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Cleaning Karl Fischer Electrodes

General

This document describes how to clean all Karl Fischer electrodes. This cleaning may be necessary due to sample contamination, consistently high results, or overall sluggishness in response. It is practical to use the KF vessel to hold the cleaning solution, as it has the added benefit of cleaning the glass and electrodes simultaneously.

Procedure

The following procedure applies to the volumetric KF electrode (6.0338.100) as well as the coulometric indicator (6.0341.100) and diaphragmless generator (6.0345.100) electrodes.

Take extreme care to not damage the platinum surfaces of the electrodes. This includes bending the two platinum wires from the indicator electrode, as well as the platinum mesh of the generator electrode.

Do **NOT** use acetone or other ketone or aldehyde containing solvents as these may cause undesirable side reactions with the KF reaction. Do **NOT** use ultrasonic baths (sonicators).

1. Remove the solvent as well as all other parts (aspiration tips, dispensing tips, etc.) from the KF vessel.
2. Prepare a solution of 50:50 Concentrated Nitric Acid and Water. Keep in mind the best practice is to add acid to water. If Nitric Acid is not available, other mineral acids such as Hydrochloric, Sulfuric, or Chromic may suffice.
3. Pour 30-40 mL of the acid solution from #2 to the KF vessel. This should be enough to cover the platinum surfaces of the electrodes.
4. Allow the electrodes to soak for 15-20 minutes, longer if there are heavy deposits on the electrode.
5. Remove the electrodes from the vessel and discard the acid solution.
6. Rinse the glass vessel and electrodes excessively first with DI water, followed by Methanol.
7. Allow the electrodes and vessel to air dry, or place in a drying oven at 50° C for 1 hour. You **CAN** simply put the electrodes back in place after cleaning; however, conditioning times may be extended significantly.

For the diaphragm generator electrode (6.0344.100) please consider the following:

Contamination	Cleaning procedure
Sticky residues on the generator	Place the generator electrode in an upright position, fill with concentrated Nitric acid and leave overnight. First rinse with water twice, then with methanol. If the contamination is particularly persistent, you may use chromic acid for cleaning. Afterwards, rinse first several times with water and then with Methanol.
Salt-like residue	First clean with water and then rinse with methanol. To rinse the diaphragm, fill the cathode chamber of the generator electrode with methanol and let the content flow out. Repeat two or three times. Carry out this procedure after each cleaning. After the last rinsing cycle, dry the cell in the drying oven at 50 °C or with a hair dryer.
Oily Contamination	Clean with solvent (e.g. hexane) and then rinse with methanol

Once the electrodes have been sufficiently cleaned, refill the vessel with the appropriate solvent (i.e. Methanol Dry, Coulomat AG, etc.) and resume testing.

Troubleshooting

If Nitric Acid is unable to clean the electrodes, using Chromic acid is suggested.

Please contact Metrohm technical support with any questions.