OPERATION KARL FISCHER WATER ANALYSIS OF DMSO
WI-QA-066

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 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 Fischer titration method.

3.2 SCOPE/BOUNDARIES
N/A

3.3 EQUIPMENT INVOLVED
- Metrohml KF Titrino autotitrator module equipped with stirrer stand/pump unit.
- AQUASTAR COMP-2 Karl Fischer titration reagent, or equivalent
- AQUASTAR SOLVENT KN, or equivalent
- 2.0 mL syringe
- A suitable sample container.

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 into the correct lab waste container.
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 located in the chemical store room. 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.

4.2 ANALYSIS

a. If needed, pump solution from titration cell into the waste container and pump in fresh solvent KN solution.
b. Before analysis begins, the solvent KN solution in the titration cell must be sufficiently conditioned. As titrant is added and the solution becomes conditioned, the drift on the KF display will begin to decrease. Once the drift is <20µL/min the display will change to “Conditioning OK”. The titrator is now ready for analysis.
c. Rinse the syringe with the sample to be tested a minimum of 2 times. Pull up 2mL of the sample to be tested and press the Start button on the titrator. 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 2mL of DMSO. If the correct weight is not listed, adjust it using the controls on the titrator.
d. Inject the sample into the reaction vessel by inserting the syringe needle through the septum in the sample injection port located at the top front of the reaction vessel. Inject the sample slowly so that no splashing occurs, as this will lead to faulty results.

e. 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.
f. When all the water is titrated, the buret will stop automatically and the LCD display will show the analysis result as ppm of water.

NOTE: The titrator automatically calculates the ppm of water found in the sample by using the volume of titrant to reach the endpoint, the titer of the comp-2 reagent and the weight of the sample tested.

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. Push the START button, inject the next sample, and push START again.
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

- Record the water content (ppm) of the DMSO in the KF usage logbook located by the instrument.

6.0 GLOSSARY

N/A

7.0 REFERENCES

N/A

8.0 TEST SPECIFICATIONS

If any product hold tank sample indicates water in excess of 1000ppm, contact operations manager and quality assurance manager immediately. A transfer of a hold tank exceeding 1000ppm of water into the bulk storage tank must be approved by the operations manager and quality assurance manager.

9.0 REVISION LOG

DATE	SECTION/PAR	CHANGE DESCRIPTION
8/21/2007	All	Last Revision Before Issued In Web-based QMS
8/18/2010	3.3	Deleted specific details on equipment involved
03/22/2011	3.3, 4.1.a, 5.0	Add Metrohml, KF, 4.1.a Add store room. Delete quotations. 5.0 delete last bullet.
2/12/2014	4.2, 5.0, 8.0	4.2 changed sink to counter. 5.0 change KFT-1 to KFT-3. 8.0 added IND and PHS DMSO
05/08/2017	All	Adjusted to mimic current analysis techniques. Adjusted specification section for notifying managers when hold tanks exceed specification. Updated waste procedure.

Comments, questions or suggestions, please e-mail

Quality Assurance Manager