Estimating the Amount of Moisture Content in Crude Oil Samples

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Abstract :- Determination of the amount of water in crude oil and petroleum products has always been important. Rather than paying crude oil prices for water, contracts have been based on "net dry oil". This is calculated by reducing the total gross standard volume (GSV) by the amount of water and sediment present as determined by analyzing a sample of the oil. Accurate analysis for the water content is usually more difficult than the determination of gross volume, temperature, and gravity of the oil.

Keywords: - oil, water, dean stark apparatus

I. INTRODUCTION

There are many Concepts that yield water as a co-product where removal of the water as it is produced is necessary to drive the reaction to completion and this is done using a Dean - Stark apparatus. The reaction is carried out under reflux in a solvent which is less dense than water. The determination of the amount of water in crude oil and petroleum products has always been important. Rather than paying crude oil prices for water, contracts have been based on "net dry oil". This is calculated by reducing the total gross standard volume (GSV) by the amount of water and sediment present as determined by analyzing a sample of the oil. Accurate analysis for the water content is usually more difficult than the determination of gross volume, temperature, and gravity of the oil.

In production areas as well as pipeline, custody transfer is based on "net dry oil". Marine facilities must determine the water content of the oil to verify the bill of lading figures in addition to determining the quantities received in their shore tanks. In the refinery, maintaining low water content is important to the operation of the crude unit, and it is a major part of quality control in the production of products like lubricating oils and transformer oils. The water content of oil can be determined either by static or dynamic methods. The static methods require that a sample of the oil be removed from the pipeline or tank; then it is analyzed. Dynamic methods determine the water content as the oil flows through, or past, an instrument located in the pipe. The following will focus on the determination of water content in the static state.

In the field method a precision micro-syringe is used to inject an exact volume of sample into the titration cell. After the titration is complete, the instrument will display the volume percentage of the water. For use in the field, a volume percentage of water is of much greater value than a mass percentage. The water content is multiplied by the volume of oil to determine the volume of water to be deducted from the total measured volume of oil. If a mass percentage (or ppm) is desired then the resulting micrograms can be divided by the specific gravity of the oil and the injected sample volume. Regardless of the method used to determine the water content, the result cannot be any better than the quality of the sample analyzed. The sample must be truly representative of the total volume of oil. If the sample to be analyzed is obtained from tank sampling, the proper procedures must be followed to assure a representative sample. If the sample is obtained from a continuous, automatic, on-line sampler, the system must be operated and maintained in accordance with the API Standard. After the sample has been obtained in a sample bottle, it must be completely homogenized to assure that the portion actually analyzed is completely representative of the total volume. High-speed, no-aerating, shear mixers are recommended in the API and ASTM D4928 standards to assure complete homogeneity of the sample before analysis. During the reaction in vapors containing the reaction solvent and the component to be removed travel out of reaction flask up into the condenser and then drip into the distilling trap. Here, immiscible liquids separate into layers. When the top less dense layer i.e. (Oil) reaches the level of the side-arm Oil Will flow back to the reactor while the bottom layer i.e. (Water) remains in the trap. The trap is at full capacity when
the Water reaches the level of the side-arm—beyond the point, The Water would start to flow back into the reactor as well. It is therefore important to drain the water from the Dean-Stark apparatus as much as needed.

A method for the measurement of fluid saturations in a core sample by distillation extraction is preferred. The water in the sample is vaporized by boiling solvent, then condensed and collected in a calibrated trap. This gives the volume of water in the sample. The solvent is also condensed then flows back over the sample and extracts the oil Crude is mixed with water immiscible solvent (xylene). The mixture is boiled (refluxed) forcing water and solvent overhead which condenses solvent and water into trap. Trap returns solvent to crude while retaining water. Designed specifically for azeotropic distillation using solvents heavier than water (e.g., chloroform). The 8 mL reservoir version is specifically designed for small scale azeotropic distillation of valuable solvents using heavy water. 2 mm bore PTFE stopcock and 300 mm overall height. Solvent vapors condense and collect in the receiver. The modified Dean-Stark trap separates the two components. Heavier than water layer sinks to the bottom of the trap leaving the water on top. The solvent is forced back through the inside tube and returned to the boiling flask. The upper water layer is drained out through the 2 mm PTFE stopcock. The Dean-Stark apparatus typically consists of vertical cylindrical glass tube, often with a volumetric graduation along its full length and a precision stopcock at its lower end, very much like a burette. The lower end of a reflux condenser fits into the top of the cylinder. Immediately below the joint between the condenser and the cylinder is a sloping side-arm that joins the cylinder to a reaction flask. The lower end of the side-arm turns sharply downward, so that the side-arm is connected to the reaction flask by a vertical tube. The reaction flask is heated. Boiling chips within it assist with the calm formation of bubbles of vapor containing the reaction solvent and the component to be removed. This vapor travels out of reaction flask up into the condenser where water being circulated around it causes it to cool and drip into the distilling trap. Here, the immiscible liquids separate into layers (water below and solvent above it). When their combined volume reaches the level of the side-arm, the upper, less dense layer will begin to flow back to the reactor while the water layer will remain in the trap. The trap will eventually reach capacity when the level of the water in it reaches the level of the side-arm. At this point, the trap must be drained into the receiving flask. The process of evaporation/condensation/collection may be continued until it ceases to produce additional amounts of water. More rarely encountered is the model for solvents with a density greater than water. This type has a tube at the bottom of the side-arm to allow the organic solvent at the bottom to flow back into the reaction vessel. The water generated during the reaction floats on top of the organic phase. This piece of equipment is usually used in azeotropic distillations. A common example is the removal of water generated during a reaction in boiling toluene. An azeotropic mixture of toluene and water distills out of the reaction, but only the toluene (density=0.865 g/ml) returns, since it floats on top of the water (density=0.998 g/cm3), which collects in the trap. The Dean-Stark method is commonly used to measure moisture content of items such as bread in the food industry.

II. EXPERIMENTAL PROCEDURE

Firstly This Apparatus Needs Continuous Flow Of Water Circulation. About 100ml Of Oil Is taken In The Round Bottom Flask Then Began By Charging The Boiling Flask with the Mixture Then Plum the Cold Water into the condenser creating zone for Cooling where vapor Will began to condense . Crude is mixed with water immiscible solvent (xylene). The mixture is boiled (refluxed) forcing water and solvent overhead condenses solvent and water into trap. Trap returns solvent to crude while retaining water.
Regardless of the water determination methodology employed, several basic points need be followed. The application should be studied to determine the specific need for water determination and whether they can best be met by static methods or dynamic methods. Then, which specific method will provide the most desirable result. Then the best equipment/instruments should be selected to meet the requirements of the application. The equipment should meet all of the appropriate standards; such as, API, ASTM, US Coast Guard, Bureau of Land Management, Minerals Management Service, etc. The manufacturer's instruction should be followed in operating and maintaining the equipment. For Static methods, the appropriate analytical procedures must be properly followed. One should remember that the determination of the water content of the oil directly affects the revenue received for the oil.

**Sample 1**: Crude of Chennai  
**Sample 2**: Crude of Gujarat  
**Sample 3**: Crude of Essar Oil (Gujrat)

**Test Method**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Moisture Content</th>
<th>Moisture Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>0.20</td>
<td>0.28</td>
</tr>
<tr>
<td>Sample 2</td>
<td>0.11</td>
<td>0.11</td>
</tr>
<tr>
<td>Sample 3</td>
<td>0.02</td>
<td>0.06</td>
</tr>
</tbody>
</table>

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III. RESULTS

It is observed that Sample 1: 0.7% of Moisture, Sample 2: 0.2% of Moisture, Sample 3: 0.4% of Moisture content present in tested crude samples. Although sample 1 consist of more amount of moisture obtained because. The crude was re-refined. Sample 2 & sample 3 has less amount of moisture.

REFERENCE