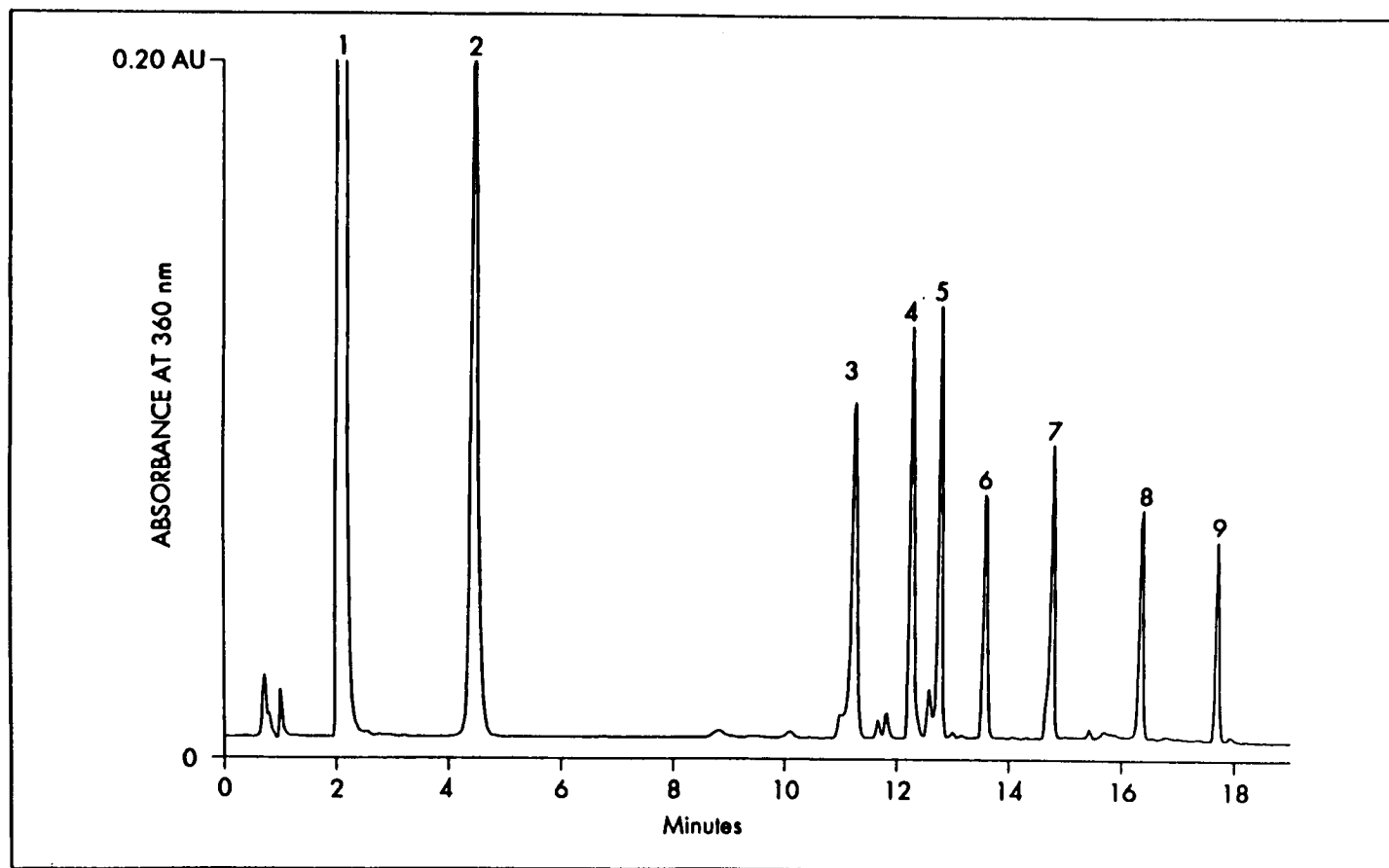


## Rapid Analysis of Aldehydes and Ketones From Water Matrices as 2,4-Dinitrophenylhydrazine (DNPH) Derivatives



### Conditions:

Sample: 2,4-Dinitrophenylhydrazine derivatives of aldehydes and ketones

Column: Nova-Pak™ C<sub>18</sub>  
(3.9mm x 150mm)

Eluent A: Water/Acetonitrile/  
Tetrahydrofuran (70:25:5)

Eluent B: Acetonitrile

Gradient:

Time	%A	%B	Curve
Init.	100	0	*
5	100	0	6
15	0	100	6

Flow Rate: 1.5 ml/min.

Detection: UV at 360 nm 0.2  
AUFS

Peak ID's:

1. 2,4-Dinitrophenylhydrazine
2. DNPH-Formaldehyde
3. DNPH-Propanal
4. DNPH-Butanal
5. DNPH-Cyclohexanone
6. DNPH-Crotonaldehyde
7. DNPH-Hexanal
8. DNPH-Octanal
9. DNPH-Decanal

The analysis of aldehydes and ketones from environmental sources, both air and water is of tremendous concern. The above application shows the ability to rapidly perform these determinations by utilizing reverse phase gradient HPLC conditions on a Nova-Pak C<sub>18</sub> column. The sample analyzed was a spiked reagent water sample which was concentrated as described in the Details section of the Note. The utilization of a photo-diode array detection system provided the ability to obtain spectral as well as chromatographic data for identification and quantitation purposes.

## Objective:

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The objective of this application was to provide analytical and sample preparation conditions to be used for the analysis of aldehydes and ketones from aqueous environmental sample sources. The conditions are modifications to EPA Method 8315 A which employs 2 Zorbax C<sub>18</sub> columns for the analysis and 3 enrichment cartridges.

## Details:

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Injection Volume: 10 $\mu$ l

Concentration: ~2ppm each component

Sample Preparation: Derivatization procedure as outlined in EPA Method 8315 A.

Add to 100 ml sample of water 4 ml 1M Sodium Citrate pH 3.0 and adjust pH to  $3.0 \pm .1$  with 6 M NaOH or 6 M HCl. Add 6 ml of a solution containing 3mg/ml 2,4-Dinitrophenylhydrazine in CH<sub>3</sub>CN. React for 1 hour at 40°C with gentle shaking. After 1 hour remove from shaker and add 10 ml of saturated NaCl solution to sample.

Prepare solid phase extraction cartridges for sample concentration as follows:

Attach 2 SepPak® C<sub>18</sub> Cartridges in series.

Wash cartridges with 10 ml CH<sub>3</sub>CN followed by 10 ml of 40 mM Sodium Citrate pH 3.0.

Add sample to extraction cartridges and apply vacuum or pressure to pass sample across cartridges

at approximately 5 ml/minute. Elute cartridges with approximately 9 ml of CH<sub>3</sub>CN into 10 ml volumetric flask. Adjust volume to 10 ml with CH<sub>3</sub>CN. Mix thoroughly and transfer a portion to a sealed vial until analyzed.

The gradient conditions employed for this separation were originally developed for the analysis of DNPH derivatized aldehydes and ketones for the monitoring of air samples, in particular auto exhaust emissions. Minor modifications to the original conditions were made to accommodate a slightly different list of components of interest. The EPA Method 8315 A as outlined requires two columns and approximately 1 hour of analysis time. The proposed alternative requires only 1 column and approximately 30 minutes for sample to sample turnaround time. Minor changes in gradient conditions can be utilized to accommodate more complex matrices which may be encountered. The use of the 991 can be helpful in component identification and detecting the possibility of co-elutions which is always present in complex sample matrices. The specificity of monitoring at a wavelength of 360 nm can eliminate a large number of possible matrix interferences.

## System:

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The System used for this application was a Model 600 Gradient System, 712 WISP autosampler and a 991 Photodiode array detector.

## References:

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R.L. Shriner et al, Systematic Identification of Organic Compounds, 6th edition; John Wiley and Sons, NY, 1980.

EPA METHOD 8315 A, DETERMINATION OF CARBONYL COMPOUNDS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC), REVISION WG 0 JANUARY 1991.

Care and use manual for Sep Pak DNPH cartridges.