

Automating DNPH Derivatisation for Aldehyde Analysis

Dan Carrier, Anatune Limited

Tel: 01223 279 210

Web: www.anatune.co.uk

Introduction

Airborne aldehydes and ketones are collected by passing air through a cartridge containing 2,4-dinitrophenylhydrazine (DNPH). The Supelco cartridge is silica based and pre-coated with DNPH. Carbonyl compounds react with the DNPH to form derivatives in the form of hydrazones which are immobilised on the cartridge. Figure 1 shows the derivatisation of aldehydes and ketones using DNPH. These compounds can be easily eluted from the cartridge with acetonitrile and analysed by HPLC with UV detection.

To automate this method, a good seal is required between the DNPH cartridge and the injection needle from the syringe attached to Multipurpose sampler (MPS). This will allow delivery of the solvent through the cartridge. Figure 2 shows how the DNPH cartridge is put together for use on the DNPH unit (Anatune 300).

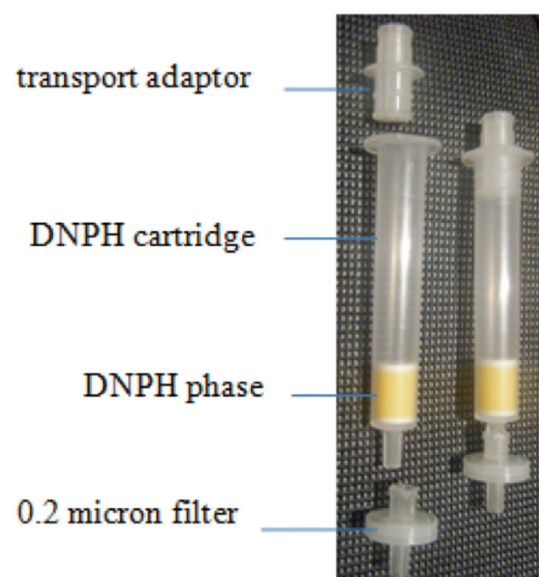


Figure 2: Picture of DNPH cartridge with sealing unit (transport adaptor) and 0.2 micron filter

Automated Method

After the DNPH cartridge has been aligned with an empty 10 ml vial, 5.6 ml of acetonitrile is added to the cartridge using a 5 ml

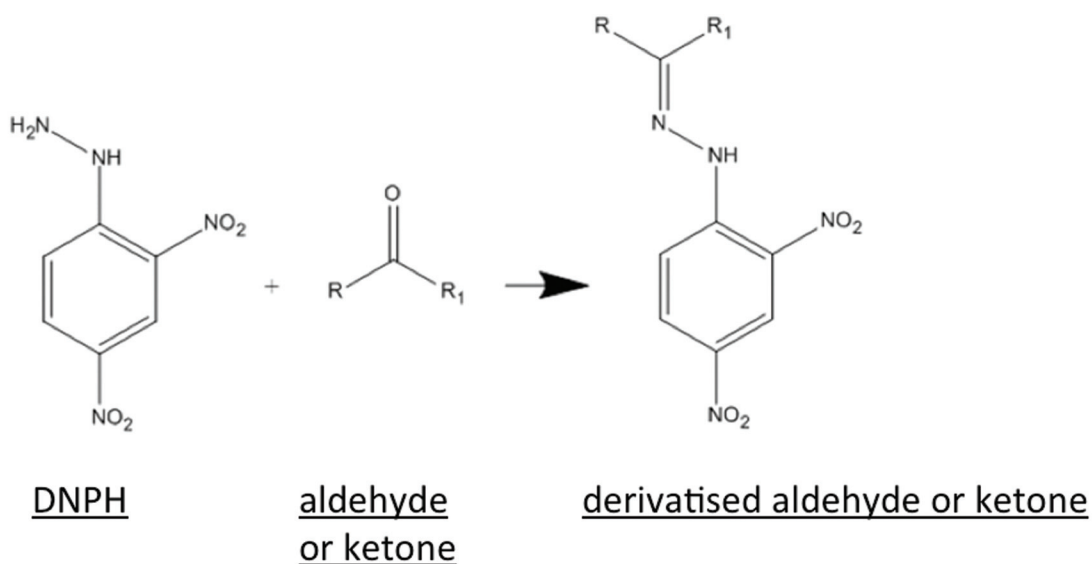


Figure 1: Reaction of DNPH with aldehydes or ketones

syringe on an MPS head. An additional air push through the cartridge performed. This is to maximise the amount of extract produced. The extract is then mixed and a 1 ml aliquot (using a 1 ml syringe) is then added to a sealed 2 ml HPLC vial which is seated within the cooled tray. This solution is then injected onto the HPLC. Up to 64 extractions can be automated. Figure 3 shows a close up photograph of the DNPH unit (Anatune 300).

For this study, 20 automated extractions were performed. Each extraction uses new transport adaptor, new SPE cartridge (LpDNPH 3ml 350 mg) and a new 0.2 micron filter (Nylon). 5.6 ml of acetonitrile was added to each cartridge.

Results and Discussion

The volume of acetonitrile was recorded for 20 automated extractions. The mean volume was calculated to be 4.94 ml and the coefficient of variation was calculated to be 1.47%.

The automation of this extraction drastically improves productivity by freeing up manual labour associated with this technique and is likely to improve any potential experimental errors associated with the method. The new system capable of automating up to 64

DNPH extractions will shortly be installed at Reckitt Benckiser for aldehyde and ketone analysis in Hull. I would like to thank Vicki Morris at Reckitt Benckiser for allowing Anatune to mention this new system, which includes the new automated DNPH unit (Anatune 300).

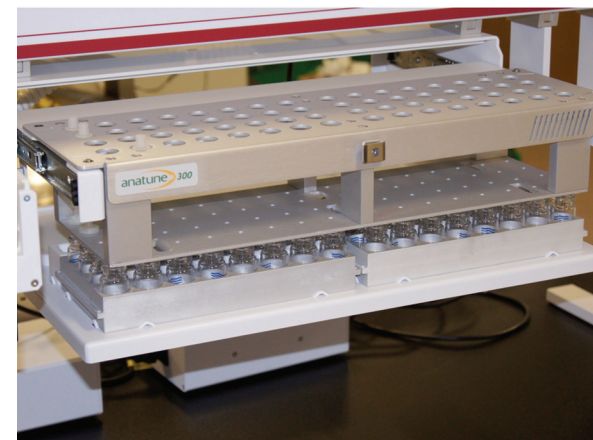


Figure 3: Anatune 300 - Automated DNPH unit

Reader Reply Card No. 127