Standard Operating Procedure

Titer determination in volumetric Karl Fischer titration

Initial Parameters:

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

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

Water Standard: Hydranal 10

Waste Container: empty

Balance: Analytical balance with readability of 0.0001g

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

Method: KFV-TiterConc_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 any water molecules that are released from a sample dissolved in methanol until all of the water is consumed. Titer concentration must be determined before water determination of a sample by volumetric Karl Fischer titration can be conducted. The titer is calibrated against a known (accurate) amount of water in a standard. The titer is volumetrically titrated to determine the amount of the KF reagent that is required to react with the known water sample to completion. The titer standardization is required to be determined against a known water standard to assess the amount of water accurately in an unknown sample.

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 the 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 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 until Conditioning reads 'Ok' at < 20uL/min
        2. Ensure the overall volume of the vessel never exceeds 125mL

III. Standards Handling and Analysis Procedure:

a. Liquid water standard
    i. Open the ampoule containing the standard as recommended by Honeywell.
    ii. Aspirate approximately 1 mL of the standard 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 standard. Then eject the standard into the waste.
    iv. Aspirate the remaining content of the ampoule 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. Twenty (20) second countdown will commence.
    viii. Inject a suitable amount of standard (0.2-0.7mL) (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. Also ensure that there are no hanging drops on the tip of the syringe as this will also lead to errors.
    ix. After injecting the standard, 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 window.
    xi. Titrant value will be automatically calculated and displayed in the report.
        1. Record the value of the titrant in mg/mL.
    xii. Repeat step vi to xi a minimum of three times.
        1. The average for the titer should have an RSD +/- 2.5%.
        2. Repeat until an acceptable RSD has been achieved.
    xiii. Once an acceptable average value for the titer has been calculated proceed to the Configuration Window.
        1. Common Variables
            a. Titer

i. Value
   1. Replace with the new acceptable average value for the titer you have calculated above.

IV. Sample Sizes using various Titrant 2* and 20 mL Buret:

a. Water standard: 10 mg/g

b. Mass of Titrant 2*: 0.4-3.6 g

NOTE: *Titrant 2: 1 mL of titrant reacts with approximately 2 mg H2O

V. Calculations of Titer concentration in mg/mL

a. By Weight

$$\text{Titer} = \frac{m_{standard} \times w(standard)}{V_{EP}}$$

Titer:	Titer of the selected titrant in mg/mL
$m_{standard}$:	Mass of standard in g
w(standard):	Certified water content of standard in mg/g (for pure water = 1000)
V_{EP}:	Titrant consumption up to the end point in mL

VI. Tips and Suggestions

a. Use fresh and certified water standards for the titer determination.
b. Carry out the titer determination at the same temperature as the water content determination of the sample. A temperature increase of 1 °C results in a titer decrease of approximately 0.1%.
c. Always aspirate the whole content of a liquid standard ampoule into the syringe. If this is not possible, give the remaining standard into the waste. In an open ampoule the standard will change its water content and lead to incorrect results.
d. It is essential that the balance is suitable for small weights is used. Otherwise, the balance error can have a significant influence on the result. Recommend is an

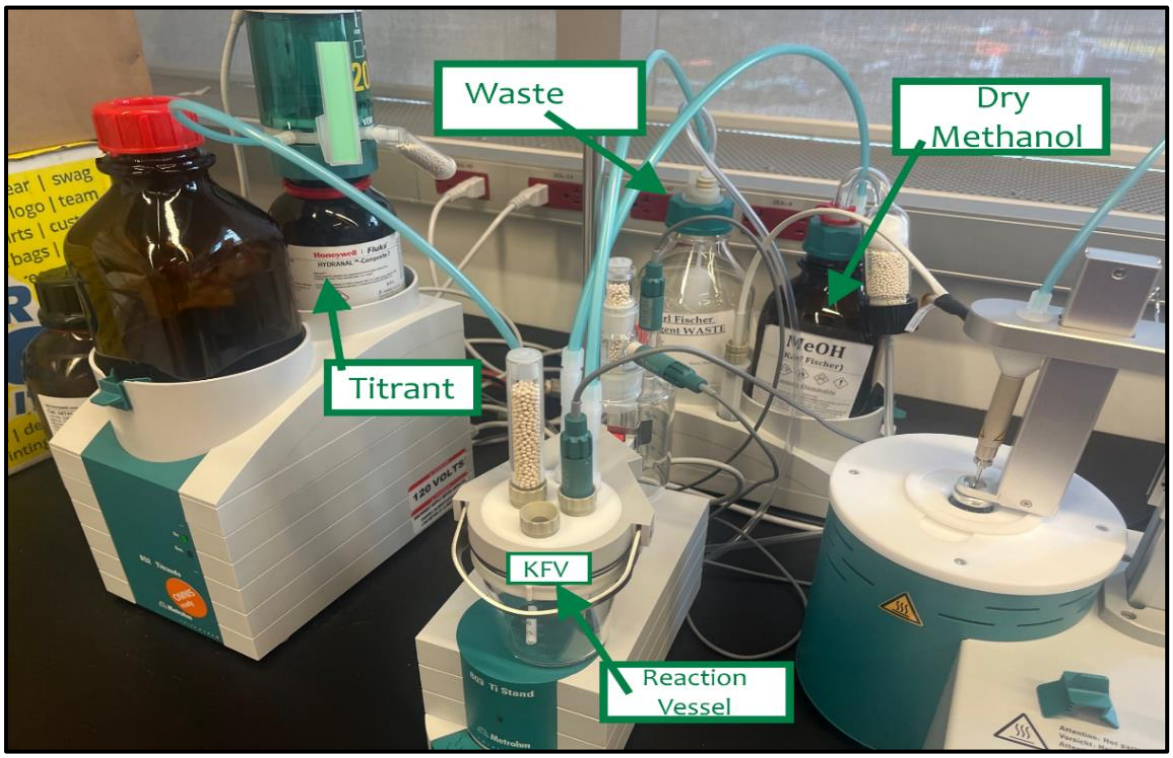
analytical balance with readability & reproducibility of 0.0001g. We also recommend that the balance have a stability period of 3-5 seconds.

e. The density of water is commonly assumed to be 1, which is accurate. In fact, the density of water depends on the temperature. Check the pressure and temperature using the barometer in TRACES to correct the sample size depending on the temperature.

f. We recommend comparing titer values with the titer history of the same titrant bottle as an approximate indicator of a reasonable value. If there are unexpected variations, the system needs to be checked.

1. Appendix

i. KFV Instrument Setup



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