# DEVELOPMENT OF A BENCHTOP NMR CRUDE OIL ANALYSER

**Despite the well demonstrated efficacy of Nuclear Magnetic Resonance (NMR) Spectroscopy for the rapid analysis and characterisation of petrochemicals, dating back to the 1960s, the oil and gas sector has been largely underserved by this powerful technique. While research labs have developed high-field $^{1}$H NMR/PLS and multivariate calibration methods to quantify many crude oil properties (e.g., API density, sulphur content, TAN), these have not been widely adopted in the field. The prevalent NMR technology is based on superconducting magnets which have a large footprint and are costly in terms of capital cost and operating expenditures and maintenance.**

The emergence of the so-called benchtop NMR spectrometer in the 2010s, has provided the unique opportunity to address these instrumentation limitations and investigate the potential for proliferating this powerful analytical technique into a series of new qualitative and quantitative applications. The benchtop NMR is compact, affordable and because the magnetic field is permanent magnet based, does not require routine cryogen fills that the superconducting magnets do. They enable accessible, affordable and automatable NMR spectroscopy.

Recently in an invited article in Magnetic Resonance in Chemistry, Sassu et. al. detail a method whereby they extrapolate approaches developed for a large database of crude oils that differ in geographical origin and/or chemical composition from high-field to low-field $^{1}$H NMR.

Crude oils and refining streams are composed of thousands of hydrocarbons – linear and branched alkanes, cycloalkanes, aromatics, olefins, asphaltenes etc.. Depending on this chemical composition, and therefore physical and chemical properties within the sample, each crude can exhibit different behaviour throughout the refining process. In order to optimise this refining process, it is necessary to understand many of these physical and chemical properties for each incoming crude oil feedstock. Traditionally these parameters were taken through a series of tests in an offline testing facility by sampling incoming crude and storing the bulk until the information was received to allow the refining process to proceed. These testing procedures typically involve cumbersome atmospheric distillations and a series of time consuming and/or expensive standard analytical methods that use large size samples destructively and often require the use copious amounts of solvent.

The primary value proposition of a benchtop NMR based method to analyse physical and chemical properties of crude feedstocks as detailed in the paper is threefold: (i) it can be done directly at the refinery; (ii) it can be performed quickly; and (iii) it can combine many tests into one simple data acquisition. For a benchtop NMR based method, data collection does not require distillation, it is non-destructive, and it can be done directly on site, requiring only about 5 minutes for each spectral acquisition and processing. While the method development was complex, requiring generation of a model with Principal Component Analysis (PCA) and Partial Least Squares (PLS) methods; the analysis is automated and simple. Once the model was built, each unknown sample that is acquired can be analysed to determine its principal components. This PC is projected onto a principle component map via their so-called PCA scores. Crudes with similar components have similar scores and therefore cluster together. The scores can be used as coefficients for the linear transformation to PLS to provide a quantitative prediction of each crude property. The modeling methods used were compared to the appropriate ASTM methods to generate a resulting root mean square error of calibration (RMSEC) to provide assurance that the low-field, benchtop NMR analysis method compares favourably to existing analytical method precision.

In order to allow this method to be used directly at the refinery, of course, it must be suitable for non-experts. Therefore, as part of the crude oil analyser application, a software interface was incorporated to allow the analyst to acquire data, and have it fit to the models in a completely automated manner that is easy, rapid and accurate. The software program ensures that the instrument is performing properly, and the data is acquired in a consistent comparable manner on each scan and processed correctly. It is then compared to the model and a data report is created, saved and printed such that integrity is maintained throughout the process. The report contains the chemical-physical properties of the crude including composition (CHNs, Aromatics, MCRT, Asphaltenes, TAN), bulk properties (API Density, Pour point, viscosity), metal content (V, Ni, etc.) and distillation yield. It also contains a measure of the reliability of the result.

The authors highlight the potential such a tool has for the increase in yield from crude oil distillation unit, the speed with which it could check crudes from incoming tanks, ships or loading terminals throughout the transportation process, the confidence in blending crudes and finally, the potential it has to improve short term planning and scheduling throughout the refining process.


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