CLASS NUMBER 6930

Examination Standard for Fire Performance of Industrial Fluids

Foreword

This standard is intended to verify that the products and services described will meet stated conditions of performance, safety, and quality useful to the ends of property conservation. The purpose of this standard is to present the criteria for examination of various types of products and services.

Examination in accordance with this standard shall demonstrate compliance and verify that quality control in manufacturing shall ensure a consistent and reliable product.

TABLE OF CONTENTS

1   INTRODUCTION

1.1   PURPOSE

1.1.1   This standard states testing and certification requirements for fire performance of industrial fluid products when used in industrial equipment systems.

1.1.2   Testing and certification criteria may include performance requirements, marking requirements, examination of manufacturing facility(ies), audit of quality assurance procedures, and a surveillance program.

1.2   SCOPE

1.2.1   This standard applies to finished industrial fluids used as, but not limited to, lubricants, hydraulic power transmission, turbine governor control, heat transfer, and cooling fluid in industrial equipment systems.

1.2.2   An industrial fluid product that is intended for use in an application that allows for contamination of the certified industrial fluid product with other fluids that are not certified is not eligible to be certified under this standard. Additionally, the use of a certified industrial fluid product in such an application will void the certification for that particular lot/batch of product.

1.2.3   Industrial fluids intended for use as transformer fluids shall meet the requirements of FM 6933, Transformer Fluids.

1.2.4   This standard is intended to evaluate only those hazards investigated. Toxicity and the suitability for the end use of the product have not been evaluated.

1.2.5   The tests conducted as part of this examination are intended to simulate conditions that may occur while the fluid is in use. This standard is not intended to be used to determine the protection requirements when containers of the fluid are placed in storage or other conditions.

1.3   BASIS FOR REQUIREMENTS

1.3.1   The requirements of this standard are based on experience, research and testing, and/or the standards of other organizations. The advice of manufacturers, users, trade associations, jurisdictions and/or loss control specialists was also considered.

1.3.2   The requirements of this standard reflect tests and practices used to examine characteristics of industrial fluids for the purpose of obtaining certification.

1.4   BASIS FOR CERTIFICATION

Certification is based upon satisfactory evaluation of the product and the fluid manufacturer in the following major areas:

1.4.1   Examination and tests on production samples shall be performed to evaluate

- the suitability of the product;

- the performance of the product as specified by the manufacturer and required for certification;

- the durability and reliability of the product.

1.4.2   An examination of the manufacturing facilities and audit of quality control procedures may be conducted to evaluate the manufacturer's ability to consistently produce the product which is examined and tested, and the marking procedures used to identify the product. These examinations may be repeated as part of the certification agency's surveillance audit program.

1.5   BASIS FOR CONTINUED CERTIFICATION

The basis for continual certification may include the following based upon the certification scheme and requirements of the certification agency:

- production or availability of the product as currently certified;

- the continued use of acceptable quality assurance procedures;

- satisfactory field experience;

- compliance with the terms stipulated by the certification;

- satisfactory re-examination of production samples for continued conformity to requirements; and

- satisfactory surveillance audits conducted as part of the certification agency's product surveillance program.

1.6   EFFECTIVE DATE

The effective date of this examination standard mandates that all products tested for certification after the effective date shall satisfy the requirements of this standard.

The effective date of this standard is eighteen (18) months after the publication date of the standard for compliance with all requirements.

1.7   SYSTEM OF UNITS

Units of measurement used in this Standard are United States (U.S.) customary units. These are followed by their arithmetic equivalents in International System (SI) units, enclosed in parentheses. The first value stated shall be regarded as the requirement. The converted equivalent value may be approximate. Conversion of U.S. customary units is in accordance with ANSI/IEEE/ASTM SI 10.

1.8   NORMATIVE REFERENCES

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the cited edition applies.

American National Standards Institute

(ANSI) B93.2 ''Glossary of Fluid Power Systems and Products''

American Society for Testing and Materials

ASTM D92, Standard Test Method for Flash and Fire Points by Cleveland Open Cup Tester

ASTM E203, Standard Test Method for Water Using Volumetric Karl Fischer Titration

ASTM D240, Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter

ASTM D808, Standard Test Method for Chlorine in New and Used Petroleum Products (High Pressure Decomposition Device Method)

ASTM D1179, Standard Test Methods for Fluoride Ion in Water

ASTM E1252, Standard Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis

ASTM D1480, Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer or Similar Method

ASTM E1642, Standard Practice for General Techniques of Gas Chromotography Infrared (GC/IR) Analysis

ASTM D4052, Standard Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

ASTM D4309, Standard Practice for Sample Digestion Using Closed Vessel Microwave Heating Technique for the Determination of Total Metals in Water

ASTM D4327, Standard Test Method for Anions in Water by Suppressed Ion Chromotography

ASTM D4868, Standard Test Method for Estimation of Net and Gross Heat of Combustion of Hydrocarbon Burner and Diesel Fuels

ASTM D5291, Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants

ASTM D5865, Standard Test Method for Gross Calorific Value of Coal and Coke

FM Approvals LLC

FM Approval Standard 6933, ''Less Flammable Transformer Fluids''

National Aeronautics and Space Administration

NASA Combustion Equilibrium and Application Code (CEA).

1.9   TERMS AND DEFINITIONS

General terminology shall be in accordance with ANSI B93.2 ''Glossary of Fluid Power Systems and Products'' unless shown below. For purposes of this standard, the following terms apply:

Adiabatic Stoichiometric Flame Temperature (T_{ad})	the critical temperature for sustained combustion
Concentrate Adiabatic Stoichiometric Flame Temperature ($T_{ad, conc}$)	A device equipped with a faucet and used for dispensing ignitable liquids.
Critical Adiabatic Stoichiometric Flame Temperature ($T_{ad, cr}$)	the critical temperature for sustained combustion for the finished fluid at the lowest water content as specified by the manufacturer.
Critical Heat Flux	the maximum heat flux at or below which there is no ignition
Emulsion	a homogeneous mixture of two or more immiscible liquid phases
Fluid Concentrate	The term as used herein defines the part of the finished fluid submitted for examination containing all the component parts except water, or with water content of 18% or less by mass percent.
Normalized SFP	The term normalized, as used herein, is defined as SFP measured at a unit flow
Spray Flammability Parameter (SFP)	a measure of the degree of flammability of a fluid in a highly atomized condition when pressurized to the stated pressure shown in this standard.

2   GENERAL INFORMATION

2.1   PRODUCT INFORMATION

Industrial fluids are liquids that may have two or more components in their composition and consist, but are not limited to, aqueous solutions, emulsions, vegetable oils, synthetic fluids, petroleum-based mineral oils, or other types of base stocks that are processed for stability and resilience. Other fluid compositions meeting the criteria of this standard may also be considered for certification. Industrial fluids are intended for use as, but not limited to, lubricants, hydraulic power transmission, turbine governor control, heat transfer, and cooling fluids. The suitability of the industrial fluid for specific use shall be determined by the manufacturer.

2.2   CERTIFICATION APPLICATION REQUIREMENTS

The manufacturer shall provide the following preliminary information for each fluid with any request for certification consideration:

- A complete list of product trade name(s) and/or formula designation(s);

- Technical Specification and/or Technical Data literature including the following elements:

    - General Fluid description (water-in-oil or oil-in-water emulsion type fluids shall be clearly identified);

    - Manufacturer's intended use/application;

    - Viscosity at °F (°C)*;

    - Specific gravity at °F (°C)*;

    - Flash and Fire point, Cleveland Open Cup, °F (°C) per ASTM D92*;

    - Appearance at 77°F (25°C)*;

- Material Safety Data Sheet(s);

- The number and location of all manufacturing facilities.

- * This information is provided to the certification agency for general information purposes only. These values will not be confirmed or evaluated as part of the examination unless deemed necessary or as required by other portions of this standard.

2.3   REQUIREMENTS FOR SAMPLES FOR EXAMINATION

2.3.1   Following authorization of a certification examination, the manufacturer shall submit samples for examination and testing based on the following:

2.3.2   • Sample requirements to be determined by the certification agency.

2.3.3   Requirements for samples may vary depending on design features, results of prior or similar testing, and results of any foregoing tests.

2.3.4   The manufacturer shall submit samples representative of production.

2.3.5   It is the manufacturer's responsibility to provide any necessary test fixtures, such as those which may be required to evaluate the product.

3 GENERAL REQUIREMENTS

3.1 MARKINGS

3.1.1 Marking on the product or, if not possible due to size, on its packaging or label accompanying the product, shall include the following information:

- name and address of the manufacturer or marking traceable to the manufacturer;

- date of manufacture or code traceable to date of manufacture or lot identification;

- model number, size, rating, capacity, etc., as appropriate.

When hazard warnings are needed, the markings should be universally recognizable.

3.1.2 The product tradename or formula identification shall correspond with the manufacturer's catalog designation and shall uniquely identify the product as certified. The manufacturer shall not place this model or type identification on any other product unless covered by a separate agreement.

3.1.3 The certification agency's mark of conformity shall be displayed visibly and permanently on the product and/or packaging as appropriate and in accordance with the certification agency's mark of conformity usage guidelines. The manufacturer shall not use this mark on any other product unless such product is covered by a separate report.

3.1.4 All markings shall be legible and durable.

3.2 MANUFACTURER'S INSTALLATION AND OPERATION INSTRUCTIONS

3.2.1 The manufacturer shall:

- prepare instructions for the installation, maintenance, and operation of the product;

- provide services to ensure proper installation, inspection, or maintenance for products of such nature that it would not be reasonable to expect the average user to be able to provide such installation, inspection, or maintenance.

3.2.2 An industrial fluid is not certified if the end use application is in conflict with the manufacturer's specifications and/or other written documentation such as technical bulletins.

3.3 CALIBRATION

3.3.1 Each piece of equipment used to verify the test parameters shall be calibrated within an interval determined on the basis of stability, purpose, and usage. A copy of the calibration certificate for each piece of test equipment is required. The certificate shall indicate that the calibration was performed against working standards whose calibration is certified and traceable to an acceptable reference standard and certified by an ISO/IEC 17025 accredited calibration laboratory. The test equipment shall be clearly identified by label or sticker showing the last date of the calibration and the next due date. A copy of the service provider's accreditation certificate as an ISO/IEC 17025 accredited calibration laboratory should be available.

3.3.2 When the inspection equipment and/or environment is not suitable for labels or stickers, other methods such as etching of control numbers on the measuring device are allowed, provided documentation is maintained on the calibration status of thus equipment.

3.4 OBSERVATION OF TEST SAMPLE PRODUCTION

3.4.1 At the discretion of the certification agency, their representative may witness the production of industrial fluids to be evaluated.

3.4.2 At the discretion of the certification agency, components of an industrial fluid may require a witness of production if the component is considered critical and may affect the results of the performance testing.

3.5   FORMULATION CHANGES

3.5.1   All formulation changes of a certified industrial fluid shall be reviewed by the certification agency prior to implementing a change. Formulation changes include, but are not limited to:

ingredient change(s),

changing a supplier of an ingredient, and/or

changing manufacturing tolerances of ingredients of an existing certified formulation.

3.5.2   A formulation change for a certified industrial fluid may require performance testing in accordance with this standard. At the discretion of the certification agency, a change to the certified industrial fluid and/or components may be evaluated by using the spray flammability parameter (SFP) estimate method in Appendix C of this standard. The estimated SFP shall be compared to the original SFP values obtained or among the SFP values calculated for each component, as appropriate.

4 PERFORMANCE REQUIREMENTS AND VERTIFICATION

The performance requirements of this standard are based on the fire performance specific to the industrial fluid type and not all tests listed under this section are applicable to every product. Industrial fluids and/or fluid concentrates submitted for examination shall satisfy each of the applicable performance criteria as listed in the table below in order to be eligible for certification under this standard:

Industrial Fluid Properties	FM 6930 Performance Requirements												
	4.1	4.2	4.3	4.4	4.5	4.6	4.7	4.8	4.9	4.10	4.11	4.12	4.13
	Fire Point Temperature[1]	Chemical Heat Release Rate	Density Determination	Critical Heat Flux	Spray Flammability Parameter (SFP)	Heat of Combustion[2]	Water Content	Elemental Composition	Adiabatic Flame Temperature	Separation Resistance[2]	Identification Testing - FTIR	Identification Testing – TD/GC/MS	Additional Tests
Fluid Has Fire Point	■	■	■	■	■	M				E	T	T	T
Fluid Does Not Have Fire Point	■	■				■	■	■	■	E	T	T	T
Water-Based Fluid (H$_2$O > 60%)	■	T				T	T	T	T	E	T	T	T

■ = Required Test for certification
M : Material evaluation for estimate of SFP on Industrial Fluid or component
T : Testing, if required, to be determined at the discretion of the certification agency
E : Required test for emulsion product. The project sponsor must state if the industrial fluid product is an emulsion or invert emulsion.
1 : An industrial fluid intended for use as a quench fluid shall not have fire point. The project sponsor must state if the industrial fluid is used as a quench fluid.
2 : Test(s) may be added as a project test requirement at the discretion of the certification agency.

4.1 DETERMINATION OF FIRE POINT

4.1.1 Requirement:

The ASTM D92 test method shall be used to determine fire point temperature. Each industrial fluid product submitted shall be screen tested to determine a fire point or verify that the fluid will boil prior to obtaining a fire point.

Industrial fluids not having a fire point shall be subjected to one (1) fire point test. An industrial fluid intended for use as a quench fluid shall not have fire point.

Industrial fluids having a fire point shall be subjected to three (3) ASTM D92 fire point tests. The average result will be determined and reported. The average result will be used in the calculation of the critical heat flux (q_{cr}) per section 4.4.

4.1.2   Test/Verification:

ASTM D92, *Standard Test Method for Flash and Fire Points by Cleveland Open Cup Tester*

4.2   DETERMINATION OF CHEMICAL HEAT RELEASE RATE

4.2.1   Requirement:

The Chemical Heat Release Rate (Q_{ch}) and massflow (m_f) of a finely atomized spray of the industrial fluid shall be determined in accordance with the procedure below for Determination of Chemical Heat Release Rate of Industrial Fluids. The test apparatus is described in Appendix A.

For industrial fluids having a fire point the chemical heat release rate and massflow results shall be used in calculation of the Spray Flammability Parameter (SFP) for the subject product per section 4.5.

For industrial fluids not having a fire point the industrial fluid chemical heat release rate (Q_{ch}) of a highly atomized spray shall be equal to, or less than, 130 kW for the subject product to be eligible for certification.

4.2.2   Test/Verification:

The test procedure for determining chemical heat release rates of industrial fluids is as follows:

Tests shall be conducted in ambient air conditions. The temperature and humidity conditions at the time of the test shall be recorded.

Fill steel container with approximately 0.5 gal (2 L) fluid sample.

Sample fluid and container shall be heated in a water bath to 140°F ±3°F (60°C ±2°C).

The heat exchanger temperature is to be set at 140°F ±3°F (60°C ±2°C).

Clear sample line to burner nozzle with nitrogen gas.

Weigh and record the weight of the heated sample.

The preheated fluid sample and container shall be inserted in insulating jacket on a weight scale to minimize heat loss. The weight of the fluid sample, nitrogen, container and accessories is recorded continuously by the data recorder for the entire test period.

The fluid sample and container shall be connected to a nitrogen gas cylinder capable to provide a constant 1000 psi ± 25 psi (6.9 MPa ± 0.17 MPa) pressure to the steel vessel container. The pressure in the sample line shall be continuous recorded by the data recorder for the entire test period.

The propane flow to the burner shall be adjusted to produce an approximate 12 kW flame. Once the required intensity of the flame has been established, it shall not be changed throughout the test. The beginning of the measurement phase of the test, the propane ring burner shall be operated alone for one minute to establish background data. The products of the combustion data for the propane are recoded by the data recorder. The propane burner shall remain in operation throughout the test. No adjustment to the propane flow rate is permitted. At the end of this period the fluid flow shall be started.

The pre heated fluid sample, approximate 0.5 gal (2 L), is pressurized to a constant 1000 psi ± 25 psi (6.9 MPa ± 0.17 Mpa) and discharged through an insulated supply line. The sample fluid flow is a continuous flow from the test sample bottle though a sample line to a heat exchanger to maintain the sample fluid test temperature of 140°F ±3°F (60°C ±2°C) located adjacent to the oil burner and then through an oil burner type 80° hollow cone spray nozzle with an exit diameter of 0.013 in. (0.33 mm), Hago 1.5 gal/h at 100 psi (5.7 L/h at 690 kPa). The nozzle shall be located 6 ft 11-3/16 in. (2.11 m) below the orifice plate of the collector. The nozzle shall be centered in a 5.5 in. (144 mm) diameter propane-air ring burner. The plane of ring burner head shall be located approximately 3/8 in. (10 mm) below the tip of the nozzle.

The during the entire test period the sample fluid temperature is measured by a thermocoupling located at the discharge to the oil burner nozzle. The temperature is recorded by the data recorder for the entire test period.

After one minute, pressurized and heated sample fluid is atomized by the Hago spray nozzle and ignited by flame from the ring burner head. The nozzle height shall be adjusted so that the tip of the spray flame is near the plane of the cone

collector rim. The test products of the combustion data for the propane and sample fluid are recorded by the data recorder for the entire test period. Record the height.

The products of the combustion entered the collection cone and duct where measures of sample fluid and propane combined temperature, CO, and CO2 generation is made. These measurements are used to calculate the combined chemical heat release rate of the sample fluid and the propane.

The test is terminated prior as the test sample container weight approaches 1600 grams. The approximate weight of an empty sample bottle. The propane ring burner flame is extinguished.

A continuous data printable digital report shall be sent to the engineer of all data measured or calculated by the data recorder and used to determine the chemical heat release rate

4.3   DETERMINATION OF FLUID DENSITY

4.3.1   Requirement:

The density of the fluids at 140°F ± 3°F (60°C ± 2°C) shall be determined and reported. The value obtained shall be used in the calculation of the Spray Flammability Parameter (SFP) per section 4.5.

4.3.2   Test/Verification:

ASTM D4052, *Standard Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter*

ASTM D1480, *Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer or Similar Method*

4.4   CALCULATION OF CRITICAL HEAT FLUX

4.4.1   Requirement:

The critical heat flux (q_{cr}) for the industrial fluid shall be calculated using the average fire point temperature determined in section 4.1 and reported.

Test/Verification:

The relationship between critical heat flux (q_{cr}) in kW/m2, and fire point temperature (T_f) is as follows:

$$q_{cr} = \alpha \times \sigma \times T_f^4 \qquad\qquad\qquad\qquad (Eq. 1)$$

where:

α is the fluid surface absorptivity, assumed to be 1,

σ is the Stefan-Boltzman constant, 5.67×10^{-11} kW/m^2 × K^4

T_f is the fire point temperature in units of K (degrees Kelvin)

4.5   CALCULATION OF SPRAY FLAMMABILITY PARAMETER (SFP)

4.5.1   Requirement:

A normalized Spray Flammability Parameter (SFP) shall be calculated and reported.

Industrial fluids having a fire point and having a normalized SFP of 5×10^4 or less are eligible for FM Approval. Calculations for determining the normalized SFP shall be rounded to the nearest whole number. For example, a result of 5.5×10^4 shall be rounded down and reported as 5×10^4. A result of 5.6×10^4 shall be rounded up and reported as 6×10^4.

4.5.2   Test/Verification:

Spray Flammability Parameter Equation:

$$SFP_{(normalized)} = 11.02 \times 10^6 \times Q_{ch} / (\rho_f\, q cr\, m_f) \qquad\qquad (Eq. 2)$$

where:

Q_{ch} is the chemical heat release rate determined per Section 4.2 and expressed in units of kW,

ρ_f is the density of the fluid, in units of kg/m^3, determined per Section 4.3,

q_{cr} is the critical heat flux for ignition determined per Section 4.4 in units of kW/m^2,

m_f is the fluid mass flow rate during the chemical heat release rate measurement, in units of g/s.

The SFP has been divided by m_f to derive SFP$_{(normalized)}$. This has been done to allow comparison of SFP values in various apparatuses having different hydraulic flow characteristics.

4.6 DETERMINATION OF GROSS HEAT OF COMPLETE COMBUSTION

4.6.1 Requirement:

The heat of combustion of the fluid concentrate shall be determined and reported. The value obtained shall be corrected for water content per the method in Appendix D and used in the determination of adiabatic stoichiometric flame temperature in section 4.9 .

Formulation Changes: In the case of evaluations of formulation changes, the heat of combustion of the industrial fluid, industrial fluid concentrate, and/or fluid component(s), as determined by the certification agency, shall be determined and reported. The results obtained shall be used in calculations in accordance with the SFP estimate method in Appendix C.

4.6.2 Test/Verification:

The heat of combustion value shall be determined by the following methods, or other procedures demonstrated to produce equivalent data at the discretion of the certification agency:

ASTM D240, *Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter* or

ASTM D4868, *Standard Test Method for Estimation of Net and Gross Heat of Combustion of Hydrocarbon Burner and Diesel Fuels* or *ASTM D5865, Standard Test Method for Gross Calorific Value of Coal and Coke*.

4.7 DETERMINATION OF WATER CONTENT

4.7.1 Requirement:

The water content of the industrial fluid and the fluid concentrate shall be determined and reported.

4.7.2 Test/Verification:

The water content of the fluid and fluid concentrate shall be determined by the following method, or other procedures demonstrated to produce equivalent data at the discretion of the certification agency:

ASTM E203, *Standard Test Method for Water Using Volumetric Karl Fischer Titration*

4.8 DETERMINATION OF ELEMENTAL COMPOSITION

4.8.1 Requirement:

The elemental composition of the fluid concentrate shall be determined and reported by percent mass for hydrogen, carbon, nitrogen, oxygen, fluorine, phosphorous, sulfur, chlorine, potassium, calcium, and bromine. Should it be determined that the above list does not include all the relevant elements in the fluid formulation, additional testing is required.

4.8.2 Test/Verification:

The elemental composition of the fluid concentrate shall be determined by the following methods, or other elemental procedures demonstrated to produce equivalent data at the discretion of the certification agency:

Perkin Elmer 240 Elemental Analyzer Method, or an equivalent analyzer having similar precession and bias

ASTM D808, *Standard Test Method for Chlorine in New and Used Petroleum Products (High Pressure Decomposition Device Method)*

ASTM D1179, *Standard Test Methods for Fluoride Ion in Water*

ASTM D4309, *Standard Practice for Sample Digestion Using Closed Vessel Microwave Heating Technique for the Determination of Total Metals in Water*

ASTM D4327, Standard Test Method for Anions in Water by Suppressed Ion Chromatography

ASTM D5291, Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants

4.9 DETERMINATION OF THE ADIABATIC STOICHIOMETRIC FLAME TEMPERATURE

4.9.1 Requirement:

The Adiabatic Stoichiometric Flame Temperature of the fluid concentrate ($T_{ad, conc}$) and industrial fluid ($T_{ad,cr}$) shall be determined and reported.

The Adiabatic Stoichiometric Flame Temperature adjusted for the total mass of water in the finished fluid ($T_{ad,cr}$), shall be equal to, or less than, 2100 K for the industrial fluid to be eligible for FM Approval.

4.9.2 Test/Verification:

NASA Combustion Equilibrium and Application (CEA) Code

4.10 SEPARATION RESISTANCE EVALUATION

4.10.1 Requirement:

The separation resistance of an emulsion based fluid shall be evaluated.

Emulsion based industrial fluids shall not exhibit more than 3 percent (by volume) separation of one immiscible phase from the remaining fluid when individual samples are conditioned and subjected to a centrifugal force (500xg) for 6 minutes.

4.10.2 Test/Verification:

A 1000 ml graduated beaker and one 40 ml centrifuge tube is filled with the test sample and placed in an environmental chamber at 40°F±5°F (4°C±3°C) for 16 hours (+0.2, -0 hrs.) and then the temperature is raised to a 70°F ±10°F (21°C±5°C) for 8 hours (+0.2, -0 hrs.). This exposure cycle is repeated for 10 complete cycles. After the final 8 hour cycle, the 1000 ml graduated beaker sample is removed and allowed to stabilize to room temperature. The 1000 ml graduated beaker sample is examined for evidence of separation.

A second 1000 ml graduated beaker and one 40 ml centrifuge tube is filled with the test sample and placed in an environmental chamber at minimum 150°F (+10°F, -0°F) [66°C (+5°C, -0°C)] environment for 24 hours (+0.25, -0 hrs.). After completion of the 24 hr. cycle in the environmental chamber the sample is removed and allowed to stabilize to room temperature. The 1000 ml graduated breaker sample is examined for evidence of separation.

The 40 ml centrifuge tube test samples from each test above plus one additional new sample is centrifuged at 500×G for 6 minutes (±5 sec). This test shall be conducted at a temperature of 70°F ±10°F (21°C±5°C). These samples are then examined for evidence of separation.

4.11 IDENTIFICATION TEST - STANDARD PRACTICE FOR GENERAL TECHNIQUES FOR OBTAINING INFRARED SPECTRA FOR QUALITATIVE ANALYSIS, ASTM E1252

4.11.1 Requirement:

For industrial fluids and/or other components as applicable, FTIR spectra shall be determined and reported at the discretion of the certification agency[1].

4.11.2 Test/Verification:

ASTM E1252, *Standard Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis*

Note 1:   These tests are conducted for identification purposes. The certification agency places no limits on the values obtained.

4.12 IDENTIFICATION TEST – THERMAL DESORPTION GAS CHROMATOGRAPHY MASS SPECTROMETRY (TD/GC/MS)

4.12.1 Requirement:

For industrial fluids and/or other components as applicable TD/GC/MS Spectrograms shall be determined and reported at the sole discretion of the certification agency[1].

4.12.2 Test/Verification:

ASTM E1642, *Standard Practice for General Techniques of Gas Chromatography Infrared (GC/IR) Analysis*

Note 1: These tests are conducted for identification purposes. The certification agency places no limits on the values obtained.

5   MANUFACTURER'S REQUIREMENTS

5.1   DEMONSTRATED QUALITY CONTROL PROGRAM

5.1.1   A quality assurance program is required to assure that subsequent bench, swab, plunger, and dispenser cans produced by the manufacturer shall present the same quality and reliability as the specific industrial fluid examined. Design quality, conformance to design, and performance are the areas of primary concern.

- Design quality is determined during the examination and tests and may be documented in the certification report.

- Continued conformance to this standard is verified by the surveillance program.

- Quality of performance is determined by field performance and by periodic re-examination and testing.

5.1.2   The manufacturer shall demonstrate a quality assurance program which specifies controls for at least the following areas:

- existence of corporate quality assurance guidelines;

- incoming quality assurance, including testing;

- in process quality assurance, including testing;

- final inspection and tests;

- equipment calibration;

- drawing and change control;

- packaging and shipping; and

- handling and disposition of non-conforming materials.

5.1.3   Documentation/Manual

There should be an authoritative collection of procedures/policies. It should provide an accurate description of the quality management system while serving as a permanent reference for implementation and maintenance of that system. The system should require that sufficient records are maintained to demonstrate achievement of the required quality and verify operation of the quality system.

5.1.4   Records

To assure adequate traceability of materials and products, the manufacturer shall maintain a record of all quality assurance tests performed, for a minimum period of two years from the date of manufacture.

5.1.5   Drawing and Change Control

- The manufacturer shall establish a system of product configuration control that shall allow no unauthorized changes to the product. Changes to critical documents, identified in the certification report, may be required to be reported to, and authorized by the certification agency prior to implementation for production.

- Records of all revisions to all certified products shall be maintained.

5.2   SURVEILLANCE AUDIT

5.2.1   An audit of the manufacturing facility may be part of the certification agencies surveillance requirements to verify implementation of the quality assurance program. Its purpose is to determine that the manufacturer's equipment,

procedures and quality program are maintained to ensure a uniform product consistent with that which was tested and certified.

5.2.2   Certified products or services shall be produced, or provided at, or provided from, location(s) disclosed as part of the certification examination. Manufacture of products bearing a certification mark is not permitted at any other location prior to disclosure to the certification agency.

5.3   INSTALLATION INSPECTIONS

Field inspections may be conducted to review an installation. The inspections are conducted to assess ease of application, and conformance to written specifications. When more than one application technique is used, one or all may be inspected at the discretion of the certification agency.

5.4   PRODUCT MODIFICATIONS

The manufacturer shall notify the certification agency of changes in product construction, components, raw materials, physical characteristics, coatings, component formulation or quality assurance procedures prior to implementation.

6 BIBLIOGRAPHY

ISO/IEC 17025, General Requirements for the Competence of Testing and Calibration Laboratories.

APPENDIX A: THE FIRE PRODUCTS COLLECTOR

Figure A-1 illustrates the overall configuration of this apparatus. The apparatus consists of a vertical steel duct connected to an air pollution control system. The duct is made up of 12 in. (305 mm) diameter sections, connected to a large collecting funnel of 72 in. (1.829 m) diameter at its widest level. The fire products generated from the fire are captured along with ambient air in the duct by the funnel (cone collector) through a 7-1/16 in. (179 mm) diameter orifice.

Measurements are made at a station several duct diameters above the orifice. In the apparatus, the fire products are diluted and well mixed with ambient air as they are captured in the sampling duct. The following measurements are made in the duct: total mass and volumetric flow rates of the mixture of fire products and air; gas temperature; and concentrations of carbon dioxide, carbon monoxide, and total gaseous hydrocarbons.
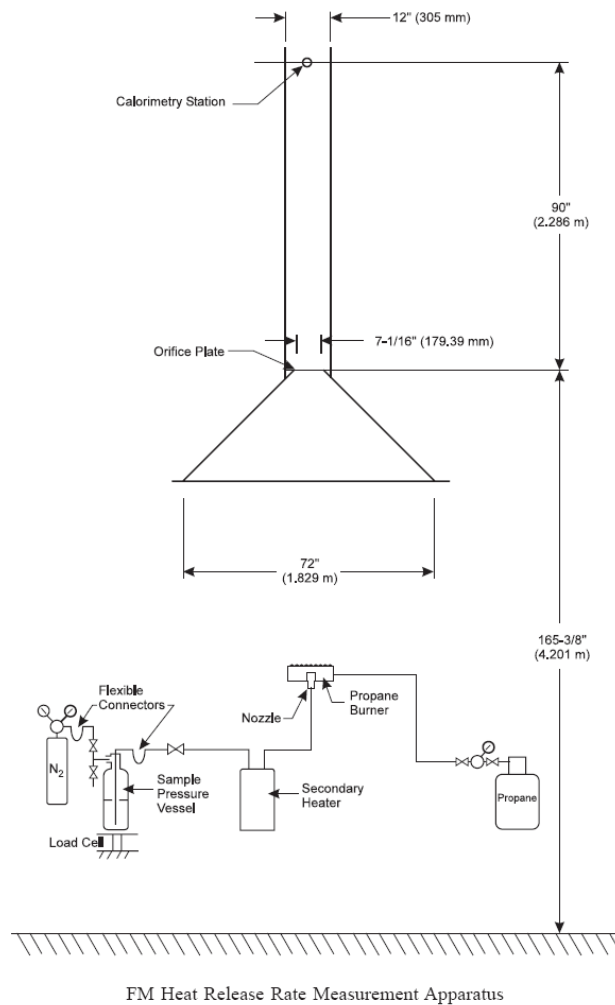
(Fig. A-1) Fire Products Collector

APPENDIX B: DETERMINATION OF HEAT RELEASE RATES

In flaming combustion, heat is released in the production of CO_2 and CO, or in the consumption of O_2. Therefore, the chemical heat release rate Q_{ch} (kW) can be calculated as:

$$Q_{ch} = k_1 \times G_{CO_2} + k_2 \times G_{CO} \qquad \text{(Eq. B-1)}$$

Where,

$k_1 = \Delta H_T/k_{CO_2}$ is the net heat of complete combustion (kJ/g) of the fuel per unit mass of CO_2 produced;

k_{CO_2} is the theoretical stoichiometric mass of CO_2 produced per unit mass of fuel converted completely to CO_2

$k_2 = (\Delta H_T - \Delta H_{CO} \times k_{CO})/k_{CO}$ is the net heat of complete combustion (kJ/g) of the fuel converted to CO per unit mass of CO produced;

k_{CO} is the theoretical stoichiometric mass of CO produced per unit mass of fuel converted completely to CO,

and ΔH_{CO} is the heat of combustion of CO (10.13 kJ/g);

and G_{CO_2} and G_{CO} are the mass generation rate (g/s) of CO_2 and CO, respectively.

Or, $\qquad Q_{ch} = k \times D_{O_2} \qquad \text{(Eq. B-2)}$

Where,

$K = \Delta H_T/k_{O_2}$ is the net heat of complete combustion of the fuel per unit of mass of O_2 consumed and D_{O_2} is the consumption rate (g/s) of O_2.

With some exceptions the values K, k_1, and k_2 are approximate constants for various types of fluids. Since it is much more difficult to measure the O2 consumption accurately for low heat release rate fires, as compared to CO_2 and CO, Equation (D1) was chosen for the determination of chemical heat release rate, using 13 and 12 for the values of k_1 and k_2, respectively.

. $\qquad Q_{ch} = 13\, G_{CO_2} + 12\, G_{CO} \qquad \text{(Eq. B-3)}$

When $G_{CO} \ll 1$, $12\, G_{CO}$ can be neglected.

The values of k_1 and k_2 can be precisely calculated if the exact elemental or chemical formula and ΔH_T, net heat of complete combustion (kg/J), of the fluid are known.

APPENDIX C: SCREENING OF FLUIDS SFP APPROXIMATION LEVELS

Most fluid manufacturers do not have ready access to either the Fire Products Collector or Critical Heat Flux test apparatuses. However, SFP approximations may be calculated from more commonly available standard ASTM bench tests. In order to determine SFP, the tests contained in the body of Examination Standard FM 6930 must be conducted. The correlation between SFP values obtained using the test method contained in the standard and that shown in this appendix is unknown

$$q_{cr} = \alpha \times \sigma \times T_f^4 \qquad\qquad\qquad (Eq.\ C1)$$

Where,

α is the fluid surface absorptivity, assumed to be 1,

σ is the Stefan-Boltzmann constant, 5.67×10^{-11} kW/m^2 × K^4

(kilowatts per square meter-degree Kelvin to the fourth power), and

T_f is the fire point temperature in units of K (degrees Kelvin).

There are no conceptual liabilities to the use of this measurement. However, there are some practical considerations which dictate caution. Some fluids do not exhibit a valid fire point. Two-component fluids, such as water-glycol mixtures, will not exhibit a fire point until all the water has been evaporated. This value is not valid for the mixture. For such fluid, a SFP cannot be determined, the flammability of the fluid should be determined using the NASA equilibrium code.

An oxygen bomb calorimeter can be used to determine net heat of complete combustion (ΔH_T). This value can be used as a proxy for chemical heat release rate (Q_{ch}), in kW. Their relationship is as follows:

$$Q_{ch} = \chi_{ch} \times \Delta H_T \times m_f \qquad\qquad\qquad (Eq.\ C2)$$

Where,

χ_{ch} is the combustion efficiency,

ΔH_T is the net heat of complete combustion in units of kJ/g (kilojoules per gram), and

m_f is the mass flow rate of the fluid in units of g/s (grams per second).

The equation used when Q_{ch} has been measured and qcr was calculated:

$$SFP_{(normalized)} = 11.02 \times 10^6 \times Q_{ch} / (\rho_f \times q_{cr} \times m_f) \qquad\qquad (Eq.\ C3)$$

If Q_{ch} and T_f have been measured, then substituting equation E1 into equation E3 provides the appropriate calculation method:

$$SFP_{(normalized)} = 1.94 \times 10^{17} \times Q_{ch} / (\rho_f \times T_f^4 \times m_f) \qquad\qquad (Eq.\ C4)$$

If Q_{ch} has not been measured, but ΔH_T is known, then equation E2 can be substituted into equation E3 to yield the third alternative calculation method. This method is only approximate and will yield an SFP higher than that calculated using equation E3 by a factor of $1/\chi_{ch}$, the combustion efficiency:

$$SFP_{(normalized)} = 11.02 \times 10^6 \times \Delta H_T / (\rho_f \times q_{cr}) \qquad\qquad (Eq.\ C5)$$

Similarly, substituting equation E1 into equation E5 yields the equation to be used when calculating SFP from ΔH_T and T_f.

Again this equation renders an approximate result, due to an assumption of a combustion efficiency of 1:

$$SFP_{(normalized)} = 1.94 \times 10^{17} \times \Delta H_T / (\rho_f \times T_f^4) \qquad\qquad (Eq.\ C6)$$

APPENDIX D: CORRECTION METHOD FOR ELEMENTAL COMPOSITION OF FLUIDS NOT HAVING A FIRE POINT

The elemental composition, i.e., mass% of industrial fluid sample is to determine for carbon, hydrogen, oxygen, calcium, phosphorous, potassium, nitrogen, bromine, chlorine, fluorine, sulfur, metals, etc. Also, the water content of the industrial fluid sample is measured by the Karl Fischer method per ASTM E203 is determined. The laboratory elemental composition results will include the presence of the hydrogen and oxygen due to water in the samples. A correction to the elemental composition is necessary to account for the effect of water on the measured mass percent of oxygen (O atom). It is strongly recommended that a concentrate be used in the elemental analysis to minimize the correction. To correct for this overlap, the following methodology is developed:

a. Suppose Y_i is the reported mass fraction of element i, where I can be carbon, hydrogen, oxygen, calcium, phosphorous, potassium, water content etc.

b. The sum of all elements including the measured water (Y_W) is:

$$\Sigma_i = Y_K + Y_P + Y_C + \ldots + Y_W \qquad \text{(Eq. D 1)}$$

c. The error in the sum is $E_{error} = \Sigma_i - 1$. Note that $E_{error} < Y_W$. This is because the reported oxygen and hydrogen is also included in the measured water mass fraction.

d. The actual mass fraction of hydrogen and oxygen is corrected to account for the measured water as follows:

$$Y_{O,a} = \frac{Y_O - \frac{16}{18}E_{error}}{1 - Y_W} \quad ; \text{and} \quad Y_{H,a} = \frac{Y_H - \frac{2}{18}E_{error}}{1 - Y_W} \qquad \text{(Eq. D 2)}$$

e. The corrected actual mass fractions of each element in the absence of any water are calculated as:

$$Y_{K,a} = \frac{Y_K}{1 - Y_W}$$

$$Y_{P,a} = \frac{Y_P}{1 - Y_W}$$

$$Y_{Ca,a} = \frac{Y_{Ca}}{1 - Y_W} \qquad \text{(Eq. D 3)}$$

and so on.

f. The gross heat of compete combustion per unit mass of the fluid consumed (ΔH_c in kJ/kg) is measured per

ASTM D4868 or ASTM D240 or ASTM D5865. The corrected gross heat of compete combustion $\Delta H_{c,a}$ is calculated as;

$$\Delta HY_{c,a} = \frac{\Delta H_c}{1 - Y_W} \qquad \text{(Eq. D 4)}$$

NASA Combustion Equilibrium and Application Code is used to calculate the Concentrate Adiabatic Stoichiometric

Flame Temperature ($T_{ad, conc}$) using the $Y_{K,a}$, $Y_{P,a}$, $Y_{C,a}$, ... $Y_{W,a}$, $\Delta H_{c,a}$. A second calculation is then made to determine the Critical Stoichiometric Flame Temperature ($T_{ad, cr}$) of the fluid at the desired water content.