Standard Operating Procedure

Coulometric Karl Fischer titration: KFC

Initial Parameters:

Reaction Cell: 75mL of Coulomat AG solution in the KFC vessel

Method: KFV_C* -no oven

Balance: Analytical balance with readability of 0.0001g

Cell type: Without diaphragm

Solubility: Samples MUST be soluble in methanol

I. Scope

The Karl Fischer (KF) Titrator is exclusively used for the quantification of water. In a coulometric KF titration, the iodine is generated from the coulometric Karl-Fisher (KFC) solution. The water reacts with the reagents to consume iodine. The titrator responds to the loss of iodine by passing current through the anode to generate more iodine from the iodide present in the KFC solution. An excess of iodine indicates the endpoint of the titration. The amount of water titrated is proportional to the amount of iodine generated at the anode and this is proportional to the product of the total current and time needed to reach the endpoint.

II. KF Instrument Setup

a. All glassware must be dry and free of water and contamination.
b. Place 50 mL of the Coulomat AG solution and waste container into the appropriate holder position[1].
c. Startup the Tiamo software, wait till all modules are discovered.
d. Select Workplace icon. Load the method and sample details (i.e., Sample1)
e. 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. 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 second countdown 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. 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 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. KFC 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 $\leq$ 20uL/min
    ii. If the electrode reads $\leq$ 0.2uL/min.
        1. Replace the entire reaction vessel solution.
        2. Wait till Conditioning reads ‘Ok’ at $\leq$ 20uL/min

IV. Cleanup:

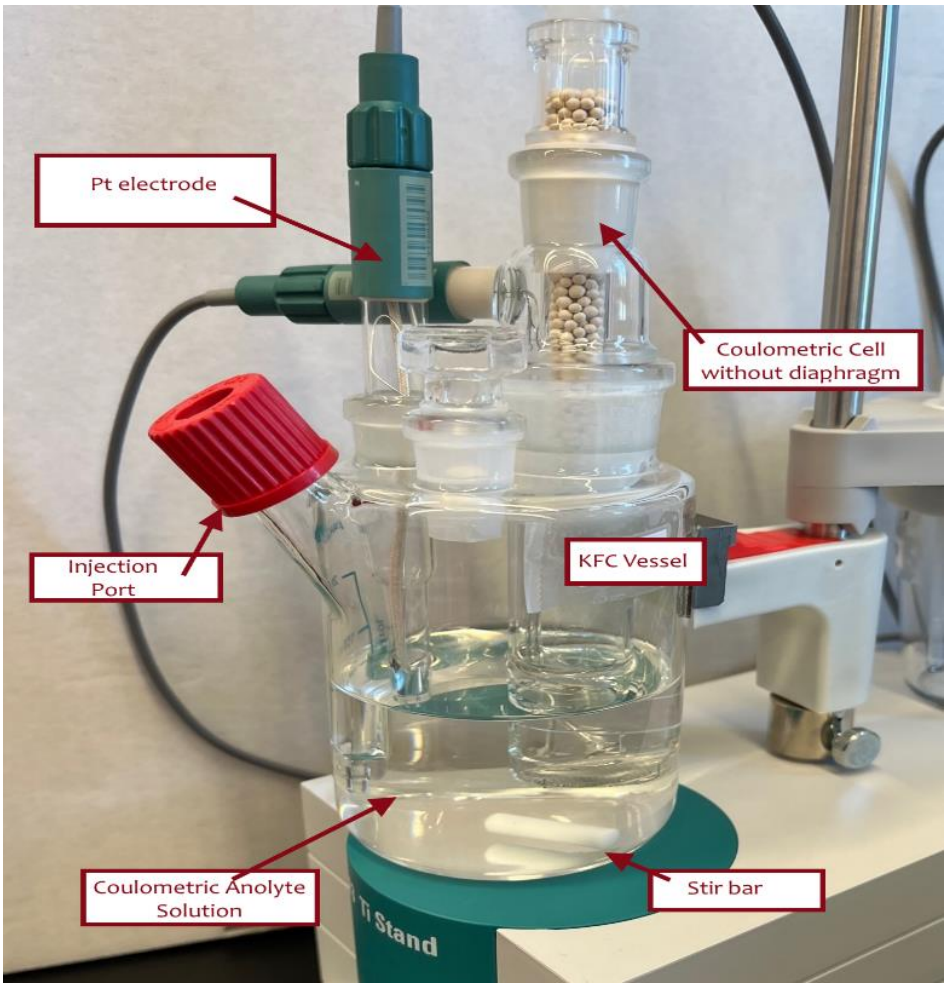
a. Vessel Cleaning
    i. Remove the vessel and rinse with methanol for cleaning.
    ii. Rinse the electrodes with methanol for cleaning.
b. Place all waste into the appropriately labeled TRACES waste container.

V. Tips and Suggestions

a. Use freshly prepared or properly stored samples for KF determination.
b. Carry out the water content determination at constant temperature.
    i. Maximum and Minimum temperature fluctuations: +/- 0.5 ^{o}C.
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. KFC Instrument Setup



ii. Table 1: Sample Size

Water Content	Sample Size
10%	10-50mg
1%	20-200mg
0.1%	100-1000mg
0.01%	1-2g
0.001%	2-5g

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