An Assessment of Three Different Fire Resistance Tests for Hydraulic Fluids

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
National Engineering Laboratory
Center for Fire Research
Washington, DC 20234

October 1981
Final Report

Sponsored in part by:
U.S. Bureau of Mines
Pittsburgh, Pennsylvania 15213

and
Mine Safety and Health Administration
Philadelphia, West Virginia 26059
AN ASSESSMENT OF THREE DIFFERENT
FIRE RESISTANCE TESTS FOR HYDRAULIC
FLUIDS

Joseph J. Loftus

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
National Engineering Laboratory
Center for Fire Research
Washington, DC  20234

October 1981

Final Report

Sponsored in part by:
U.S. Bureau of Mines
Pittsburgh, Pennsylvania  15213

and

Mine Safety and Health Administration
Triadelphia, West Virginia  26059
TABLE OF CONTENTS

LIST OF TABLES ........................................ iv
Abstract ........................................... 1
1. INTRODUCTION ..................................... 1
2. FIRE TEST METHODS ................................ 2
   2.1 Autogenous Ignition Temperature Test ....... 2
   2.2 Temperature-Pressure Spray Ignition Test .... 2
   2.3 Effect of Evaporation on the Flammability of Hydraulic Fluids (Wick Test) ......... 3
3. TEST MATERIALS ................................... 3
4. TEST RESULTS ...................................... 4
   4.1 Autogenous Ignition Temperature Test ....... 4
   4.2 Temperature-Pressure Spray Ignition Test .... 5
   4.3 Effect of Evaporation on the Flammability of Hydraulic Fluids .......... 5
5. TEST OBSERVATIONS ................................ 6
   5.1 Autogenous Ignition Temperature Test ....... 6
   5.2 Temperature Pressure Spray Ignition Test .... 6
   5.3 Effect of Evaporation on the Flammability of Hydraulic Fluids .......... 7
6. RECOMMENDATIONS .................................. 8
   6.1 Autogenous Ignition Temperature Test ....... 8
   6.2 Temperature Pressure Spray Ignition Test .... 9
   6.3 Effect of Evaporation on the Flammability of Hydraulic Fluids .......... 10
7. CONCLUSIONS ...................................... 10
8. ACKNOWLEDGEMENTS ................................ 12
9. REFERENCES ....................................... 13
APPENDIX A. Autogeneous Ignition Temperature Test
   for Hydraulic Fluids ............................... 14
APPENDIX B. Temperature Pressure Spray Ignition Test
   for Flammability Resistance of Hydraulic Fluids .......................... 20
APPENDIX C. Test for the Effect of Evaporation on the
   Flammability of Hydraulic Fluids .......................... 38
LIST OF TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Autoignition Temperatures for Hydraulic Fluids</td>
<td>4</td>
</tr>
<tr>
<td>2.</td>
<td>Comparison of MSHA and NBS Wick Test Data</td>
<td>8</td>
</tr>
<tr>
<td>3.</td>
<td>Results of Spray Ignition Tests on Hydraulic Fluids Using MSHA Test Procedures</td>
<td>11</td>
</tr>
</tbody>
</table>
An Assessment of Three Different Fire Resistance Tests for Hydraulic Fluids

Joseph J. Loftus

Abstract

Three different fire resistance tests for hydraulic fluids were evaluated in tests on fourteen different hydraulic fluid materials. Special emphasis was placed on procedural details pertinent to the repeatability and reproducibility of the tests. Recommendations for changes in the test methods and procedures are made based on results in this evaluation study.

Key words: Autoignition temperature; coal mines; fire resistance tests; fire tests; flammability; hydraulic fluids; ignition.

1. INTRODUCTION

The Center for Fire Research at the National Bureau of Standards (NBS), at the request of the Mine Safety and Health Administration (MSHA) and the Bureau of Mines (BOM), made an evaluation or assessment of three different flammability tests used by MSHA for measuring the fire resistance of hydraulic fluids intended for use in underground coal mining operations. The test methods described in the Code of Federal Regulations Schedule 30, Part 35,
are: an Autogenous Ignition Temperature Test, a Temperature-Pressure Spray Ignition Test and a Test to Determine the Effect of Evaporation on the Flammability of Hydraulic Fluids.

This report summarizes the results of the evaluation studies and includes recommendations for changes or improvements in the three different test procedures. Each of these test methods are discussed briefly in section 2.

2. FIRE TEST METHODS

2.1 Autogenous Ignition Temperature Test

This test is used to determine the lowest temperature for ignition of a hydraulic fluid material. The method calls for injecting measured fluid volumes from a hypodermic syringe into a preheated erlenmeyer flask mounted in a heated tube furnace and observing the temperature at which the fine fluid spray ignites in the flask. Tests are made for a number of different temperatures until the minimum temperature and volume of fluid needed for ignition are determined for the hydraulic fluid material.

2.2 Temperature-Pressure Spray Ignition Test

In this test a hydraulic fluid, under a pressure of 1030 KPa (150 psi) and at a temperature of 65°C (150°F) is sprayed into each of three different ignition sources: a flaming trough (cotton waste wetted with kerosine and ignited), a propane torch flame and electric sparks. Observations for continuous flaming are made. Any fluid which shows continuous flaming for 6 seconds or more fails to meet the fire resistant requirements of the test.
2.3 Effect of Evaporation on Flammability of Hydraulic Fluids (Wick Test)

This test seeks to determine whether a hydraulic fluid on losing some of its components, e.g., water, glycol, etc., as a result of use in mining equipment, may become ignited when exposed to an open flame. In this test, original and oven conditioned fluid specimens [2 and 4 hours in an oven at 65°C (150°F)] are soaked into pipe cleaner wicks and the wicks are then cycled into a bunsen burner flame until the wick is ignited. A count is made of the number of times a wick cycles into the flame without igniting. If the count meets or exceeds an established minimum number of cycles to ignition, a fluid meets the wick test requirements.

3. TEST MATERIALS

A total of 14 different hydraulic fluids were selected for testing. Included were two water glycols, seven synthetics and five invert emulsions. The fluids were obtained from manufacturers who normally supply these materials to the underground coal mining industry and who report that the fluids are fire resistant under the Code of Federal Regulations, Schedule 30, Part 35, Flammability Regulations.

Invert emulsions are hydraulic fluids that consist of oil in a water emulsion in which the water content may vary from 40 to 95 percent.

Synthetics are fluids which contain organic esters, e.g., phosphates or synthesized hydrocarbons, while water glycols consist of a water-glycol solution with at least 35 percent water.
4. TEST RESULTS

4.1 The Autogenous Ignition Temperature Test

Table 1 lists (AIT) temperatures and minimum fluid volumes for the lowest ignition temperatures for the 14 different hydraulic fluids.

Table 1. Autoignition Temperatures for Hydraulic Fluids

<table>
<thead>
<tr>
<th>Fluid No.</th>
<th>Type</th>
<th>Autoignition Temperatures</th>
<th>Min. Volume</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Deg F</td>
<td>Deg C</td>
</tr>
<tr>
<td>1</td>
<td>Water glycol</td>
<td>884</td>
<td>440</td>
</tr>
<tr>
<td>2</td>
<td>Water glycol</td>
<td>770</td>
<td>410</td>
</tr>
<tr>
<td>3</td>
<td>Synthetic</td>
<td>761</td>
<td>405</td>
</tr>
<tr>
<td>4</td>
<td>Synthetic</td>
<td>1013</td>
<td>545</td>
</tr>
<tr>
<td>5</td>
<td>Invert Emulsion</td>
<td>743</td>
<td>395</td>
</tr>
<tr>
<td>6</td>
<td>Synthetic</td>
<td>1013</td>
<td>545</td>
</tr>
<tr>
<td>7</td>
<td>Invert Emulsion</td>
<td>716</td>
<td>380</td>
</tr>
<tr>
<td>8</td>
<td>Synthetic</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>9</td>
<td>Synthetic</td>
<td>932</td>
<td>500</td>
</tr>
<tr>
<td>10</td>
<td>Synthetic</td>
<td>1032</td>
<td>555</td>
</tr>
<tr>
<td>11</td>
<td>Invert Emulsion</td>
<td>702</td>
<td>375</td>
</tr>
<tr>
<td>12</td>
<td>Synthetic</td>
<td>1022</td>
<td>550</td>
</tr>
<tr>
<td>13</td>
<td>Invert Emulsion</td>
<td>752</td>
<td>400</td>
</tr>
<tr>
<td>14</td>
<td>Invert Emulsion</td>
<td>770</td>
<td>410</td>
</tr>
</tbody>
</table>

An examination of the above data shows that all of the test hydraulic fluids easily passed the minimum allowed AIT level established by this test at 315°C (600°F).
4.2 Temperature-Pressure Spray Ignition Test

Table 3 (see page 11) shows results for spray ignition tests on 14 different hydraulic fluids using the MSHA spray ignition testing procedures. An examination of this table shows that 13 of the 14 fluids passed the electric sparks test while 11 fluids failed every propane torch and flaming trough test. Fluids no. 1, 2 and 3 water glycol, water glycol and synthetic respectively were the only fluids found to pass all spray ignition tests.

4.3 Effect of Evaporation on the Flammability of Hydraulic Fluids

The MSHA wick test uses three different sets of test samples:

1. Original state - all 14 test fluids passed the wick test.

2. Two hour conditioned samples - all invert emulsion and synthetic fluids passed all tests. Water glycol fluids failed the test.

3. Four hour conditioned samples - results duplicate (2) above.

Oven conditioning had no effect on the wick test performance of invert emulsions and synthetic fluids simply because these fluids remained unchanged (i.e., showed negligible weight loss) as a result of oven conditioning, and the fluid samples performed in the same manner as original state fluids.
Oven conditioning had a considerable impact on the flammability of water glycol fluids. These fluids lost 30 to 40 percent of original sample weight and the residual fluid materials were easily ignited in the wick tests.

5. TEST OBSERVATIONS

5.1 Autogenous Ignition Temperature Test

- The AIT test uses the same test apparatus and procedures as ASTM Standard D-2155-66 Autoignition Temperature Test for Liquid Petroleum Products.

- The test was found to require an inordinate amount of time to conduct because attainment of uniform temperatures in the test flask required numerous adjustments to three manually controlled heaters in the test apparatus.

- Much of the testing protocol used in the (AIT) test appears to be unnecessary since the objective of the test is simply to determine whether a hydraulic fluid ignites at 315°C (600°F) or below.

- Repeatability of the AIT test was found excellent.

5.2 Temperature-Pressure Spray Ignition Test

- The electric sparks ignition source did not provide a severe test for the hydraulic fluid types tested here.
• It appears unnecessary for the spray ignition test to require two different open flame ignition sources (i.e., propane torch and flaming trough) since each ignition source provided for identical test results.

• The MSHA Spray Ignition Test conditions of 65°C temperature, pressure of 1030 Kpa, spray nozzle orifice diameter of 0.63 mm and spray nozzle to ignition source distance of 45 cm were found to be appropriate for conducting spray ignition tests on hydraulic fluids.

Reproducibility of the test results; e.g., comparison of the Center's test results with MSHA showed poor agreement. The Center showed 11 failures for 14 fluids. MSHA approved all 14 fluids for use in coal mines.

5.3 Effect of Evaporation on the Flammability of Hydraulic Fluids

• Repeatability of the wick test was found poor in the NBS laboratory.

• Reproducibility of the wick test between laboratories was also found poor as evidenced by a comparison of MSHA and NBS data for 6 of the hydraulic fluids shown in table 2.
Table 2. Comparison of MSHA and NBS Wick Test Data

<table>
<thead>
<tr>
<th>Fluid No.</th>
<th>Oven Conditioning</th>
<th>MSHA*</th>
<th>NBS</th>
<th>MSHA*</th>
<th>NBS</th>
<th>MSHA*</th>
<th>NBS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0 hrs.</td>
<td>2 hrs.</td>
<td>4 hrs.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>13</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>9</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>12</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*MSHA data obtained by the Center for Fire Research in a review of test results recorded over a period of time.

6. RECOMMENDATIONS

Recommendations for changes in each of the MSHA fire test methods based on these evaluation test results are made here and these are included in revised and rewritten versions of the MSHA testing procedures in appendices A, B and C of this report.) Highlights of these changes are listed in the following sections.

6.1 Autogenous Ignition Temperature Test

(For new procedure see appendix A.)

- Furnace air temperature - The furnace air temperature should be maintained at one test temperature only: 315°C (600°F).
• All tests should be made at this temperature since the test criteria is satisfied when a hydraulic fluid does not ignite at this temperature.

6.2 Temperature-Pressure Spray Ignition Test

(For new procedure see appendix B.)

• Spray ignition test nozzles should be calibrated and those having tolerances of ± 10 percent of the flow rate specified for the nozzles by the spray test method should be used for test.

• System integrity - Before beginning tests a 0-200 psi pressure gage should be substituted for the spray nozzle in the test apparatus and the system pressurized (without fluid) to 150 ± 5 psi. All tubing, hoses and valve connections should be inspected for leaks and all necessary adjustments should be made to ensure that the spraying system will maintain its pressurized integrity for testing.

• Ignition sources - The spacing between electrodes in the Electric Spark Test should be 12.5 mm (0.5 in).

    The propane torch nozzle orifice diameter should measure 2.5 ± 0.25 mm.

    The flaming trough should be fueled with standard cotton cheesecloth material (federal supply no. 8305-00-205-3496).

    Fifty grams of material should be wetted with 50 cc of kerosine and ignited for test.

• Heat losses - If metal tubing or other material used to transport fuel to the spray nozzle is subject to heat losses it should be wrapped in heating tape or other material to ensure that the fluid exits the spray nozzle within 3°C of the specified test temperature.
6.3 Effect of Evaporation on the Flammability of Hydraulic Fluids "Wick Test"

(For new procedure see appendix C.)

- The bunsen burner orifice should be specified. Recommend 1.25-1.60 mm (.06-.08 in) diameter.
- Methane gas (96 ± 1 percent methane) should be used.
- Gas flow to the burner should be monitored with a flowmeter having a measurement range of 1130-1880 cc/min (.04-.07 cu ft/min).
- Gas flow for test should be 1440 cc/min.
- Wicks should be located 7.5 cm (3 in) from the top of the burner.
- Wicks (pipe cleaner stems) should have a wire thickness of 0.75-1.0 mm (.03-.04 in) and have a combustible content of .015-.025 g/in of stem length.

7. CONCLUSIONS

- The Autogenous Ignition Temperature Test produced repeatable test results in the laboratory.
- Testing at one furnace temperature 315°C (600°F) appears to be adequate for obtaining ignition temperature information by this test.
Table 3. Results of spray ignition tests on hydraulic fluids using MSHA test procedures

<table>
<thead>
<tr>
<th>No.</th>
<th>Fluid Type</th>
<th>Ignition Source</th>
<th>Torch (Benzomatic)</th>
<th>Electric Sparks</th>
<th>Flaming Cotton</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Water Glycol</td>
<td></td>
<td>N</td>
<td>N</td>
<td>N</td>
</tr>
<tr>
<td>2</td>
<td>Water Glycol</td>
<td></td>
<td>N</td>
<td>N</td>
<td>N</td>
</tr>
<tr>
<td>3</td>
<td>Synthetic</td>
<td></td>
<td>N</td>
<td>N</td>
<td>N</td>
</tr>
<tr>
<td>4</td>
<td>Synthetic</td>
<td></td>
<td>I</td>
<td>N</td>
<td>I</td>
</tr>
<tr>
<td>5</td>
<td>Invert Emulsion</td>
<td></td>
<td>I</td>
<td>N</td>
<td>I</td>
</tr>
<tr>
<td>6</td>
<td>Synthetic</td>
<td></td>
<td>I</td>
<td>N</td>
<td>I</td>
</tr>
<tr>
<td>7</td>
<td>Invert Emulsion</td>
<td></td>
<td>I</td>
<td>N</td>
<td>I</td>
</tr>
<tr>
<td>8</td>
<td>Synthetic</td>
<td></td>
<td>I</td>
<td>N</td>
<td>I</td>
</tr>
<tr>
<td>9</td>
<td>Synthetic</td>
<td></td>
<td>I</td>
<td>N</td>
<td>I</td>
</tr>
<tr>
<td>10</td>
<td>Synthetic</td>
<td></td>
<td>I</td>
<td>I</td>
<td>I</td>
</tr>
<tr>
<td>11</td>
<td>Invert Emulsion</td>
<td></td>
<td>I</td>
<td>N</td>
<td>I</td>
</tr>
<tr>
<td>12</td>
<td>Synthetic</td>
<td></td>
<td>I</td>
<td>N</td>
<td>I</td>
</tr>
<tr>
<td>13</td>
<td>Invert Emulsion</td>
<td></td>
<td>I</td>
<td>N</td>
<td>I</td>
</tr>
<tr>
<td>14</td>
<td>Invert Emulsion</td>
<td></td>
<td>I</td>
<td>N</td>
<td>I</td>
</tr>
</tbody>
</table>

N - Nonignition - Pass
I - Ignition - Failure
Temperature-Pressure Spray Ignition Test

• Reproducibility of the MSHA tests at NBS was poor.

• The propane torch test might easily be dropped from the MSHA test procedures without reducing the severity of the spray ignition test, since the flaming trough test produces identical test results.

• From a safety viewpoint the flaming trough test is preferred over the propane torch ignition source because it is easier to control, reproduce, and is safer to operate.

• The electric spark test does not provide a severe ignition source for hydraulic fluid spray tests.

The Effect of Evaporation on the Flammability of Hydraulic Fluids

• This wick test showed poor repeatability in laboratory tests and poor reproducibility between MSHA and NBS test results.

• The test is severe on water glycols but had no effect on fluids which did not lose weight on oven conditioning; synthetics and invert emulsions.

8. ACKNOWLEDGEMENTS

Thanks are due to the following co-op Student Assistants whose considerable contributions helped to complete these evaluation studies: R. Torres, J. Simenauer, E. Ruiz, A. Maldonado, N. Juarez, and P. Allen.
9. REFERENCES

Flammability measurements on fourteen different hydraulic fluids
using a temperature--pressure spray ignition test, Nat. Bur. Stand.
(U.S.), NBSIR 81-2247 (March 1981).


Temperature-Pressure Spray Ignition Test for Hydraulic Fluids, Nat.
APPENDIX A

Autogenous Ignition Temperature Test for Hydraulic Fluids

.1 Scope and application.

The Code of Federal Regulations, Title 30, Part 35, lists three different tests for measuring the flammability resistance of hydraulic fluids.

- Temperature-pressure spray ignition test;
- Autogenous ignition temperature test;
- Test to determine the effect of evaporation on flammability of hydraulic fluids.

Candidate hydraulic fluids which meet all the flammability requirements of these tests are considered acceptable for use in all underground coal mining operations.

Under Title 30, flammability resistant hydraulic fluids must be used in the following mining operations:

(a) Where electrically powered equipment is used in attended operations but where no fire suppression equipment is readily available.

(b) Where electrically powered equipment is used in unattended operations.
The following, provides procedures for test no. 2, "The Autogenous Ignition Temperature Test" for hydraulic fluids.

.2 Definitions.

(a) **Attended operations.** Under Title 30, Chapter 1, Mining Enforcement Safety Admin Sec 75.1107-1 (A-D), attended operations means (1) any machine or device which is regularly operated by a miner and (2) any machine or device which is in direct line of sight of a job site and within 500 feet of a miner working the job site.

(b) **Unattended operations** means all operations not covered by the above definition for attended operations.

(c) **Fire suppression equipment** means any equipment capable of discharging a fire extinguishing medium such as dry powder or other type of material in sufficient quantity on a fire to bring it under control and to extinguish it.

(d) **Hydraulic fluid** means any fluid which is used under pressurized conditions to operate mining machines or devices.

(e) **Fire resistant hydraulic fluid** means any (d) fluid which meets all requirements of the three tests cited in .1 Scope and application.
.3 Summary of test.

Small sample volumes of hydraulic fluid (<1 cc), are sprayed or injected by a hypodermic syringe into an electric heated tube furnace operated and maintained at a temperature of 351°C (600°F) to determine whether a fluid ignites or resists ignition at this specified temperature.

(a) Test criteria. An hydraulic fluid meets the requirements of the "Autogenous Ignition Temperature Test" if it does not ignite at the test temperature of 315°C (600°F).

.4 Test procedure.

(a) Apparatus. The tube furnace used for test, consists of a vertical cylinder [having ID x H dimensions of 12.5 x 12.5 cm (5 x 5 in)] which is surrounded by electric heaters; i.e., sides, base, and top (removable). The cylinder is insulated and contained in a retaining shell. (Note: One furnace type found suitable for test is that described in ASTM Standard Method of Test for Autoignition Temperature of Liquid Petroleum Products, ASTM D 2155-66.)

(b) Test flasks (which are supported within the refractory cylinder of the tube furnace and which are used to receive samples injected into the furnace) shall be commercial 200 cu cm Erlenmeyer borosilicate glass containers.
(c) **Thermocouples.** Three thermocouples (chromel alumel 28-gauge, or equivalent) shall be used to measure flask temperature. Two of these shall be mounted so as to contact the walls of the flask at 2.5-5.0 cm (1 and 2 in) locations from the neck of the flask and one shall be located under the base of the flask at its center.

(d) **Hypodermic syringe.** A 0.25 or 1 cc capacity hypodermic syringe equipped with a no. 18 stainless steel needle and calibrated in units of 0.01 cu cm shall be used to inject fluid samples into the test flask.

(e) **Timer.** An electric timer or stopwatch capable of at least 0.2-second interval measurements shall be used to determine times to ignition for tests.

.5 Testing.

(a) Adjust the temperature of the furnace so that the temperatures at the top, center, and bottom of the furnace flask are within $\pm 1^\circ C$ ($2^\circ F$) of the initial test temperature of 315$^\circ C$ ($600^\circ F$).

(b) Ensure that the furnace flask is at equilibrium test temperature for at least 5 minutes before starting a test.

(c) Position a mirror at an appropriate location above the furnace so that the inside of the furnace flask can be clearly observed.
(d) Fill the hypodermic syringe to a volume of 0.07 cu cm with test fluid.

(e) Darken the testing room area so that ignitions may be more easily observed.

(f) Place the hypodermic needle into the furnace top opening and immediately discharge the test sample into the furnace flask and start the timer.

(g) Observe the inside of the flask to determine if an ignition occurs. The maximum time for observation is 5 minutes. (Note: Dry air shall be used to flush the furnace flask after each test. Should the flask become visibly coated with residue at any time it shall be replaced with a new flask.)

(h) A new test shall be started, only after the furnace flask has reached test temperature and remains at this equilibrium temperature for at least 5 minutes before test.

.6 Test observations.

(a) If the 0.07 cu cm sample of test fluid does not ignite after 5 minutes of test at the test temperature of 315°C (600°F), other fluid volumes, e.g., 0.03, 0.05, 0.13, and 0.15 cu cm, shall also be subjected to the same test at the same temperature.
(b) If none of these fluid volumes ignite at this test temperature, the test fluid shall be considered to have met the requirements for ignition resistance as promulgated by the Autogenous Ignition Temperature Test.

(c) If any of the fluid volumes do ignite at the test temperature of 315°C (600°F), the test fluid fails to meet the requirements of test.

(d) If the 0.07 cu cm sample of test fluid produces an ignition at the test temperature of 315°C (600°F), the candidate hydraulic fluid fails to meet the requirements of the ignition test and no further testing shall be required of the fluid.

.7 Report.

A report of test results shall include:

(a) Individual test results for each fluid volume tested, i.e., ignition or nonignition.

(b) For samples which ignited in the test, report the time at which ignition occurred.

(c) A statement on whether a candidate hydraulic fluid meets the requirements of the "Autogenous Ignition Temperature Test."
APPENDIX B

Temperature-Pressure Spray Ignition Test for Flammability Resistance of Hydraulic Fluids

1. Scope and application.

The Code of Federal Regulations, Title 30, Part 35, lists three different tests for measuring the flammability resistance of hydraulic fluids.

- Temperature-pressure spray ignition test.
- Autogenous ignition temperature test.
- Test to determine the effect of evaporation on flammability of hydraulic fluids.

Candidate hydraulic fluids which meet all of the flammability requirements of these tests are considered acceptable for use in all underground coal mining operations.

Under Title 30, flammability resistant hydraulic fluids must be used in the following mining operations:

(a) Where electrically powered equipment is used in attended operations but where no fire suppression equipment is readily available.
(b) Where electrically powered equipment is used in unattended operations.

The following, provides procedures for test no. 1, "Temperature-Pressure Spray Ignition Test," for measuring the flammability resistance of hydraulic fluids.

.2 Definitions.

(a) Attended operations. Under Title 30, Chapter 1, Mining Enforcement Safety Admin Sec 75.1107-1 (A-D), attended operations means--(1) any machine or device which is regularly operated by a miner and (2) any machine or device which is in direct line of sight of a job site and within 500 feet of a miner working the job site.

(b) Unattended operations means all operations not covered by the above definition for attended operations.

(c) Fire suppression equipment means any equipment capable of discharging a fire extinguishing medium such as dry powder or other type of material in sufficient quantity on a fire to bring it under control and to extinguish it.

(d) Hydraulic fluid means any fluid which is used under pressurized conditions to operate mining machines or devices.
(e) **Fire resistant hydraulic fluid** means any (d) fluid which meets all requirements of the three tests cited in .1 Scope and application.

.3 Summary of test.

Candidate hydraulic fluids are sprayed under specified temperature and pressure conditions, i.e., $65 \pm 3^\circ C \ (150 \pm 5^\circ F)$ and $150 \pm 5$ psi, into each of three different ignition sources: (1) torch flame, (2) electric sparks, and (3) flaming cotton.

The ignition sources are located at each of three different distances from the spray nozzle, i.e., 45, 60, and 90 cm (18, 24, and 36 in). Determinations of flammability resistance are made, based on a fluid's ability to resist ignition as defined below.

(a) **Test criteria.** A candidate hydraulic fluid resists ignition and meets the flammability requirements of the spray test if none of the ignition sources, at any test location ignites the hydraulic fluid and causes a continuous flaming condition for 6 seconds or more when fluid is sprayed across it.
.4 Test procedure.

(a) **Apparatus** (the spray ignition test apparatus is shown in figure 1). (1) **Test chamber or enclosure.** Minimum dimensions for the test chamber shall be 90 cm wide \( \times \) 120 cm deep \( \times \) 90 cm high (3 x 4 x 3 ft) (see figure 2). The chamber shall be equipped with an exhaust system capable of removing fumes and smoke produced by testing.

(2) **Spray nozzle.** An atomizing round spray nozzle having a discharge orifice of 0.025 \( \pm \) 0.003 in diameter, capable of discharging 3.20 \( \pm \) 0.30 gallons of water per hour (GPH) with a spray angle of 90 degrees at a pressure of 100 \( \pm \) 5 psi shall be used for test (one suitable nozzle is Binks model F-12-25 or M-12-25). The nozzle shall be located or positioned at least 30 cm (12 in) from the base or floor of the test enclosure. Note: If a cabinet is used as the enclosure, the nozzle may be conveniently located or mounted on the sidewall of the cabinet.

(3) **Spray nozzle calibration.** Each spray nozzle used for test shall be calibrated for rate of fluid discharge. A preweighed plastic bag of approximately 1 liter capacity shall be attached to the discharge end of the spray nozzle and water at nominal room temperature of 18-26°C (65-80°F) shall be discharged from the nozzle into the bag under 100 \( \pm \) 5 psi for 60 \( \pm \) 0.05 sec. The weight or volume of water (specific gravity of water = 1 g per cu cm) shall be determined by difference (weight of bag + water minus weight of bag) and this
FIGURE 1
SPRAY IGNITION TEST APPARATUS
FIGURE 2.
SPRAY IGNITION TEST CHAMBER
(6) **Thermocouples.** Two shielded thermocouples (chromel-alumel or equivalent) shall be used for fluid temperature measurement. One thermocouple shall be located in the test fluid in the cylinder or tank and the other in the tubing line just before the spray nozzle.

(7) **Propellant medium.** Test fluid shall be discharged through the spray nozzle at 150 ± 5 psi pressure by any means capable of developing and maintaining that pressure. Two methods found suitable for providing adequate discharge pressure are: (i) a circulating pumping system and (ii) nitrogen, in commercially pressurized cylinders.

The spray test setup shall be equipped with the customary regulators valves, tubing, and connectors and shall have the capability of applying constant pressure over a wide range, e.g., 100-1000 psi. Note: In the interest of safety all nonmetallic tubing and hoses used in the setup shall have a safety factor rating of at least 2 for temperature and 10 for pressure, i.e., above operating test conditions.

(8) **Timer.** An electric timer or stopwatch capable of at least 0.2-second interval measurement shall be used to determine the duration of continuous flaming exhibited by a test fluid.

(9) **Ignition devices.** Three ignition devices shall be used to provide three different ignition sources for test.
(i) **Propane torch flame.** This ignition source consists of a propane gas cylinder (one size found useful is a 14.1 oz by weight or 1 pt. 10.7 fluid oz cylinder (bernzomatic or equivalent). The cylinder shall be fitted with a burner head with an ID of $12.5 \pm 1.3$ mm ($0.5 \pm 0.05$ in) and an orifice of $2.5 \pm 0.25$ mm ($0.1 \pm 0.01$ in). The torch flame used for test shall be 10-15 cm (4-6 in) in length.

(ii) **Electric sparks.** Electric sparks shall be provided for test by a 12,000 volt transformer (Donegan type or equivalent) which is allowed to discharge across a 0.5 in (12.5 mm) air gap or space separating two horizontally mounted copper electrodes (straight pencil type soldering iron tips) measuring $25 \pm 2.5$ mm ($1.0 \pm 0.1$ in) in length and having a thickness of $3 \pm 0.3$ mm ($0.12 \pm 0.01$ in). (See figure 3 for illustration.)

(iii) **Flaming cotton.** The flaming cotton ignition source consists of 50 ± 1 gram of "cloth cotton cheesecloth (Type II, Class 2, 36 in) Fed. Supply No. 8305-00-205-3496" which is layered or folded into a sample measuring 45 cm long x 5 cm wide (18 x 2 in); i.e., to fit inside a metal trough container (see figure 4) having similar 1 x w dimensions and measuring 5 cm (2 in) in depth. For test the entire exposed gauze surface is wetted with 50 cu cm of kerosene streaming from a 50 cu cm capacity glass pipette.

When ignited the ignition source shall provide an 45 cm (18 in) long flame front with flame heights ranging from 10-15 cm (4-6 in).
FIGURE 3.
ELECTRIC SPARKING DEVICE
Note: If after continuous testing the ignition source fails to develop the desired flame height, inspect the ignition source and determine whether additional kerosene is required or if the entire cotton cheesecloth sample needs to be replaced.

(iv) Ancillary equipment includes, a pressure gauge (0-200 psi) graduated in at least 5 psi steps, a linear scale, laboratory hot plate, glass thermometer (0-100 °C), glass pipette (50 cc capacity), beakers (1 liter capacity), a wide mouth funnel, and bench stand with clamps and kerosene.

(b) Testing. (1) Before beginning tests the spraying system shall be checked for integrity and pressure leaks. A pressure gauge (0-200 psi) shall be installed in the system in place of the spray nozzle and the system shall be pressurized (without fluid) to $150 \pm 5$ psi. All tubing, hoses, and valve connections shall be inspected for leaks and all necessary adjustments shall be made to ensure that the spraying system shall be capable of maintaining its pressurized integrity throughout a testing series.

(2) The system shall then be depressurized and the pressure gauge replaced in the system with the spray test nozzle.

(3) Test fluid shall be poured into the sample cylinder, tank, or other type of vessel used for test. Note: The quantity of fluid used for test may vary with the size or capacity of the vessel. If the entry port to the vessel is small or narrow and the test hydraulic
fluid is viscous, it may be necessary to heat the test fluid to an approximate test temperature of 65°C (150°F) before attempting to add it to the vessel. Heating the fluid in beakers on a hot plate to the approximate temperature should help reduce the time required to fill the vessel.

(4) After filling, the fluid in the vessel shall be heated to the test temperature of 65 ± 3°C (150 ± 5°F) and this temperature shall be maintained for all tests.

(5) All necessary tubing and hose connections shall be made and the system shall be pressurized to the test pressure of 150 ± 5 psi.

(6) Open the spray nozzle and discharge fluid through the system for 5-15 seconds. Note: This is done to ensure that the tubing leading from the vessel to the spray nozzle location is filled with fluid which can then be heated (in the tubing) to the testing temperature.

(c) The propane torch flame test. (1) The propane fuel cylinder used for test shall be supported on a bench stand (with the aid of clamps) and the stand shall be positioned in the test chamber with the burner tip of the cylinder set to produce a horizontal flame that will be perpendicular to the path of a spray pattern.
(2) Tests shall be made with the burner tip located at three different distances downstream from the spray nozzle: i.e., 18, 24, and 36 in (45, 60, and 90 cm).

(3) Ignite and adjust the gas flame to produce a flame length of 4-6 in (10-15 cm).

(4) With the burner tip positioned at the 18 in (45 cm) test location, open the spray nozzle and start the timer.

(5) Cycle the torch flame into and out of the spray pattern at a rate of approximately 15-20 cycles per min and observe whether the fluid spray resists ignition (for 1 minute of spray time) or ignites as evidenced by flaming for six continuous seconds or more. Note: No spray test is run longer than 1 minute. In cases where an ignition is observed the test shall be terminated immediately by stopping the spray and timer.

(6) Record results for the torch test at the 18 in (45 cm) test location.

(7) Reposition the torch burner tip at the 24 in (60 cm) and then the 36 in (90 cm) test locations downstream from the spray nozzle tip and repeat the same spray test (as described above) at each of these test locations.
(8) Record results for the torch tests at the 24 and 36 in (60 and 90 cm) test locations.

(9) Report results for all tests made at each test location in the propane torch flame tests.

(d) Electric spark test. (1) The electric sparking device shall be positioned in the test cabinet, perpendicular to the spray pattern and with its electrodes the same distance from the floor of the cabinet as the tip of the spray nozzle.

(2) Spark tests shall be made with the electrodes located at distances of 18, 24, and 36 in (45, 60, and 90 cm) downstream from the spray nozzle.

(3) Turn on the sparking device and ensure that it is working properly.

(4) Position the electrodes at the 18 in (45 cm) test location. Open the spray nozzle and start the timer.

(5) Observe whether the fluid spray is ignited by the sparks (i.e., flames continuously for 6 seconds or more) or resists ignition for 1 minute of spray. Note: No spray test shall be run longer than 1 minute. In cases where an ignition is observed, the test shall be terminated immediately, i.e., by stopping the sparking device, spray, and timer.
(6) Record results for the electric spark test at the 18 in (45 cm) test location.

(7) Reposition the sparking device with the electrodes located at the 24 in (60 cm) and then the 36 in (90 cm) test locations downstream from the spray nozzle and repeat the same spray test (as described above) at each of these test locations.

(8) Record the results for the electric spark tests at the 24 in (60 cm) and 36 in (90 cm) test locations.

(9) Report results for all tests made at each test location in the electric spark test.

(e) **Flaming cotton test.** (1) The metal trough containing the prepared ignition source (kerosine soaked cotton cheesecloth) shall be positioned in the test cabinet or enclosure, perpendicular to the fluid spray pattern. In relation to the spray nozzle the top edge of the trough shall be located 3.0 ± 0.1 in (7.5 ± 0.25 cm) below the tip of the spray nozzle.

(2) Tests shall be made with the trough located at distances of 18, 24, and 36 in (45, 60, and 90 cm) downstream from the nozzle.

(3) With the trough positioned at the 18 in (45 cm) test location, ignite the cotton and ensure that 4-6 in (10-15 cm) high flames are generated along the full length of the trough.
(4) Open the spray nozzle and start the timer.

(5) Observe whether the fluid spray ignites (as evidenced by continuous flaming for 6 seconds or more) or resists ignition for 1 minute of spray time. Note: No spray test shall be run longer than 1 minute. In cases where an ignition is observed the test shall be terminated immediately by stopping the spray and extinguishing the ignition source by closing the metal cover (attached to the side of the trough) over the flames.

(6) Record the results for the flaming cotton test at the 45 cm (18 in) test location.

(7) Reposition the trough at 60 and then 90 cm (24 and 36 in) distances downstream from the spray nozzle and repeat the spray test (as described above) at each test location.

(8) Record results for the flaming cotton tests at the 60 and 90 cm test locations.

(9) Report results for all tests made at each test location in the flaming cotton test.

(f) Acceptance. (1) Candidate hydraulic fluids meet the requirements of .3(a) Test criteria if they produce nonignition
results for all three ignition sources (i.e., propane torch flame, electric sparks, and flaming cotton) at all test locations.

(2) Report whether the candidate hydraulic fluid meets the requirements of the Temperature—Pressure Spray Ignition Test.
Test for the Effect of Evaporation on the Flammability of Hydraulic Fluids

.1 Scope and application.

The Code of Federal Regulations, Title 30, Part 35, lists three different tests for measuring the flammability resistance of hydraulic fluids.

- Temperature—pressure spray ignition test;
- Autogenous ignition temperature test;
- Test to determine the effect of evaporation on flammability of hydraulic fluids.

Candidate hydraulic fluids which meet all of the flammability requirements of these tests are considered acceptable for use in all underground coal mining operations.

Under Title 30, flammability resistant hydraulic fluids must be used in the following mining operations:

(a) Where electrically powered equipment is used in attended operations but where no fire suppression equipment is readily available.
(b) Where electrically powered equipment is used in unattended operations.

The following, provides procedures for test no. 3, "Test for the Effect of Evaporation on the Flammability of Hydraulic Fluids."

.2 Definitions.

(a) **Attended operations.** Under Title 30, Chapter 1, Mining Enforcement Safety Admin Sec 75.1107-1 (A-D), **attended operations** means--(1) any machine or device which is regularly operated by a miner and (2) any machine or device which is in direct line of sight of a job site and within 500 feet of a miner working the job site.

(b) **Unattended operations** means all operations not covered by the above definition for attended operations.

(c) **Fire suppression equipment** means any equipment capable of discharging a fire extinguishing medium such as dry powder or other type of material in sufficient quantity on a fire to bring it under control and to extinguish it.

(d) **Hydraulic fluid** means any fluid which is used under pressurized conditions to operate mining machines or devices.
(e) **Fire resistant hydraulic fluid** means any (d) fluid which meets all requirements of the three tests cited in .1 Scope and application.

.3 Summary of test.

(a) The test uses a cycling device, a bunsen burner (methane gas) flame ignition source and fluid soaked wicks for measurement of the effect of evaporation on the flammability resistance of hydraulic fluids.

In the test, samples are cycled (at a prescribed rate) into the burner flame until a sustaining flame is observed on the wick. The number of cycles required to ignition are counted and fluids which resist ignition for the number of cycles prescribed in .3(b) Test criteria, meet the flammability requirements of the test.

(b) **Test criteria.** A candidate hydraulic fluid meets the flammability requirements of the "Wick Test," if the material meets or exceeds the minimum (5 set) test average for each test condition. (See table 4.)
Table 4. Wick Test Criteria

<table>
<thead>
<tr>
<th>Sample</th>
<th>Minimum No. of Cycles (5 Test Ave.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As received fluid</td>
<td>24</td>
</tr>
<tr>
<td>2-Hour oven conditioned fluid*</td>
<td>18</td>
</tr>
<tr>
<td>4-Hour oven conditioned fluid*</td>
<td>12</td>
</tr>
</tbody>
</table>

* Oven evaporation temperature $65 \pm 1^\circ C$ ($150 \pm 2^\circ F$).

.3 Test procedure.

(a) **Apparatus.** (1) The cycling device used for Wick Test shall have the capability of cycling samples in a horizontal plane (through a 90 degree arc) into a bunsen flame ignition source at the rate of $25 \pm 2$ cycles per minute. Note: An automobile windshield wiper mechanism or other device capable of producing the specified cycling rate may be used for test.

(2) **Ignition source.** A bunsen burner with an orifice of $1.25$-$1.60$ mm ($0.06$-$0.08$ in) and with ID x H dimensions of $1.3$ x $16.5$ cm ($0.5$ x $6.5$ in) fired with methane gas ($96.1 \pm 1$ percent methane) shall be used as the ignition source for test.

(3) **Flowmeter.** Gas flow to the bunsen burner shall be monitored by a flowmeter calibrated for methane gas delivery over a range of $1133$-$1882$ cc per min ($0.04$-$0.07$ cu ft per min) at STP.
(4) **Oven.** A non-air-circulating gravity convection oven operated and maintained at a temperature of 65 ± 1°C (150 ± 2°F) shall be used for test.

(5) **Wicks.** Pipe cleaner stems (one type found suitable—Dill U.S. Tobacco Company) having a base wire thickness of 0.75-1.0 mm (0.03-0.04 in) and a combustible content of 0.006-0.01 g per cm of stem length shall be used for test. Note: The combustible content is determined by weighing a full length pipe cleaner (=15 cm) before and after burning away the tufting material on the cleaner. The weight difference divided by stem length equals weight loss per cm of stem.

(6) **Petri dishes.** Standard laboratory heat resistant glass petri dishes 13 mm (0.5 in) deep and 88 mm (3.5 in) in diameter fitted with matching lid covers shall be used to contain fluid samples for oven evaporation and testing. Note: The lids shall be used to cover the dishes immediately after their removal from the oven.

(7) **Ancillary equipment.** An electric timer or stopwatch capable of at least 0.2 sec interval measurement shall be used for timing the cycling device. Tweezers shall be used for handling all wick samples.

(b) **Sample preparation.** (1) Prepare a total of 15 wicks, each 7.5 cm (3 in) long, for tests on each candidate hydraulic fluid.
(2) Pour 30 cc of test fluid into each of three different petri dishes. Set one dish aside for tests on the fluid in its as received condition and place the other two dishes (uncovered) into the oven [operated and maintained at 65 ± 1°C (150 ± 2°F)].

(3) After 2 hours, remove one of the dishes from the oven and cover it immediately with its matching lid and set the dish aside to cool to room temperature 17-26°C (65-80°F).

(4) After 4 hours, remove the remaining dish from the oven and repeat the above procedures for covering and cooling the fluid sample.

(5) A total of five wicks shall be allotted for tests on each of the fluid samples, i.e., as received and 2 and 4 hours.

(6) Use tweezers to place the wicks into each of the dishes containing the test fluids and allow each wick to soak in the fluid sample for at least 2 minutes. Note: To ensure complete wetting, use tweezers to move the wicks around in the test fluid.

(7) After soaking for 2 minutes or more, each test wick shall be removed from the dish and shall be supported in a holder to drain for at least 2 minutes but no longer than 5 minutes, before testing.
(c) **Testing preparations.** (1) All testing shall be conducted in a draft-free location equipped with facilities for exhausting smoke and fumes produced by testing.

(2) With a spare wick mounted in the wick holder ensure that the vertical distance from the wick to the burner top is $7.5 \pm 0.3$ cm ($3.0 \pm 0.125$ in).

(3) Turn on and monitor the cycling device to ensure that the wick cycling rate is $25 \pm 2$ cycles per minute and that the midpoint of a cycle ends with the wick directly over the middle of the burner top.

(4) Turn on and adjust the methane gas flow to the burner to $0.05$ cu ft per min (1444 cc per min). Note: The burner flame shall be blue, have no appreciable inner cone, and be 10-15 cm (4-6 in) in height.

(5) At the start of a test the wick holder arm shall be located at one extreme end of a cycle, i.e., farthest away from the burner flame. Mount a fluid soaked wick sample into the holder (use tweezers) and ensure that a $5 \pm 0.2$ cm ($2 \pm 0.1$ in) length of stem extends horizontally from the holder end.

(d) **Testing.** (1) Start the cycling device and record the number of times that the test wick cycles into the burner flame until a self-sustaining flame is observed on the wick. Note: A
self-sustaining flame is defined here as one in which the wick becomes ignited and travels while flaming to the extreme end of a cycle (away from the burner) and is still flaming on its return and reentry into the burner flame.

(2) A total of five replicate tests shall be made on each of the three samples (as received and 2 and 4 hours) of each fluid.

(3) Average values for each (5 test) set on each of the above test samples shall be taken as test results.

(e) Acceptance. To be classified as meeting the flammability requirements of the wick test, candidate hydraulic fluids must meet or exceed the minimum number of cycles cited for the different samples of fluid as outlined in .3(b) Test criteria.

(5) Report. A report of test results shall include:

(1) Individual test values (no. of cycles to ignition for each test).

(2) The average value for five replicate tests on each different sample of a test fluid.

(3) A statement on whether a candidate hydraulic fluid meets the requirements of the "Test for the Effect of Evaporation on the Flammability of Hydraulic Fluids."
AN ASSESSMENT OF THREE DIFFERENT FIRE RESISTANCE TESTS FOR HYDRAULIC FLUIDS

Joseph J. Loftus

NATIONAL BUREAU OF STANDARDS
DEPARTMENT OF COMMERCE
WASHINGTON, D.C. 20234

U.S. Bureau of Mines
Pittsburgh, Pennsylvania 15213

Mine Safety and Health Administration
Triadelphia, West Virginia 26059

The Center for Fire Research (CFR) at the National Bureau of Standards (NBS) at the request of the Mine Safety and Health Administration (MSHA) and the Bureau of Mines (BOM) made an evaluation or assessment of the three different flammability tests used by MSHA for measuring the fire resistance of hydraulic fluids intended for use in underground coal mining operations. The methods described in the Code of Federal Regulations Schedule 30, Part 35, consist of the following: an Autogenous Ignition Temperature Test, a Temperature—Pressure Spray Ignition Test, and a Test to Determine the Effect of Evaporation on the Flammability of Hydraulic Fluids.

This report summarizes the various studies conducted in the evaluation and provides recommendations for improvement of the three test procedures.

Key Words: Autoignition temperature; coal mines; fire point; fire resistance tests; fire tests; flammability; flash point; hydraulic fluids; ignition.

Unlimited


Order From National Technical Information Service (NTIS), Springfield, VA. 22161

$6.50