Cleaning the Karl Fischer Reaction Cell

General Cleaning

Various cleaning procedures are possible, with a general method consisting of washing the cell with a detergent solution, rinsing with de-ionized water then rinsing with methanol or acetone. Sometimes, the frit must be cleaned more vigorously by drawing reagents through the frit by applying a vacuum to the inner chamber of the cell.

Cleaning the frit

1. Block the hole in the upper glass portion of the Generator Assembly, where the platinum wire goes from the inside to outside of the Generator Assembly. A recommended method is to wrap a piece of Parafilm around the generator assembly covering the hole, other adhesive tapes may also work.
2. Have a vacuum source available. One method is to use a vacuum aspirator.
3. Attach the aspirator to a faucet. Connect the Generator Assembly to the aspirator.
4. Place the Generator Assembly into a beaker of reagent containing a solvent that the samples that were analyzed will dissolve in. WARNING! Use caution and protection when using any reagents.

Type of Samples	Reagents for Cleaning
Hydrocarbons	Chloroform
Oils	Chloroform
Amines	Methanol
Ketones	Acetone

5. Draw the solvent up through the frit. WARNING! Verify that the reagents that are being used for cleaning do not cause a chemical reaction with any sample material remaining in the frit!
6. Repeat until the frit appears to be cleaned:
   - Discoloration in the frit may be removed.
   - Solvent drawn through appears "cleaner".
   - An oil layer is no longer being removed.
   - Any precipitate visible is removed.
   - Solvent is drawn through the frit easier.
7. More than one reagent may be drawn through the frit for cleaning purposes.
8. The last solvent to be drawn through the generator assembly should be either methanol or acetone.
9. Final cell drying should be done in an oven at 50 - 80° C for 1- 2 hours. WARNING! Let the cell cool before filling it with fresh Karl Fischer reagent, which is mostly methanol and is flammable!

After placing fresh coulometric Karl Fischer reagents in the cell and Generator Assembly, place the cell on the titrator and begin the automatic cell and reagent drying process. It may take the newly cleaned frit some time for reagent to diffuse into the frit and provide full titration current.

If the above procedure does not clean or unclog the glass frit, it may be necessary to use strong cleaning agents. For severely clogged frits, a chromic acid or concentrated nitric acid cleaning process is recommended.

WARNING! These are strong acids and must be used with appropriate care and protection!

Technical information provided by Denver Instrument Co.