Improved Separation of Aldehydes and Ketones by replacing a Solvent Gradient with an Isocratic High Temperature Method

Introduction

Aldehydes and ketones are air pollutants that are emitted by internal combustion engines. The California Air Resources Board (CARB) has a method for the determination of these compounds in automotive source samples. The aldehydes and ketones in air are trapped using cartridges or impingers treated with 2,4dinitrophenylhydrazine (DNPH). The DNPH derivatives are collected and analyzed by reversed phase HPLC. The standard HPLC method is a solvent gradient method that takes approximately 30 minutes. This application note outlines a shorter method achieved using high temperature and two HPLC columns. The new method is an isocratic method and the run time was reduced to 15 minutes.

Table 1:
Conditions for Analysis of Aldehydes and Ketones in Auto Emissions

Column:	Selerity Blaze$_{200}$ C_{18}, 3 um, 150 x 4.6 mm
Solvent gradient Method:	10:90 Acetonitrile:Water, 10 to 60% ACN over 30 minutes, ambient temperature, one column
Isocratic High Temperature Method:	25:75 Acetonitrile:Water, 150°C, two columns in series
Flow Rate:	1.0 mL/min solvent gradient method, 3.0 mL/min isocratic method
Detection:	UV-VIS at 365 nm
Injection:	5 µL

Experimental

HPLC conditions are summarized in Table 1. A Dionex pump and detector was used with an Alltech vacuum degasser and a Selerity Series 9000 Polaratherm Total Temperature Controller.

Results and Discussion

Figure 1 shows the solvent gradient analysis, and Figure 2 shows the isocratic high temperature analysis. Figure 3 shows an overlay of the two chromatograms. The solvent gradient method has an analysis time of 30 minutes and requires additional re-equilibration before the next sample can be run. The high temperature method, in which two columns were connected in series to provide increased resolution, had a total analysis time of only 15 minutes. The isocratic separation could be run at 3.0 mL/min even though two columns were used. The higher flow rate was possible because of the reduction of mobile phase viscosity and system back pressure at the high temperature conditions. There is also a temperature induced selectivity change for methylethylketone and methacrolein.

Conclusions

A solvent gradient method was replaced by a high temperature isocratic method to reduce the analysis time for the analysis of DNPH derivatives of aldehydes and ketones in auto emission samples.

Acknowledgement

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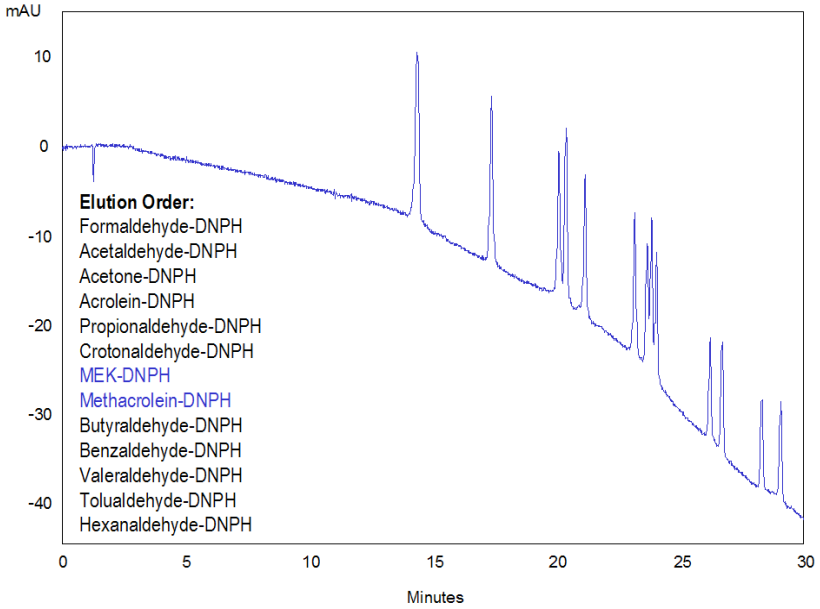

Figure 1: *Solvent gradient analysis of aldehydes and ketones.*

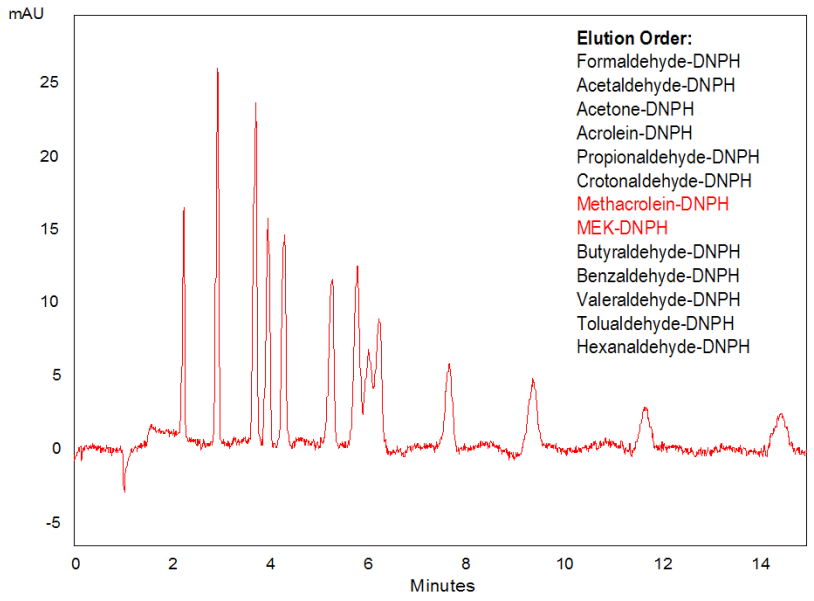

Figure 2: *Isocratic high temperature method for aldehydes and ketones using two columns in series at 150°C.*

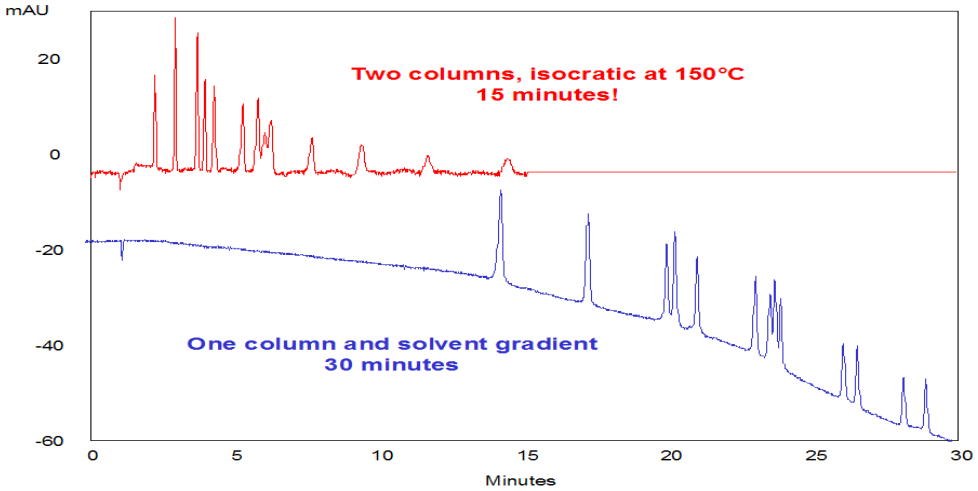

Figure 3: *Overlay of the two analysis methods. Run time has been reduced to 15 minutes using the high temperature isocratic method.*