METHODS DEVELOPMENT FOR MEASURING AND CLASSIFYING FLAMMABILITY/COMBUSTIBILITY

Everett W. Heinonen and Robert E. Tapscott
Center for Global Environmental Technologies (CGET)
New Mexico Engineering Research Institute (NMERI)
The University of New Mexico (UNM)
901 University Blvd. SE
Albuquerque, New Mexico 87106-4339
(505) 272-7255

For the past four years, the New Mexico Engineering Research Institute (NMERI) has conducted research into the ability of halocarbons to inert explosive environments. The flammability of refiigerants and refiigerant blends has also been investigated as part of a recently completed program*. Both efforts have involved the measurement of the explosion overpressure resulting from the ignition of a flammable gas, either alone or with inertants added to prevent explosion or lower the explosion overpressure. The refiigerant flammability study examined the test procedures used to measure the flammability of refrigerants and established conditions under which refiigerants and refrigerant blends exhibit flammability as a fbnction of composition and test conditions. A second goal of the program was to recommend changes to test procedures and equipment which would make flammability results more consistent. While this program specifically addressed refiigerants, the results and conclusions are applicable to all uses of halocarbons for inertion and explosion prevention.

NMERI utilized two separate apparatuses to perform this work. The first, the NMERI explosion sphere, was designed to screen various HFCs for potential replacement of ozone-depleting Halon 1301 in the North Slope of Alaska oil and gas producing facilities. The apparatus and results have been reported during a previous conference'. Control and data acquisition were performed by a Hewlett-Packard (HP) HP 85 Data computer. While this system performed admirably for several years, it was apparent even fiom the start of testing that a modified data acquisition system based on a PC could better perform the tasks. The existing system could only record one channel of data, which was stored in a HP format incompatible with DOS. As part of the recently-completed program, a new test flask based on American Society for Testing and Materials (ASTM) Standard E681 was constructed and an updated data acquisition system (DAS) was developed. This paper describes the two apparatuses and the new DAS, and provides generalized procedures applicable to explosion testing in both the explosion sphere and flask.

NMERI EXPLOSION SPHERE

The NMERI explosion sphere was originally constructed to investigate the ability of halocarbons to inert propane and methane. It was designed to screen large numbers of

*This effort was sponsored by the Air-conditioning and Refrigeration Technology Institute, Inc., 4301 North Fairfax Drive, Suite 425, Arlington VA 22203.

halocarbons to determine which required the least weight and volume to reduce the explosive overpressure to 1 psi (6.9 kPa) or less, which was considered the definition of an explosion. In addition to its intended use, it has also been used to test inertants using refrigerants such as R-32, R-152a, and R-142b as fuels. As part of this screening, approximate upper and lower flammability limits for these flammable refrigerants were measured, although precise limits were not determined. In all cases, the flammability limits were narrower than reported using other test facilities, reflecting the trend that less inerting agent was required in the NMERI explosion sphere than in other apparatuses. A potential explanation of these differences was presented in 1993 paper.[2]

The NMERI explosion sphere' (Figure 1) was modeled after the Fenwal Explosive

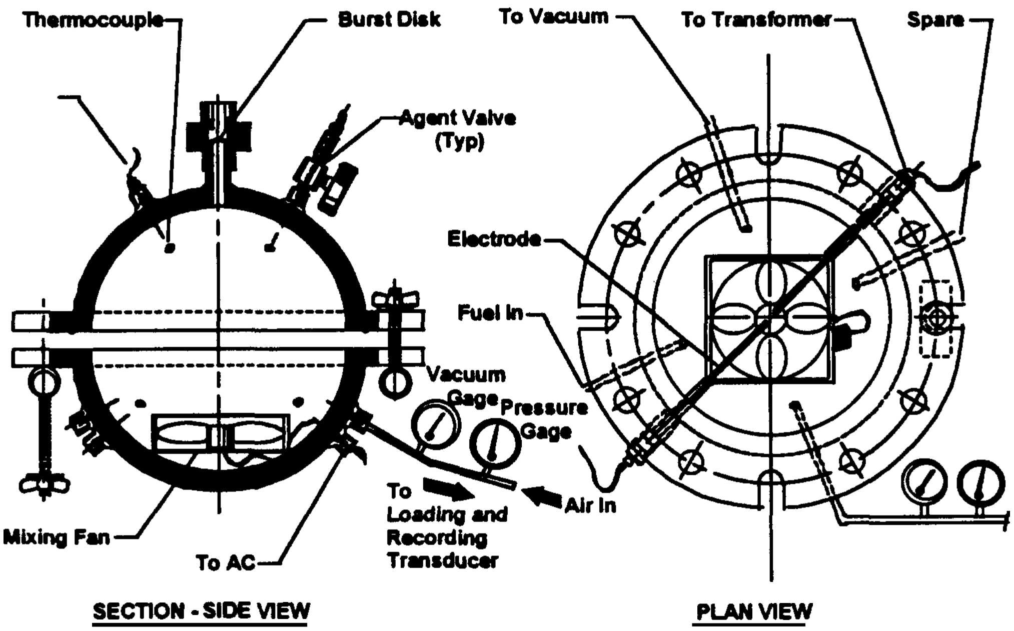

Figure 1. NMERI Explosion Sphere.

Sphere.' It consists of two 9-3/4-inch (25-cm) diameter 304-stainless steel hemispheres welded on stainless steel flanges that could be fastened to form a sphere with a measured volume of 7930 cm^3. Overpressure relief was provided by a 3/4 in (19 mm) safety vent disc (200 psi (1378 kPa), Fike model number 3/4-inch PV-UT Nickel) installed in a rupture disk holder on top of the sphere. The internal pressure was measured by two Druck pressure transducers; a 0- to 2.5-psi (0 to 17.2 kPa) transducer was used to measure the partial pressures of the components as they were added, and a 0-to 30-psi (0 to 207 kPa) transducer recorded the overpressure due the flammable event. Two thermocouples were installed, one above and one below the ignition point. An electronic cooling fan inside the sphere provided mixing. Eight pipe nipples, four on the top hemisphere and four on the bottom, provided the inlets for the fuel and refiigerants, pressure transducer openings, and the vacuum exhaust port, as well as for two thermocouples and one fan power penetration. A manifold connected to one of the penetrations on the bottom hemisphere contained the air inlet, the two transducers, an analog gauge, an auxiliary vacuum pump port, and a vent to the atmosphere.

Two igniters (Eclipse model number 18193), consisting of ceramic jackets and stainless steel rods, were installed in the sphere. The ends of the electrodes were pointed and separated by ¼ in (6-mm). The mixture was ignited across a spark gap; a wide variety of different types of ignition sources were possible using these electrodes, including AC- and DC-electrical sparks, heated and exploding wire, and matches. A high-voltage DC source, an AC source, and the wire and match ignition sources were developed during this program.

Two separate vacuum systems were used, the house vacuum for exhausting the by-products of the explosion through the cryotrap, and a second pump for pulling a vacuum prior to loading the sphere. The house vacuum could not pull sufficient vacuum to reduce the vacuum to a sufficiently low and repeatable level. The house vacuum was used to exhaust combustion by-products because its exhaust was outside the building. Figure 2 is a photograph of the sphere as set up for test.

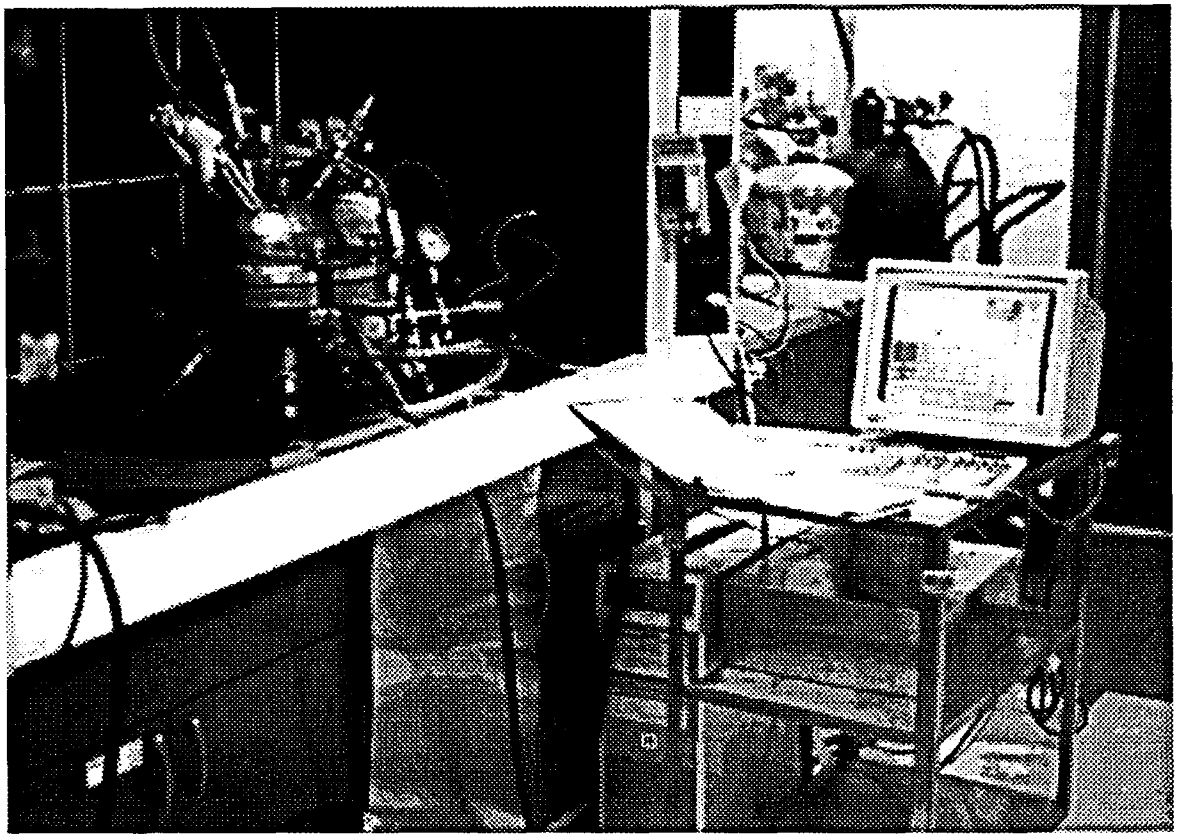

Figure 2. Explosion Sphere Setup for Test.

ASTM E681 FLASK

The most commonly used method to determine the flammability of flammable gases is described in ASTM Standard E681-85. The general technique for testing in this flask is similar to that used in the explosion sphere, namely addition of known amounts of fuel, air, and inertants into a 5-liter flask, providing a source of ignition, and evaluating the resultant explosion. The major difference is that the ASTM method uses a visual criterion to determine the flammability of a substance instead of an overpressure criterion.

A test apparatus, consisting of a flask and vessel cover, an oven enclosure, and a manifold for inlets and gauges, was constructed according to the specifications of ASTM E681. A major addition to the apparatus was a pressure transducer to measure the explosive overpressure.

Flask and Test Vessel Cover

The flask was a standard 5-liter boiling flask with short ring neck (Fisher number 10-065F). The test vessel cover was the ASTM E681-specified No. 14 rubber stopper with five penetrations, two each for the electrode rods and the thermocouples, and one for the gas inlet and exhaust. Two type-K thermocouples were located 3/4 in (19 mm) above and below the electrodes, approximately 314 in (19 mm) to each side. The electrode rods were stainless steel rods, threaded on the top and bottom. The connectors to the transformer wires were attached to the top, and a holder to allow insertion of removable tungsten electrodes was screwed on the bottom. The electrodes themselves were held in place by set screws, and the separation could be adjusted; one end of the electrodes was flat, and the other pointed. The same ignition sources available in the explosion sphere can be used in this apparatus. Both the thermocouples and electrodes were inserted in glass rods and epoxied on each end to prevent loss of vacuum. A laboratory stirring egg was placed inside the flask, allowing stirring by a magnetic stirring bar located just below the flask in the enclosure. Figure 3 illustrates the interior of the flask.

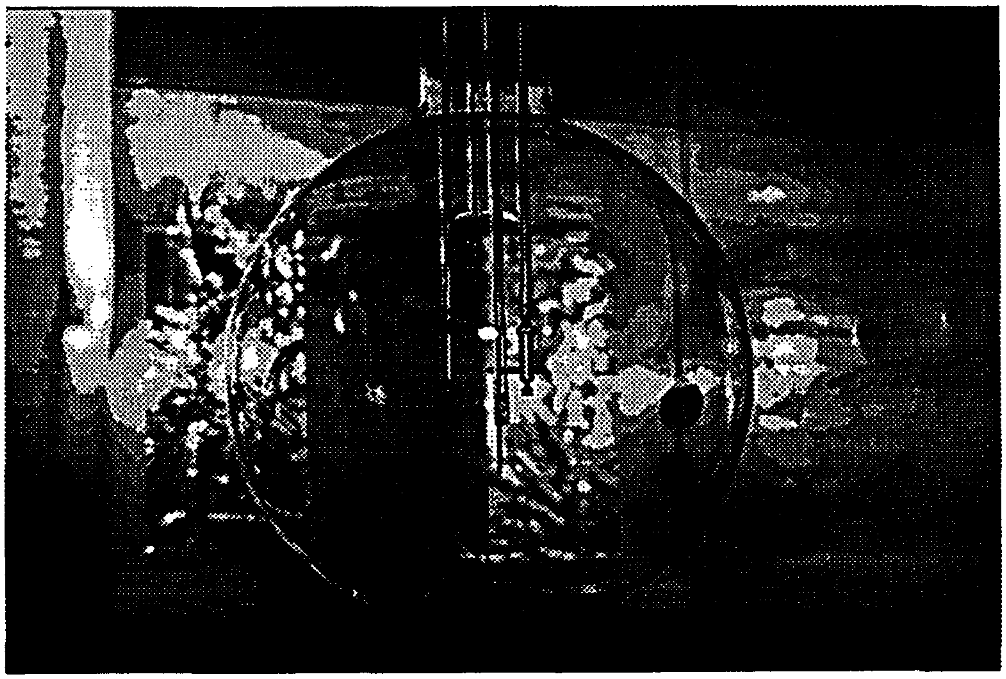

Figure 3. Interior of Flask.

Enclosure

An existing laboratory oven was used for the enclosure. The oven, which has an internal volume of 13 in by 13 in by 13 in (33 cm by 33 cm by 33 cm), was disassembled and thoroughly cleaned. The old insulation was removed and discarded, as was the door seal. A hole was cut in the top for the flask opening. The rear of the oven was cut out and re-installed as a blow-out panel. A circular hole was cut in a removable shelf to act as a holder for the rounded bottom of the flask. The window on the door was replaced with a ½ in (1.27 cm) lexan panel. Bolts, springs, and nuts were installed in the top for the hold down, although their use was not required during testing. New Kaolite® insulation and a home oven door seal were installed. All wiring was replaced and a 400-W strip heater was installed as the oven's heating element. A 4½ in (22.5 cm) box fan was installed on the bottom of the oven to mix the air and ensure consistent temperatures. A laboratory stirrer was installed underneath the oven, and the shaft extended into the oven area to position a stirrer magnet just below the flask. Figure 4 shows the enclosure and flask assembly.

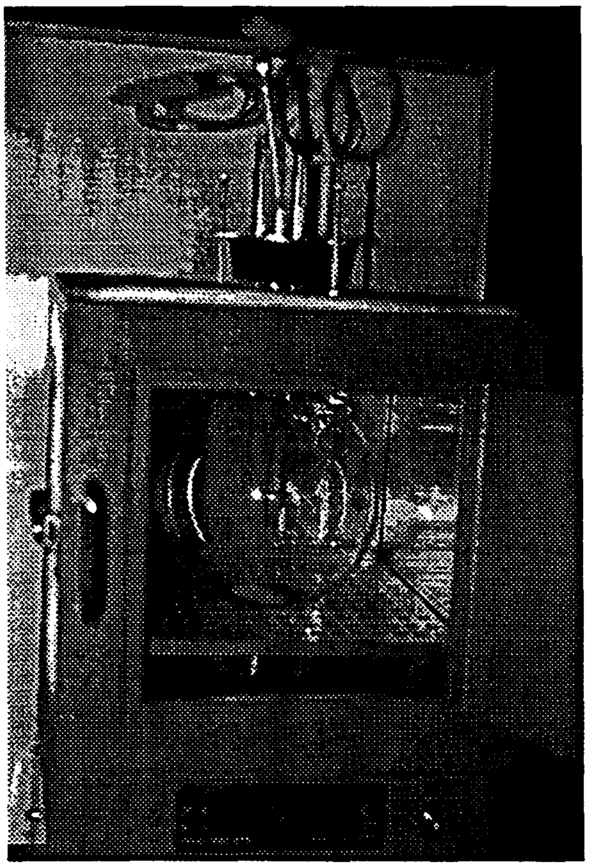

Figure 4. ASTM E681 Enclosure and Flask.

Manifold

The fifth penetration in the test vessel cover was for the gas inlet and exhaust. Because of the limited space on the test vessel cover, all penetrations and transducers which were located on the explosion sphere were moved to the manifold that was redesigned to accommodate all required components. The new manifold consisted of (1) four inlet ports, (2) the loading and overpressure transducers, (3) two vacuum exhausts (one to house vacuum and one to a vacuum pump), (4) one air inlet, (5) an analog gauge; and (6) an exhaust to ambient air. All components except the overpressure transducer and the house vacuum exhaust were located to the side of the enclosure; these were located just above the enclosure and were connected to the outlet tube of the flask and the remainder of the manifold by tubing and Swagelok® fittings. A flexible tube allowed sufficient movement for the test vessel cover to be rotated for installation of the flask. One fuel and two diluent input lines were connected to three of the four inlet ports (the fourth was designed for an additional fuel such as propane), and bottled air was connected to the air inlet.

DATA ACQUISITION SYSTEM

The goal of the new DAS was to automate all facets of testing, except the physical addition of the fuel and other components; to record and store all test parameters including the amounts and concentrations of all components; and to provide a real-time plot of the test temperatures and overpressure within the test device.

Software

The software was developed using the National Instruments LabWindows® CVI Version 3.0.1 run-time system for windows. This system permits a customized set of functions, including all the required test control and data acquisition functions, as well as the appropriate screens, to be developed on one computer and an executable program exported to any other windows-compatible computer. With the appropriate data boards, any computer with Microsoft Windows™ and sufficient memory has the capability to function as the data acquisition computer. Specific code for this application was written in C language.

Hardware

The software was installed on a 486-33mHz computer. The input/output board was a National Instruments AT-MIO-16F-5 high-performance, multifunctional analog, digital, and timing board with 8 A-D converters and 12-bit resolution. A National Instruments SC-2070 general purpose breadboard connected the analog and digital signals to the AT-MIO-16F-5. A National Instruments SC-205X cable adapter board interfaced the signals to the computer.

Six data channels were monitored as follows: three thermocouples, the loading transducer, the overpressure transducer, and a humidity probe. Data were take sequentially one channel at a time at a rate of 1000 points per channel over 16 seconds, or 62 Hz. In addition, the computer controlled the charging of capacitors in the NMERI DC spark ignition source and the discharge of the capacitors to form the arc. Figure 5 illustrates the data acquisition screen, and Figure 6 illustrates the screen displayed after completion of a test.

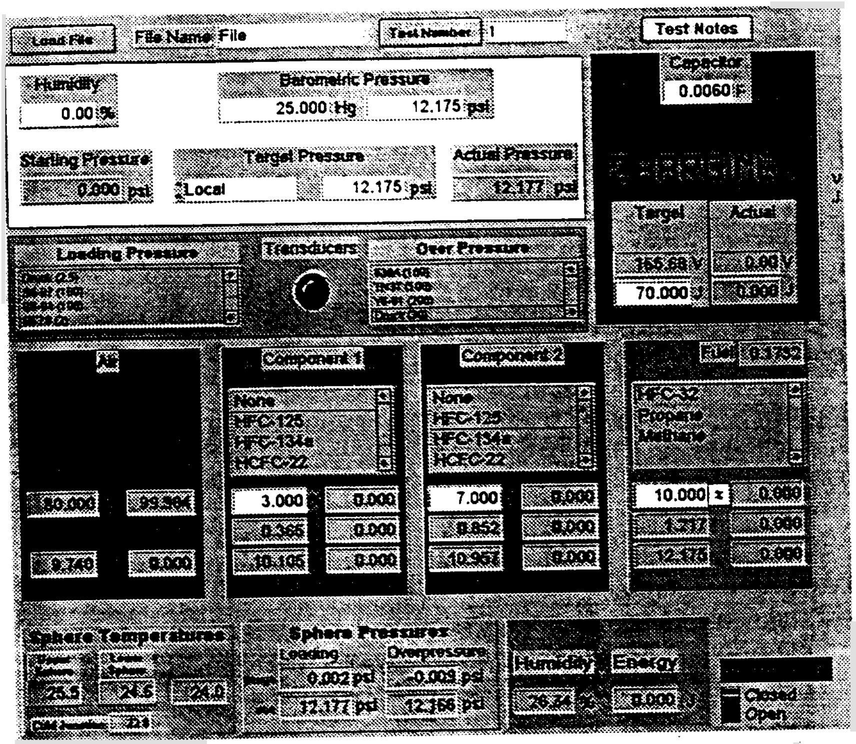

Figure 5. Data Entry Screen.

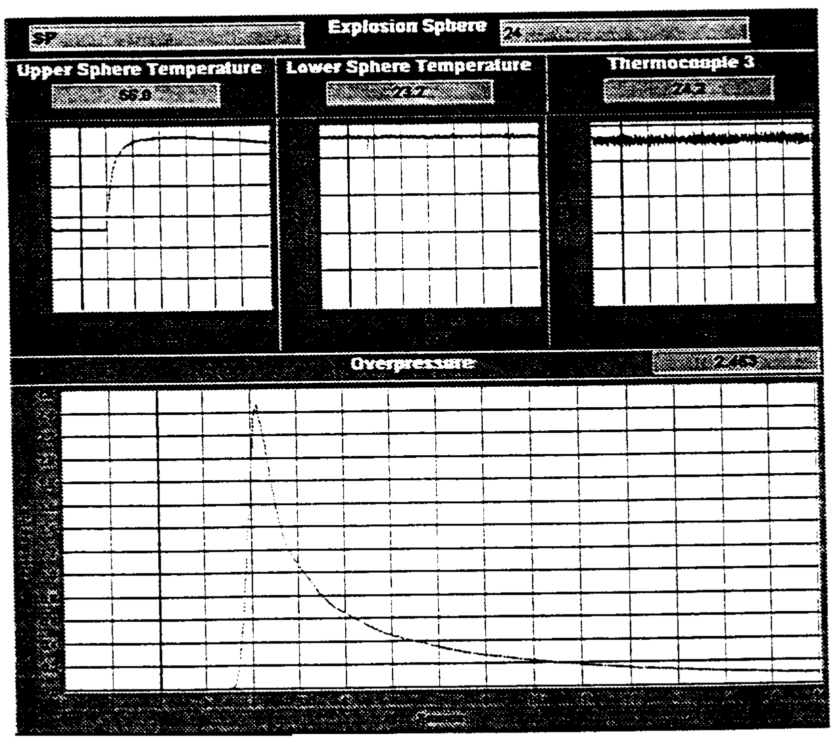

Figure 6. Data Screen.

RESULTS FROM METHODS DEVELOPMENT

Prior to the start of the test program, it had been hypothesized that several parameters, including the following, could affect flammability:

Apparatus
Composition of refrigerant
Humidity
Starting pressure
Starting temperature
Ignition type and strength
Turbulence in apparatus
Mixing of components
Altitude of testing

· ace limitations prevent description of the test techniques in this paper. A complete ..cription of these techniques may be found in Appendices A through C in the final report for the completed program.[5] The following general results and conclusions are from the effort and are applicable to all explosion and flammability testing in the explosion sphere and ASTM flask.

The goal of this program was to investigate the degree to which each of the parameters affected results. The first investigation was a correlation between the visual indication of flammability as determined by the ASTM E681 method and overpressure measurements from the transducer.

Visual Flammabilitv vs. Overpressure.

A mixture was considered flammable if flame propagated beyond an imaginary truncated cone bounded by the ends of the electrodes and the neck of the flask. Using this criteria, fluoromethane (HFC 32) was flammable at an overpressure reading of 0.3 psi. In past testing in the explosion sphere, we used the more severe criterion of 1 psi for flammability. We have not ... tests using other flammable mixtures to ascertain whether the 0.3 psi equivalent will .. other flammable gases.

Humidity.

In previous testing a combination of room air and compressed air from the laboratory compressed air system was used. The humidity was neither measured nor controlled, although the compressed air was if relatively low humidity. Literature searches indicated that relative humidity of the air affected the flammability characteristics of some refrigerant blends; our results with R-32 confirmed this. For all testing under this program, we used purified natural air with most moisture removed (relative humidity of 1.7%) as the air source. We also produced humidified air for certain tests by bubbling the air through water, increasing its relative humidity to 82%. The amount of non-flammable refrigerant required to make a refrigerant blend non-flammable increased up to 35 percent as the relative humidity increased from 1.7% to 82%.

Starting Pressure.

As starting pressure is increased and all other factors are held constant, the explosive overpressure also increases. This has an effect on the altitude of testing, as explained later.

Starting Temueerature.

All the previous testing at **NMERI** was performed at room temperature. Literature searches revealed significant differences in the flammability limits and the inertion concentrations at higher temperatures, which our testing at 100 "C confirmed. Flammability limits changed slightly and the amount of inertant required to make a refrigerant blend non-flammable increased by up to 22 percent as the test temperature was raised fiom room temperature (25 "C) to 100°C.

Ignition Type and Strength.

Bench tests indicated that conically shaped electrodes required the least voltage for the spark to arc, and should be used in all electric arc discharges. The separation of 0.25 in (6 mm) used previously appears to be adequate.

The NMERI low-voltage DC spark ignition, which generates a spark by the discharge of capacitors charged to 70 J, actually delivers only 0.25 J across the gap for an efficiency factor of 0.35%. The reason for this low efficiency **is** the saturation of the iron core of the transformer used to boost voltage. The energy content of the spark could be increased if required by using a transformer with a core material that will not saturate as fast. Depending upon the configuration of the ignition system used, the ignition energies claimed by other researchers may also be different than reported.

***An* AC** ignition source, which was developed for this program, has been demonstrated to be effective for explosion testing. The duration of discharge was set at 0.2 seconds, and was adequate to repeatably ignite flammable materials.

Turbulence and Mixing.

Tests were conducted varying the time for which the components were mixed. It **was** determined that the components must be fully mixed to ensure repeatable results. The 2-minute mixing time of **ASTM** E681 appears adequate, although 5 minutes is recommended when final inertion or flammability limit values are being determined. Several tests were conducted when the mixing fan or stirrer were moving; results were not consistent.

Altitude of Testing

A significant increase in both the width of the flammability limits and the flammability was observed as the starting test pressure was increased fiom local atmospheric pressure at Albuquerque (12.0 psia [620 torr]) to sea level atmospheric pressure (14.7 psia [760 torr]). The effect of altitude should be considered as a special case of the starting pressure parameter.

RECOMMENDATIONS

The following general recommendations were provided to help standardize flammability and explosion results in the NMERI explosion sphere and ASTM E681 flask.

Ignition Source

The AC source developed for this program appeared to be satisfactory as a nominal source. A duration for this spark should be specified. The duration used in this program, 0.2 seconds, appeared to work well. The electrode rod points should be conical. The 0.25-in (6-mm) spacing appears to be satisfactory.

When using the low-voltage DC source, the electrode points should be cleaned between every test, especially when final trials are being conducted. The need for cleaning between tests has not been demonstrated for the AC source, although it appears logical to perform the same procedures between tests.

Humidity of the Air

The humidity of the air must be specified and controlled. It is suggested that a source of air readily available throughout the country (such as is distributed by Matheson or other large supplier) be specified, and that the humidity of the air be varied to investigate the impacts on individual flammable gases.

Altitude of Test

A method to normalize the altitude of the test facility, if the trends observed during this sphere testing program are confirmed, should be developed. This may involve modification of the ASTM flask and technique.

Temperature at Start of Test

Since the temperature of the mixture at ignition affects the flammability of a substance, it should be controlled to within ±2°C (±4°F). If it can reasonably be assumed that the temperature can reach above room temperature, it should be tested at 100°C in the ASTM E681 flask.

REFERENCES

1. Heinonen, E. W., and Skaggs, S. R., "Fire Suppression Testing of Halon 1301 Inertion Agents," Proceedings of the Halon Alternatives Technical Working Conference 1992, pages 213-224, New Mexico Engineering Research Institute, Albuquerque, NM 87106, 1992.

2. Heinonen, E. W., "The Effect of Ignition Source and Strength on Sphere Ignition Results," Proceedings of the Halon Alternatives Technical Working Conference 1993, pages 565-576, New Mexico Engineering Research Institute, Albuquerque, NM 87106, 1993.

3. Skaggs, S.R., Heinonen, E.W., Moore, T.A., and Kirst, J.A., Low Ozone-Depleting Halocarbons as Total-Flood Agents: Volume 2: Laboratory-Scale Fire Suppression and Explosion Prevention (Draft Report), New Mexico Engineering Research Institute OC 92/26, pages 18-31, New Mexico Engineering Research Institute, Albuquerque, NM 87106, Sep 1993.

4. Dalzell, Warner G., A Determination of the Flammability Envelope of Four Ternary Fuel-Air-Halon 1301 Systems, Fenwal Report No. PSR-624, Fenwal Incorporated, Ashland, MA, January 1977.

5 Heinonen, E. W., and Tapscott, R. E., Methods Development for Measuring and Classifying Flammability/Combustibility of Refrigerants: Task3 - Laboratory Test Results, Air-Conditioning and Refrigeration Technology Institute, Arlington, Virginia, December, 1994. DOE/CE/23810-50, NMERI 1994/44.