

# REMSPEC Application Note

## # 00-01 The Reduction of *n*-Heptaldehyde with Sodium Borohydride

### Introduction

The synthesis of alcohols by the reduction of aldehydes with sodium borohydride is a reaction that is widely used in industrial organic chemistry. Before the development of fiber-optic IR spectroscopy there was no simple, real-time method for following the course of these reactions in large and small reaction vessels. To obtain maximum conversion of aldehyde, it is common to use a reaction time determined by the individual chemist's experience coupled with generally accepted practice, and this is often much longer than necessary. Apart from the inefficient use of time, this approach allows for the development of unwanted byproducts from side reactions.

### Real-Time In-Situ Monitoring

Real-time, in-situ monitoring of reaction progress is the ideal answer to questions about realistic reaction times. When the reactants, products or intermediates are infrared active and are present in adequate concentrations, the integrated Remspec Mid-IR Fiber-Optic Reaction Monitoring System provides the answer. With this system, any liquid-phase reaction can be monitored, even when a solid catalyst is present. The Remspec system uses an ATR (attenuated total reflectance) probe directly coupled to a fiber optic cable that is linked to the spectrometer. The use of the ATR technique largely eliminates the problem of signal absorption by the solvent, or of

scattering by suspended solids. The sampling errors and delays associated with manual methods are avoided by in-situ measurement of the spectrum. Both room temperature and refluxing reactions can readily be monitored using the Remspec probe, allowing the chemist to know the status of a reaction in real-time. An added advantage is the slim design of the Remspec probe which allows it to be used with standard laboratory glassware – no special fittings are needed. The result is enhanced yields, higher-purity end products, and improved productivity.

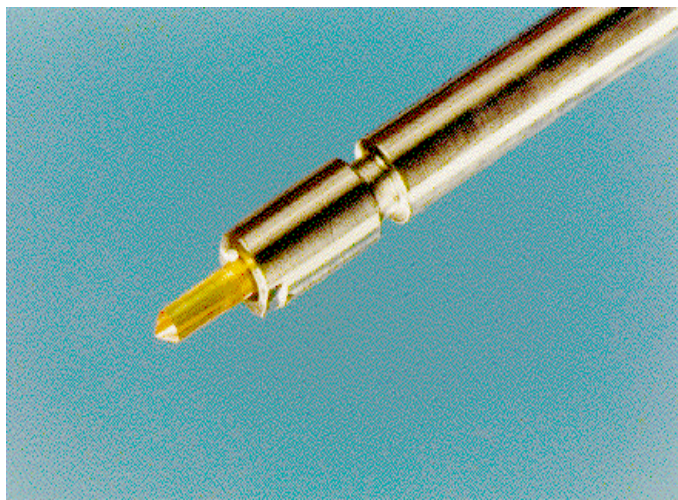


Figure 1: The ATR head of a Remspec Mid-IR Fiber-Optic Immersion Probe

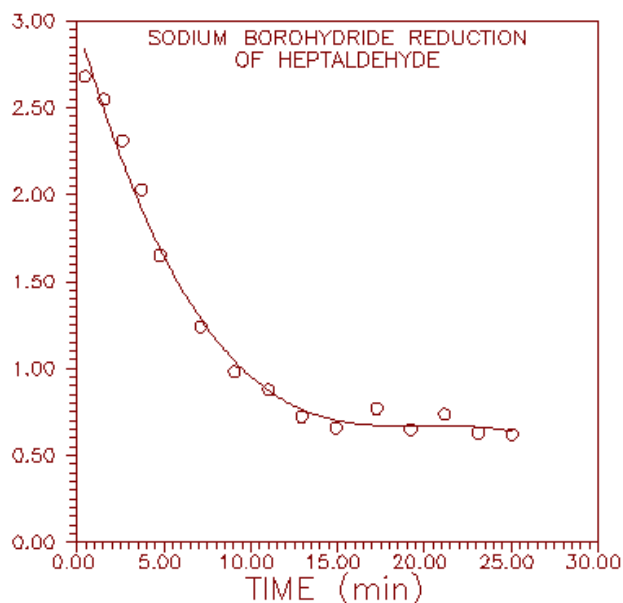
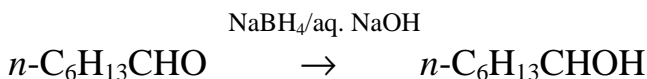


Figure 2. Concentration of *n*-heptaldehyde as a function of time.

## Experimental

Preparation of *n*-heptanol from *n*-heptaldehyde:



This experiment was performed using a Remspec Mid-IR Fiber-Optic Probe with a ZnSe ATR element. The reaction was carried out in a three necked flask equipped with a magnetic stir bar and purged with nitrogen. The probe was inserted to fully immerse the tip using a simple ring stand and clamp. The FTIR instrument was a standard unit equipped with a "flipper mirror" for diverting the signal to the collimated beam port and the capability to accept the output from an external detector.

Reagents:

*n*-heptaldehyde, 3.40g (0.0298 mole)  
 sodium borohydride, 0.070g (0.0019 mole)  
 ethanol, 4.0 ml

The ethanol and *n*-heptaldehyde were charged into the magnetically stirred flask, which was purged with nitrogen. Spectral scans were then initiated and the sodium borohydride was added.

## Discussion

Spectra were determined every minute during the initial part of the run and less frequently later. The carbonyl absorption band for *n*-heptaldehyde at 1732 cm<sup>-1</sup> is clearly isolated and its intensity was monitored over time to follow the reaction.

Figure 2 is a plot of the peak area for the carbonyl band as a function of time normalized to the concentration of *n*-heptaldehyde at *t* = 0. It can be clearly seen from the plot of that the reaction is over rather quickly -- within fifteen minutes, it is almost complete.

## Commentary

This reaction is typical of hydrogen reductions that are routinely performed in chemical laboratories every day. By selecting the strong, isolated carbonyl stretch of the *n*-heptaldehyde as the peak to be monitored, it was easy to plot the concentration/time graph for the starting material. For routine use, or for industrial process monitoring, automatic, real-time display of concentration/time curves for either reactants or products can be programmed using the spectrometer control software.

The use of the Remspec Mid-IR Fiber-Optic Immersion Probe gives the scientist or engineer the ability to follow the course of any reaction, organic or inorganic, rapid or slow, and in any liquid medium desired -- aqueous or non-aqueous -- for improved analytical and synthetic productivity.

**For more information contact:**

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