Standard KFC-Oven Procedure

Water Determination using Karl Fischer Coulometric Titration with Oven

Initial Parameters:

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

Oven attachments: Oven connected to the KFC vessel[1]

Method: KFV_C* -oven

Balance: Analytical balance with readability of 0.0001g

Cell type: Without diaphragm

Solubility: Samples may be soluble in methanol

1. 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. Water in a water or air sensitive solid is determined by heating a sample in an oven at 150 °C to evaporate water which is conveyed to a coulometric Karl Fisher titrator which measures the amount of water evolved. As water is released by the sample it is carried by a constant flow of dry air to the coulometric Karl-Fisher titration cell. As the gas bubbles through the Karl-Fisher solution the water reacts with the reagents to consume I_2. The titrator responds to the loss of I_2 by passing current through the anode to generate more I_2 from the I^- present in the reagents. An excess of iodine indicates the endpoint of the titration. As the amount of water titrated is proportional to the total current (current x time), the water content is determined from the current required for the titration.

2. KF Instrument Setup

a. All glassware must be dry and free of water and contamination.
b. Place 75 mL of the Coulomat AG-oven solution into the KFC vessel[1].
c. Power up the oven.
    i. Select the OK button.
    ii. Set the temperature.
        1. T1: Temperature (50°C is standard for heat sensitive material)

2. T2: final temperature (150°C and over is recommended)

iii. Set up the flow (50mL/min is standard for many applications)
iv. Set the gas type: air.
v. Set the flow source: pump.

d. Startup Tiamo software, wait till all modules are discovered.
e. Select Workplace icon. Load the method and sample details (i.e., Hydranol 1)

3. Sampling Handling and Analysis Procedure:

a. Blank Determination (amount of water in a blank sample vial)
   i. Crimp four empty sample vials.
      1. Three vials will be your blanks.
      2. One vial will be for conditioning the system.
         a. **This vial may have to be replaced if initial conditioning values are not achieved.**
   ii. Place the conditioning vial into the oven[2].
   iii. Place the needle into the vial[3].
      1. Press the needle holder lever.
      2. Push down on the needle holder.
      3. Peirce the septum with the oven needle.
   iv. From the Workplace window, select Start.
      1. Wait until Conditioning reads 'Ok' at ≤ 8ug/min.
      2. Wait till the oven temperature and flow values are achieved.
   v. From the Workplace window, select Start.
      1. 10 second countdown will commence.
   vi. Replace the conditioning vial with a blank vial in the oven.
      1. **Caution the vial is VERY HOT.**
      2. Place the needle into the vial.
   vii. Once the reaction/titration is completed
      1. Return the conditioning vial to the oven.
      2. Peirce the septum with the oven needle.
      3. Wait until Conditioning reads 'Ok' at ≤ 8ug/min.
   viii. Repeat (v to vii) with next two blank vials
      1. Blank samples should provide values below the highest expected water concentrations.
      2. RSD ≤5%
      3. Enter an average of the three values into the Blank value in the Tiamo
         a. Database Window
            i. Configuration Window

1. Blank Value
4. Repeat till acceptable RSD has been achieved.

b. Sample Determination (amount of water in a sample vial)
   i. Crimp an empty vial for conditioning the system.
      a. **This vial may have to be replaced if initial conditioning values are not achieved.**
   ii. Weigh approximately 0.02-0.08 grams of sample into a vial and crimp[4].
   iii. Place the conditioning vial into the oven.
   iv. Place the needle into the vial[3].
   v. From the Workplace window, select Start.
      1. Wait until Conditioning reads 'Ok' at ≤ 8ug/min.
      2. Wait till the oven temperature and flow values are achieved.
   vi. From the Workplace window, select Start.
      1. 10 second countdown will commence.
   vii. Replace the conditioning vial with a sample vial in the oven.
      1. **Caution the vial is VERY HOT.**
      2. Place the needle into the vial.
   viii. Once the reaction/titration is completed
      1. Return the conditioning vial to the oven.
      2. Peirce the septum with the oven needle.
      3. Wait until Conditioning reads 'Ok' at ≤ 8ug/min.
   ix. Repeat (ii to viii) with the sample vials at least two additional times.
      1. RSD ≤5%
      2. Repeat till acceptable RSD has been achieved.

c. KFC Reaction Vessel Concerns
   i. If moisture in the air is a concern, all samples, blanks, and conditioning vials must be prepared in a glove box (inert gas filled).
      1. *Dry the vials in the oven for at least two hours at 120°C.*
      2. *After the vials have been dried remove them from the oven **Caution the vial(s) is VERY HOT**, place them in a desiccator.*
         a. *Leave vials in a desiccator for 10-15 minutes before using.*
         b. *Use vials within the next 4 hours.*
      3. *Take the sample into the glove box.*
      4. *Place a 'dry' vial onto the balance and use as above.*
   ii. If the colour of the reaction vessel solution becomes too dark
      1. Replace the entire solution.
      2. Wait till Conditioning reads 'Ok' at ≤ 8ug/min.
   iii. If the electrode reads ≤ 0.2ug/min.
      1. Replace the entire reaction vessel solution.

2. Wait till Conditioning reads ‘Ok’ at at ≤ 8ug/min.

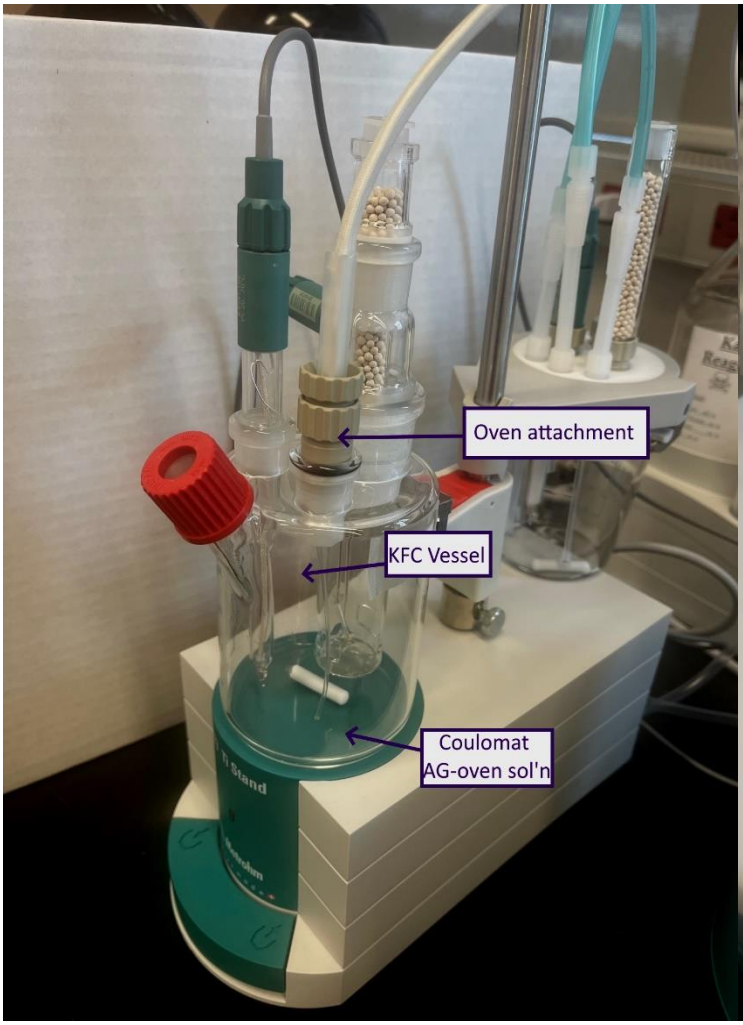
4. Calculations in ug water/ sample g

a. The instrument will calculate the water concentration in ppm, but the following calculation can be done manually if necessary.

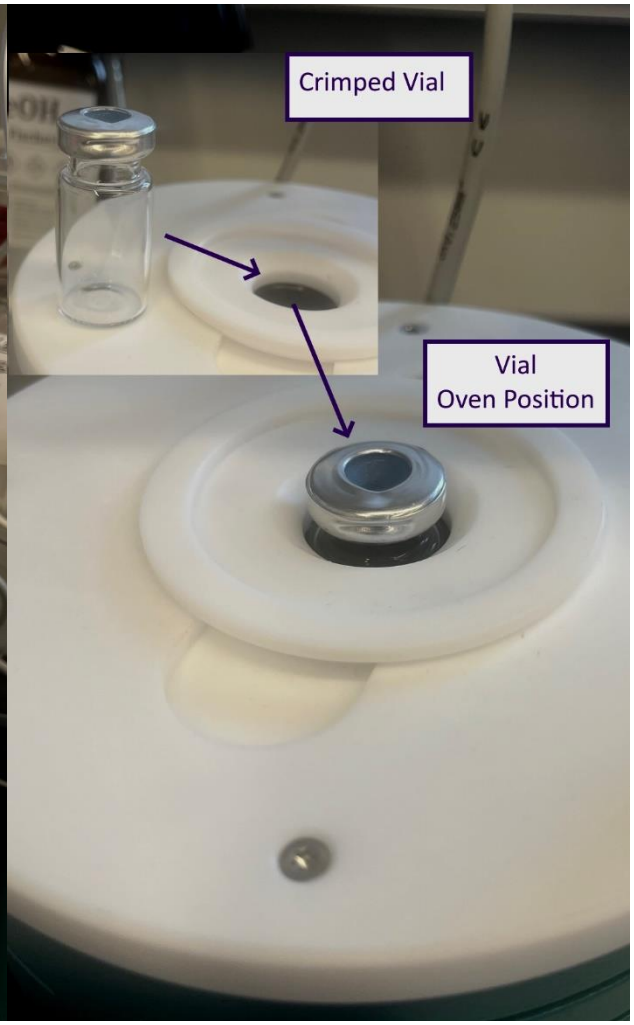
$$\text{ppm water} = \frac{\mu g \text{ water} - \mu g \text{ water from the averaged blanks}}{\text{sample weight, g}}$$

5. Appendix

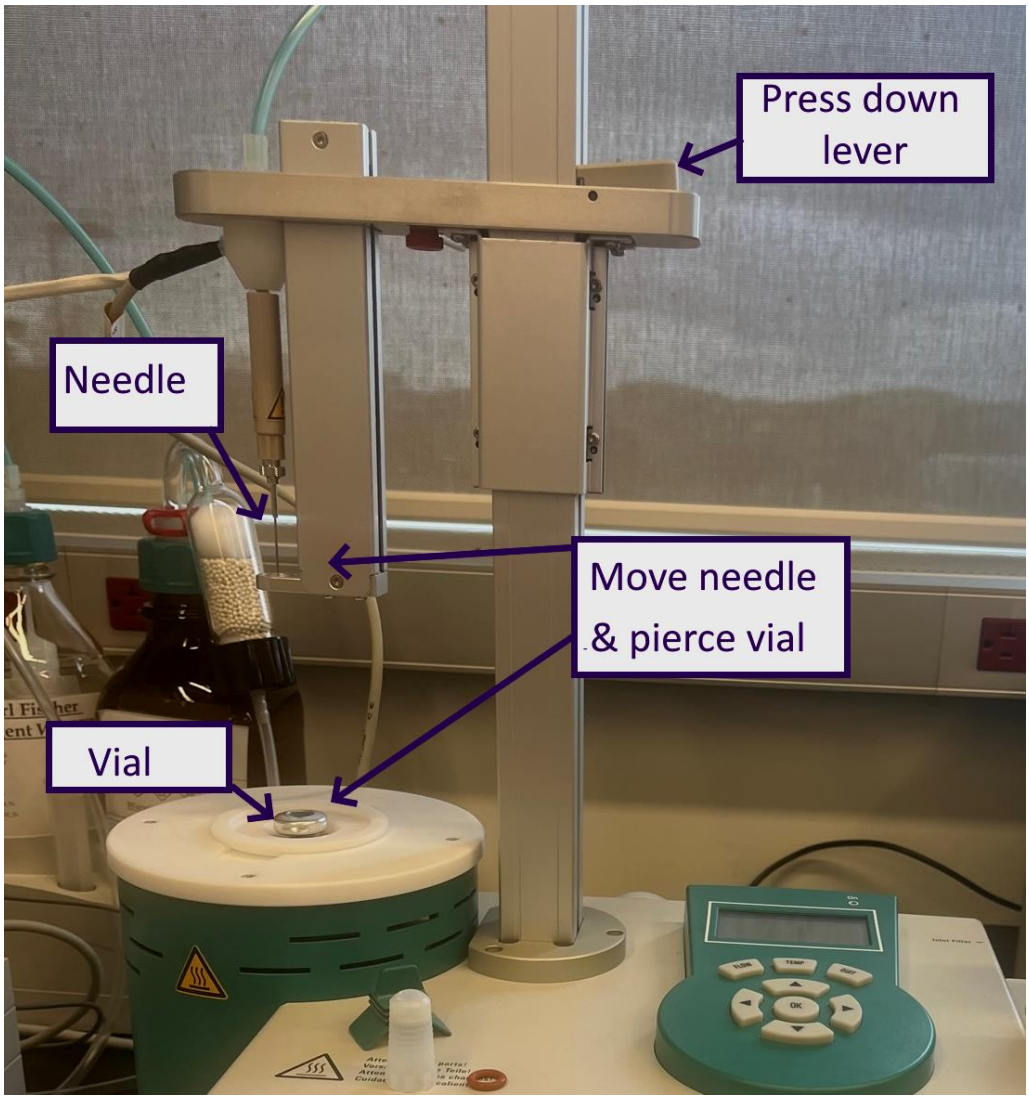
[1]KFC-Oven Setup:



[2]KFC-Oven Vial Setup



[3]KFC-Oven Needle Introduction



[4]KFC-Oven Sample size table

Water Content	Sample Size
≤40%	10-50mg
10%	20-100mg
1%	100-350mg
0.1%	200-1000mg

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