTERY GRADE PHOSPHORIC ACID – A MINTEK PACKAGE

By

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ABSTRACT

The current work evaluates the optimisation of an analytical method for the determination of fluoride and chloride impurities in a high phosphoric acid matrix. A typical battery grade acid is expected to contain meagre level of impurities to better enhance the capacity, voltage, specific energy, energy density, and thermal stability of lithium ion batteries, latter being the most crucial. Accurate determination of the species in the final product as well as streams generated throughout the three major phases of pre-treatment, solvent extraction and post-treatment is the only means to monitor the process effectiveness.

A robust inter-linked technique is required for preparation and analysis of various commodities and species during the process to meet the stringent impurity thresholds at each stage. These include determination of density, total suspended solids, P₂O₅, total organic carbon, critical base metals (arsenic, chromium, sulphur and boron), chloride and fluoride.

A precise determination of chloride and fluoride in the process is critical to ensure that the corrosiveness of the acid is minimised. The chloride and fluoride contents are expected not to exceed 20 and 10 ppm, respectively. These ions can be analysed using either a UV-Vis or an ion chromatography. This paper therefore, details how analyses of such limited range were conducted, the development and optimization of the exclusion methods to suit the high phosphorus matrix of these samples. International certified reference material was used for validation purposes. The high phosphoric acid matrix resulted in challenges with both the UV-Vis and the Ion chromatography methods however, the ion chromatography exclusion method allowed for the determination of these ions at low concentrations. This comparative study showed good recoveries for both elements of interest (F⁻ and Cl⁻) when using the ion chromatography exclusion method and the data obtained indicated good repeatability and reproducibility.

Keywords: Battery Grade Phosphoric Acid, Solvent Extraction, Ion Chromatography Exclusion, UV-Vis