

Improve Analysis of Aldehydes and Ketones in Air Samples with Faster, More Accurate Methodology

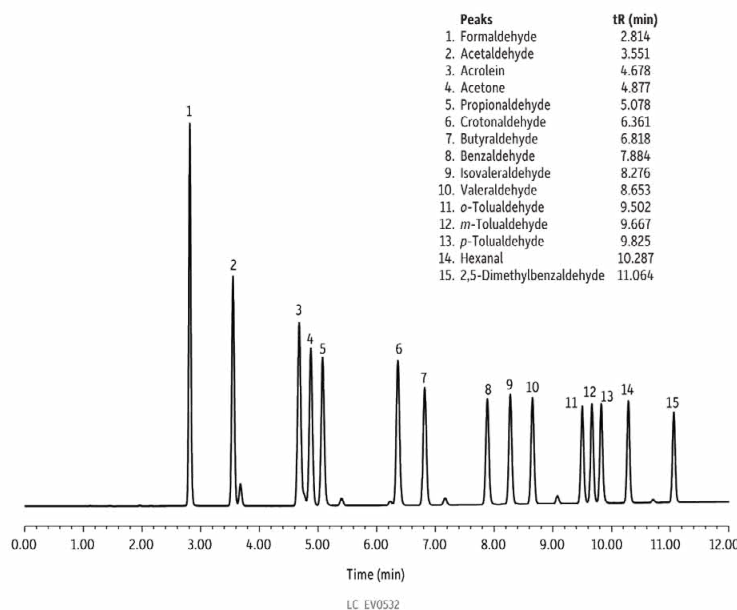
- Baseline separation ensures accurate determination of 15 commonly analyzed aldehydes and ketones in air samples.
- Raptor™ LC columns give you results in minutes—not hours—for increased sample throughput.
- Restek® certified reference materials are specially formulated for ambient air and automotive emissions testing.

Accurate analysis of aldehydes and ketones in air samples is an important part of monitoring programs that are used to control air pollution and to protect human health around the world. These airborne carbonyls from industrial and vehicle emissions are precursors to ozone in smog and are associated with respiratory and pulmonary problems. In addition, specific carbonyls—formaldehyde and acetaldehyde—have been identified as known or suspected carcinogens. Prior to analysis, aldehydes and ketones are converted to hydrazone derivatives when they are captured on a sampling tube composed of silica gel coated with 2,4-dinitrophenylhydrazine (DNPH). Then, the aldehyde/ketone DNPH derivatives are eluted from the sampling tube with solvent and analyzed by HPLC-UV. Typically, carbonyl LC methods require long analysis times (often over an hour), but many still fail to provide adequate resolution for some aldehyde/ketone DNPH derivatives.

To help labs improve analytical speed and accuracy in the analysis of aldehydes and ketones in air samples, Restek has developed LC columns, instrument conditions, and certified reference materials that reliably provide baseline separation of commonly analyzed aldehyde/ketone DNPH derivatives in just minutes. For example, airborne carbonyls LC method U.S. EPA Method TO-11A uses a conventional C18 column and requires an analysis time of over one hour, yet acrolein and acetone partially coelute and the *o*-, *m*-, and *p*-tolualdehyde isomers cannot be fully separated. In contrast, using a Raptor™ C18 column under the conditions shown below, all 15 carbonyls—including the tolualdehyde isomer cluster—are separated to baseline in a fast 14-minute analysis.

Similarly, airborne carbonyl LC method CARB 1004 produces extremely long analysis times; in fact, this method requires two separate chromatographic analyses in order to quantify all compounds. The first is a 37-minute analysis using two columns connected in series. Even with two columns and a long analysis time, methyl ethyl ketone and butyraldehyde coelute in this analysis. To report these two compounds, another 30-minute chromatographic analysis with a different column is needed. However, this second analysis cannot be used to report all compounds due to the coelution of formaldehyde with a non-carbonyl compound. This same list of CARB 1004 aldehyde/ketone DNPH derivatives can be fully resolved by the second Restek® method shown below using a Raptor™ ARC-18 column in a fast 8.5-minute analysis—an 8-fold reduction in analysis time with a single column.

Both of these methods from Restek are compatible with conventional 400 bar HPLC systems and benefit from the larger, low-clog frit used in Raptor™ column hardware. Using either Raptor™ LC column—along with Restek's airborne carbonyl reference standards—assures fast, accurate analysis of aldehydes and ketones in air and offers an opportunity for improved sample throughput compared to typical columns and methods.



Column
 Dimensions: Raptor™ C18 (cat.# 9304A65)
 150 mm x 4.6 mm ID
 Particle Size: 2.7 µm
 Pore Size: 90 Å
 Temp.: 30 °C
Sample
 Aldehyde-ketone-DNPH TO-11A calibration mix (cat.# 31808)
 Diluent: Acetonitrile
 Conc.: 15 µg/mL
 Inj. Vol.: 2 µL
Mobile Phase
 A: Water
 B: Methanol:acetonitrile (650:50)*

Time (min)	Flow (mL/min)	%A	%B
0.00	0.8	30	70
5.00	0.8	25	75
11.00	0.8	10	90
11.01	0.8	0	100
12.00	0.8	0	100
12.01	0.8	30	70
14.00	0.8	30	70

Detector Notes

UV/Vis @ 365, 4.8 nm
 *Mobile phase B was prepared by combining 650 mL methanol and 50 mL acetonitrile.

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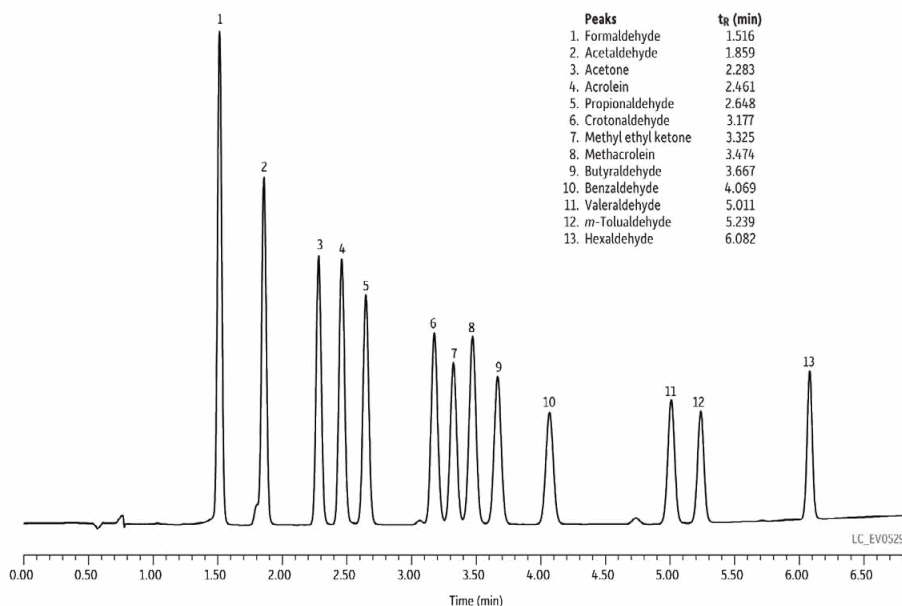


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Column Raptor™ ARC-18 (cat.# 9314A1E)
Dimensions: 100 mm x 3.0 mm ID
Particle Size: 2.7 µm
Temp.: 40 °C
Sample CARB 1004 aldehyde/ketone-DNPH calibration standard (cat.# 33093)
Diluent: Acetonitrile
Conc.: 3 µg/mL
Inj. Vol.: 3 µL
Mobile Phase
 A: Water:tetrahydrofuran (5:2)
 B: Acetonitrile

Time (min)	Flow (mL/min)	%A	%B
0.00	0.6	70	30
3.5	0.6	65	35
6.5	0.6	30	70
6.51	0.6	70	30
8.5	0.6	70	30

Max Pressure: 240 bar
Detector UV/Vis @ 365, 4 nm
Notes Using stainless steel tubing and fittings instead of PEEK material is recommended when the mobile phase contains high concentrations of tetrahydrofuran.

Raptor™ C18 LC Columns (USP L1)

Description	cat.#
2.7 µm Columns	
150 mm, 4.6 mm ID	9304A65

Raptor™ ARC-18 LC Columns (USP L1)

Description	cat.#
2.7 µm Columns	
100 mm, 3.0 mm ID	9314A1E

EXP® Direct Connect Holder

Description	qty.	cat.#
EXP Direct Connect Holder for EXP Guard Cartridges (includes hex-head fitting & 2 ferrules)	ea.	25808



Raptor™ EXP® Guard Column Cartridges

Description	Particle Size	qty.	5 x 2.1 mm cat.#	5 x 3.0 mm cat.#	5 x 4.6 mm cat.#
Raptor ARC-18 EXP Guard Column Cartridge	2.7 µm	3-pk.	9314A0252	9314A0253	9314A0250
Raptor C18 EXP Guard Column Cartridge	2.7 µm	3-pk.	9304A0252	9304A0253	9304A0250

Maximum cartridge pressure:
 600 bar/8,700 psi (2.7 µm) or
 400 bar/5,800 psi (5 µm).

Aldehyde-Ketone-DNPH TO-11A Calibration Mix (15 components)

Acetaldehyde-DNPH (1019-57-4)	n-Butyraldehyde-DNPH (1527-98-6)	Hexaldehyde-DNPH (1527-97-5)	o-Tolualdehyde-DNPH (1773-44-0)
Acetone-DNPH (1567-89-1)	Crotonaldehyde-DNPH (1527-96-4)	Isovaleraldehyde-DNPH (2256-01-1)	p-Tolualdehyde-DNPH (2571-00-8)
Acrolein-DNPH (888-54-0)	2,5-Dimethylbenzaldehyde-DNPH (152477-96-8)	Propionaldehyde-DNPH (725-00-8)	Valeraldehyde-DNPH (2057-84-3)
Benzaldehyde-DNPH (1157-84-2)	Formaldehyde-DNPH (1081-15-8)	m-Tolualdehyde-DNPH (2880-05-9)	
15 µg/mL each in acetonitrile, 1 mL/ampul*			
cat.# 31808 (ea.)			

CARB 1004 Aldehyde/Ketone-DNPH Calibration Standard (13 components)

Acetaldehyde-2,4-DNPH (1019-57-4)	n-Butyraldehyde-2,4-DNPH (1527-98-6)	Hexaldehyde-2,4-DNPH (1527-97-5)	Propionaldehyde-2,4-DNPH (725-00-8)
Acetone-2,4-DNPH (1567-89-1)	Crotonaldehyde-2,4-DNPH (1527-96-4)	Methacrolein-2,4-DNPH (5077-73-6)	m-Tolualdehyde-2,4-DNPH (2880-05-9)
Acrolein-2,4-DNPH (888-54-0)	Formaldehyde-2,4-DNPH (1081-15-8)	Methyl ethyl ketone-2,4-DNPH (958-60-1)	Valeraldehyde-2,4-DNPH (2057-84-3)
Benzaldehyde-2,4-DNPH (1157-84-2)			
3 µg/mL each in acetonitrile, 1 mL/ampul*			
cat.# 33093 (ea.)			

*The reported concentrations reflect the amount of aldehyde or ketone in the mixture. The concentration of derivatized aldehyde or ketone is not reported.



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Lit. Cat.# EVSS2393-UNV



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