NANOCOLOR® HC 300
Hydrocarbons

Test 0-57

REF 985 057 / 918 571 / 918 572

Tube test

Method: Patented photometric determination of hydrocarbons as chemical oxygen demand (COD) after pentane extraction from water and soil samples

Ranges:		Method
0.5 – 5.6 mg/l HC		**0571**
30 – 300 mg/kg HC		**0572**

NANOCOLOR® reagent sets: HC 300 (REF 985 057) and HC extraction from water (REF 918 571) or HC extraction from soil (REF 918 572)

Wavelength: **436** nm

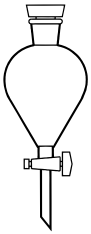
Interferences: A fat content exceeding 1000 mg/l results in high hydrocarbon values. Residual pentane also causes high hydrocarbon results. For this reason the evaporation time for the solvent has to be strictly observed and all glassware has to be free of fat. Hydrocarbons with boiling temperature < 120 °C (e. g. petrol) are not covered by the test.

This method can also be used for analyzing sea water.

Procedure: Requisite accessories: 2 separation funnels 500 ml (REF 916 08), Soxhlet apparatus 30 ml (REF 916 05), extraction thimbles 23 Ø x 100 mm (REF 645 008), 2 columns CHROMABOND® ALOX N (REF 730 250), plastic syringe 50 ml with syringe adaptor (REF 916 09 and 916 03), volumetric flask 25 ml (REF 916 61), volumetric flask 50 ml (REF 916 06), measuring cylinder 50 ml (REF 916 84), piston pipette 1–5 ml with disposable tips and additional stop valve (REF 916 21), heating block *NANOCOLOR*®, reaction tubes (REF 916 80), threaded union (REF 916 04)

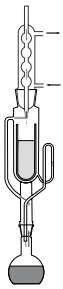
1a. Extraction of water samples

In the separation funnel add 25 g magnesium sulphate to 400 ml water sample *(the pH value of the sample must be between pH 1 and 10)*. Shake for about 1 min, until the magnesium sulphate has dissolved. Add 25 ml n-pentane and shake for about 5 min with frequent careful ventilation. Let phases separate. Discard lower aqueous layer. Apply organic layer to the CHROMABOND® ALOX N column and collect solvent in the volumetric flask 25 ml. Rinse the column with n-pentane, until the volumetric flask is filled slightly below the ring mark and then top up the volumetric flask to the ring mark. Close volumetric flask and mix by shaking slightly.



1b. Extraction of soil samples

Sieve 50 g of the moist soil sample (2 mm mesh size). Grind 15 g of the sieved sample with 15 g sodium sulphate in a mortar and transfer mixture into the extraction thimble. Place the extraction thimble into the Soxhlet extractor and fill the flask with 50 ml n-pentane. Set up apparatus, adjust the temperature of heating unit (hotplate or water bath) to 70 °C and reflux for 1 h. Apply organic extract to the CHROMABOND® ALOX N column and collect solvent in the volumetric flask 50 ml. Rinse the column with n-pentane, until the volumetric flask is filled slightly below the ring mark and then top up the volumetric flask to the ring mark. Close volumetric flask and mix by shaking slightly.



2. Blank value
Apply about 20 ml n-Pentan to the second CHROMABOND® ALOX N column and collect solvent in a beaker.

3. Evaporation of the extraction solvent
2 ml each of the pentane extracts are transferred into an empty reaction tube with the aid of a pipette with stop valve. Place reaction tube in the heating block (programme 70 °C, 30 min) and evaporate the pentane.

4. COD determination of the hydrocarbons
After evaporation of the extraction solvent each reaction tube is tightly joined to a HC 300 test tube - which contains the acid reagent - with the aid of a threaded union. Turn the joined tubes top-down and place them into the heating block (reaction tube below, HC 300 tube on top). Set heating block to 148 °C and 2 h and start. After 2 h remove tubes from the heating block, allow to cool for 15 min. Remove upper tube and carefully add 4.0 ml COD-free water on top of the lower tube *(do not mix)*. Again screw the upper tube onto the reaction tube, and shake carefully ***(Caution: tubes become hot)***. For photometric measurement equilibrate the temperature of the test tubes to 20 °C.

Measurement: Insert test tube.

Source of error	Result [1]	Correction
Evaporation time of the solvent was not observed → residual pentane	+	Observe evaporation time of 30 min
Use of wrong pipette when dosing the extracts → pipette drips a) drop is lost b) drop too much	- +	Use pipettes with direct displacement or use stop valve
Unclean operation, reagent impurities → higher HC content	+	Determine blank value
Losses due to evaporation → concentration of the sample	+	Uninterrupted speedy work, keep vessels closed
Error when volumetric flask is topped up a) above the ring mark b) below the ring mark	- +	Precise work
Dilution error during addition of 4.0 ml COD-free water a) volume too low b) volume too high	+ -	Precise work, exact pipetting
High content of volatile hydrocarbons	-	HC with boiling temperature < 120 °C cannot be determined

[1] Error causes high (+) or low (-) results.