

Determining Water Content of Crude Oils

How its done, what can go wrong.

Introduction

- Overview of the tests methods used for water determination in and factors affecting their precision and accuracy
- Bruce Williams, ITS Caleb Brett

Topics of Discussion

- Why is water important:
 1. Money – Water displaces crude which costs money
 2. Processability – Water content can cause difficulties in refining and transportation; corrosion
 3. Environmental – Water must be treated before it can be disposed of

Test Methods for Determining Water

- **D 96 - Water and Sediment in Crude Oil by Centrifuge Method (Field Procedure)**
- **D 4006 -Water in Crude Oil by Distillation**
- **D 4377 - Water in Crude Oils by Potentiometric Karl Fischer Titration**

D96 – Centrifuge Method

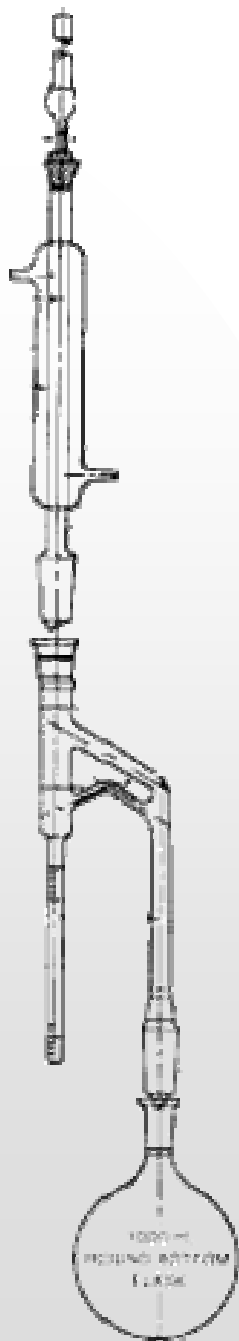


- Heated Crude is mixed with solvent (generally water saturated toluene)
- De-emulsifier added if needed
- Crude is centrifuged at 500X gravity
- Amount of sediment and water read at bottom of tube

D 96 – Factors Effecting Precision and Accuracy

- Incomplete Centrifugation
- Improper reading of Results
- Formation of an emulsion (rag) layer

D 4006 – Water by Distillation



- Crude is mixed with water immiscible solvent (xylene)
- The mixture is boiled (refluxed) forcing water and solvent overhead
- Condenser condenses solvent and water into trap
- Trap returns solvent to crude while retaining water

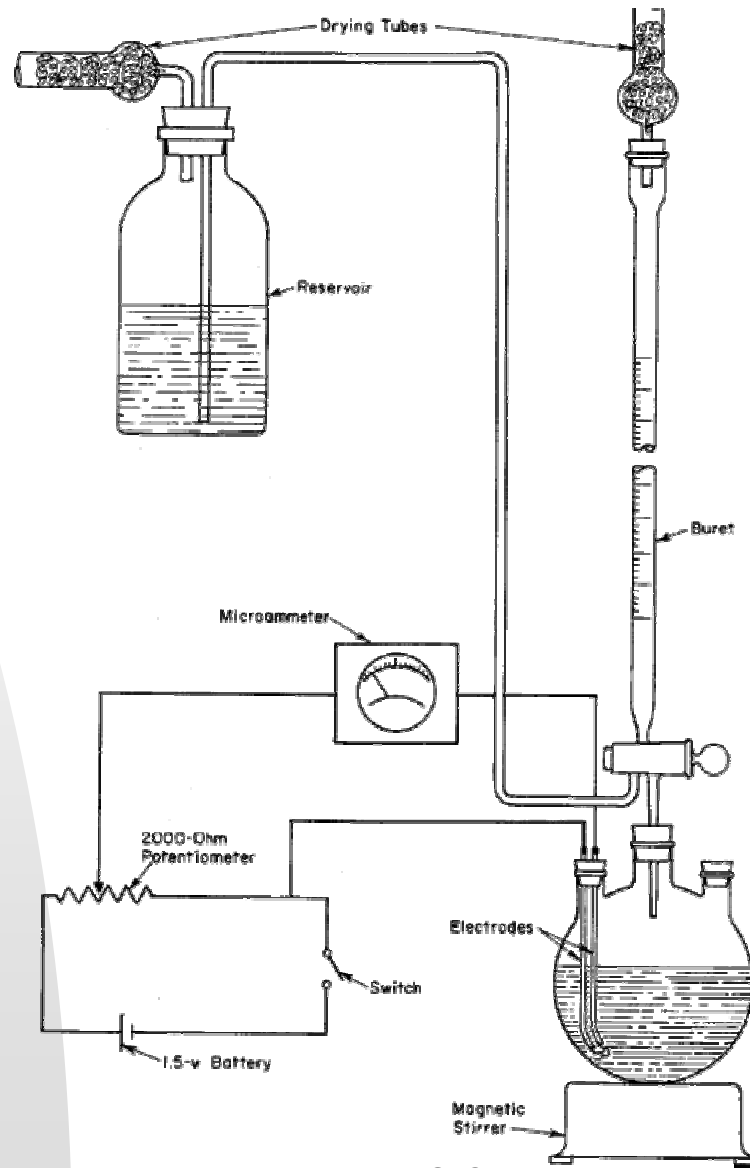
D 4006 Factor which affect precision and accuracy

- Water can adhere to glassware if it's not cleaned properly
- Incomplete refluxing of crude
- Improper reading of trap

D 4377 – Water by Karl Fischer

- Titration cell solvent is titrated (zeroed) with Karl Fischer (KF) solution
- Sample is injected into cell
- Water in sample reacts with Iodine from KF solution raising cell resistance
- Fresh KF solution is added until original resistance restored
- Amount of KF solution added is directly proportional to water in sample

D 4377 Water by Karl Fischer Apparatus



ITS Caleb Brett

D 4377 – Factor affecting Karl Fischer Precision and Accuracy

- Strength of KF titrant
- Accuracy of sample weight introduced
- Interferences in crude such as sulfur compounds, metals, and others
- Improper determination of cell equilibrium (Zero)

The Big Problem!!

- Sampling, Sampling, and Sampling
- Water settles out making it hard to obtain samples which are truly representative of the bulk crude
- Water will settle in samples over a very short time making it necessary to homogenize some samples
- Water will form emulsions with crude oil which are very hard to break and can go undetected

Questions?

- Bruce Williams
- Email bwilliams@itscb.com
- 832-771-6251