Volumetric Karl Fischer Troubleshooting & Routine Maintenance

This document details Karl Fischer troubleshooting tips, good practices and routine maintenance.

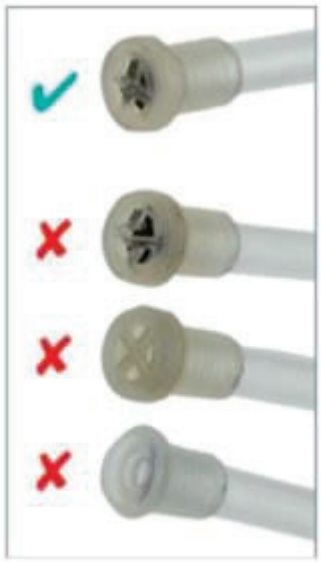
Technology: Titration

Inspection and Maintenance

Volumetric Vessel

1. Inspect the indicator electrode (6.0338.100).
    a. The pins should be 3mm apart.
    b. The pins should be parallel.
    c. Routine cleaning is recommended, see 'Advanced Cleaning' section below.
2. Molecular sieve replacement.
    a. It is difficult to make a general statement concerning how often the molecular sieve should be replaced, because this is dependent on the humidity in the laboratory. Our experience has shown us that replacement every six weeks is appropriate in the presence of medium humidity. An increase in drift indicates that the leak-tightness of the cell should be inspected and that the molecular sieve should possibly be replaced.
    b. The molecular sieve can be regenerated at 300 °C in the drying oven. The regeneration time is at least 24 hours. The molecular sieve is then placed in a desiccator for cooling down and afterwards stored in a glass bottle with an airtight seal.
3. Cleaning and inspecting the vessel.
    a. Ensure that o-ring is intact and not corroding.
    b. Verify that the electrode is fitted tight in the titration head.
    c. Wipe clean the bottom of the titration head.

d. Inspect the anti-diffusion buret tip. This should be replaced at least annually or as needed if leaking or blocked.



Antidiffusion Tips

e. Properly empty the vessel and clean:
    i. Clean with soap and water.
    ii. Flush with water 3 – 5 times to remove soap residue.
    iii. Rinse with methanol (or acetone for ketone system).
    iv. Allow to dry.
    v. Fill with appropriate solvent to ensure coverage of platinum electrode or solvability of samples. Please note that the solvent level should not be greater than 80-90% of vessel volume.
f. Tighten all connections.
g. Inspect septum. This should be replaced regularly.
h. Don't forget the stir bar.
    i. Stirring should create dimple on surface, not a vortex exposing pins of indicator electrode.

Good Volumetric Practices

1. Run a Manual Prepare of the dosing or exchange unit to ensure fresh titrant and no bubbles in cylinder or tubing.

2. If starting drift is 0 or if solution is brown/red, do not start sample analysis – refer to 'Inspection and Maintenance' section above to remedy.
    a. It may be necessary to replace electrode and/or cable.
3. Determine the KF reagent (i.e. Composite 5) titer daily or as directed by QA or SOP. Metrohm recommends use of Hydranal - Water Standard 10 for titer determination.
4. Run a certified reference standard to check system suitability.
    a. KF system is not calibration during this check, instead proper functionality is confirmed.
5. Ensure proper sample size is used: The sample size depends on the water content of the sample. In principle, the sample size should be selected in such a way that the titrant consumption lies between 10% and 90% of the buret volume. For a 10 mL buret, the consumption of titrant should therefore be between 1 and 9 mL.

		Titer 5 mg/mL			Titer 2 mg/mL			Titer 1 mg/mL		
		Burette volume			Burette volume			Burette volume		
		5 mL	10 mL	20 mL	5 mL	10 mL	20 mL	5 mL	10 mL	20 mL
		Recommended sample size (g)			Recommended sample size (g)			Recommended sample size (g)		
Expected water content	90%	X	0.04	0.08	0.007	0.015	X	X	X	X
	75%	X	0.05	0.1	0.01	0.02	X	X	X	0.02
	50%	X	0.08	0.16	0.015	0.03	0.05	X	0.015	0.025
	20%	0.08	0.125	0.25	0.025	0.05	0.1	X	0.025	0.05
	10%	0.125	0.25	0.5	0.05	0.1	0.2	0.025	0.05	0.1
	5%	0.25	0.5	1	0.1	0.2	0.4	0.05	0.1	0.2
	2.5%	0.5	1	2	0.2	0.4	0.8	0.1	0.2	0.4
	0.25%	5	10	20	2	4	8	1	2	4
	0.1% (1000 ppm)	12.5	25	25	5	10	20	3	6	12
	0.01% (100 ppm)	25	25	X	25	25	X	25	25	X
	0.001% (10 ppm)	X	X	X	25	X	X	25	X	X

Consumption >1/2 burette volume
Consumption 1/2 burette volume
Consumption <1/2 burette volume
X = not recommended

Troubleshooting

1. 'Overtitration' error generated or vessel solvent is brown/red – this indicates that too much iodine is being added or the sample is producing iodine.
    a. Replace solvent.
    b. Clean/inspect vessel/electrode per 'Inspection and Maintenance' section above.
    c. Replace electrode and/or electrode cable.

2. ‘Sample unfit’ error is generated – this indicates that the maximum polarity has been exceeded.
    a. Replace solvent.
    b. Check stir rate.
    c. Clean/inspect vessel/electrode per ‘Inspection and Maintenance’ section above.
    d. Replace electrode and/or electrode cable.
3. Stop volume is reached during Conditioning.
    a. Replace solvent.
    b. Clean/inspect vessel/electrode per ‘Inspection and Maintenance’ section above.
    c. Replace electrode and/or electrode cable.

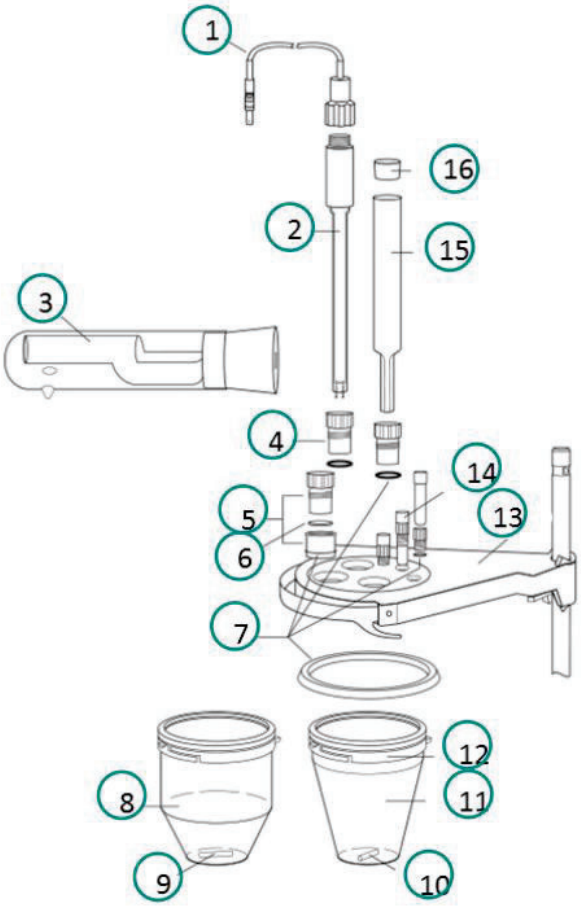
Advanced Cleaning – Excerpt from Metrohm KF Leaflet

Metrohm does not recommend the use of any solvents containing ketone or aldehyde for cleaning Karl Fischer accessories.

- Cleaning the indicator electrode:
    a. 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.

Metrohm USA Inc.

Consumable Parts



1. 6.2104.020
2. 6.0338.100
3. 6.2412.000
4. 6.2730.010
5. 6.2730.020
6. 6.1448.010
7. 6.1244.040
8. 6.1415.250
9. 6.1903.030
10. 6.1903.020
11. 6.1415.220
12. 6.2036.000
13. 6.1414.030
14. 6.2730.030
15. 6.1403.040
16. 4.4202.440

Other ideas

Review: FAQs – Karl Fischer Titration | Metrohm

'Submit a request' for further assistance from Metrohm Technical Support at support.metrohmusa.com.