FAST FACTS

Aldehyde/Ketone DNPH Analysis

For CARB 1004, ASTM Method D5197, and US EPA Methods TO-11A and 8315

- Convenient concentrations for most ambient air methods.
- Certificate of Analysis lists both aldehyde/ketone and DNPH derivative concentrations.
- Fast analysis — Allure® AK HPLC column separates 13 carbonyl compounds in less than 15 minutes.

Carbonyl compounds, including low molecular weight aldehydes and ketones, are receiving increased attention from the regulatory community. Formaldehyde in automobile exhaust accounts for 50-70 percent of the total atmospheric carbonyl burden. Motor vehicles emit reactive hydrocarbons that undergo photochemical oxidation in the atmosphere, producing formaldehyde and other carbonyls. Short-term exposure to formaldehyde and other specific aldehydes (acetaldehyde, acrolein, crotonaldehyde) causes irritation of the eyes, skin, and mucous membranes of the upper respiratory tract. Formaldehyde is also a major promoter in the formation of photochemical ozone.

CARB (California Air Resources Board) Method 1004 is used by the automotive industry to monitor a range of carbonyl compounds in engine exhaust. In this method, sample collection cartridges impregnated with 2,4-dinitrophenylhydrazine (DNPH), or impingers containing acidified DNPH, are used to sample the exhaust. After conversion to the DNPH derivatives, the carbonyl compounds are analyzed by HPLC. US EPA Compendium Method TO-11A and Method 8315 target formaldehyde, but at least 14 other carbonyl compounds also can be detected and quantified. Method TO-11A modifies the sampling procedures outlined in earlier Method TO-5; the newer method is based on the specific reaction between carbonyl compounds and the DNPH coating on a silica gel adsorbent (packed in cartridges), in the presence of a strong acid catalyst. The reaction produces stable, colored hydrazone derivatives.

To meet the needs of analysts monitoring these compounds, Restek offers a 13-component calibration standard, suitable for CARB 1004 testing. Additionally, we offer a 15-component calibration mix for US EPA Compendium Method TO-11A and Method 8315. The compound concentrations in these mixes are optimized for the respective methods and are suitable for most ambient air work. For convenience, our Certificate of Analysis lists concentrations for both the aldehydes/ ketones and the DNPH derivatives.

We developed the Allure® AK HPLC column specifically for the analysis of aldehydes and ketones. With a single 200mm column, excellent resolution of 13 carbonyl compounds can be achieved in less than 13 minutes. While C18 phases often cannot separate butyraldehyde and methyl ethyl ketone (MEK), the Allure® AK column shows excellent resolution of this difficult pair.

Additional Information

Formaldehyde and Other Aldehydes Committee on Aldehydes, Board of Toxicology and Environmental Hazards, National Research Council, National Academy Press, Washington, DC, 1981.

Excellent resolution of 13 carbonyls on an Allure® AK column in less than 13 minutes.

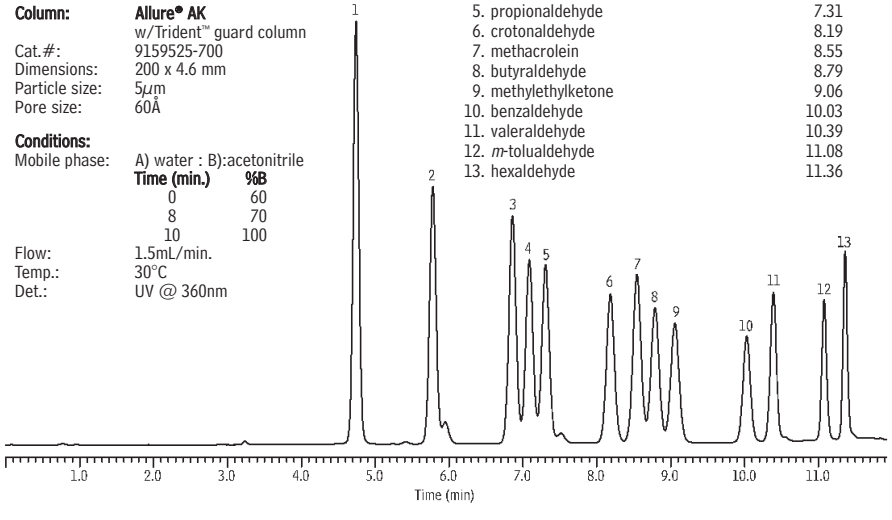
Sample:

Sample:	dinitrophenylhydrazine (DNPH) derivatives of aldehydes/ketones
Inj.:	10µL
Conc.:	3µg/mL each analyte, as aldehyde/ketone
Sample diluent:	acetonitrile
Column:	**Allure® AK** w/Trident™ guard column
Cat.#:	9159525-700
Dimensions:	200 x 4.6 mm
Particle size:	5µm
Pore size:	60Å
Conditions:	
Mobile phase:	A) water : B):acetonitrile

Time (min.)	%B
0	60
8	70
10	100

Flow:	1.5mL/min.
Temp.:	30°C
Det.:	UV @ 360nm

Peak DNPH derivative of:	Ret. Time (min.)
1. formaldehyde	4.74
2. acetaldehyde	5.78
3. acrolein	6.86
4. acetone	7.09
5. propionaldehyde	7.31
6. crotonaldehyde	8.19
7. methacrolein	8.55
8. butyraldehyde	8.79
9. methylethylketone	9.06
10. benzaldehyde	10.03
11. valeraldehyde	10.39
12. *m*-tolualdehyde	11.08
13. hexaldehyde	11.36



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

acetaldehyde-2,4-DNPH
acetone-2,4-DNPH
acrolein-2,4-DNPH
benzaldehyde-2,4-DNPH
n-butyraldehyde-2,4-DNPH
crotonaldehyde-2,4-DNPH
formaldehyde-2,4-DNPH
hexaldehyde-2,4-DNPH
methacrolein-2,4-DNPH
methyl ethyl ketone-2,4-DNPH
propionaldehyde-2,4-DNPH
m-tolualdehyde-2,4-DNPH
valeraldehyde-2,4-DNPH

3µg/mL each in acetonitrile, 1mL/ampul
cat. # 33093 (ea.)

DNPH Reference Materials

100µg/mL in acetonitrile, 1mL/ampul

Compound	CAS#	Individual cat.#
acetaldehyde-2,4-DNPH	1019-57-4	33074
acetone-2,4-DNPH	1567-89-1	33075
acrolein-2,4-DNPH	888-54-0	33076
benzaldehyde-2,4-DNPH	1157-84-2	33077
2-butanone-2,4-DNPH	958-60-1	33078
n-butyraldehyde-2,4-DNPH	1527-98-6	33079
crotonaldehyde-2,4-DNPH	1527-96-4	33080
2,5-dimethylbenzaldehyde-2,4-DNPH	152477-96-8	33081
formaldehyde-2,4-DNPH	1081-15-8	33082
glycolaldehyde-2,4-DNPH	—	33091
hexaldehyde-2,4-DNPH	1527-97-5	33083
isobutyraldehyde-2,4-DNPH	2057-82-1	33084
isovaleraldehyde-2,4-DNPH	2256-01-1	33085
methacrolein-2,4-DNPH	5077-73-6	33095
propionaldehyde-2,4-DNPH	725-00-8	33086
m-tolualdehyde-2,4-DNPH	2880-05-9	33088
o-tolualdehyde-2,4-DNPH	1773-44-0	33087
p-tolualdehyde-2,4-DNPH	2571-00-8	33089
valeraldehyde-2,4-DNPH	2057-84-3	33090

Formaldehyde-DNPH Mix

formaldehyde-DNPH
500µg/mL in acetonitrile, 1mL/ampul
cat. # 31837 (ea.)

Aldehyde-Ketone-DNPH TO-11A Calibration Mix

(15 components)

acetaldehyde-DNPH
acetone-DNPH
acrolein-DNPH
benzaldehyde-DNPH
n-butyraldehyde-DNPH
crotonaldehyde-DNPH
2,5-dimethylbenz aldehyde-DNPH
formaldehyde-DNPH
hexaldehyde-DNPH
isovaleraldehyde-DNPH
propionaldehyde-DNPH
m-tolualdehyde-DNPH
o-tolualdehyde-DNPH
p-tolualdehyde-DNPH
valeraldehyde-DNPH

15µg/mL* each in acetonitrile, 1mL/ampul
cat. # 31808 (ea.)

*Concentration calculated as aldehyde.

Allure® AK HPLC Columns

Physical Characteristics:

particle size: 5µm
pore size: 60Å
endcap: yes
pH range: 2.5 - 7.5
temperature limit: 80°C

5µm Column, 3.2mm	cat. #
200mm with Trident™ Inlet Fitting	9159523-700
5µm Column, 4.6mm	**cat. #**
200mm with Trident™ Inlet Fitting	9159525-700

Ultra C18 HPLC Columns (USP L1)

Ultra C18 HPLC columns separate aldehydes and ketones well and are also suitable for a wide range of other analyses. See our website for more information.

Physical Characteristics:

particle size:
3µm or 5µm, spherical
pore size: 100Å
carbon load: 20%
endcap: fully endcapped
pH range: 2.5 to 7.5
temperature limit: 80°C

5µm Column, 4.6mm	cat. #
150mm	9174565
150mm with Trident™ Inlet Fitting	9174565-700

also available

For guard cartridges for these columns, visit our website at **www.restek.com**.

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ISO 9001:2000 cert.# FM80397