

Free formaldehyde content in water soluble polymers

Analytical Regulations by Association TEGEWA

Principle

Quantitative determination of free formaldehyde (FD) in water soluble polymers after precipitation of the polymer and derivatization with 2,4-Dinitrophenylhydrazine.

Reagents

Water	Milli-Q Water System	(WEM)
Acetonitrile	HPLC Grade, Merck	(ACNL)
Ammoniumacetate	p.A. Merck	(AMAC)
Sep-Pak DNPH-Silica Cartridges Plus-Short Body (360 mg)	Waters AG (Part No. WAT037500)	(DNPH-Silica Cartridges)
Formaldehyde 2,4-dinitrophenylhydrazone	(QC-Nr.: 21097)	(FD-DNPH)

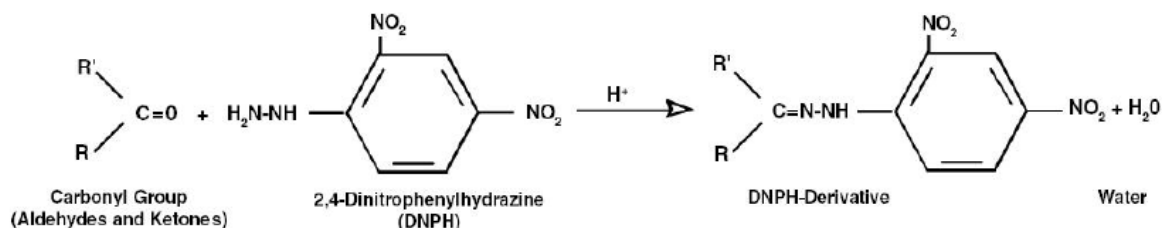
Calibration

Dissolve 45-55 mg FD-DNPH in 50.0 ml ACNL (stock solution, always prepare fresh solution). Dilute the stock solution 1:10, 1:100 and 1:1000 with ACNL and inject 2 µl.

Sample preparation

Put 20 ml ACNL into a 50 ml volumetric flask. Under agitation add drop wise 2500 – 3500 mg of the sample and agitate to hours. The polymer should precipitate. Fill the flask to the mark with ACNL. If necessary, adapt the sample amount to the capacity of the cartridge (see QA).

Put an 1 ml aliquote of the solution on the Waters DNPH-Silica Cartridge. Flow the cartridge with 5 ml ACNL into a 10 ml volumetric flask and fill to the mark with ACNL. Inject 2 µl.



HPLC conditions

Binary Solvent Manager:

Gradient

A:WEM + 0.05 mol/L AMAC

B:ACNL

Time: [min]	A [%]	B [%]	Flow [ml/min]
0.0	80.0	20.0	0.30
2.0	80.0	20.0	0.30
8.0	0	100.0	0.30
8.5	0	100.0	0.30
9.0	80.0	20.0	0.30
10.0	80.0	20.0	0.30

Column Manager:

Column: Acquity UPLC BEH C18

Particle size: 1.7 µm

Length: 50 mm ID: 2.1 mm

ID: ID: 2.1 mm

Supplier: Waters

Temperature: 30 °C

Sample Manager:

Method: Partial loop with needle overfill

Weak wash: 600 µl ACNL

Strong wash: 200 µl ACNL

Detector: (Waters PDA 2996)

Wavelength range: 210 – 450 nm

Extracted wavelength: 360 nm

Spectral resolution: 4.8

Sample rate: 20 pt/sec

Digital filter: 0.2

Instrument control and data processing with Empower 3.0.

Processing

Calibration (3 points) with external standard (linear including origin).

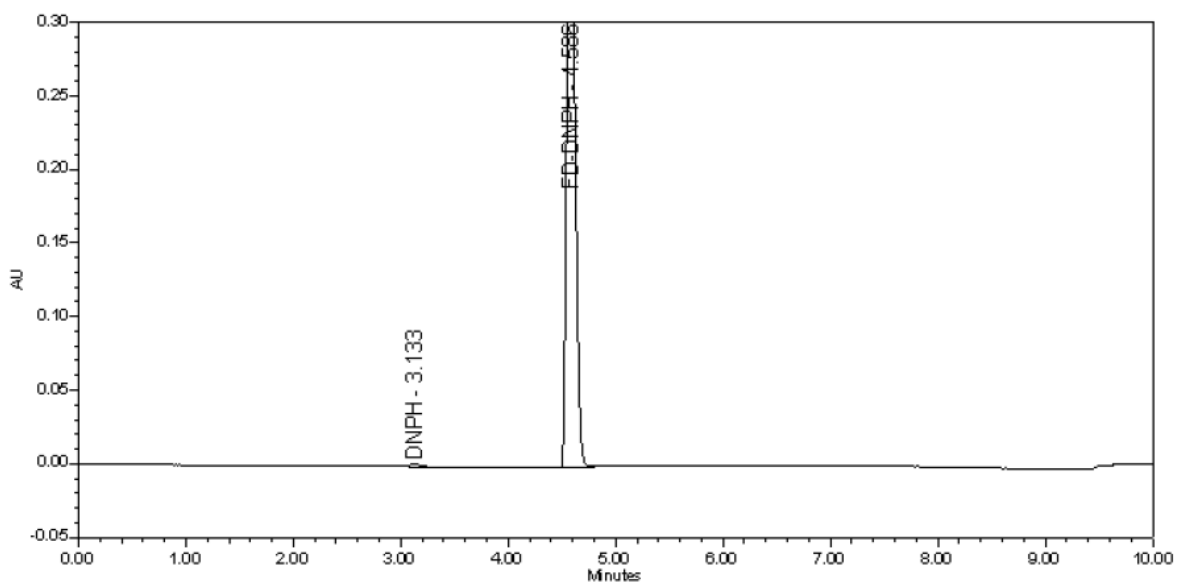
Content FD = Content FD-DNPH * 0.1428

QA

The capacity of the cartridge is limited. If on the chromatogram of the sample the intensity of the DNPH peak is lower than 0.25 times the intensity of the FD-DNPH peak, the analysis must be repeated using a smaller amount of the sample.

Retention times und chromatograms:

Standard:



RT 3.1 min DNPH

RT 4.6 min FD-DNPH

Sample: