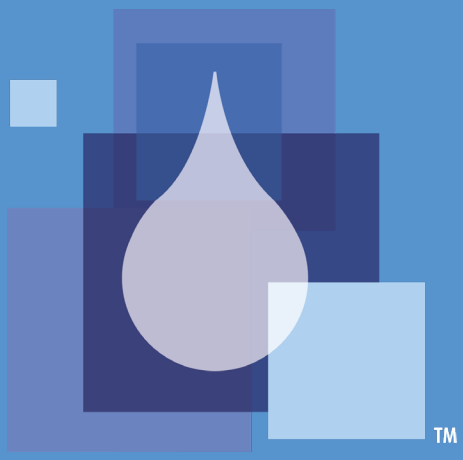
Sample Preparation for MEA-Triazine and MEA Analysis in Crude Oil



OndaVia

Disclaimer

The OndaVia Raman Spectrometer with OndaVia Raman Controller (ORC) Software is intended to provide an initial determination and to be used as an information resource or tool, and not as an absolute or conclusive identification of unknown substances. Results provided by the OndaVia Raman Spectrometer and associated Software should be verified using other appropriate laboratory techniques. OndaVia makes no recommendations nor does it assume any liability for how the information is used.

The Raman Spectrometer has a high energy laser that may burn some samples and targets. It is the user's responsibility to verify that the Spectrometer will not damage the sample or target. OndaVia makes no recommendations nor does it assume any liability for how the information is used or any damage it may cause.

The Raman Spectrometer includes the Advanced ORC Software, which allows the user to adjust the laser power settings. These settings are approximate, and may change over time and with repeated use. OndaVia makes no warranties or representations as to the accuracy or stability of the laser settings. For precise determinations, a laser power meter should be used. With our spectrometry technology, no reference beam is used and the Spectrometer is not intended for absolute or relative quantification of substances.

Use of the Spectrometer is at the user's own risk.

Chemical Safety Statement: The chemicals used in this measurement process can be dangerous to the user and surroundings if inappropriately used. Exercise caution and use appropriate personal protective equipment to protect yourself and your environment.

Disposal Statement: Dispose of chemicals in accordance with the guidelines of your local, state, or federal authority

Purpose

The extraction and analysis procedures outlined herein will create an aqueous sample of amines present in a crude oil sample through acid extraction. Instructions for optional sample cleanup are presented and may be necessary for some oil varieties. By using the included reagents and OndaVia Analysis System any technician can produce accurate & reliable results for MEA or MEA-triazine content in crude oil matrices in the 1-100 ppm range.

Materials & Supplies

- 0.1– 1 mL pipettor & tips
- 0.1 – 10 µL pipettor & tips
- 10 – 100 µL pipettor & tips
- 1 M NaOH (aq.) (1 mL)
- 10 mM HCl (aq.)
- pH paper or meter for ranges 1-4 and 11-14
- OndaVia Software with the appropriate calibration curve
- 50 mL centrifuge tube or 2 mL microcentrifuge tube
- Centrifuge
- *Optional*: Aqueous extract pretreatment kit, xylene

Extraction

Prepare at least 10 mL of 10 mM HCl in water.

Large volume method – for bigger centrifuges

- Weigh 2 to 10 g of crude oil into a 50 mL centrifuge tube. Note the mass of oil. If oil is viscous, add 2- to 10-ml xylene or toluene to aid mixing.
- Add 10 mM HCl equal to the mass of oil added in previous step. Do not add extra acid solution to account for xylene or toluene (if used).
- Vortex, sonicate, or aggressively mix the oil-acidified water mixture for at least two (2) minutes.
- If possible, centrifuge the entire 50 mL tube at a minimum 1000 g for 5 minutes. Some mixtures will emulsify more or less, and the centrifugation time may need to be adjusted. Speeds up to 2000 g will help to accelerate the separation. The goal is a separation of oil and water, allowing the user to extract a clean water aliquot from the aqueous (lower) phase. Transfer the aqueous phase to a separate vial. This vial now contains the 'sample'.

Small volume method – for microcentrifuges

- Weigh 0.6 to 0.8 g of crude oil into a 2 mL microcentrifuge tube. If oil is viscous, add 10-30 µL xylene to aid mixing.
- To the centrifuge tube, add 10 mM HCl equal to the weight of oil added in previous step. Do not add extra acid solution to account for xylene (if used).
- Vortex, sonicate, or aggressively mix the oil-acidified water mixture for at least 2.5 minutes.
- Centrifuge at minimum 1000 g for 2.5 minutes. The goal is a separation of oil and water, allowing the user to extract a clean water aliquot from the aqueous (lower) phase. Transfer the aqueous phase to a separate vial. This vial now contains the 'sample'.

This method may be adapted to other centrifuges and vials as required.

Sample Pretreatment

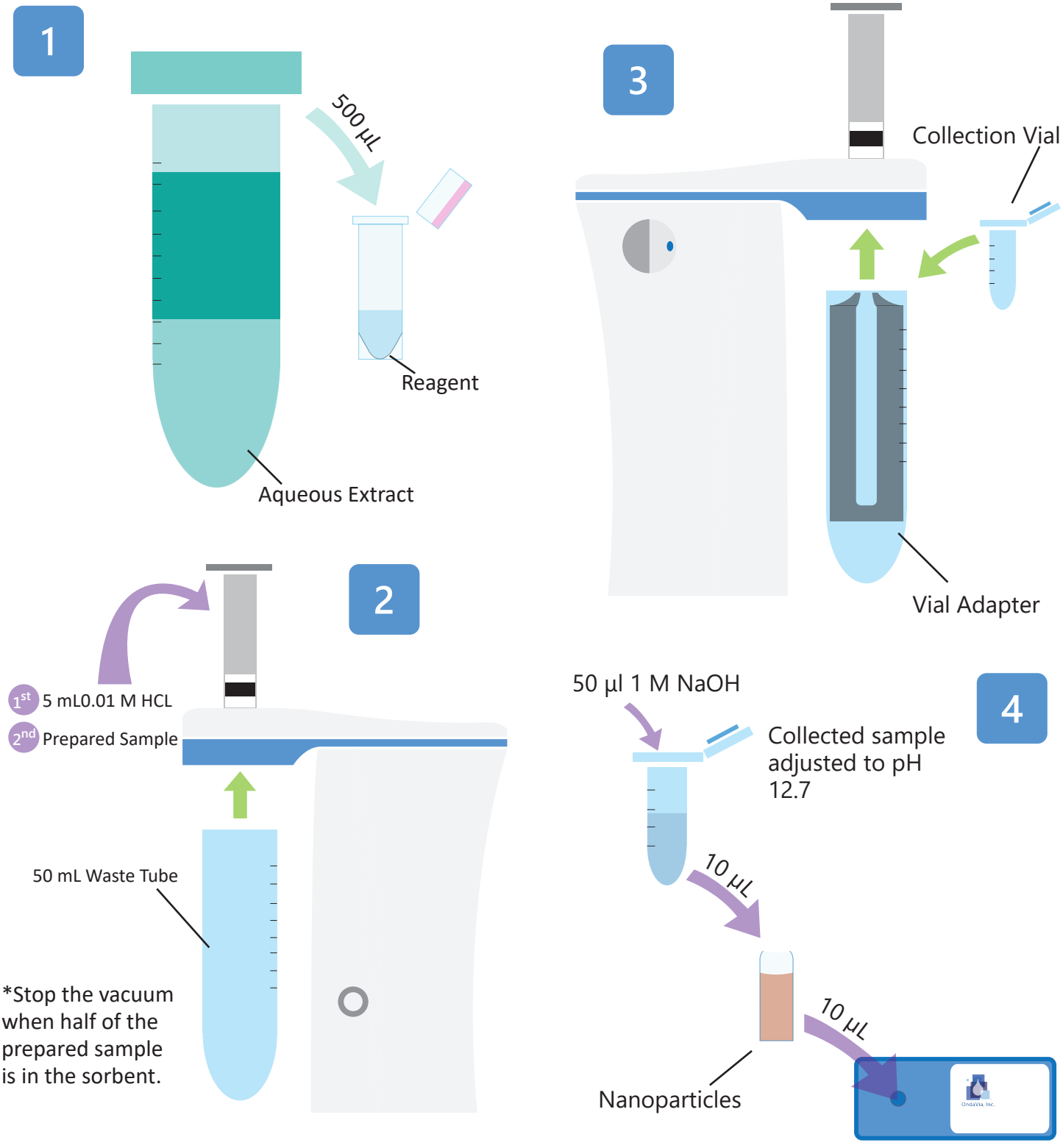
Obtain the appropriate analyte cartridge and reagent packets

- Using a pipette, dispense 500 µL of the aqueous extract into the **reagent** vial.

To obtain accurate results and achieve maximum performance from the column vacuum only use supplied columns

- Remove a column from the clear bag and insert it tip-first through the top opening on the vacuum apparatus.
- Screw an empty 50 mL centrifuge tube into the column vacuum (*this tube will be used to capture the liquid from the conditioning cycles*). **Refer to Step 2 in the graphic guide**
- Pass 10 mL of 10 mM HCl (aq.) solution through the column, do not allow the sorbent bed to completely dry. Allow the fluid level to drop so that it is just above the sorbent bed, and then switch off the vacuum.
- Add 1 mL of the prepared sample into the column. When half of the sample solution has passed into the sorbent bed, switch off the vacuum.
- Exchange the 50 mL centrifuge tube used for the column conditioning with the centrifuge tube adapted to fit a 1.5 mL vial. Power on the vacuum to collect the prepared sample in the 1.5 mL vial. **Refer to Step 3 in the graphic guide**
- Adjust the collected sample to pH 12.7 with NaOH. Typically, 50 µL 1 M NaOH can be used. Scale volume of NaOH to the volume of collected sample.
- Pipette 10 µL of the solution from the capture vial into the vial containing the nanoparticle solution and mix thoroughly. **Refer to Step 4 in the graphic guide**
- Pipette 10 µL of the sample-reagent-nanoparticle solution onto the cartridge as shown on the right. Insert the cartridge into the holder for the Raman spectrometer and begin analysis.

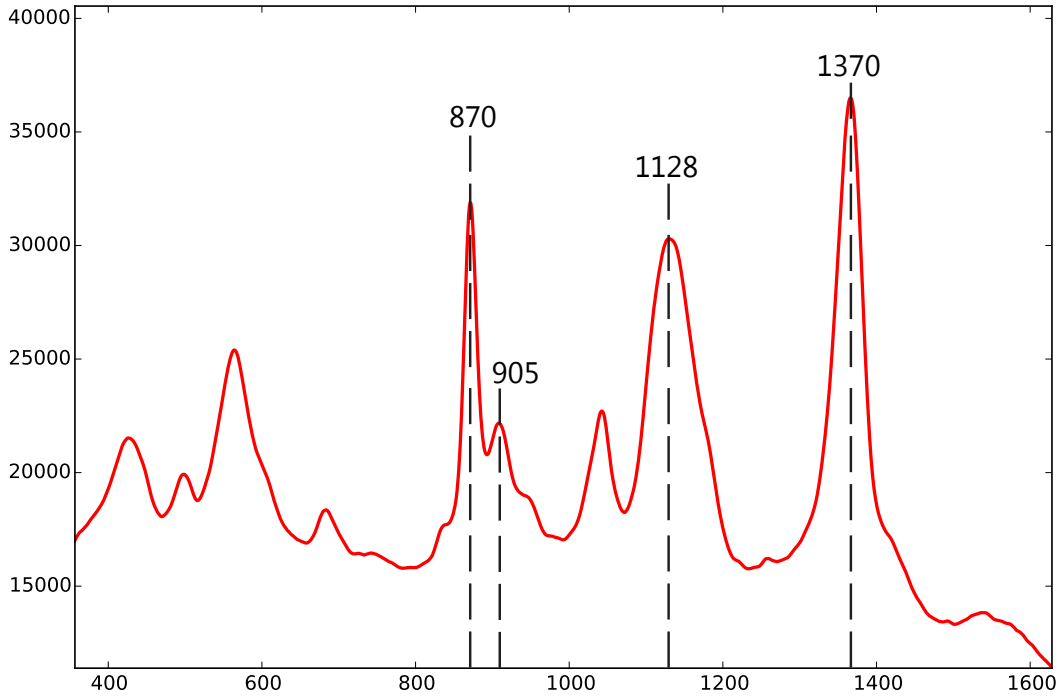
Sample Pretreatment



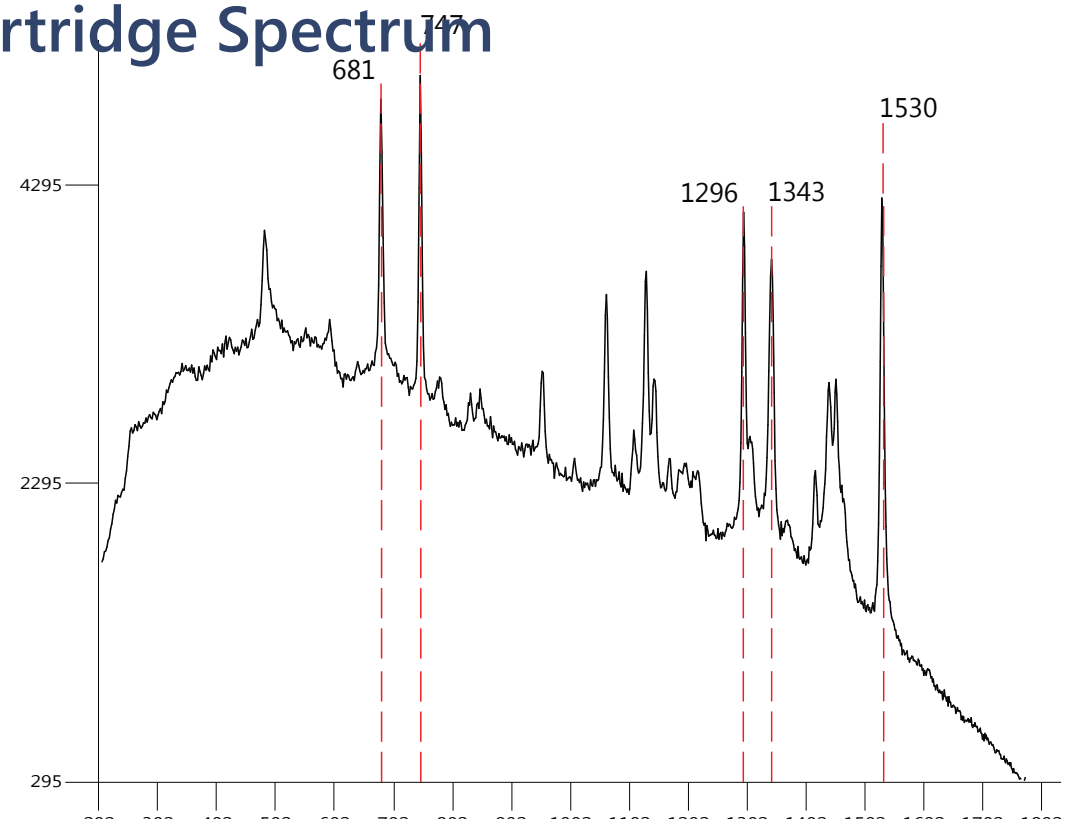
Analysis

- Insert cartridge into the cartridge holder on the OndaVia Analysis System. It will “click” into place when it is securely in position.
- In **Settings**>**Contaminant** choose the either **MEA** or **MEA-triazine-ppm**, then click **Focus**. After a few seconds, a spectrum should become visible in the lower pane of the OndaVia Raman Controller (ORC). Use the zoom housing to focus on the sample and maximize the signal (peaks) while minimizing the noise (jaggedness of background). Click **Abort** when finished.
- Click the **Start Analysis** button. A serial number dialog box will appear where you can enter a name or code for this sample run.
- Click the **OK** button to begin collecting data. During data collection a progress bar runs in the lower left of the ORC window.
- You may click the **Abort** button at any time during data collection to stop the process. No measurement data will be processed.
- When data collection is complete, an updated measurement will appear in the notes box and a dialog screen with the heading **Additional Notes** also appears which allows the operator to enter any other relevant information.

MEA-Triazine Spectrum



Cartridge Spectrum



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