

REF 985057

en

Test 0-57

01.19

NANOCOLOR® HC 300

(Hydrocarbons)

Procedure for soil samples:

Requisite accessories: Soxhlet apparatus 30 mL (REF 91605), extraction thimbles 23 mm dia. x 100 mm (REF 645008), measuring cylinder 50 mL (REF 91684), 2 columns CHROMABOND® ALOX N (REF 730250), plastic syringe 50 mL with syringe adaptor (REF 91609 and 91603), volumetric flask 50 mL (REF 91606), Erlenmeyer flask 100 mL (REF 91638), piston pipette 1–5 mL with disposable tips and additional stop valve (REF 91621), NANOCOLOR® heating block, reaction tubes (REF 91680), threaded union (REF 91604)

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 according to the figure, adjust the temperature of the heating unit (hotplate or water bath) to min. 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.

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* not necessary for the determination of lipophile substances

Common procedure for water and soil extracts:**2. Blank value**

Apply about 20 mL n-pentane to the second CHROMABOND® column and collect solvent in a Erlenmeyer flask.

3. Evaporation of the extraction solvent

2.0 mL each of the pentane extracts and of the blank value 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 (cf. figure 2). 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 room temperature.

Method:

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

	1a. water samples	1b. soil samples
Range:	0.5–5.6 mg/L HC	30–300 mg/kg HC
Sample:	400 mL	15 g
Wavelength (HW = 5–12 nm):	436 nm	
Evaporation:	30 min at 70 °C	
Decomposition:	2 h at 148 °C	

Contents of reagent set HC 300:

20 test tubes HC 300

Contents of reagent set Extraction HC from water (REF 918571):

500 mL n-pentane 500 g magnesium sulfate
200 mL n-pentane 100 mL COD-free water

Contents of reagent set Extraction HC from soil (REF 918572):

2 x 500 mL n-pentane 300 g sodium sulfate
200 mL n-pentane 100 mL COD-free water

Hazard warning:

Test tubes contain sulfuric acid 80–98 % and potassium dichromate 0.0–0.1 %.

H314, H317 Causes severe skin burns and eye damage. May cause an allergic skin reaction.

P260sh, P280sh, P303+361+353, P305+351+338, P310 Do not breathe dust/vapors. Wear protective gloves/eye protection. IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water [or shower]. IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. Immediately call a POISON CENTER/doctor. For further information ask for a safety data sheet. When shaking test tubes use safety bottle (REF 91637).

n-pentane 90–100 %:

H224, H304, H336, H411, EUH066 Extremely flammable liquid and vapor. May be fatal if swallowed and enters airways. May cause drowsiness or dizziness. Toxic to aquatic life with long lasting effects. Repeated exposure may cause skin dryness or cracking.

P210, P233, P260D, P273, P301+310, P331 Keep away from heat/sparks/open flames/hot surfaces. No smoking. Keep container tightly closed. Do not breathe vapors. Avoid release to the environment. IF SWALLOWED: Immediately call a POISON CENTER/ doctor. Do NOT induce vomiting. For further information ask for a safety data sheet.

Interferences:

A fat content > 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 a boiling temperature < 120 °C (e.g. petrol) are not covered by the test.

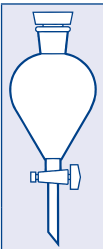
This method can also be used for analyzing sea water.

Procedure for water samples:

Requisite accessories: 2 separation funnels 500 mL (REF 91608), measuring cylinder 50 mL (REF 91684), 2 columns CHROMABOND® ALOX N (REF 730250), plastic syringe 50 mL with syringe adaptor (REF 91609 and 91603), volumetric flask 25 mL (REF 91661), Erlenmeyer flask 100 mL (REF 91638), piston pipette 1–5 mL with disposable tips and additional stop valve (REF 91621), NANOCOLOR® heating block, reaction tubes (REF 91680), threaded union (REF 91604)

1a. Extraction of water samples

In the separation funnel add **25 g** magnesium sulfate 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 sulfate 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.



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Measurement:

For MACHEREY-NAGEL photometers see manual, test 0-57.

Photometers of other manufacturers:

For other photometers check whether measurement of round glass tubes is possible. Verify factor for each type of instrument by measuring standard solutions.

Possible errors:

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	-	Use pipettes with direct displacement or use stop valve
b) too many drops	+	
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.