1.0 PURPOSE

This document establishes MSHA's Standard Test Procedure (STP) for the Determination of Water in Hydraulic Fluid Using the Karl Fischer Titration Method.

2.0 SCOPE

This document applies to MSHA approved Fire-Resistant Hydraulic Fluids (FRHF), audits of MSHA approved FRHFs, and accident investigations involving MSHA approved FRHFs. This document also applies to other materials/products that may need titrated to determine their water content.

3.0 REFERENCES

3.1. 30 CFR, Part 35, Subpart A

3.2. Operating Instructions for the Mettler Toledo V20/30 Karl Fischer Titrator

4.0 DEFINITIONS

4.1. Fire-resistant hydraulic fluid - means a fluid of such chemical composition and physical characteristics that it will resist the propagation of flame.

5.0 TEST EQUIPMENT

a. Mettler Toledo V20 Karl Fischer Titrator

b. Mettler Toledo AG245 Balance

6.0 TEST SAMPLES

Three samples of the hydraulic fluid are tested; the size of the sample is typically 3 or 4 drops injected through the septum and into the burette. The total typical weigh of the injected should be between 0.02g to 0.05g.

7.0 PROCEDURES

7.1. Pretitration & Drift Determination

7.1.1. Turn power switch on (located on the front panel of the unit).

7.1.2. Start the Pretitration and/or Drift Determination by pressing the "Titrate" button on the LCD screen. After the titrator performs a pretitration and/or drift determination the titrator will display "Standby Mode" on the LCD screen.

7.1.3. In the event that "Overtitration" appears in the Title Bar of the LCD screen, the operator should shake the burette in an effort to wet the sides of the burette glass. If after shaking, "Overtitration" continues to appear in the Title Bar, the operator should remove the septum and exhale one warm moist breath into the burette. This procedure should remove the "Overtitration" from the Title Bar & reset the titrator into the "Standby Mode" (ready for a concentration determination &/or titration).

7.1.4. **Note**: **For very accurate titrations and determinations of small amounts of water (less than 1 mg), it should be determined periodically.** When the drift determination is completed, the titrant drift value is entered in the setup of the titration stand. The system then generates an automatic printout containing the sample data, raw results, and resource date. If the drift falls below a defined value, the system automatically switches to the Standby mode. The sample size should be selected so that between 30% and 70 % of the burette volume can be titrated.

7.2. Concentration Determination

7.2.1. After the titrator has completed the pretitration process and the titrator is in the "Standby Mode" press the Concentration button.

7.2.2. Place the syringe on the balance and "tare" the weight after it has stabilized.

7.2.3. Insert syringe needle into septum and inject 3 or 4 drops (0.02g to 0.05g) of distilled water into the burette.

7.2.4. Immediately press the "OK" button, and then place the syringe back onto the balance. The weight of the injected water will be display in the Title Bar of the LCD screen.

7.2.5. After the titrator completes the Concentration Determination, the results will appear on the LCD screen - press "OK".

7.2.6. Repeat Steps 2 through 5 two or three additional times (or Series) in order to ensure repetitive results; then press the "More" button from the following menu: Result, Sample, More, Start Concentration, Start Sample.

7.2.7. The titrator will then display the following menu: Start Drift Determination, End Series, Stop Method, Axes, Sample Size Calculation - press "End Series".

7.2.8. The titrator will then display the following menu: Yes, No - press "Yes".

7.2.9. The titrator will then print the results of each Concentration Determination along with the average results for the complete series of Concentrations Determinations.

7.2.10. **Note**: **For accurate titrations, the concentration should be determined periodically.** The determined concentration or the mean value for a series of several concentration determinations will be entered in the Setup of the relevant titrant, if it falls within the limits. If the mean value falls outside the specified limits, it will not be transferred to the Setup, but will still switch to the Standby Mode.

7.3. Water Titration Determination

7.3.1. After the titrator has completed the pretitration process and the titrator is in the "Standby Mode" press the Titration button.

7.3.2. Place the syringe on the balance and "tare" the weight after it has stabilized.

7.3.3. Insert syringe needle into septum and inject 3 or 4 drops (0.02g to 0.05g) of the test sample into the burette.

7.3.4. Immediately press the "OK" button, and then place the syringe back onto the balance. The weight of the injected water will be display in the Title Bar of the LCD screen.

7.3.5. When the titrator completes the titration, the results will appear on the LCD screen - press "OK".

7.3.6. Repeat Steps 2 through 5 two or three additional times in order to ensure repetitive results; then press the "More" button from the following menu: Result, Sample, More, Start Concentration, Start Sample.

7.3.7. The titrator will then display the following menu: Start Drift, Determination, End Series, Stop Method, Axes, Sample Size Calculation - press "End Series".

7.3.8. The titrator will then display the following menu: Yes, No - press "Yes".

7.3.9. The titrator will then print the results of each Water Titration Determination along with the average results for the complete series of Water Titration Determinations.

8.0 TEST DATA

Generally, titration results are reported as the percent water based on the average of 3 samples. Record the results of titration on the Karl Fischer Titration Test Sheet (see page 5).

9.0 PASS/FAIL CRITERIA

Generally, the titration results will fall within ±1% of the water content specified on the company's application for approval of the Fire Resistant Hydraulic Fluid according to the Code of Federal Regulations, Title 30, Part 35.

Karl Fischer Titration Test Sheet

PAR No.	File No.
Company:	
Hydraulic Fluid Trade Name:	
Hydraulic Fluid Type:	
Tested By:	
Date:	
Test Sheet _____ of _____	
Sample 1 Mass of water injected (g): _____ Weight Percent of Water: _____	
Sample 2 Mass of water injected (g): _____ Weight Percent of Water: _____	
Sample 3 Mass of water injected (g): _____ Weight Percent of Water: _____	
Sample 4 Mass of water injected (g): _____ Weight Percent of Water: _____	
Sample 5 Mass of water injected (g): _____ Weight Percent of Water: _____	
Average Weight Percent of Water: _____	
Comments	