

Karl Fisher Titration and the 20 Most Critical Questions

1. "Our Karl Fisher says 'OVER TITRATION' and the reagent is turning really dark. Why?"

"Over Titration" simply means that the reagent called "anolyte" has released more than enough iodine to consume the amount of moisture that exists inside the vessel. Since there is too much iodine we need to try to equalize the situation by introducing more moisture. More importantly we should also investigate why this happened. Was the stirrer turned off? Is the Detector electrode not working?

2. "Why won't the instrument go into "Ready mode"?"

The instrument is having trouble reducing the drift (background moisture) to a low and steady state. Is the generator electrode producing a current to cause the reagent to produce iodine to neutralize the moisture?

3. "Results seem "all over the place", what should I do?"

Repeatability is important. Is there anything that the operator is doing that might be causing this variability? Try running three tests, increase the sample size, and see if the results become more repeatable.

4. "We use a solids evaporator and we are getting ZERO moisture results. Why?"

Is the evaporator introducing ANYTHING into the vessel? Sometimes a visual inspection can determine this by examining the bubbling action inside the vessel. If you don't see some bubbles appearing at the opening of the bubbler tube inside the vessel you know there is something wrong.

5. "What kind of reagent should I use?"

There are a variety of reagents that can be used. Talk with us and we can help you determine the best choice of reagent for your application.

6. "What is a dual vs. a single reagent setup?"

Dual-reagent means that two reagents are used where one reagent (referred to as the anolyte) is used to fill the vessel and the second reagent (referred to as the catholyte) is used to fill the generator electrode. Single-reagent simply means that only one reagent (anolyte) is used. The generator electrode is manufactured as either single or dual. If the generator electrode has a "frit" or "wafer" positioned at the bottom of the generator electrode to form a bottom, then, it is designed to accommodate a second reagent and is known as a dual-reagent generator electrode.

Generator electrodes that do not have a “frit” or “wafer” at the bottom of the electrode are known as single-reagent generator electrodes.

7. "Is an inner-burette the same thing as a Generator Electrode?"

Yes. Some people refer to a “generator electrode” as an “inner-burette”.

8. "What is a detector electrode?"

A detector electrode is the second electrode that sits down inside the vessel. The detector electrode is about the diameter of a pencil and has two platinum prongs extending outward from the end. The detector electrode has only one function and that is to “detect” an electrical current inside the vessel.

9. "Should I use Karl Fisher Grease?"

Some vessels are designed differently and have ground glass fittings instead of screw on caps. If your working with a vessel with ground glass fittings then you will require the use of karl fisher grease to seal the fittings.

10. "How many tests can I run and when should I change out my reagents?"

Different reagents can have a different useful life. Depending on the type of reagents being used, frequency of testing and sample size, the amount of tests that can be performed over a specified period of time can vary. Check with us and we can review this with you.

11. "How can I make sure my instrument is giving me good results?"

Check yourself periodically with water standards. Water standards can help you verify that the instrument is performing properly.

12. "What are water standards? How do I use them?"

Depending on your type of karl fisher setup there are several types of water standards that can be used. Generally the operator will use the water standards as a replacement for a real sample and run a test as usual. The water standard has a known amount of water content and therefore allows the operator to compare results with expected amounts.

13. "How and where can I get my instrument serviced and calibrated?"

Scientificgear can perform both service and calibration service. Give us a call for help at 800-998-6429 or email: service@scientificgear.com.

14. "What kind of syringe and needle do I need?"

There are a variety of syringes and needles that can be used. We often recommend using a disposable syringe (4mL) and a disposable needle (at least 4" long) for testing.

15. "Should I use an analytical balance or use Specific-gravity?"

Depending on the parameters your using to evaluate moisture both can be used. However, the Analytical Balance will provide the best results as they eliminate subjectivity. Using the specific gravity method requires the operator to visually determine the volume of sample being introduced.

16. "How dry does my glassware have to be before I run a test?"

Well, we say very dry. Some operators try to clean and use their glassware in only a few minutes - just prior to running tests. Giving the glassware time to properly dry is always recommended.

17. "Parts Per Million (PPM) vs. Percent (%)"

Depending on the type of evaluation being performed both PPM or % may be appropriate. Usually historical data requirements dictate how the data is reported. However, we like to use the PPM approach as often as possible as it provides a more specific "whole number" to use as a comparison. Sometimes communicating a % reading is less precise for comparison purposes when the results need to be as accurate as possible.

18. "Can I use a Karl Fisher Titrator to measure moisture in Solids?"

Yes! Karl Fisher Titrators work well with evaporator ovens. Evaporator ovens allow for the solid sample to be heated to the point where only the moisture from the sample is introduced into the vessel.

19. "What's the difference between Volumetric and Coulometric Karl Fisher?"

Volumetric Karl Fisher is better for measuring larger amounts of moisture (~3%+ where also the sample size required is larger). Coulometric Karl Fisher Titrators are better for measuring lower moisture levels (around 1% and under).

20. "How much sample should I use?"

Depending on the type of Karl Fisher Titrator (Volumetric vs. Coulometric) the sample size can be determined that best utilizes the instrument's moisture testing capabilities. Scientificgear can assist with determining sample sizes that fit the testing conditions and type of titration application.