OPERATOR KARL FISCHER WATER ANALYSIS OF DMSO

1.0 Purpose

To analyze for the moisture content in DMSO using the Karl Fischer titration method. Because DMSO is very hygroscopic, sample handling is very important in achieving accurate results.

2.0 Safety/Health

2.1 PPE

Wear standard laboratory safety equipment consisting of safety glasses or goggles, gloves and protective clothing.

2.2 Safety Systems

N/A

2.3 Health Hazards

Refer to the Material Safety Data Sheets located in the Catalog of Chemicals.

3.0 Process Description

3.1 Process Overview

This method is used to analyze for the moisture content of DMSO using the Karl Fisher titration method.

3.2 Scope/Boundaries

N/A

3.3 Equipment Involved

Brinkmann 701 KF Titrino autotitrator module equipped with a 701TI stirrer stand/pump unit.
AQUASTAR COMP-2 Karl Fischer titration reagent.
AQUASTAR SOLVENT KN
2.0 cc syringe equipped with a rubber stopper
A suitable sample container. (A 4 oz glass bottle with septum top is recommended.)

4.0 Procedure

4.1 Pre-Analysis Instrument Checks

A. Check that the waste bottle has enough room to hold the used reagent. If it does not, pour the contents down the sink under the hood. (Make sure hood is on and some water is running in the sink.) Re-install tightly to insure proper waste pump operation.

B. Check the levels in the COMP-2 reagent bottle and the SOLVENT-KN reagent bottle. If either of these require filling, do so using the reagent bottles on the counter next to the instrument. (These reagents are also stored in the DMS test shack for use in the DMS dehydrator moisture analysis. Get bottles from this shack if none are on the counter.) Re-install tightly to insure proper solvent and waste pumping.

NOTE: IF THE COMP-2 REAGENT BOTTLE IS REFILLED, LEAVE A NOTE FOR THE LAB PERSONNEL. THEY WILL RE-CALCULATE THE TITER OF THE REAGENT AND CHANGE THE FORMULA CONSTANT AS SOON AS POSSIBLE.

C. Pump the SOLVENT-KN that is in the reaction vessel to the waste container using the integrated pump unit. When the reaction vessel is empty, use the pump unit to pump fresh SOLVENT-KN into the reaction vessel until the solvent covers the platinum tips on the probe.

D. Check that the Dv / dt rheostat on the front of the autotitrator is on "3", and that the stirrer speed rheostat on the front of the stirrer / pump unit is on "3" also.

4.2 Sampling And Handling

Sampling is performed by DMSO operations personnel and is the responsibility of the operator to insure that the sample is from the proper tap and is not contaminated prior to analysis. Rinse sample bottle two or three times with sample to purge any contaminated DMSO. Fill with sample and quickly replace septum top. Insert 2.0 cc syringe's needle through the septum and below the surface of the DMSO. Fill syringe two or three times, ejecting each into the sink or into a waste container (A 250 ml Erlenmeyer flask works well. Just empty it into the sink under the hood when finished with analysis.) Fill the syringe to the 2.0 cc line and remove from sample bottle. (2.0 cc = 2.0 ml) Quickly insert needle into green stopper to minimize contact with water in the air. Sample is now ready to inject into the autotitrator.

4.3 Analysis

A. Push the START button on the front of the autotitrator. The green indicator light on the front of the unit will start blinking, and the unit will show "WAIT" on the LCD display. This indicates that the instrument is titrating any moisture in the SOLVENT-KN or "conditioning" the reaction vessel. When the SOLVENT-KN is dry, the LCD display will show the word "CONDITIONING", and the green indicator lamp will stop blinking.

B. Push the START button again. The buret on the autotitrator will refill and the LCD display will show the sample weight to be "2.2 grams." This is the weight of the 2.0 cc of DMSO.

C. Remove the stopper from the needle and inject the sample into the reaction vessel through the sample injection port located at the top front of the reaction vessel.

D. Push the START button once again. The autotitrator will now start titrating the water in the injected DMSO sample and the LCD display will show a running display of the milliliters of the AQUASTAR COMP-2 reagent used. A blinking "pound" sign in the upper left corner indicates that the autotitrator is operating normally.

E. When all the water is titrated the buret will stop automatically and the LCD display will show the ppm of water.

NOTE: THE AUTOTITRATOR AUTOMATICALLY CALCULATES THE ppm OF WATER IN THE SAMPLE USING THE TITRATION VOLUME, THE TITER OF THE COMP-2 REAGENT, AND THE WEIGHT OF THE SAMPLE.

F. Push the STOP button.

G. If performing multiple titrations in a short time span (two or three titrations within less than 5 minutes), it is acceptable to re-use the same SOLVENT-KN. Just push the START button, inject the next sample, and push START again. Push the STOP button after the last sample has been titrated.

H. If the autotitrator is not going to be used for an extended period (an hour or so), or if finished with multiple titrations, pump the used SOLVENT-KN to the waste container and refill the reaction vessel with fresh SOLVENT-KN.

5.0 Documentation

5.1 Report the water content (ppm) of the DMSO in the appropriate space on the DMSO Plant Operations Log Sheet and in the KFT-1 GMP logbook located next to the instrument.

5.2 Log all analyses into USP Karl Fisher Usage Logbook located by instrument.

6.0 Glossary

N/A

7.0 References

N/A

8.0 Test Specifications

The current manufacturing spec for water in DMSO is 1000 ppm. If any product hold tank sample indicates water in excess of this spec, it must be reprocessed through the DMSO purification system.