

Nota applicativa

Fast Analysis of Aldehydes and Ketones by ACQUITY UPLC

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Abstract

This application note demonstrates that with ACQUITY UPLC System, analysis times can be reduced by as much as 75 percent, thereby increasing throughput and productivity in the lab.

Introduction

Aldehydes and ketones are products of combustion that permeate the environment. A number of these compounds are known carcinogens and as a result, several US EPA and state methods have been developed for their analysis. These methods, US EPA TO5 (air), 554 (drinking water), 8315 A, Options 1 and 2, (waste water, soil, and air), along with California method 1004 (carbonyl compounds as alcohol oxidants in auto exhaust), describe the derivatization of these compounds using dinitrophenylhydrazine (DNPH) followed by HPLC separation and ultraviolet (UV) detection at 360 nm. Target analytes vary slightly by method. Present methodologies describe run times of over 40 minutes and multiple column setups to achieve acceptable analyte resolution.

This application note demonstrates that with ACQUITY UPLC System, analysis times can be reduced by as much as 75 percent, thereby increasing throughput and productivity in the lab. For example, in a typical 8-hour day, one could run 12 40-minute, traditional HPLC analyses. Using the ACQUITY UPLC System, this becomes 48 ten-minute analyses or 36 more runs per day.

In addition to the increased throughput, excellent chromatographic resolution is achieved for the indoor air method (particularly for acetone, acrolein and propanal), and the auto exhaust method, particularly for methacrolein and methyl ethyl ketone). For air sampling, Waters offers DNPH coated cartridges including Sep-Pak DNPH-Silica (WAT037500 <<https://www.waters.com/nextgen/us/en/shop/sample-preparation--filtration/wat037500-sep-pak-dnph-silica-plus-short-cartridge-350-mg-sorbent-per-cart.html>>) for outdoor air and XPoSure Sampler (WAT047205 <<https://www.waters.com/nextgen/us/en/shop/sample-preparation--filtration/wat047205-sep-pak-xposure-plus-short-cartridge-350-mg-sorbent-per-cartridg.html>>) for indoor air. To eliminate ozone interferences, Scrubber cartridges are also available from Waters (WAT054220).



ACQUITY UPLC System.

Experimental

Chromatographic Conditions

| | |
|-----------------|--|
| System: | ACQUITY UPLC System |
| Injection mode: | Full loop |
| Loop size: | 5 μ L (5 μ L injection volume) Use 15 μ L needle |
| Weak wash: | 5% aqueous acetonitrile - 800 μ L |
| Strong wash: | 50% aqueous acetonitrile - 500 μ L |
| Sample temp.: | 25 $^{\circ}$ C |
| Detection: | UV @ 360 nm |

Allow 2 minute equilibration between injections.

Column: Waters ACQUITY UPLC BEH Phenyl 2.1 x 100 mm,
1.7 μm @ 35 °C

Eluent: A - 90:10 water - THF (stabilized)*
B - acetonitrile

*Mix 900 mL water and 100 mL stabilized tetrahydrofuran (THF), filter and degas.

Flow rate: 0.5 mL/min

Software

Data were acquired and processed with Waters Empower Chromatography Software.

Results and Discussion

The chromatographic gradient profiles are unique per the method used due to the differences in the selectivity of target analytes. EPA method 554 (drinking water), and 8315 A Option 1 (collected by method 0011), target the same 12 analytes, including the strongly retained C5-C10 compounds (pentanal-decanal). The gradient profile (Table 1) and the UPLC chromatogram (Figure 1) are shown.

| | Time | Flow | %A | %B | Curve |
|---|---------|------|------|------|---------|
| 1 | Initial | 0.5 | 70.0 | 30.0 | Initial |
| 2 | 6.5 | 0.5 | 53.0 | 47.0 | 6 |
| 3 | 9.5 | 0.5 | 70.0 | 30.0 | 11 |

Table 1. Gradient profile for EPA method 554.

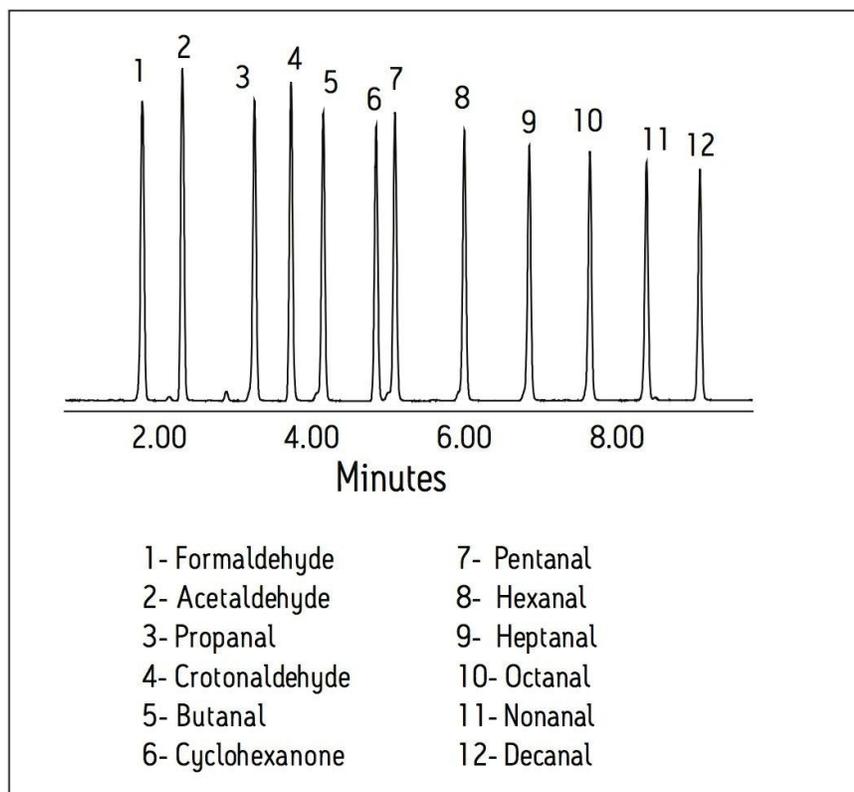


Figure 1. EPA method 554,8315 A-Opt. 1 analytes 20 ppm as DNPH derivatives.

EPA method 8315 A Option 2 targets 15 analytes including the difficult baseline triad of acetone, acrolein and propanal, along with the three tolualdehyde isomers (o, p, m). This is an indoor air method using collection protocol 0100. The gradient profile (table 2) and UPLC chromatogram (Figure 2) are shown.

| | Time | Flow | %A | %B | Curve |
|---|---------|------|------|------|---------|
| 1 | Initial | 0.5 | 70.0 | 30.0 | Initial |
| 2 | 9.0 | 0.5 | 36.0 | 64.0 | 6 |
| 3 | 9.5 | 0.5 | 70.0 | 30.0 | 11 |

Table 2. Gradient profile for EPA method 8315.

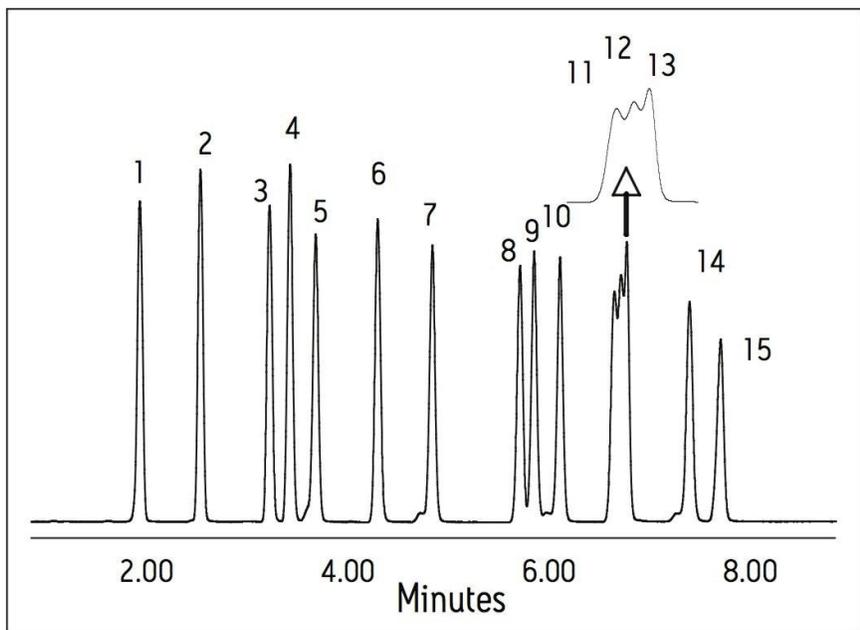


Figure 2. EPA method 8315 A-Opt. 2 analytes 20 ppm as DNPH derivatives.

California method 1004 targets 14 analytes contained in the alcohol oxidation products emitted from auto engines exhausts. Here, the critical pair is methyl ethyl ketone and methacrolein. The gradient profile (Table 3) and UPLC chromatogram (Figure 3) are shown.

| | Time | Flow | %A | %B | Curve |
|---|---------|------|------|------|---------|
| 1 | Initial | 0.5 | 70.0 | 30.0 | Initial |
| 2 | 8.0 | 0.5 | 70.0 | 30.0 | 6 |
| 3 | 9.0 | 0.5 | 50.0 | 50.0 | 6 |
| 4 | 11.0 | 0.5 | 70.0 | 30.0 | 11 |

Table 3. Gradient profile for California method 1004.

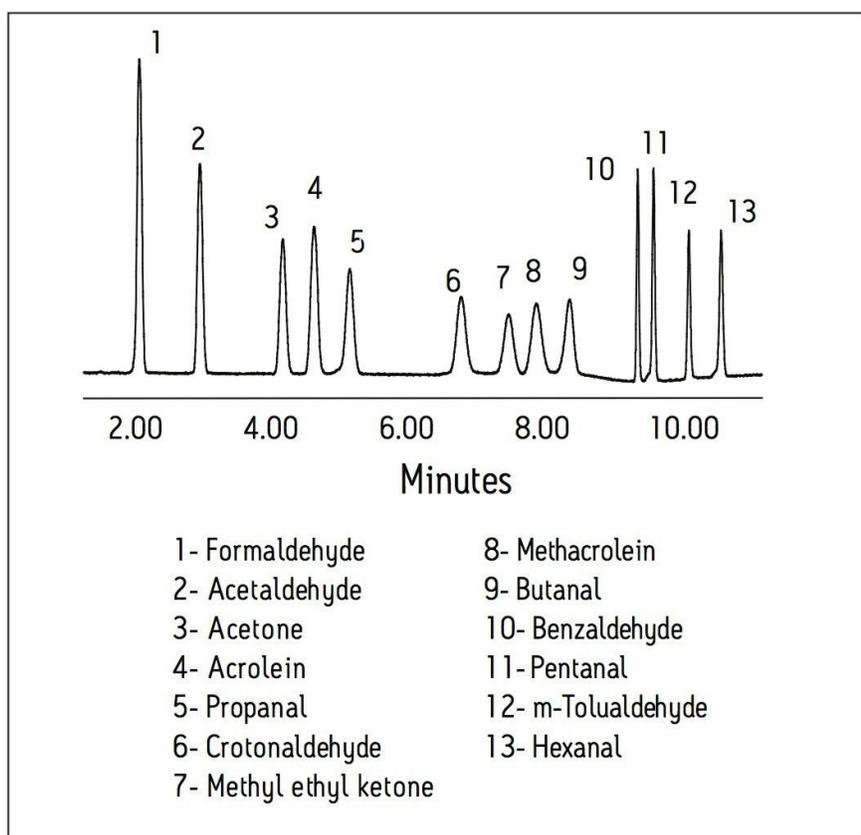


Figure 3. California method 1004 analytes, 0.75 ppm as parent compounds.

As in any separation, retention time and area reproducibility are essential. Figure 4 is an overlay of 5 injections of the EPA 554 standard. As demonstrated in Table 4, RT and area reproducibility are less than 0.2% and 0.5% for

all analytes respectively.

| Analyte | % RSD RT | % RSD Area |
|----------------|----------|------------|
| Formaldehyde | 0.163 | 0.398 |
| Acetaldehyde | 0.119 | 0.408 |
| Propanal | 0.078 | 0.410 |
| Crotonaldehyde | 0.071 | 0.377 |
| Butanal | 0.061 | 0.336 |
| Cyclohexanone | 0.053 | 0.308 |
| Pentanal | 0.103 | 0.471 |
| Hexanal | 0.075 | 0.296 |
| Heptanal | 0.049 | 0.288 |
| Octanal | 0.039 | 0.267 |
| Nonanal | 0.026 | 0.333 |
| Decanal | 0.024 | 0.338 |

Table 4. Reproducibility data for 5 injections EPA 554 analytes.

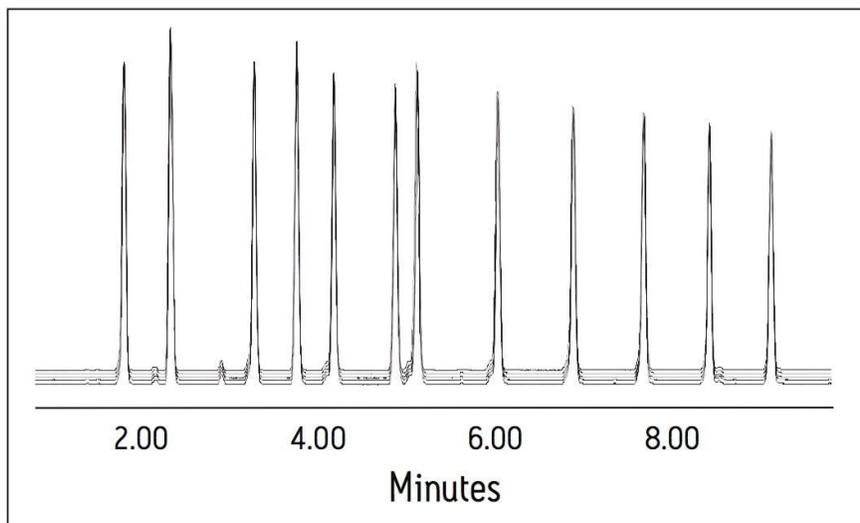


Figure 4. Overlay of 5 injections EPA 554 analytes.

Conclusion

Aldehydes and ketones can be analyzed as DNPH derivatives rapidly and efficiently using ACQUITY UPLC System. Results can be obtained several times faster than conventional HPLC technologies with corresponding cost savings, which allows more samples to be processed in a given day. By a simple variation of gradient conditions, the separation requirements of the major EPA methods 554 and 8315 A, Options 1 and 2, along with California method 1004 can be achieved.

Warning

Peroxide contaminants in THF can spontaneously and destructively explode when you partially or completely evaporate the THF.

System recommendation

THF, hexane, ethyl acetate, and acetone can be used as the mobile phase in ACQUITY UPLC H-Class, H-Class Bio, and I-Class Series Systems. As with many nonaqueous solvents, however, they can shorten system and instrument life compared with equipment running typical reversed-phase solvents. If you use THF, hexane, ethyl

acetate, or acetone, you must install a hexane/THF compatibility kit.

Featured Products

[ACQUITY UPLC System <https://www.waters.com/514207>](https://www.waters.com/514207)

[Empower Chromatography Data System <https://www.waters.com/10190669>](https://www.waters.com/10190669)

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