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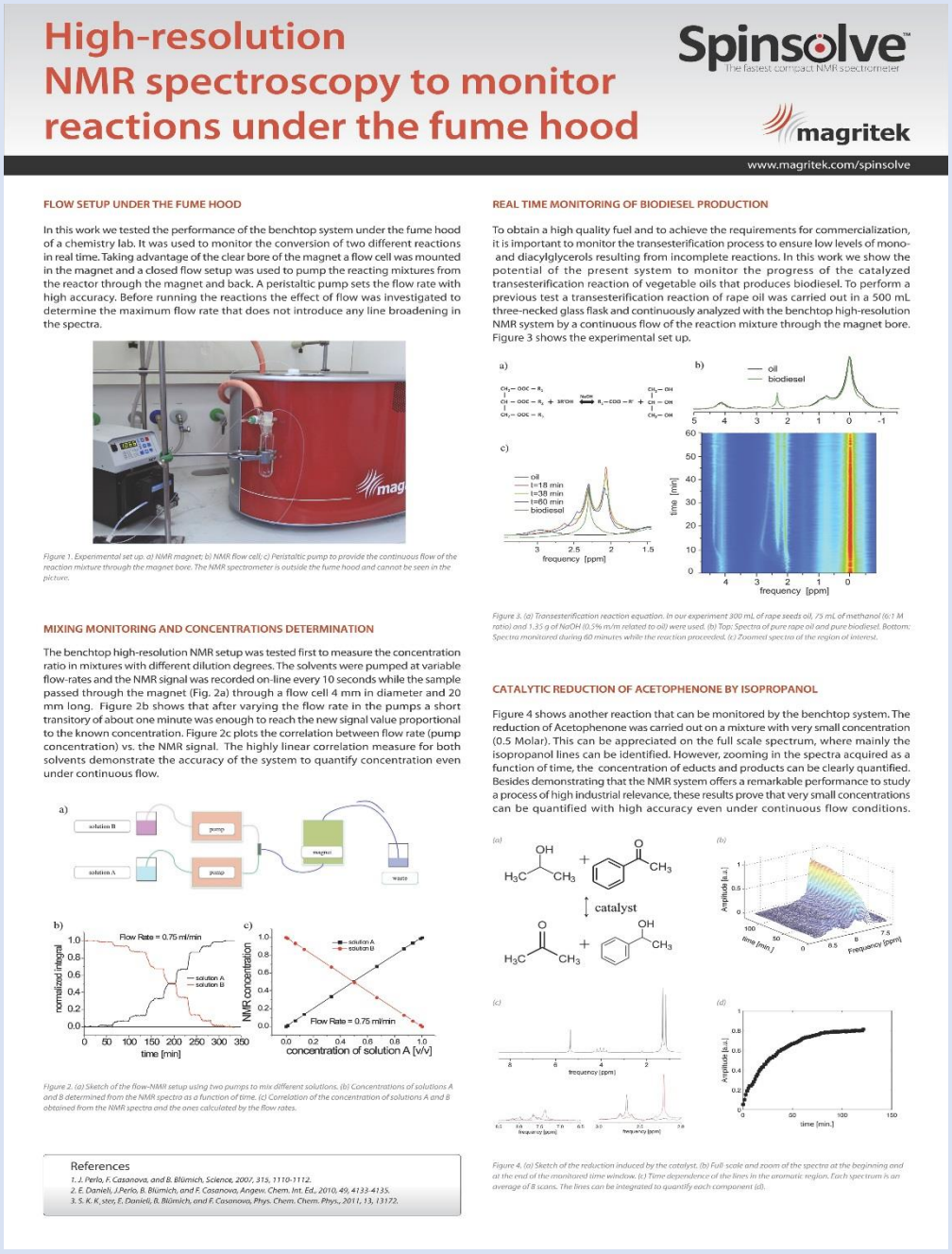
機種：下記 4 種です。
^{1}H 専用
^{1}H, ^{19}F　　2D NMR
^{1}H, ^{19}F, ^{13}C 2D NMR
^{1}H, ^{19}F, ^{31}P 2D NMR

主な仕様：
周波数 42.5MHz(^{1}H)
寸法：L58xW43xH40cm
重量：約 55Kg
磁石：永久磁石



High-resolution NMR spectroscopy to monitor reactions under the fume hood

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FLOW SETUP UNDER THE FUME HOOD

In this work we tested the performance of the benchtop system under the fume hood of a chemistry lab. It was used to monitor the conversion of two different reactions in real time. Taking advantage of the clear bore of the magnet a flow cell was mounted in the magnet and a closed flow setup was used to pump the reacting mixtures from the reactor through the magnet and back. A peristaltic pump sets the flow rate with high accuracy. Before running the reactions the effect of flow was investigated to determine the maximum flow rate that does not introduce any line broadening in the spectra.

Figure 1. Experimental set up. a) NMR magnet; b) NMR flow cell; c) Peristaltic pump to provide the continuous flow of the reaction mixture through the magnet bore. The NMR spectrometer is outside the fume hood and cannot be seen in the picture.

MIXING MONITORING AND CONCENTRATIONS DETERMINATION

The benchtop high-resolution NMR setup was tested first to measure the concentration ratio in mixtures with different dilution degrees. The solvents were pumped at variable flow-rates and the NMR signal was recorded on-line every 10 seconds while the sample passed through the magnet (Fig. 2a) through a flow cell 4 mm in diameter and 20 mm long. Figure 2b shows that after varying the flow rate in the pumps a short transitory of about one minute was enough to reach the new signal value proportional to the known concentration. Figure 2c plots the correlation between flow rate (pump concentration) vs. the NMR signal. The highly linear correlation measure for both solvents demonstrate the accuracy of the system to quantify concentration even under continuous flow.

Figure 2. (a) Sketch of the flow-NMR setup using two pumps to mix different solutions. (b) Concentrations of solutions A and B determined from the NMR spectra as a function of time. (c) Correlation of the concentration of solutions A and B obtained from the NMR spectra and the ones calculated by the flow rates.

References

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REAL TIME MONITORING OF BIODIESEL PRODUCTION

To obtain a high quality fuel and to achieve the requirements for commercialization, it is important to monitor the transesterification process to ensure low levels of mono- and diacylglycerols resulting from incomplete reactions. In this work we show the potential of the present system to monitor the progress of the catalyzed transesterification reaction of vegetable oils that produces biodiesel. To perform a previous test a transesterification reaction of rape oil was carried out in a 500 mL three-necked glass flask and continuously analyzed with the benchtop high-resolution NMR system by a continuous flow of the reaction mixture through the magnet bore. Figure 3 shows the experimental set up.

Figure 3. (a) Transesterification reaction equation. In our experiment 300 mL of rape seeds oil, 75 mL of methanol (6:1 M ratio) and 1.35 g of NaOH (0.5% m/m related to oil) were used. (b) Top: Spectra of pure rape oil and pure biodiesel. Bottom: Spectra monitored during 60 minutes while the reaction proceeded. (c) Zoomed spectra of the region of interest.

CATALYTIC REDUCTION OF ACETOPHENONE BY ISOPROPANOL

Figure 4 shows another reaction that can be monitored by the benchtop system. The reduction of Acetophenone was carried out on a mixture with very small concentration (0.5 Molar). This can be appreciated on the full scale spectrum, where mainly the isopropanol lines can be identified. However, zooming in the spectra acquired as a function of time, the concentration of educts and products can be clearly quantified. Besides demonstrating that the NMR system offers a remarkable performance to study a process of high industrial relevance, these results prove that very small concentrations can be quantified with high accuracy even under continuous flow conditions.

Figure 4. (a) Sketch of the reduction induced by the catalyst. (b) Full scale and zoom of the spectra at the beginning and at the end of the monitored time window. (c) Time dependence of the lines in the aromatic region. Each spectrum is an average of 8 scans. The lines can be integrated to quantify each component (d).

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