

Spinsolve

Fully Automated Compound Screening and Verification Using Spinsolve and MestReNova

Sample screening to verify the identity or integrity of a compound or raw material is a key function of many quality control (QA/QC) labs. NMR spectroscopy is extremely well suited to many QA/QC applications because of the very high degree of specificity and valuable quantitative information it can provide. Furthermore, compared to many other analytical methods, such as liquid chromatography (LC), the sample preparation is minimal, the data collection is rapid, and the consumable requirements (e.g. solvents) are low. With the advent of **high-performance benchtop NMR spectrometers such as Spinsolve**, NMR systems can be easily deployed in QA/QC labs for routine analyses.

Depending on the questions that need to be addressed by the QA/QC lab, NMR-based compound screening can be done in a variety of ways. In many cases, the question to be answered is simply "Do I have what I think I have?". One of the simplest and most robust approaches here is to compare the NMR spectrum of the sample under investigation with a reference spectrum or set of spectra. Another important question to answer is "What is the purity of this compound?". Quantitative NMR (qNMR) provides reliable methods for purity analysis that will be discussed in a separate Application Note.

In this Application Note we show how **Spinsolve** benchtop NMR is used to perform peakbased compound screening in a **rapid**, **reliable** and **fully automated** way, using the analysis of pesticides as an example. Additional sample screening applications being developed include those for pharmaceutical drugs (APIs), nutraceuticals, petrochemicals and others.



Sample Screening Workflow

Figure 1 shows a typical workflow used for benchtop NMR-based compound screening. Note that the sample preparation for the method is straightforward and the data collection, analysis and reporting are rapid and fully automated.



Figure 1. Typical workflow for benchtop NMR-based compound screening. The steps enclosed in the dashed box are fully automated.

Method Setup

Before the system can be used to perform routine sample screening according to the work, there are two tasks that need to be carried out by the system's administrator. The first task is to create a database of reference spectra of different compounds against which routine sample measurements will be compared. The second task is to automate the data collection and analysis using scripting tools in Spinsolve and MestReNova (Mnova). Note that both tasks are one-time undertakings, though the database and scripts can be modified at any time to accommodate more library compounds or adjust the data analysis, for example.

Building a Spectral Database

Figure 2 shows a series of proton spectra of ten different commercially-available pesticides, collected on a **Spinsolve 60 system**. As the figure illustrates, the spectrum of each pesticide provides a distinct "fingerprint" of that pesticide. It is important to note that the quality of the NMR spectrum is critically important to the reliability and robustness of any sample screening method that relies on matching routine spectra to those in a database. If the resolution of the spectrum is poor (the lines are broad) or the spectrum shows poor signal-to-noise ratio, this will degrade the reliability of the analysis. The spectra in Figure 2 all show excellent resolution and sensitivity, making them ideally suited to the pesticide screening application described here.



Figure 2. Stacked plot of proton spectra of ten different commercially available pesticides

Once the reference spectra have been collected, the next step is to create a database in Mnova and add to it a unique record for each compound (Figure 3). **Mnova DB** is a flexible and powerful database utility that is fully integrated with Mnova. In our pesticide screening example, each record comprises a proton spectrum, the experiment parameters, a molecular structure (MOL file) and some explanatory text. Note, however, that the items and fields stored in the database are not fixed and can be fully customized to suit the application's needs. Additionally, the database need not be restricted to proton spectra: it may be useful or desirable in some applications, for example, to include carbon-13 and/or 2D (e.g. HSQC) spectra.



Figure 3. Saving spectra and molecular structures to the Mnova DB database

Automating Data Collection and Analysis

Now that the database has been set up, the next step is to configure Spinsolve to perform routine sample screening. Spinsolve software features flexible scripting tools that allow sample measurements to be fully automated, so that technicians can easily run the instrument with the minimum of training. In addition, Spinsolve scripts can be combined with Mnova's powerful scripting tools to automate and customize the data processing and analysis, and the reporting of the results. In the pesticide example shown here, the Mnova script processes the data, generates a peak list from the spectrum and sends the peak list to Mnova DB to find a match. Finally, the script generates a report that shows the results of the analysis.

Routine Sample Screening

The Spinsolve is now fully set up to carry out routine sample screening. Figure 3 shows a simple user interface for carrying out the analysis. To carry out the measurement, the user simply selects the solvent, enters the sample name and clicks the "Start" button. As a final check, the system prompts the user to confirm that the sample has been inserted into the Spinsolve.



Figure 4. Spinsolve user interface for pesticide screening.

Once the measurement has completed, the data are automatically processed and analyzed and a report is generated. Figure 5 shows a typical report obtained.



Figure 5. Two-page pesticide analysis report, automatically generated using Mnova.

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Conclusions

It has been shown how the high-quality NMR data from Spinsolve, combined with powerful software scripting and database tools, is used to perform peak-based screening for sample ID verification of pesticides. The sample measurements, analysis and reporting of results can be fully automated, making it easy for a technician or non-expert to perform the analysis in a routine QA/QC lab. Complementary to sample ID verification is the determination of sample purity that will be described in a separate Application Note.



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