Standard Operating Procedure

Volumetric Karl Fischer titration: KFV

Initial Parameters:

Titrant: Titrant 2: Hydranal Composite 2 (150mL minimum)

Dry Methanol: Hydranal Methanol or equivalent (350mL minimum)

Waste Container: empty

Reaction Cell: 50-75mL of dry methanol in the KFV vessel

Balance: Analytical balance with readability of 0.0001g

Method: KFV_V* -no oven

Cell type: Without diaphragm

Solubility: Samples and titer MUST be soluble in methanol

I. Scope

The Karl Fischer (KF) Titrator is exclusively used for the quantification of water. In a volumetric KF titration, the iodine solution, also known as the "titer", will react with a sample that contains water in a methanolic solution until all the water is consumed. Before proceeding with a sample measurement, ensure the **Titer concentration has been determined (SOP-KFV003) and updated into the Tiamo software.** The known titer concentration is then used to stoichiometrically determine the amount of water in the sample by calculating the volume of titer required for the reaction.

II. KF Instrument Setup

a. All glassware must be dry and free of water and contamination.
b. Place the titrant, methanol, and waste container into the appropriate holder position[1].
c. Startup Tiamo software, wait till all modules are discovered.
d. Select the Manual icon. Under the Dosing Device select the Prepare tab.
    i. Start (this will prime and purge the titrant in the Dosino).
    ii. Close the Manual
e. Select Workplace icon. Load the method and sample details (i.e., Hydranol 10)
    i. Fill the reaction cell with approximately 50-75ml of dry methanol ensuring the electrode is submerged in the liquid.
f. In the Run window select Start

i. Live Window will illustrate Conditioning of reaction vessel
    1. Wait till Conditioning reads 'Ok' at < 20uL/min
    2. Ensure the overall volume of the vessel never exceeds 125mL

III. Sampling Handling and Analysis Procedure:

a. Liquid sample
    i. Determine the amount of sample required using Table 1.
    ii. Aspirate approximately 1 mL of the sample into the syringe.
    iii. Take the tip of the needle out of the liquid and pull back the plunger to the maximal volume. Sway the syringe to rinse it with sample. Then eject the sample into the waste.
    iv. Aspirate further content of the sample into the needle (in case air is aspirated, eject the air out of the syringe).
    v. Remove excess liquid from the outside of the needle with a paper tissue.
    vi. Place the needle on a balance and tare the balance.
    vii. Select Start from the Workplace window to start the determination.
        1. 20 seconds counter will commence.
    viii. Inject a suitable amount of sample (not the whole content!) through the septum into the titration vessel. Please take care that the standard is injected into the reagent and not at the electrode or the wall of the titration vessel. This leads to unreproducible results.
    ix. After injecting the sample, place the syringe again on the balance.
    x. Enter the injected sample weight in the software.
        1. There is no concern if you enter the wrong value. This value can be reprocessed in the Database.
    xi. Sample water content value will be displayed in the report.
        1. Either in ppm or %.
    xii. Repeat step vi to xi for a minimum of three times.
        1. The average for the sample should have an RSD +/- 2.5%.
        2. Repeat till acceptable RSD has been achieved.
b. KFV Reaction Vessel Concerns
    i. If the colour of the reaction vessel solution becomes too dark
        1. Replace the entire solution.
        2. Wait till Conditioning reads 'Ok' at < 20uL/min
    ii. If the electrode reads < 0.2uL/min.
        1. Replace the entire reaction vessel solution.
        2. Wait till Conditioning reads 'Ok' at < 20uL/min

IV. Cleanup:

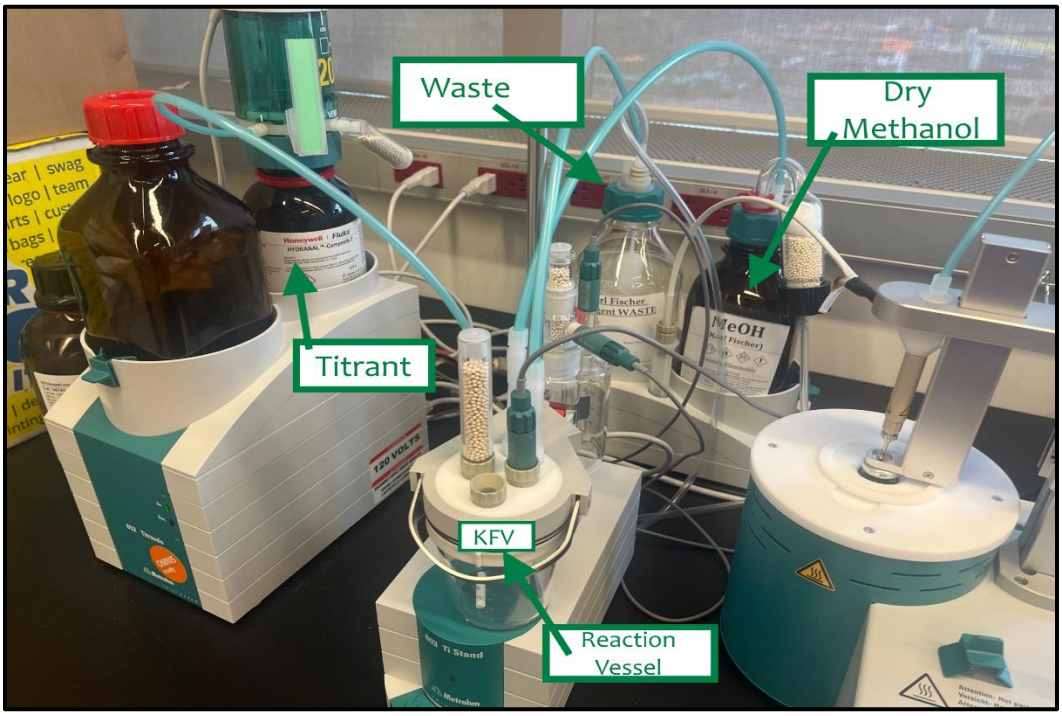
a. Titer Cleaning
    i. Remove the titer solution and replace it with methanol for cleaning.
    ii. Select the Manual icon. Under the Dosing Device select the Empty tab.
        1. Start (this will prime and purge the titrant out of the Dosino).
        2. Under the Dosing Device select the Prepare tab.
        3. Start.
        4. Repeat step (ii) until the Dosing is clean.
            a. Leave it empty.
b. Vessel Cleaning
    i. Remove the vessel and rinse with methanol for cleaning.
c. Place all waste into the appropriately labeled TRACES waste container.

V. Tips and Suggestions

a. Use freshly prepared or properly stored samples for KF determination.
b. Carry out the water content determination of the sample at the same temperature as the titer determination.
    i. Maximum and Minimum temperature fluctuations: +/- 0.5 ^{o}C.
    ii. Temperature increase of 1 °C results in a titer decrease of approximately 0.1%.
c. Always aspirate the whole content of a liquid sample into the syringe. If this is not possible, place the remaining sample into the waste. If possible, store the extra sample content in a crimped septa sealed vial.
d. All determination of the sample size are done by weighing. It is essential that a balance suitable for small weights is used. Otherwise, the balance error can have a significant influence on the result. It is recommended to use an analytical balance with readability & reproducibility of 0.0001g. We also recommend that the balance have a stability period of 3-5 seconds.
e. The pressure and temperature can be monitored by using the barometer in TRACES to correct the sample size depending on the temperature.

1. Appendix

i. KFV Instrument Setup



ii. Table 1: Sample Size

Water Content	Sample Size
≤40%	50-100mg*
10%	50-100mg
1%	100-750mg
0.1%	200-1000mg

*The TRACES Manager will provide full details during hands-on training.