Case Study

Oxford Instruments benchtop NMR helps high-end chemical production

An important industrial application of NMR spectroscopy is quality control of incoming raw materials, manufacturing processes and end products in the fine and speciality chemicals industries.

An Oxford Instruments **X-Pulse** broadband benchtop NMR spectrometer is being used for quality control of fine chemicals in a chemical enterprise in Shaanxi Province, China. The company's Quality Control Manager, highlighted how important high quality inspection standards are for their export products. Structural identification NMR ensures that the impurity composition and content meet their end customer requirements and regulatory standards.

Figure 1: ^{1}H NMR spectrum of a mixture of typical solvents contained in lithium-ion batteries: ethylene carbonate (EC), dimethyl carbonate (DMC) and diethyl carbonate (DEC) demonstrating the quantification of multiple species in sample. The spectrum also includes trace vinylene carbonate (VC) at 7.4 ppm.

Previously, they sent samples to universities for testing, with turnaround times of 2 - 3 days. The COVID-19 outbreak closed these external testing facilities, negatively affecting both the production volume and delivery times.

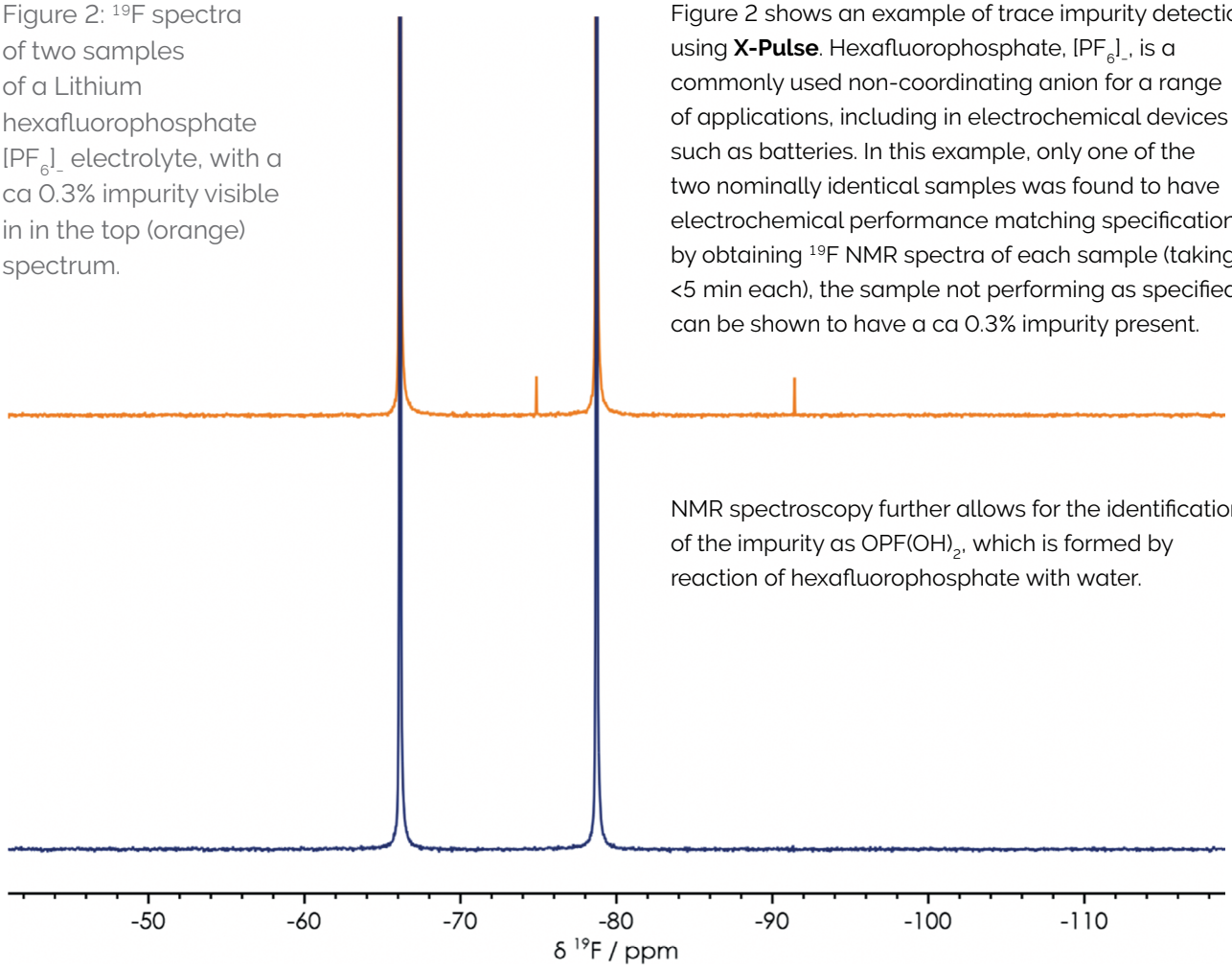
Now all the trace targets and impurities can be clearly identified in their own labs by acquiring quantitative ^{1}H NMR spectra in under 4 hours. The test data from their in-house benchtop **X-Pulse** is directly comparable to the results they previously obtained externally using high-field NMR.

Figure 1 shows an example of how different signals in an NMR spectrum can be integrated, allowing for the concentrations, and ratios of components in solution to be accurately determined as the area beneath the peaks is proportional to the number of nuclei present.

Figure 2: ^{19}F spectra of two samples of a Lithium hexafluorophosphate $[PF_6]^-$ electrolyte, with a ca 0.3% impurity visible in in the top (orange) spectrum.

Figure 2 shows an example of trace impurity detection using **X-Pulse**. Hexafluorophosphate, $[PF_6]^-$, is a commonly used non-coordinating anion for a range of applications, including in electrochemical devices such as batteries. In this example, only one of the two nominally identical samples was found to have electrochemical performance matching specification; by obtaining ^{19}F NMR spectra of each sample (taking <5 min each), the sample not performing as specified can be shown to have a ca 0.3% impurity present.

NMR spectroscopy further allows for the identification of the impurity as $OPF(OH)_2$, which is formed by reaction of hexafluorophosphate with water.



The company's cryogen-free **X-Pulse** benchtop system is now a critical detection method for on-site quality control across the production process, shortening the analysis time, solving the pain point of impurity detection, and providing a traceable guarantee for their exported high-quality chemicals.

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