Standard Test Procedure for Conducting the Autoignition Temperature Test on Hydraulic Fluids Submitted for MSHA Approval: 30 CFR, Section 35.20

1.0 Purpose:

1.1 This document establishes MSHA's Standard Test Procedure (STP) for Conducting the Autoignition Temperature Test on Hydraulic Fluids Submitted for MSHA Approval: 30 CFR, Section 35.20.

1.2 The purpose of the test is to determine the lowest autoignition temperature of a hydraulic fluid at atmospheric pressure when using the syringe-injection method.

2.0 Scope:

2.1 This document applies to all applications for MSHA approval of Fire-resistant Hydraulic Fluids (FRHF) and audits involving MSHA approved FRHFs.

3.0 Reference:

3.1 This document supersedes CDS document ASTP4014 (undated).

3.2 30 CFR, Part 35, Subpart A & B

4.0 Definitions:

4.1 Autoignition – the minimum self-ignition temperature, at atmospheric pressure, at which a hydraulic fluid will burst into flame.

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

4.3 Emulsion – A mixture of oil and water in which water is the continuous phase, and having a water content as high as 95% by volume.

4.4 Invert Emulsion – A mixture of oil and water in which oil is the continuous phase, and usually having a water content between 40% and 45% by volume.

4.5 Glycol – A mixture of water and a dihydric alcohol such as ethylene glycol.

4.6 Synthetic – A mixture of chemical compounds, containing no water.

5.0 Test Equipment:

5.1 The equipment used to conduct the autoignition temperature test consists of the following components:

a.	Autoignition Test Furnace-	Built to ASTM Standard D-2155-66 Furnace Specifications
b.	Fume Hood	METB Laboratory
c.	Ring Stand	30 - inch
d.	Mirror and Clamp	Mounted on the ring stand
e.	Erlenmeyer Flask -	200-milliliter capacity, borosilicate glass or Pyrex
f.	Syringe-	0.25-millimiter capacity, calibrated in .01cc divisions
g.	Syringe Needle-	No. 18, 2-inch stainless steel
h.	Stopwatch or Timer	5 - minute
i.	Compressed Air	Available within METB fume hood

6.0 Test Procedure:

6.1 <u>General Description:</u>

The method for testing hydraulic fluids described in this procedure is to inject a small amount of fluid from a syringe into a flask that has been preheated in the autoignition test furnace (Figure 1, page 10) to a desired temperature at atmosphere pressure. The fluid is then observed for 5 minutes to determine whether autoignition occurs. The temperature of the flask is then raised or lowered and the size of the sample is varied until the lowest autoignition temperature is determined.

6.1.1 Prior to beginning a test to determine the autoignition temperature of a fluid the investigator should:

a. review the “Autoignition Test Procedure Flow Diagram” (Figure 2, page 11),
b. and complete the appropriate information on the Autoignition Temperature Test Data Sheet (Figure 3, page 12).

6.12 Place the autoignition test furnace under the fume hood and turn the ventilation fan on.

6.13 Remove the furnace cover and place a clean 200-ml Erlenmeyer flask inside the furnace as shown in Figure 4, page 13.

6.14 Ensure that the thermocouples at the base, center and neck of the flask touch the flask.

6.15 Replace the furnace cover and center the neck of the flask in the hole of the furnace cover.

6.16 Adjust the observation mirror (mounted on the ring stand above the Erlenmeyer flask) so that the interior of the flask can be seen in the mirror.

6.17 Set the three digital temperature controllers (Figure 1, page 10) at 600°F.

Warning: The following test involves the use of a high-temperature electric furnace. Exercise extreme caution when working near it to avoid contact. Surfaces may be as hot as 1000°F.

6.18 Turn on the autoignition test furnace and subdue the lighting in the hood and/or laboratory to facilitate observing any ignition within the flask.

6.2 <u>The Basic Test (0.07 cc):</u>

6.2.1 Fill the syringe with 0.07 cc of hydraulic fluid.

6.2.2 Wait until the three temperature indicators read an average of 600°F (± 2°F) for the base, center and neck thermocouples.

Warning: Toxic gases may be released during this test. Do not inhale fumes. If ignition occurs, turn on the fume hood fan to evacuate the fumes, than turn

the fan off.

6.2.3 Quickly inject the fluid into the flask, remove the syringe, and start the timer.

6.2.4 Observe in the mirror the reflected inside of the flask until either 5-minutes has passed or an ignition has occurred.

6.2.5 Record the following information on the Autoignition Temperature Test Sheet (Figure 3):

a. The size of the fluid sample.
b. The average temperature displayed on the three temperature indicators, rounded off to the nearest whole number.
c. Whether or not an ignition occurred.

6.2.6 Flush the flask with clean compressed air.

6.2.7 Refill the syringe with the sample of the same size.

6.2.8 If ignition did not occur, proceed to step 6.3 (Increasing the Temperature to the Autoignition Point). If ignition occurred, skip to step 6.4 (Decreasing the Temperature to the Autoignition Point).

6.3 <u>Increasing the Temperature to the Autoignition Point:</u>

6.3.1 If ignition did not occur, increase the temperature setting on the temperature controllers 50°F.

6.3.2 Wait at least 15 minutes or until the temperature indicators read an average of the selected temperature, whichever is the longer period.

6.3.3 Repeat steps 6.2.3 through 6.2.7.

6.3.4 Continue testing the fluid at higher temperatures (that is, repeating steps 6.3.1, 6.3.2, and 6.3.3), until either ignition occurs or another temperature increase would put the temperature over 1000°F.

6.3.5 If the fluid does not ignite (even at 1000°F), record 1000°F on the test sheet and skip to step 6.5 (Second Test - 0.10 cc).

6.3.6 If ignition occurred, a range of 50°F has been determined for the autoignition temperature. Proceed with step 6.4 (Decreasing the Temperature to the Autoignition Point) to determine more precisely the non-ignition temperature for the 0.07 cc sample.

6.4 Decreasing the Temperature to the Autoignition Point:

6.4.1 If ignition occurred, decrease the temperature setting on the temperature controllers 5°F.

6.4.2 Wait at least 15 minutes or until the temperature indicators read an average of the selected temperature, whichever is the longer period.

6.4.3 Repeat steps 6.2.3 through 6.2.7.

6.4.4 Continue testing the fluid at lower temperatures (that is, repeating steps 6.4.1, 6.4.2, and 6.4.3), until a temperature is reached at which ignition does not occur.

6.4.5 When the point is reached at which ignition does not occur, record the average temperature displayed on the three temperature indicators on the Test Data Sheet rounded off to the nearest whole number. (This is the non-ignition temperature for the 0.07 cc sample). Proceed with 6.5 (Second Test - 0.10 cc).

6.5 Second Test (0.10 cc):

6.5.1 Starting at a temperature 50°F higher than the 0.07 cc non-ignition temperature determined previously, test a 0.10 cc sample of the fluid in accordance with the steps 6.2.3 through 6.2.8, and 6.3 or 6.4. If the non-ignition temperature of the 0.10 cc sample is higher than the 0.07 cc non-ignition temperature, record this temperature on the Test Data Sheet and proceed with Step 6.6 (Decreasing Sample Sizes); if it is lower than the 0.07 cc non-

ignition temperature, skip to step 6.7 (Increasing Sample Sizes).

6.6 Decreasing Sample Sizes:

6.6.1 If the non-ignition temperature of the 0.10 cc sample was higher than that for the 0.07 cc sample (step 6.5.1), retest the fluid as follows with increasingly smaller samples and higher temperatures to determine their effects on the autoignition temperature.

6.6.2 Starting at a temperature 50°F higher than the 0.07 cc non-ignition temperature determined previously, test a 0.05 cc sample of the fluid in accordance with steps 6.2.3 through 6.2.8, and 6.3 or 6.4. Record the 0.05 cc non-ignition temperature on the Test Data Sheet. If the non-ignition temperature of the 0.05 cc sample is higher than the 0.07 cc non-ignition temperature, skip to step 6.8 (Test Data Evaluation); if it is lower than 0.07 cc non-ignition temperature, proceed to the next smaller sample size.

6.6.3 Starting at a temperature 50°F higher than the 0.05 cc non-ignition temperature determined in step 6.6.2, test a 0.03 cc sample of the fluid in accordance with steps 6.2.3 through 6.2.8, and 6.3 or 6.4. Record the 0.03 cc non-ignition temperature on the Test Data Sheet and skip to step 6.8 (Test Data Evaluation).

6.7 Increasing Sample Sizes

6.7.1 If the non-ignition temperature of the 0.10 cc sample was lower than that for the 0.07 cc sample (step 6.5.1), retest the fluid as follows with increasing larger samples and higher temperatures to determine their effects on the autoignition temperature.

6.7.2 Starting at a temperature 50°F higher than the 0.10 cc non-ignition temperature determined in step 6.5.1, test a 0.12 cc sample of the fluid in accordance with steps 6.2.3 through 6.2.8 and 6.3 and 6.4. Record the 0.12 cc non-ignition temperature on the Test Data Sheet. If the non-ignition temperature of the 0.12 cc sample is higher than the 0.10 cc non-ignition temperature, skip to step 6.8 (Test Data Evaluation); if it is lower than 0.10 cc non-ignition temperature, proceed to the next larger sample size.

6.7.3 Starting at a temperature 50°F higher than the 0.12 cc non-ignition temperature determined in step 6.7.2, test a 0.15 cc sample of the fluid in accordance with steps 6.2.3 through 6.2.8 and 6.3 and 6.4. Record the 0.15 cc non-ignition temperature on the Test Data Sheet. If the non-ignition temperature of the 0.12 cc sample is higher than the 0.10 cc non-ignition temperature, skip to step 6.8 (Test Data Evaluation); if it is lower than 0.10 cc non-ignition temperature, proceed to the next larger sample size.

6.7.4 Starting at a temperature 50°F higher than the 0.15 cc non-ignition temperature determined in step 6.7.3, test a 0.18 cc sample of the fluid in accordance with steps 6.2.3 through 6.2.8, and 6.3 or 6.4. Record the 0.18 cc non-ignition temperature on the Test Data Sheet and proceed with step 6.8 (Test Data Evaluation).

6.8 Test Data Evaluation

6.8.1 Locate the lowest non-ignition temperature recorded on the Test Data Sheet. If the lowest non-ignition temperature is higher than 600°F, the sample has passed the autoignition temperature criteria as defined in 30 CFR, Subpart B, Section 35.20. If the lowest non-ignition temperature is lower than 600°F, the sample has failed. Record the “Pass” or “Fail” tests results on the Test Data Sheet.

7.0 Maintenance:

7.1 After test completion, the Erlenmeyer Flask must be cleaned and placed within the furnace.

8.0 Test Modifications:

8.1 Since all possible materials / products, compositions, physical properties, and applicable methods cannot be foreseen, MSHA reserves the right to modify the above test procedures.

9.0 Responsibility:

9.1 The Quality Assurance and Materials Testing Division (QA&MTD) is responsible for the maintenance and operation of the autoignition

temperature test.

10.0 Notofication:

10.1 The Quality Assurance and Materials Testing Division (QA&MTD) will notify all appropriate Approval and Certification Center personnel.

11.0 Distribution:

11.1 This document will be distributed to all appropriate Approval and Certification Center personnel.

12.0 Results:

12.1 Test results are summarized in MSHA's approval and audit documentation files of fire-resistant hydraulic fluids. Test results regarding accident and other investigations requiring autoignition temperature test data will be summarized where appropriate.

13.0 Review:

13.1 This document will be reviewed at least once every three years.

14.0 Authority:

14.1 30 CFR, Part 35, Subpart A & B

Autoignition Temperature Test Furnace

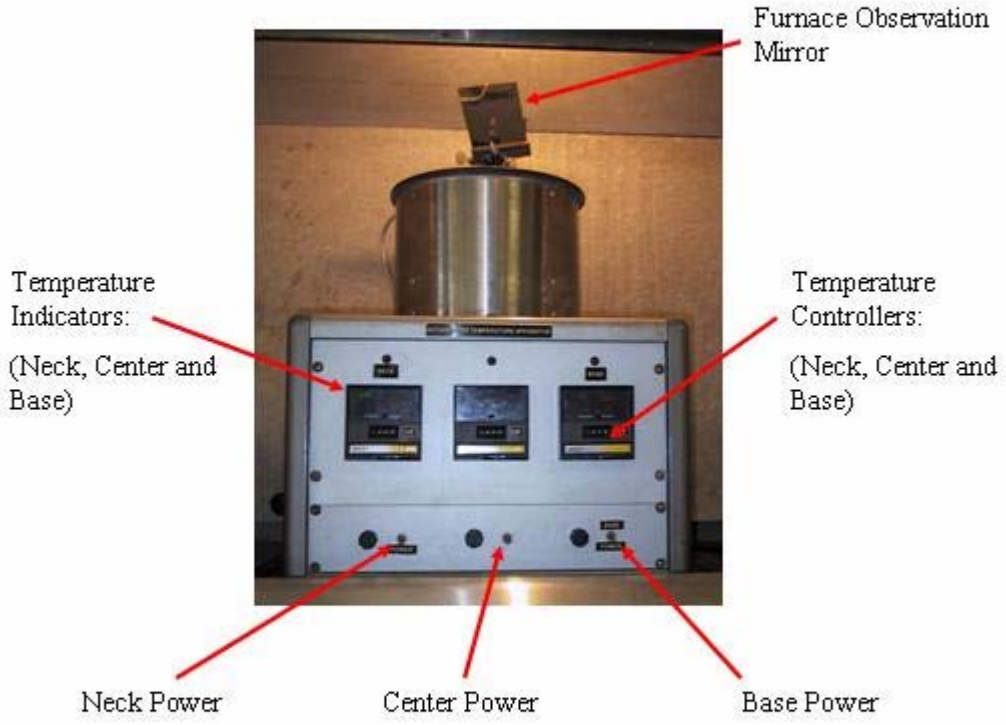

Figure 1, Page 10

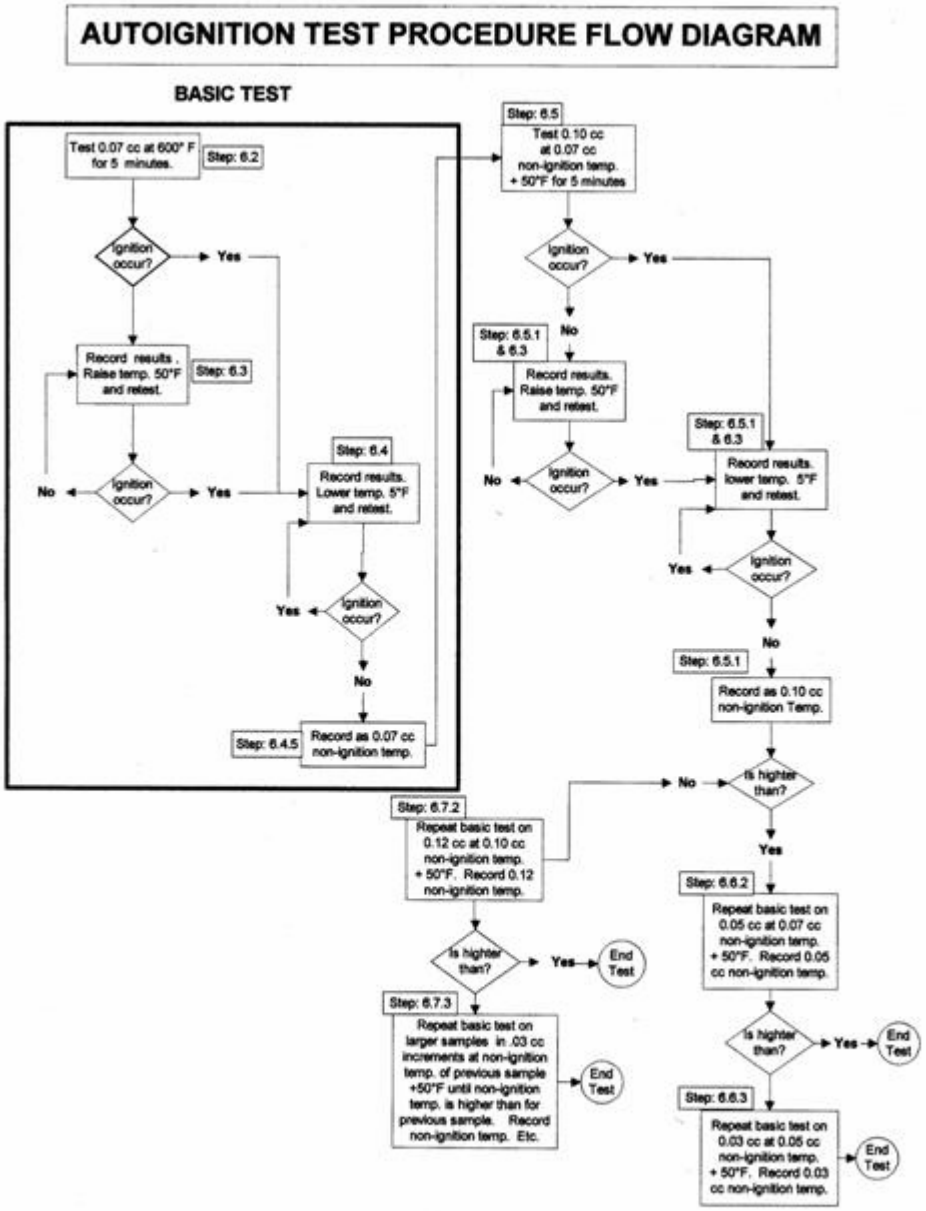

Figure 2, Page 11

AUTOIGNITION TEMPERATURE TEST DATA SHEET

PAR (or MSRS): _____ Data Sheet No. _____

Company: _____

Date: _____

Sheet No. _____ of _____ Investigator: _____

Test Results: _____

Reference: _____ Ambient Temperature: _____ at _____

Project: _____

Specimen _____

Sample Size [cc]	Flask Temp. [°F]	Ignition [yes/No]	Sample Size [cc]	Flask Temp. [°F]	Ignition [yes/No]	Sample Size [cc]	Flask Temp. [°F]	Ignition [yes/No]

AUTOIGNITION TEMPERATURE (°F)

0.07 cc	0.05 cc	0.12 cc
0.19 cc	0.03 cc	0.15 cc
		0.18 cc

Figure 3, Page 12

Flask Position Inside Furnace

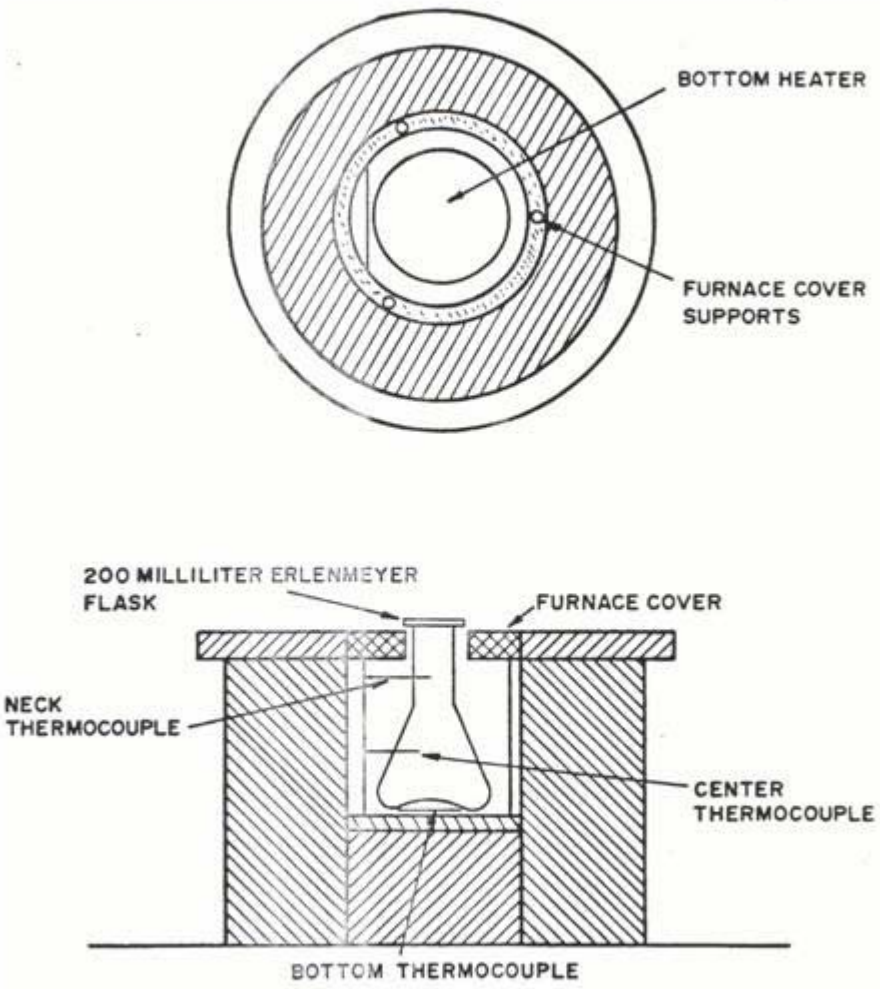

Figure 4, Page 13

Document Information Form

CDS No. _ASTP5002_____ (RCO Assigns)

____ Enter (Original)
__X__ Supersede CDS No. ASTP4014 (undated)
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Title: Standard Test Procedure for Conducting the Autoignition Temperature Test on Hydraulic Fluids Submitted for MSHA Approval: 30 CFR, Section 35.20

Category: _XX_ A (PAR related) ____ N (Not PAR related)

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List of 30 CFR References:

Part	Section(s)
35	All Subpart A & B

Key Words: Autoignition Temperature, Part 35, Fire-Resistant Hydraulic Fluid
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Concurrence:
Technical Review By: _____ Committee Representative

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Division Chief Concurrence (Initials)

	Yes	No
AED	___	___
ESD	___	___
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QA&MTD	___	___

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