

General

This document details KF volumetric troubleshooting tips and routine maintenance.

Procedure**A. KF cell**

1. Inspect the Indicator [double platinum wire, 6.0338.100] electrode
 - a. The pins should be 3 mm apart. Adjust with care since the pins are fragile
 - b. The pins should be straight. Adjust with care since the pins are fragile.
 - c. Routine cleaning is recommended at least every month or depending on the number and type of analyzed samples, see Appendix I.
2. Molecular sieve replacement
 - a. Every 4 – 8 weeks for controlled humidity room.
 - b. Every 1 – 2 weeks if humidity is greater than 75 %.
 - c. The sieve can be regenerate in a 200 C oven for 48 hrs or 300 C oven for 24 hrs.
3. Cleaning and Inspecting the vessel and titration lid
 - a. Ensure that all o-rings are intact and not corroded.
 - b. Ensure that the platinum electrode is fitted tight in the titration head.
 - c. Wipe clean the bottom of the titration head.
 - d. Inspect the anti-diffusion tip. This should be replaced annually or as needed if leaking or blocked.
 - e. Completely empty the vessel and clean the vessel.
 - i. Soap and water.
 - ii. Flush with water 3 – 5 times to remove soap residue.
 - iii. Rinse with about 20 mL methanol or acetone for ketone system.
 - iv. Vessel and sensor can be dried with hair dryer or placed in 60C oven for 1 hr.
 - v. Fill with appropriate solvent to ensure coverage of platinum electrode or solvability of samples. Please note that the solvent level shouldn't be greater than 80-90% of vessel volume.
 - vi. Tighten all connections.
 - vii. Replace septum regularly

B. Good KF practices

1. Prepare the Dosing unit/buret or Exchange unit/cylinder to ensure fresh titrant and no air bubbles.
2. Determine the KF reagent, ex. Comp 5, Titer daily or as directed by QA or SOP. Metrohm recommends using Hydranal Water Standard 10.0.
3. The initial stir rate should ensure complete dissolution of titrant. Meaning, there should be a titrant trail in the vessel.
4. The stir rate must be increased as volume increases in vessel.
5. The maximum volume in the vessel during a titration should not exceed 80-90% of the vessel volume.
6. Run a standard to check the functionality of the KF system.
 - a. When preparing standard or samples always wear clean gloves.
 - b. Metrohm recommends Hydranal Water standards see Appendix II
 - c. The KF system isn't calibrated, instead the functionality is confirmed.

7. For direct injection KF system, the stir rate should create a slight vortex but not expose the pins of the platinum electrode.
8. If the drift is 0 or the color is brown or reddish, then don't start a sample.
 - a. Check for a leaking antidiffusion tip
 - b. Clean and inspect electrode
 - c. Replace cable
 - d. Replace electrode
 - e. Review Part A for root source of error.

Troubleshooting

1. **If overtitration error is generated or vessel solvent is dark brownish or reddish, then this indicates that too much Iodine or the sample is producing Iodine.**
 - a. Replace solvent
 - b. Clean/inspect electrode
 - c. Clean vessel.
 - d. Replace electrode cable.
2. **If sample unfit error is generated, then this indicates that the maximum polarity has exceed. Try the following:**
 - a. Clean electrode
 - b. Replace solvent
 - c. Check stirring
3. **If the stop volume is reached during Conditioning mode, then the electrode and/or cable are damaged.**
 - a. Clean and inspect electrode
 - b. Clean vessel and replace solvent
 - c. Replace electrode and/or cable.
4. **If an oven is connected, then the Heatable transfer tube.**
 - a. The tube should be warm to touch.
 - b. The connection of the buret tip to the transfer tube should be inspected for loss of seal and moisture.
 - c. The heatable transfer tube should be positioned in the vessel such that it isn't bubbling into the generator. It should be positioned over the stir bar for best delivery.
 - d. The heatable transfer tube is a consumable part which should be replaced based on sample through put and loss of seal, typically, 12-18 months
5. **If the drift is excessively greater than titration rate, then the cable is damaged. Replacement is required.**

Appendix I Cleaning Karl Fischer Electrodes – excerpt from Metrohm KF leaflet**2.2 Cleaning the indicator electrode****CAUTION**

Use caution when cleaning the indicator electrode in order not to bend the Pt pins.

Contaminated indicator electrodes can be cleaned with an abrasive agent such as aluminum oxide powder (6.2802.000 polishing set) or toothpaste. Afterwards, rinse first with water and then with methanol.