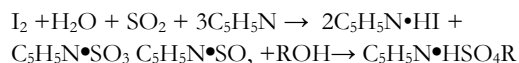


## Moisture content of tall oil by Karl Fischer titration

### Scope

This test method covers the determination of water in liquid organic materials containing 0.5 to 10% water. It is based on the reduction of iodine by sulfur dioxide in the presence of water. The moisture content of tall oil is determined by automatic, volumetric titration with Karl Fischer reagent. In the Karl Fischer reaction, iodine reacts with water in the presence of sulfur dioxide, an organic nitrogen base like pyridine and an alcohol:



The Karl Fischer reagent is the titrant. It normally consists of iodine, pyridine and sulfur dioxide, in a molar ratio of 1 : 10 : 3, dissolved in anhydrous 2-methoxyethanol.

### Safety precautions

Always operate the titrator in a fume hood. Most of the reagents, including some of the pyridine-free systems are noxious or toxic and inhalation or direct skin contact with them should be avoided.

### Apparatus

1. Automatic Karl Fischer titrator.
2. Sample vials, 8-mL or 4-dram.
3. Medicine droppers.
4. Disposable plastic syringes, 1-cc with 16, 18 or 20 gauge needles.
5. Syringe, 10- and 25-uL.
6. Weighing pipet (optional).

### Reagents

Karl Fischer reagent or Karl Fischer pyridine-free reagent Reagents are available from the manufacturer of the apparatus.

### Instrument preparation

Prior to sample analysis the analyst must become familiar with the operation of the titrator which differs slightly depending on the manufacturer of the apparatus.

1. Prepare and calibrate the apparatus as specified in the operating manual supplied by the manufacturer. This generally includes the following steps:
  - a. Clean and assemble the titration chamber.
  - b. Introduce a volume of anhydrous titration solvent into the titration vessel. This solvent is normally ethylene glycol- pyridine and is supplied by the manufacturer.
  - c. Titrate the solvent to the instrument end point with Karl Fischer reagent. This insures that all water due to the solvent and due to moisture on the surfaces of the apparatus is consumed.
  - d. Now standardize the Karl Fischer reagent.
2. Standardize the Karl Fischer reagent each day. Inject exactly 10 uL or exactly 25 uL of water below the surface of the titration solution. Larger volumes of water are undesirable because they require large volumes of titrant which fills the vessel so that the solvent has to be poured out and new solvent introduced. A weighing pipe may also be used to introduce the water.
3. Put the instrument in the TITRATE or RUN position and record the volume of titrant used when the END light comes on or a message appears that the titration is complete.

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4. Calculate the weight of water in milligrams per mL of titrant. This is the equivalency factor and will be used later in the calculation of the water content of the sample:

$$F = \frac{A}{B}$$

where:

- F = equivalency factor, mg of water per mL of titrant  
A = weight of water used, mg (10 or 25)  
B = volume of Karl Fischer titrant used, mL

### Procedure

Add the sample to the titrator after the standardization has been completed.

1. Put approximately 4 mL of tall oil into a small sample vial. Do not allow the sample to touch the outside of the vial.
2. Put a medicine dropper into the vial and weigh the vial, sample and medicine dropper together on an analytical balance to the nearest 0.001 g. Record this weight.
3. Using the medicine dropper, introduce 0.5 to 1.0 mL of the sample into the titrator.
4. Put the medicine dropper back into the vial and weigh to the nearest 0.001 g. Record this weight.
5. Begin the titration and record the end point in mL as before.
6. Calculate the water content of the sample as shown below.

### Calculation

$$\text{Moisture, \%} = \frac{(V \times F)}{10 \times W}$$

where:

- V = volume of Karl Fischer reagent required to titrate the sample, mL  
F = equivalency factor, mg of water per mL of titrant  
W = weight of sample added to the titrator, g

Report to the nearest 0.01%.

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### Alternate methods

PCTM 4A, PCTM 4B

### References

ASTM D890 "Water in Liquid Naval Stores."  
ASTM D1364 "Water in Volatile Solvents."