



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

2.0 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.

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 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.