DOE/CE/23810-50
NMERI 1994/44

METHODS DEVELOPMENT FOR MEASURING AND CLASSIFYING FLAMMABILITY/COMBUSTIBILITY OF REFRIGERANTS

Final Report
TASK 3–LABORATORY TEST RESULTS

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DECEMBER 1994

Prepared for:
The Air-Conditioning and Refrigeration Technology Institute
Under
ARTI MCLR Project Number 660-52400

This program is supported, in part, by U.S. Department of Energy (Office of Building Technology) Grant Number DE-FG02-91CE23810: Materials Compatibility and Lubricants Research (MCLR) on CFC-Refrigerant Substitutes. Federal Funding supporting this program constitutes 93.57% of allowable costs. Funding from non-government sources supporting this program consists of direct cost sharing of 6.43% of allowable costs, and significant in-kind contributions from the air-conditioning industry.

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The data and conclusions contained in this report should not be construed to represent or imply the safety or safe use of a refrigerant. The methods and procedures used in this report are for the most part not contained in current refrigerant safety classification standards such as published in ANSI/ASHRAE Standard 34-1992 or Underwriters Laboratories Standard UL 2182 and have not been verified or accepted by industry, code, or standard experts as a means of adequately determining or describing the flammability or safety of commercial refrigerants.

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PREFACE

This final report was prepared by the Center for Global Environmental Technologies (CGET), New Mexico Engineering Research Institute (NMERI), University of New Mexico, Albuquerque, New Mexico 87131-1376 under Contract 660-52400 for the Air-Conditioning and Refrigeration Technology Institute (ARTI), 4301 N. Fairfax Drive, Suite 425, Arlington VA 22203.

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ACKNOWLEDGMENTS

The efforts of Glenn M. Mattson, Jacob A. Kaizerman, and Jimmy D. Watson are gratefully acknowledged. Without their efforts, this phase of the project would not have been completed. Likewise, the efforts of the software developers, Neale Pickett, John C. Logothetis, and Brian N. Wylie, are also acknowledged. Finally, the work of the late Jon A. Kirst, who pioneered the development of the software system under a previous program and who ably assisted in the early part of this program, will live on in the hardware and software produced for this effort.

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LIST OF ABBREVIATIONS AND ACRONYMS

AC	Alternating Current
ANSI	American National Standards Institute
ARTI	Air Conditioning and Refrigerating Technology Institute
ASHRAE	American Society of Heating, Refrigerating, and Air-Conditioning Engineers, Inc.
ASTM	American Society for Testing and Materials
CFC	Chlorofluorocarbon
CFR	Critical Flammability Ratio
DC	Direct Current
FTIR	Fourier-Transfer Infrared
GVEq	Gas Volume Equivalent
HF	Hydrogen Fluoride
IR	Infrared
LFL	Lower Flammability Limit
MIE	Minimum Ignition Energy
MIPV	Mutual Inductance Particle Velocity
NFR	Non-Flammability Ratio
NMERI	New Mexico Engineering Research Institute
PC	Personal Computer
SCR	Silicon Controlled Rectifier
SVEq	Storage Volume Equivalent
UFL	Upper Flammability Limit
UL	Underwriters Laboratory
UV	Ultraviolet
VAC	Volts AC
V_{bank}	Capacitor Bank Charging Voltage
Weq	Weight Equivalent

LIST OF SYMBOLS

ºC	Degrees Celsius
$d_{\parallel}$	Quenching Distance
cm	Centimeters
E	Energy
F	Capacitance
F	Farads
J	Joules
kPa	Kilopascals
Hz	Hertz (cycles per second)
mA	Milliamps
mH	Millihenries
mL	Milliliters
mm	Millimeters
mg/m^3	Milligrams per Cubic Meter
P	Power
psi	Pounds per Square Inch
psia	Pounds per Square Inch Absolute
t	time
torr	mm of Hg
V	Voltage
ε_{eff}	Minimum Energy Required to Ignite Flammable Mixture

1.0 INTRODUCTION

1.1. Background

Because of concerns for the effect that chlorofluorocarbon (CFC) fluids currently in use as refrigerants have on the environment, the refrigeration industry is considering the use of natural refrigerants, many of which are potentially flammable. In some cases, these flammable fluids may result in the least environmental damage when considering ozone depletion, global warming, efficiency, and photochemical reactivity. Many potentially flammable fluids have been proven to be effective when used either by themselves or as a part of a binary or ternary mixture. However, despite favorable initial test results, these fluids may not be acceptable to the general public if questions of safety cannot be adequately addressed. Significant research is being conducted to investigate the flammability of these materials.

American National Standards Institute/American Society of Heating, Refrigeration, and Air-Conditioning Engineers (ANSI/ASHRAE) Standard 34-1992[1] currently classifies refrigerants based on the lower flammability limit (LFL) and the heat of combustion. The flammability limit is determined by using the test method described in ANSI/ASTM E681-85.[2] This test method uses a visual criterion for determining whether the refrigerant is flammable and is generally only valid at ambient pressure and below. While this standard specifies an alternating current (AC) spark generated between electrodes inside the test flask or an exploding copper wire, ANSI/ASHRAE 34-1992 and Underwriters Laboratory (UL) 2182 recommends an electrically ignited paper match. These ignition types may or may not be representative of a realistic threat. The use of the ASTM E681 test method to determine the flammability of refrigerants has been questioned.

To resolve some of these questions, the Air-Conditioning and Refrigeration Technology Institute, Inc. (ARTI) has commissioned the New Mexico Engineering Research Institute (NMERI) to conduct a test program. The purpose of this project is to experimentally determine the impact and variability of eleven different parameters which may affect flammability and/or combustibility of refrigerants and refrigerant blends, as a function of composition and test conditions, and to develop a better understanding of methods and conditions to measure the flammability of refrigerants. The refrigerants used in this study are being considered as new refrigerants and reviewed published data on these materials is scarce. The data contained herein should not be considered complete and should be used only to make relative comparisons of the impacts of the test parameters, not to represent the flammability characteristics of the materials.

This report documents Task 3 of the test program. During Task 1, Gather and Review Available Data, technical literature was thoroughly reviewed and a database of available documents was constructed.[3] During Task 2, the test plan for this task was written.[4] The goals of Task 3 are to investigate the flammability characteristics of selected blends of refrigerants R32, R134a, and R125 using an existing explosion sphere and a newly-constructed ASTM E681 apparatus.

1.2. Flammability Parameters

Not all combustible mixtures are considered flammable, and one of the difficulties in determining flammability is the definition of what constitutes flammability. According to Zabetakis,[5]

> A combustible gas-air mixture can be burned over a **wide** [emphasis author] range of concentrations - when either subjected to elevated temperatures or exposed to a catalytic surface at ordinary temperatures. However, homogeneous combustible gas-air mixtures are flammable, that is, they can [only]propagate flame freely within a limited range of compositions.

We will use this definition for flammability, namely, that flames propagate freely through the gas-air mixture. The most dilute mixture that is flammable is known as the lower flammability limit (LFL) or lower limit, and the most concentrated mixture is known as the upper flammability limit (UFL) or upper limit. The paramount concept is that flame must propagate for the material to be flammable, and, with that in mind, we have borrowed liberally from the large body of combustion data to develop this test plan.

A second difficulty is the definition of the flammability limits themselves. Some researchers consider the LFL a unique fundamental material property[6] while other consider that there "is no convincing evidence for the existence of fundamental limits of inflammability, although theory suggests that there probably are such limits."[7] It is known that flames which can propagate from bottom to top do not always propagate from top to bottom, and that "carefully nurtured flames can be maintained well outside conventional limits"[8] using flat-flame burners. Pressure, temperature, catalytic effects, and other external factors influence flammability limits. One goal of this program is the development of a test technique which will determine realistic limits that accurately reflect the "true" flammability of refrigerants. One possible definition of "true" limits is that the limits should reflect the behavior of the flammable mixture in the environment in which the refrigerant is used.

Little has been published on the flammability of refrigerants. More data are available on the flammability of combustible hydrocarbon fuels. Even more information is available on the ignition and propagation of flames through combustible mixtures. Therefore, for planning the test program, the flammable refrigerant will be considered to have flammability properties similar to those of a hydrocarbon fuel and other refrigerants in the mixture will be considered to be diluents or inertants. Although the flammability characteristics of halogenated refrigerants are known to differ from those of hydrocarbons, this will allow the application of combustion theory.

Combustion, and therefore flammability, generally cannot occur without the four legs of the fire tetrahedron—fuel, oxygen, heat, and sufficient free radicals to sustain the reaction. Unless sufficient fuel is available (at the LFL) or sufficient oxygen is available (at the UFL), the mixture is non-flammable. Unless the temperature reaches the ignition temperature, and heat is conveyed to the next layer of unburned gas, the mixture is non-flammable. And unless an adequate number of free radicals are available to sustain the reaction, the mixture is also non-flammable.

Gaseous fuels can burn in one of two ways. Fuel and air may be intimately mixed prior to burning (pre-mixed flames) or they may be initially separate and burned in the zone where they mix (diffusion flames)[9]. While flammable refrigerants may burn under either condition, most testing uses a pre-mixed flame rather than the diffusion flame (flowing, constant pressure systems are the exception). In reality, a realistic scenario for a flammable refrigerant could be an unconfined vapor cloud, which is not truly representative of either type of testing. For the purpose of this test program, only pre-mixed flames will be investigated due to the general acceptance of flammability results based on this type of testing.

Visual indications are only one of many indications that may be used to verify flammability. This may not be the most reliable method due to reactions at low temperatures that may produce cool flames or low-temperature explosions which, while producing luminosity, do not propagate on their own and thus do not meet the definition of flammability. Other indications of flammability that could potentially prove to be more reliable include the following:

(1) Temperature rise. Does the ignition source raise the temperature above the limiting flame temperature of the mixture, which is required to sustain combustion?

(2) Light. Are there non-visual methods - infrared or ultraviolet(IR or UV) - that could be more reliable and repeatable than visual?

(3) Pressure rise. Both the magnitude of the pressure rise and the rate of the rise could be indicators of flammability.

(4) Presence of radicals. It is known that combustion cannot occur without the presence of an adequate number of free radicals to sustain the reaction.

(5) Presence of combustion products. Combustion could be indicated by the ratio of certain combustion products in the mixture.

(6) Heat of Reaction. Combustion can be indicated by the presence of a temperature rise detected in bomb calorimetry.

(7) Flame Velocity. Combustion waves travel with a specific flame velocity which can be measured and analyzed to determine combustion.

(8) Electrical properties. Electrical conductivity and other electrical properties of the mixture may change after combustion.

Conceivably, each of these techniques could result in different limits of flammability depending upon the criteria. It is the purpose of this test program to propose and assess one or more test techniques to reliably and repeatable determine the flammability of refrigerants and refrigerant blends. Any experimental method should minimize the following quantities[10]:

(1) Natural convection

(2) Conductive/convection losses to walls
(3) Radiative losses to walls
(4) Selective diffusional demixing
(5) Non-linear flow gradients (flame stretch).

Many parameters affect flammability and must be considered in the design and analysis of test methodology. Each of the factors below could affect the flammability limits:

(1) Ignition source
(2) Temperature of the mixture
(3) Pressure of the mixture
(4) Humidity of the air
(5) Size and shape of the test vessel
(6) Test vessel materials
(7) Turbulence in the test vessel
(8) Concentration of the components of the mixture
(9) Reactivity of the components
(10) Mixing of the components
(11) Altitude of testing

Each parameter will be discussed below.

1.2.1. Ignition Source

The ignition source may be among the most critical parameters in determining repeatable flammability limits. Richard and Shankland[11] found differences of 2.4% in the LFL for R32 (13.8% vs. 11.4% at 95 °C, a 17% variability) when ignited with copper wire as compared to a match, and much greater differences have been found for more marginally flammable refrigerants such as R-141b. Potential ignition sources include matches, pyrotechnic igniters (including electric matches), electric sparks, mechanical sparks, glowing wires, and hot surfaces. Alternating current (AC) sparks must be examined as well as the more traditional direct current (DC) sparks. Any potential source (for example nichrome wire) must be examined to ensure that it does not have a catalytic effect on the reaction. Of the above sources, the two most likely to be repeatable are the match and the electric spark (AC or DC), and most flammability testing has been accomplished using those two ignition sources. The task is to determine whether a flame would be self-sustaining once the effects of the ignition source has dissipated. The proper test method would have any effect of the ignition source dissipated prior to the flame propagation to the wall.

The electric spark is a very fast-acting ignition source, on the order of 10^{-8} to 10^{-7} seconds discharge time, and, therefore, the energy is highly concentrated. Sparks have been studied for years, primarily because of their importance in the internal combustion engine.

Variables in this technique include:

(1) AC vs. DC. Testing at NMERI involving inertion of propane and methane by Halon 1301 has indicated that 120 volts AC (VAC) boosted through a transformer can ignite mixtures that cannot be ignited by a DC spark at a stored energy (in the capacitors) of up to 100 J. However, the duration of the AC spark was not controlled.

(2) Electrodes. The shape, diameter, separation distance, and materials may be critical. Most references indicate that above the quenching distance $d_{\parallel}$—the minimum gap between electrodes that will successfully quench ignition—the shape of the ends of the electrodes is not important.[12] However, Lewis and von Elbe also state that for large spark energies, $d_{\parallel}$ actually increases, due to the increased heat transfer produced by the turbulence of the larger spark.[13]

(3) Position of the ignition source. It appears as if the majority of flammability testing, including the NMERI inertion work, has been conducted with the electrodes located approximately in the center of the apparatus. However, the procedures for testing in the ASTM flask do not specify a location of the electrodes, and indeed, indicate that the location should be somewhat lower than the center of the flask. Since ignition is measured by the upward propagation of flame, this would seem logical. On the other hand, Crescetti and others have shown a correlation between the vertical location of the electrodes and the location of the flame front as a function of time.[14] Therefore, the vertical location of the electrodes in the ASTM flask will be considered as a variable.

(4) Energy. Most electric sparks are produced by a capacitive discharge with many also having an inductive component. The energy level in a capacitive spark is defined by the stored electrical energy in the capacitors, $\frac{1}{2}FV^2$, where F is the capacitance and V is the voltage to which the capacitors are charged (actually, the voltage before and after discharge must be considered). If there are no losses between the capacitor and the electrodes, all energy is transferred into the spark. However, even in this case, some of that energy will be required to initiate the spark and will not be available to ignite the flammable mixture. The energy deposited at a sufficient temperature to initiate a freely propagating flame is called ε_{eff} and may be up to two orders of magnitude less than the stored energy, depending upon the voltage to which the capacitors were charged and the chamber volume size.[15] The energy loss due to the high-voltage transformer used in previous NMERI testing has been estimated at 15%, however it is believed that this figure is much lower than the actual value, and a significant portion of the Task 3 effort will revolve around determining this value.

(5) Circuit parameters. It is known that inductance in the ignition circuit results in a different type of spark than that without inductance[16] and that ignition energy is dependent not only on the resistance and capacitance of the circuit, but also on the product of the two, the discharge time constant.[17]

Matches have also been used for flammability testing. Matches are easily ignited using low voltage batteries or power supplies. Under most test conditions, matches have a higher energy content (one source reported 176 Joules)[18] than a spark with a time duration much longer than a spark and have resulted in wider flammability limits than electric sparks or heated wires. It must be assessed whether matches provide a realistic ignition source in the small test volumes used in flammability testing.

Lewis and von Elbe describe ignition by hot-wires and heated metal bars.[19] These sources will be considered as potential ignition sources in this program.

The minimum ignition energy (MIE) for various hydrocarbons has been extensively studied for years.[20,21] According to Bradford and Finch,[22] "in all cases which have been examined, more electrical energy is necessary to bring about ignition of mixtures near the limits than in the middle zone of inflammability." Therefore the MIE must be examined not only at the stoichiometric fuel-to-air ratio, but at the limits as well. However, it is believed that rather than devote significant effort to precisely determining the MIE for various concentrations of agents, it is more critical to develop a representative source that provides repeatable and reliable ignition of the mixture with a known energy.

1.2.2. Temperature

In general, the higher the initial temperature, the wider the flammability limits. This occurs because less energy is required to bring the flammable mixture to its flame temperature. The mixture will ignite without an external source when raised to its auto-ignition temperature. Zabetakis[23] has suggested that the LFL of a hydrocarbon at any temperature can be estimated by drawing a line between the room temperature LFL and 0% concentration at 1300 ºC (Figure 1). If this estimation can be extrapolated to flammable refrigerants, or if a similar estimation can be made, the amount of testing at elevated temperature can be minimized.

1.2.3. Pressure

Pressure effects are among the most difficult of all the factors affecting flammability to quantify, and in some cases trends presented in different sources are contradictory. It is fairly well understood that higher pressures affect the UFL much more than the LFL. For example, Drysdale reports the UFL of methane as 60% and the LFL as 4% at 200 atmospheres (as compared to 15% and 5% at 1 atm)[24], indicating a significant widening of the limits. On the other hand, Coward and Jones state that increases in pressure above that of atmospheric do not always widen the limits and for some mixtures, the range of flammability is lowered with increasing pressure.[25]

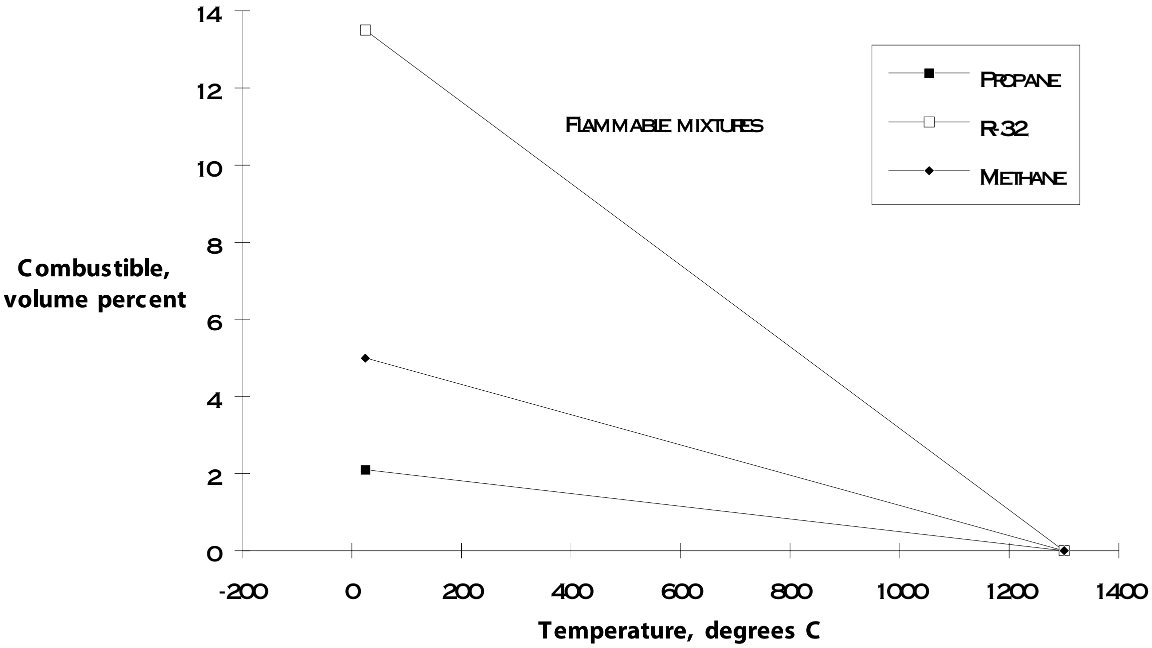

Figure 1. Effect of Temperature on Lower Limits of Paraffin Hydrocarbons in Air at Atmospheric Pressure (from Reference 23).

At lower pressures some disagreements also occur. Drysdale indicates that pressures below atmospheric do not affect the flammability limits providing that the pressure is above 0.1 atmosphere and the compound remains either a gas or a liquid.[26] Egerton states that "a reduction of the pressure below 760 mm always causes both limits to converge until they coincide at some critical pressure below which no propagation can occur"[27] (although he does not state how rapidly this convergence occurs). However, Lovachev[28] has reported that Lewis and von Elbe felt it was uncertain that a lower pressure limit independent of vessel size could exist. He also reported instances of flammability limits at extremely low pressures, although he also felt that ignition effects may have played a part in those tests. While these three statements may not be totally inconsistent (the conditions under which the conclusions were made were not described), the fact that some controversy appears to occur indicates that difficulty in measuring the flammability limits at low pressures exits. Therefore, care must be taken to define upper and lower pressure requirements that are reasonable and will not impact the flammability limits.

1.2.4. Humidity

While it has long been known that water vapor can affect the kinetics of a reaction, it has been only recently that the flammability behavior of R-245ca has been analyzed with respect to the moisture content of the air.[29,30] It has been postulated that more than one combustion reaction is possible depending upon whether adequate water vapor is present. This dichotomy occurs primarily where the number of fluorine atoms is greater than the number of hydrogen atoms and the flammability of refrigerants such as R134a, R245ca, and R245fa may be affected by this phenomenon. Effectively, as refrigerant concentration, temperature and pressure were kept constant, the flame characteristics intensified as the moisture content is increased from 10 to 60% relative humidity. Therefore, any test methods developed must consider the relative humidity of the air.

Dr. N. D. Smith of the EPA has indicated in a personal correspondence that he found some interesting observations regarding attempts to obtain known moisture levels in the ASTM E681 flammability tests. In one series of 21 consecutive flammability tests, the same amount of water (96 microliters) was injected into the evacuated flask and allowed to evaporate and the resulting pressure exerted by the water vapor was recorded. For the first 20 runs, when the sphere was not cleaned between runs, the pressure of the water vapor declined after each test. When the flask was rinsed with di-ionized water and dried in the oven for several minutes, the vapor pressure returned to the original value. In a similar discovery, the actual pressure was 37% lower than the predicted value 0.228 psia vs. 0.362 psia (11.8 torr vs. 18.7 torr).

One potential explanation involves the adsorption of water vapor by the inside glass surface of the flask. The glass surface itself adsorbs water vapor, accounting for the discrepancy between the predicted and actual values. When hydrogen fluoride (HF) is formed as a result of combustion, it clings to the surface, providing radicals for the water vapor to be adsorbed. As more tests are run without washing the sphere between tests, more HF builds up. This observation underscores the importance of washing the inside of glass flasks, especially when zeroing in on the LFL or the UFL.

In a similar area, the flammability limits of ammonia in dry air and 100% humidity were reported in a recent report.[31] The authors report that flammability tests of ammonia in 100% humid air result in narrower flammability limits than in dry air, and that tests at 50% relative humidity exhibit the same limits as tests at dry air. This behavior was also seen in earlier tests at BASF.

1.2.5. Test Vessel Size and Shape.

Much of the accepted flammability results were developed in the Bureau of Mines in the 59 in (150 cm) high by 2 in (5 cm) diameter explosion (or flame) tube. In several studies it was determined that flammability limits were affected by the quenching effects of the vessel walls under 2 in (5 cm), but were generally unaffected over 2 in (5 cm).[32] Likewise, it was determined that explosion spheres of 5 liters (8 in (20.2 cm) diameter) give similar results to larger vessels for R32 flammability testing.[33]

However, the behavior of flammability limits in free space have not been studied extensively. Lovachev indicates that "the flammability limits of ammonia-air flames in free space were found to be wider than for a standard tube. This indicated that there are mixtures capable of burning in free space only".[34] Therefore, any limits determined in the confined spaces of the ASTM flask or explosion sphere must be regarded as approximate if the true flammability situation is an unconfined cloud of refrigerant.

1.2.6. Test Vessel Material

Although for the most part, flammability has been determined by the time that the flame front has reached the walls of the test vessel, two different properties could affect the flame front after that point. First, different materials have different heat conduction values, affecting the temperature of the flame front. Second, various types of materials tend to affect the free radicals differently, promoting different kinetics at the wall of the vessel.

One additional parameter to be considered is the cleanliness and condition of the ASTM flask. Repeated testing may eventually etch the Pyrex, affecting both the actual results of the flammability testing and the visual observation of the tests. The results of Dr. Smith reported in section 1.2.4 illustrate this point.

1.2.7. Turbulence in the Test Vessel

Turbulence affects the development of the flame front. Drysdale[35] indicates that turbulence increases the rate of flame propagation through a mixture, but the effect is difficult to quantify. Significant research has been conducted on the effect of turbulence on coal dust explosions[36] and to a lesser extent flammable gases.[37] Any testing needs to consider that turbulence is a variable and should most likely reduce turbulence to as low a level as possible.

1.2.8. Composition of the Components of the Mixture

Two factors determine the flammability of the mixture in air—the weight (or volume) fraction of each constituent element in a binary or ternary mixture, especially when only one of the constituents is flammable, and the total concentration of the mixture with air. In order to discuss the flammability of a mixture, the concept of the critical flammability ratio (CFR) has been developed. The CFR is the ratio of non-flammable refrigerant required to render the mixture non-flammable. For non-binary blends, methods such as the critical flammability parameter[38] can provide a good estimate of the flammability of any ratio of constituents if the CFR is known for each individual constituent. One additional factor which must be considered is the purity of the individual constituents.

1.2.9. Reactivity of the Components

For the most part, the refrigerants used are very stable. However, even stable components may react with outside chemicals such as lubricants or other fluids. In inertion testing using ethylene oxide and R12 in the NMERI explosion sphere, a regular pressure decrease was noted prior to ignition. This was believed to be due to the ability of the ethylene oxide to polymerize. Reactivity is not considered to be a problem either in test or field situations and will be evaluated on a case-by-case basis rather than to conduct testing for each mixture.

1.2.10. Mixing of the Components

The ASTM E681 method requires stirring for at least 5 minutes to obtain complete mixing and thermal equilibrium, with final trials to be made at longer mixing times. Researchers in the flammable refrigerant field have reduced this requirement to 2 minutes for some testing. During initial inertion testing in the NMERI sphere, it was determined that repeatable results required thorough mixing of the fuel, air, and inertant. A electronic box fan, which created greater mixing than the mixing bar in the flask, was installed inside the sphere and allowed to run at least one minute to ensure proper mixing. Total mixing of all components is required in any test technique for consistency in the evaluation of flammability in a laboratory environment.

1.2.11. Altitude

Coward and Jones state that the normal variations of atmospheric pressures do not appreciably affect the limits of flammability[39]. However, the local atmospheric pressure at some locations may differ considerably from the 14.7 psia(760 torr) sea level value. For example, the average barometric pressure at Albuquerque, with an altitude in excess of 5000 feet, is approximately 12.0 psia (620 torr). All NMERI sphere tests prior to this program were conducted at 14.7 psia (760 torr), which required the addition of approximately 2.5 psi (17 kPa) additional air to the sphere to compensate for the altitude. This air had always been compressed air from the laboratory compressor system. For this program, the sphere or flask is fully evacuated and all air used in the testing comes from a cylinder of "zero-gas air", purified natural air consisting of 78.084% nitrogen, 20.946% oxygen, 0.934% argon, 0.033% carbon dioxide, 0.003% rare gases, and not more than 2 parts per million hydrocarbons. The ASTM method, which is performed at local atmospheric due to the requirement to rest the stopper on the top of the flask to allow proper venting of the explosion, does not accommodate additional pressure.

1.3. Refrigerant Properties

The three refrigerants of interest in this program—R32, R134a, and R125—have been considered as replacements for chlorine- and bromine- containing compounds in applications other than refrigerants. A NMERI-conducted program screened fluorinated halocarbon as replacements to Halon 1301 for application in fire and explosion protection in Alaskan North Slope oil and gas production facilities.[40]

1.3.1. Fire Extinguishment and Explosion Prevention Effectiveness

As part of the Halon 1301 replacement program, all three refrigerants were tested for fire extinguishment effectiveness in the NMERI cup burner apparatus, and for explosion prevention ability in the NMERI explosion sphere. Their effectiveness was ranked against Halon 1301 for fire extinguishment using n-heptane and for explosion prevention using both propane and methane as fuels. Tables 1, 2, and 3 report the NMERI-generated extinguishing (or inerting) concentrations for the cupburner and the explosion sphere. The gas volume equivalent (GVeq) is the ratio of the gas volume of refrigerant required to extinguish a fire normalized to a value of 1 for Halon 1301. Likewise, the weight equivalent (WEq) and the storage volume equivalent (SVEq) are the weight and volume requirements normalized to Halon 1301. The inerting concentration is the amount of refrigerant required to reduce the explosion overpressure to 1 psi (6.9 kPa) using a stoichiometric fuel-to-air ratio.

TABLE 1. FIRE EXTINGUISHMENT EFFECTIVENESS.

Refrigerant	Extinguishment % - volume	GVEq	WEq	SVEq
Halon 1301	2.9	1.00	1.00	1.00
R32	8.8	3.03	1.06	1.62
R134a	10.5	3.62	2.48	3.10
R125	9.4	3.24	2.61	3.19

TABLE 2. EXPLOSION PREVENTION PERFORMANCE EFFECTIVENESS, PROPANE.

Refrigerant	Inertion % - volume*	GVEq	WEq	SVEq
Halon 1301	4.3	1.00	1.00	1.00
R32	17.8	4.14	1.45	2.21
R134a	14.1	3.28	2.25	2.81
R125	14.7	3.42	2.76	3.36

*stoichiometric fuel-to-air ratio

TABLE 3. EXPLOSION PREVENTION PERFORMANCE EFFECTIVENESS, METHANE.

Refrigerant	Inertion % - volume*	GVEq	WEq	SVEq
Halon 1301	4.3	1.00	1.00	1.00
R134a	7.8	1.81	1.24	1.55
R125	9.7	2.26	1.82	2.22

*stoichiometric fuel-to-air ratio

1.3.2. Flammability

The refrigerant R32 is often referred to as “mildly flammable”.[41] While this may be partially true in that it is difficult to ignite using low energy ignition sources, it is definitely not true in terms of its explosion overpressure and temperature rise. NMERI has also tested a stoichiometric fuel-to-air mixture of R32 in the explosion sphere using the NMERI DC ignition source with 70 J stored energy. The explosion overpressure measured 103 psi (710 kPa), similar to propane and higher than methane, and the temperature rise inside the sphere was similar to that of propane. The terms “mildly flammable” or “marginally flammable” should be used with caution when referring to R32 (or other potential flammable refrigerants) because these terms might be misleading and infer that the potential for a damaging explosion does not exist when indeed it does.

Neither R134a nor R125 has been tested in the NMERI explosion sphere for flammability, although there is no reason to believe that either would be flammable under normal environmental conditions. The presence of two hydrogen atoms in the R134a molecule does indicate, however, a potential for flammability, and under extraordinary conditions it might prove flammable. The Du Pont Material Safety Data (MSD) sheet has been modified to include the following statement: "HFC-134a should not be mixed with air for leak testing. In general, it should not be used or allowed to be present with high concentrations of air above atmospheric pressure."[42]

1.4. Results of Tasks 1 and 2

Tasks 1 and 2 were performed and documented prior to beginning the testing. During Task 1, a search was made for references containing data on flammable refrigerants, general flammability, and ignition technology. The results of this search were incorporated into a Microsoft Access™ database, and an assessment of the state of the art of flammable refrigerant measurement technology was made and included in the task report.[43] During Task 2, the test plan for this testing was developed and documented.[44]

2.0 TEST APPARATUSES

Three separate apparatuses were used to conduct the testing on this project.

2.1. Bench Test

The first apparatus was developed to conduct bench tests on the electrical ignition systems and measure the energy in the spark gaps. A Plexiglas stand to hold the electrodes was constructed. One end of the stand was movable and attached to a micrometer which allowed the electrodes to be moved a precise distance apart. This apparatus was used to evaluate the shape of the ends of the electrodes, determine the minimum voltage required to initiate an arc, and to determine the energy in the spark gap for both the low and the high voltage sources. Figure 2 illustrates this apparatus as it was used to measure the energy in the spark gap.

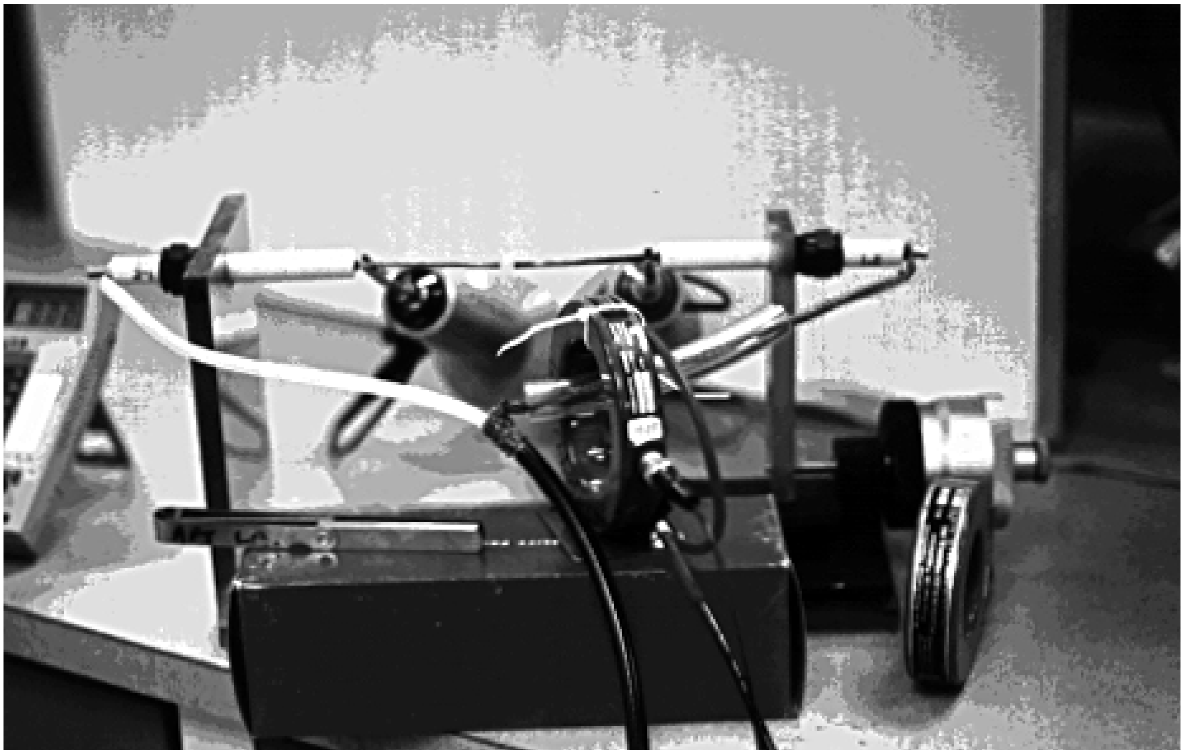

Figure 2. NMERI Electrode Simulator.

The measurement of the energy in the spark gap involved the use of two Tektronix P6015A high voltage probes, one on the end of each electrode. These probes measured the high frequency voltage data which, when integrated with the current data, provided the actual energy in the spark gap. Three different current probes, a Pearson, a Stanganese, and a Rogowski, provided the current data. The data was recorded on a four-channel Tektronix recording oscilloscope and stored in a format compatible with manipulation by standard spreadsheet software. The current and voltage for each side of the electrode was multiplied for each time increment and summed to give the total energy in the gap during discharge.

It is important to note that this methodology uses the only procedure to precisely determine the energy in the spark. The energy and power in the gap are measured only after the voltage in the gap collapses to 100 volts (the voltage between the electrodes has broken down and the arc occurs). Eventually, the voltage between the electrodes is reduced to a level such that the gap will not conduct, and the arc ceases to exist. This equipment was used to measure the energy in both the NMERI standard DC spark and the high-voltage DC spark using the system developed for this program.

2.2. 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 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 R32, R-152a, and R-142b as fuels. As part of this screening, rough 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. The difference in NMERI results, which is not significant for the work conducted here, was the subject of a paper presented in 1993.[45]

The NMERI explosion sphere[46] (Figure 3) was modeled after the Fenwal Explosive

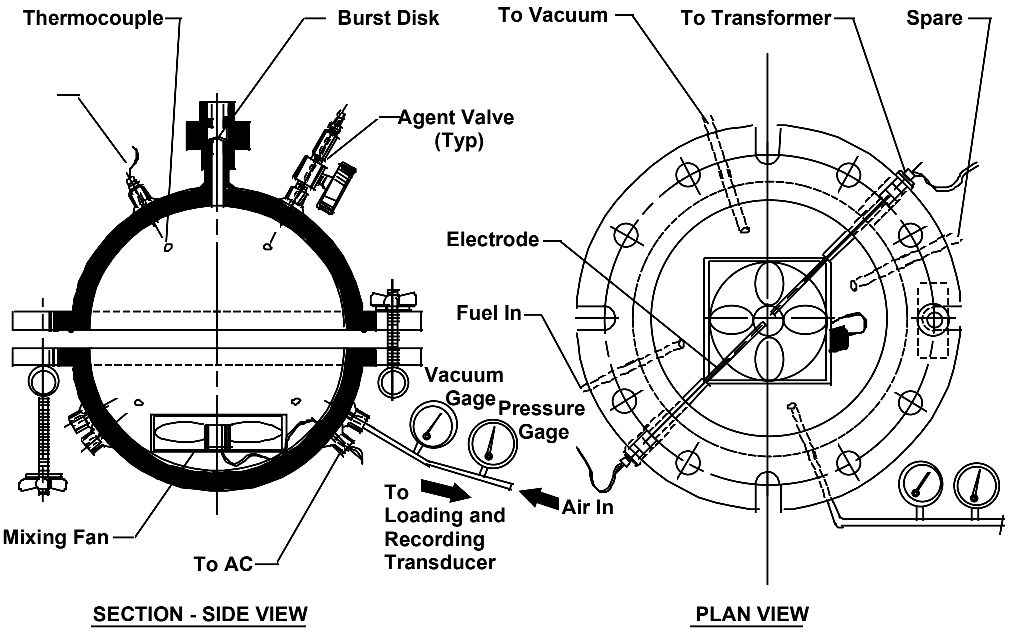

Figure 3. NMERI Explosion Sphere.

Sphere.[47] 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-(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 refrigerants, 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 gage, 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; various types of ignition sources were possible using these electrodes. These ignition sources will be detailed in the methodology section.

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 4 is a photograph of the sphere as set up for test.

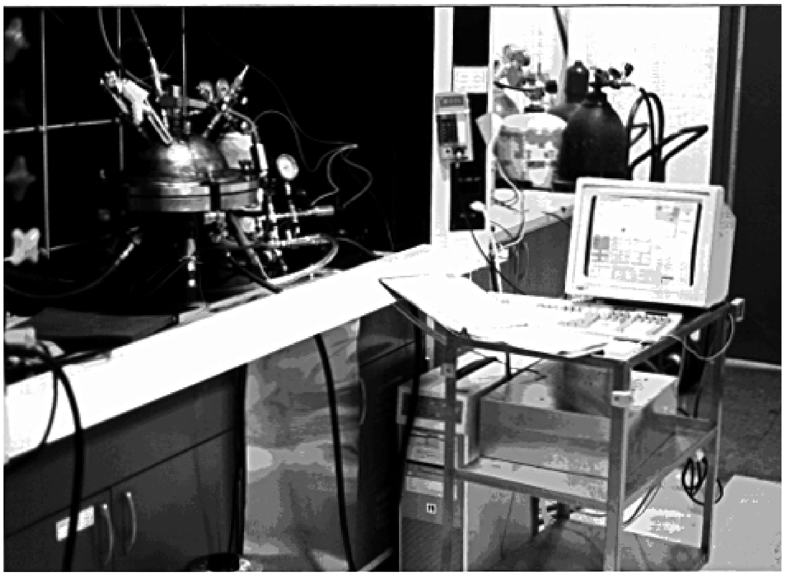

Figure 4. Explosion Sphere Setup for Test.

2.3. ASTM E681 Test Apparatus

A flask and test apparatus was constructed according to the specifications of ASTM E681.

2.3.1. 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 3/4 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 were flat, and the other pointed. In addition, the holder blocks constructed for the sphere could be installed to allow testing with the match. Both the thermocouples and electrodes were inserted in glass rods and epoxied on each end to prevent loss of vacuum. Figure 5 illustrates the interior of the flask.

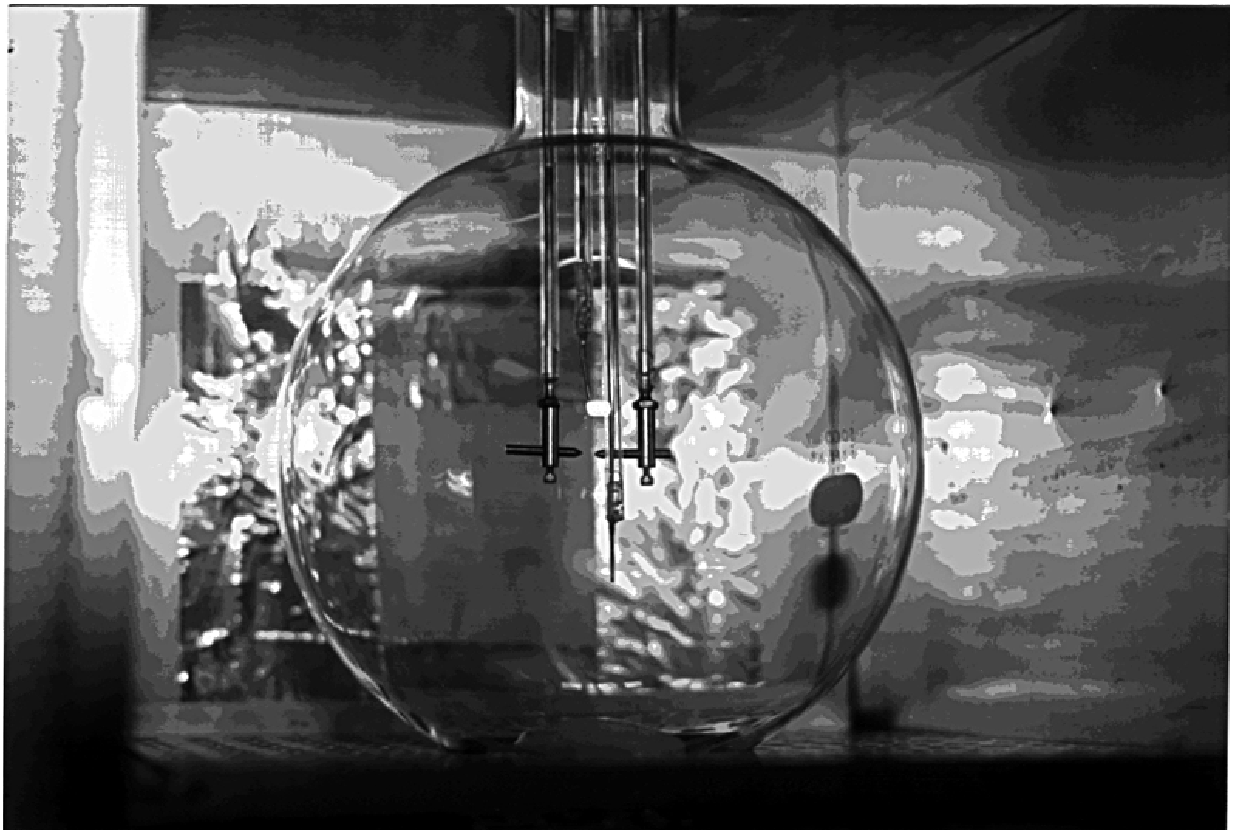

Figure 5. Interior of Flask.

2.3.2. Enclosure

An existing laboratory oven was modified in lieu of fabricating an insulated chamber. 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 to supplement the oven's heating element. This was chosen instead of blowing heated air into the chamber as a means of better controlling the variations of the temperature. 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 modified to extend into the oven area and position the stirrer magnet just below the flask. Figure 6 shows the enclosure and flask assembly.

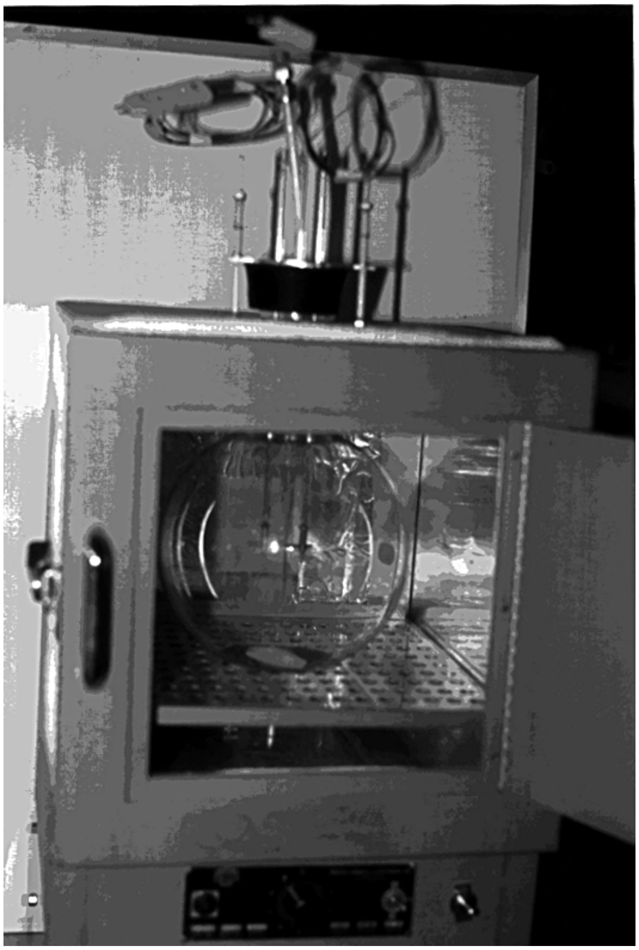

Figure 6. ASTM E681 Enclosure and Flask.

2.3.3. Manifold

The fifth penetration in the cover was for the gas inlet and exhaust. Because of the limited space on the test vessel cover, all penetrations transducers were moved to the manifold which 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 gage; and (6) an exhaust to ambient air. All components but the overpressure transducer and the house vacuum exhaust were located to the side of the enclosure. These two components 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. The flexible hose allowed sufficient movement for the test vessel cover to be rotated for installation of the flask. The R32, R134a, and R125 inlet lines were connected to three of the four inlet ports (the fourth was designed for an additional fuel such as propane), and the bottled air was connected to the air fitting. When testing moved from the flask to the sphere, the manifold was kept intact except for the installation of the overpressure transducer and house vacuum on penetrations on the top of the sphere. While this was a slightly different configuration than earlier sphere tests, it did not affect results.

2.4. Ignition Methods

There are several different types of ignition sources for determining the explosion limits of mixtures of materials that range from dust-air mixtures, to propane and other flammable gases, to refrigerants. Current methods of ignition include match heads, electrical discharges (both AC and DC), exploding wires, and heated wires. While match heads provide a very energetic ignition, their potential energy can vary from match to match, they introduce foreign products of combustion into the reaction, and their brightness can obscure the flammability effect. Heated or exploding wires do not give very reproducible results. Electrical ignition sources, on the other hand, are more definable, and, therefore, ignition is more repeatable from test to test than with other methods.

The electrode geometry is an important consideration when testing materials. Before the arc occurs, the resistance of the air must be broken down. Three major geometries are considered. All of them use a round stainless steel rod with only the ends differing in shape. The shape of the ends affects the breakdown of the air gap.

The first type of electrode has a flat end (Figure 7) which is a standard geometry. This type of electrode will produce less predictable results that the other two types of electrodes because of the flat surface and the relative sharp edges at the ends of the rods. There will be electrical field enhancement at the edges but none on the face (except due to the non-smooth surface).

Figure 7. Flat Type Electrode.

The second type of electrode is the hemispherically shaped rod (Figure 8). The ends of this type of electrode should be made so that the radius of the end of the electrode is the same as the radius of the body of the electrode. This type of rod eliminates field enhancement so that breakdown of the gap occurs more uniformly.

Figure 8. Hemispherical Type Electrode.

The third type of electrode is the conical electrode (Figure 9). This rod is specifically designed to provide field enhancement at the tips forcing a breakdown to occur more often at the tips. The conical electrodes in these tests had their ends machined at 45 degrees.

Figure 9. Conical Type Electrode.

2.4.1. Direct Current (DC) Electrical Arcs

Electrical ignition sources can be well defined in terms of the ignition circuit components and, therefore, can produce electric arcs of repeatable energy. Since the electrical components are typically "off the shelf," different researchers can test using similar test equipment. While there are many different categories of DC discharge circuits, only two different types are considered here. Both are capacitive discharge circuits driving a pair of electrodes with a specified electrode spacing. The difference between them is the voltage at which the capacitors are charged. Low voltage is typically considered as having a capacitor charging bank voltage (V_{bank}) of under 500V, while high voltage is considered as having a V_{bank} of over 500 V.

Both the low and high voltage sources have advantages and disadvantages. Figure 10 illustrates a typical circuit schematic diagram of either type. The electrical ignition used in this testing consisted of two entirely different configurations, one each in the low voltage and high voltage region.

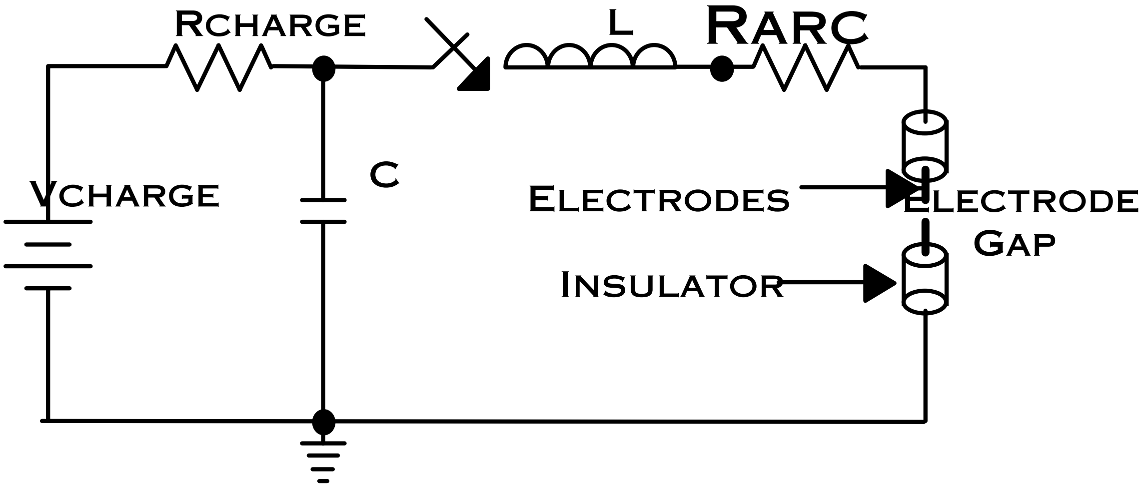

Figure 10. Typical Discharge Circuit.

(1) Low Voltage Ignition Source. The low voltage source is typically inexpensive. The physical layout of the circuit design can be minimized while achieving high energy storage. The safety of the operator is also maximized due to the low operating voltage. The disadvantage of this circuit is the requirement of a step up transformer to increase the electrode gap voltage sufficiently to cause the gap to break down. A typical oil burner or illumination ignition transformer presents problems due to saturation of the core material causing a lower output voltage, and energy lost in the transformer core material. Using non-metallic materials for the core will minimize this effect.

The low voltage configuration was a capacitive discharge using a NMERI-designed Mutual Inductance Particle Velocity (MIPV) box (Figure 11). This box contained three 2000-mF capacitors connected in parallel (approx. 7000 mF measured), a 1.3 mH series inductance, and a relay control interface. The output switch is an ECG5548 silicon controlled rectifier (SCR). It had an interface to the computer which allowed the computer to charge the capacitors to the correct voltage and discharge the capacitors when required.

The bank is charged to the appropriate voltage (up to 200 V) to achieve the desired stored energy and discharged through a Franceformer® ignition transformer (model LA4V) to the electrodes. The conical electrodes were used due to their more reliable ignition performance. The advantage of this system is a low voltage pulse is sent to the transformer, and only a short run of automobile ignition wire with the high voltage pulse is present. While safety was a factor in this configuration (the capacitors stored up to 100 joules energy), no special precautions were required.

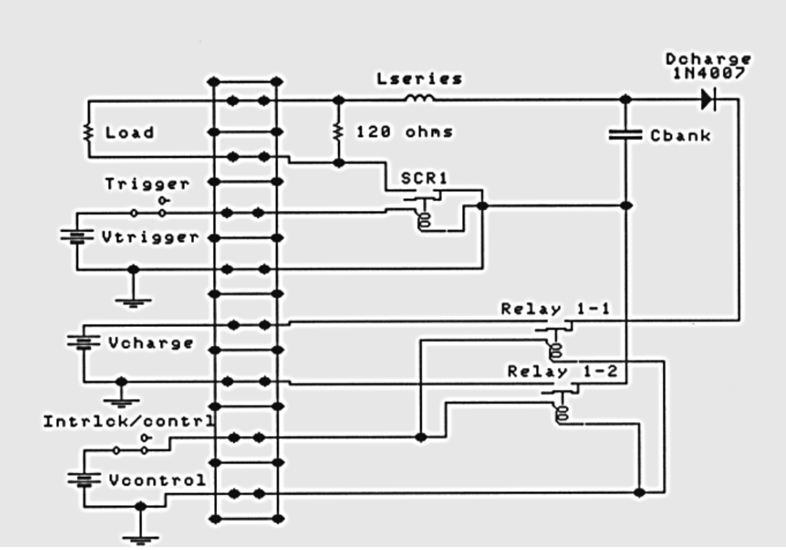

Figure 11. Mutual Inductance Particle Velocity (MIPV) Box Schematic.

(2) High voltage Ignition Source**.** The high voltage source is typically more expensive due to the materials involved in the circuit design. Additionally, because of the greater energy stored in the capacitors, the components are physically larger and physical layout must be a priority. The higher voltages require greater care when operating, both for human and equipment safety. High voltage should only be handled by specially trained personnel. There are, however, several advantages to this type of circuit, the first being that higher charging voltages give higher stored energy with smaller capacitors based on

$$E = \frac{1}{2} F V^2 \tag{1}$$

where E = stored energy (in joules), F = capacitance (in farads), and V = charging voltage (in volts). This smaller capacitance can be combined with smaller inductances to provide short discharge times. This results in higher peak discharge currents so that the peak power is greater while the stored energy is the same as the low voltage source.

$$P = \frac{E}{t} \tag{2}$$

where P = power (in watts), E = energy (in joules), and t = the time that energy is flowing (in seconds). The main electrical disadvantage to this circuit is that if long discharge times are desired, physically large and costly inductors are necessary.

The high voltage ignition system developed for this testing consisted of two separate configurations. The first consisted of a standard 1 mF capacitor bank and a series inductance and a series inductance of approximately 6 mH. The second configuration, which was used in the refrigerant testing, added a 2.5 W series wirewound resistance to elongate the discharge pulse. No step-up transformer was necessary due to the capacitor ratings, and, therefore, there are no transformer losses. Experimental diagnostics were sufficient to determine gap voltage, discharge current, gap power, and gap energy.

While the ignition was simple in terms of the number of components, it required large and expensive parts. The four capacitors were approximately 0.245 μF and were charged by a Glassman constant voltage/current power supply with a peak voltage of 20 kV and peak current of 15 mA. Two high-voltage relays were used, one for discharging the capacitors across the load and one for an emergency "dump" in case the capacitors could not be discharged through the load.

Safety was of paramount concern with this configuration. The system was enclosed in a 3-ft (1 m) plywood cube, with all high-voltage components mounted on Plexiglas and separated for safety. All wiring was high-voltage wires. After all tests, the capacitors were grounded using a grounding rod, and were grounded between tests to prevent buildup of charge.

2.4.2. Alternating Current (AC) Electrical Arcs

An ignition device similar to that outlined in ASTM E681 was constructed to provide an alternative to the DC sources above. A single-shot timer was constructed using a 120/140-V solid state relay (Potter and Brumfield SSRT-240D25), and an integrated timer chip (RCA CA555CE). Additionally, an 1100 μF, 30 VDC tank capacitor was used for power filtering, and a 150 microfarad ceramic capacitor, a precision 1000 ohm resistor, and a variable resistor (Trimpot 3009P-1-203) formed the timing circuit. A similar multiple-shot circuit was constructed on the same circuit board but was not used. The timer could provide a pulse length between 0.09 to 2.62 seconds, and was set at 0.208 seconds as measured on an oscilloscope. This period was not changed throughout the testing. Any similar timer circuit which can accurately control AC can be used for this function.

The AC is boosted in voltage through the same transformer used in the NMERI standard DC spark and transmitted through automobile ignition wires to the electrode rods. The AC was manually activated by a pushbutton during the data acquisition time interval. The box holding the timer board was approximately the size of a thick paperback book, and the only other components were the wires and plugs to connect the timer to AC and the transformer. Since only 110 V current flowed up to the transformer, no special safety precautions other than those normally required for handling 110 V were required, in contrast to the stringent regulations placed on the high-voltage DC.

2.4.3. Matches

Matches have long been used as an ignition source in refrigerant flammability testing. While a very energetic source, the match energy is not reproducible and the matches introduce extraneous by-products to the combustion process. In addition, the light from the match often obscures the actual ignition.

The experimental procedure using a match head was similar for both the sphere and the flask. Two stainless steel blocks were constructed to slide over the ends of the electrodes and were attached by a set screw. A hole the size of the match stick was drilled into one block. The match, an “Ohio Bluetip” strike anywhere, was cut off just below the flammable material and inserted into the hole. A 4½-in (11.4-cm), 7.95-mil diameter Nichrome wire was wrapped around the set screw of the block twice, around the match three times, and around the second set screw twice, connecting the wire across the spark gap. A 22.5-V battery was connected to each outside ends of the electrodes, heating the wire and igniting the match.

2.4.4. Heated Wire

A technique similar to that developed to ignite the match was developed for the heated wire. Instead of inserting the match in the block, the same type and length of wire was wrapped around each of the set screws and the voltage applied. In some cases the wire broke, while in others it glowed brightly but remained intact.

2.4.5. Exploding Wire

Experiments with AC current using a single strand of copper wire across the gap were conducted on the bench test simulator. The resulting explosion caused a bright light—too intense to view—, a very loud noise, and pieces of copper to fly outward. It was determined that the light was too intense for visual recording of the event, and this technique was not used during any testing.

2.5. Instrumentation

A new data acquisition system and accompanying software were developed for this program. The goal of the new system 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.

2.5.1. 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. A major advantage of this technique is that the program can be developed on one computer and an executable program exported to any other windows-compatible computer. With the appropriate data boards, the computer has the capability to run all functions within the program. Specific code for this application was written in C language.

2.5.2. 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 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.

3.0 TEST METHODOLOGY

The procedures and conditions under which testing is conducted may be as critical in determining final results as is the equipment. Knowledge of the procedures by which the refrigerants and air were introduced into the test apparatus and mixed, techniques of cleaning the apparatuses between tests, and data interpretation are all potential factors which could have a great deal of influence on the determination of whether a test is flammable or not.

To aid in the understanding of the methodology employed in this program, the operating procedures for the data acquisition process, the NMERI explosion sphere and the ASTM E681 flask are included as Appendices A through C. These appendices explain in detail each step required to set up and conduct the tests and record data in each of the two devices and provide insight into the operation of the equipment and environmental state of the refrigerants at the time of ignition.

3.1. Data Reduction

Results are presented as a function of flammability (yes or no—see 3.2 for criteria) or overpressure with respect to the concentration of the refrigerant. The total concentration of refrigerant, either a single component of propane or R32 or a blend of R32 and other refrigerants, is reported as a volume percent of the total volume of the mixture. For example, a 20% concentration of a R32 and R134a blend would correspond to 20% R32/R134a and 80% air by volume. The amount of flammable refrigerant within that blend is expressed as a weight percent of the total refrigerant. Using the above example, a 57% concentration of R32 would correspond to 57% of the blend *by weight* of R32. This corresponds to a 72.2% concentration of R32 *by volume*. The use of weight percent to report the concentration of R32 follows the protocol established by other researchers. Since the concentrations of all components are measured and loaded by volume percent, the desired weight concentration of R32 is first converted to volume percent by using the molecular weights prior to entering the data on the computer screen.

3.2. Definition of Flammability

The definition of what constitutes a flammable test is one of the areas of uncertainty within the ASTM E681 method. For this program, a definition of flammability determined by the degree to which the flame propagates within an imaginary frustum of cone within the flask is used. The flammability of a test is rated in six classes according to a scale from 1 to 6 (Figure 12), according to the protocol of N.D. Smith. The dashed line indicates the outline of the cone extending from the edges of the electrodes to the edge of the neck of the flask. A test was considered flammable if the flame extended beyond the boundaries of that imaginary cone. Classes 1 and 2 are non-flammable, and the remainder are considered flammable.

(1) A Class 1 test either does not propagate a flame or propagates flames upward but not as high as the neck of the flask. A percent of the distance from the electrodes to the top of the flask that the flame progresses in percent is given to indicate a degree of flame spread.

(2) A Class 2 test propagates the flame upward to the top of the flask but the flame does not spread once it reaches the neck. It can extend up into the neck, however.

(3) A Class 3 test propagates flame beyond the cone. Flame cannot be seen below the height of the electrodes.

(4) A Class 4 test is similar to Class 3 but the flame reaches the level of the electrodes.

(5) A Class 5 test propagates flame below the level of the electrodes but does not completely fill the flask.

(6) A Class 6 test propagates flames below the electrodes immediately after ignition.

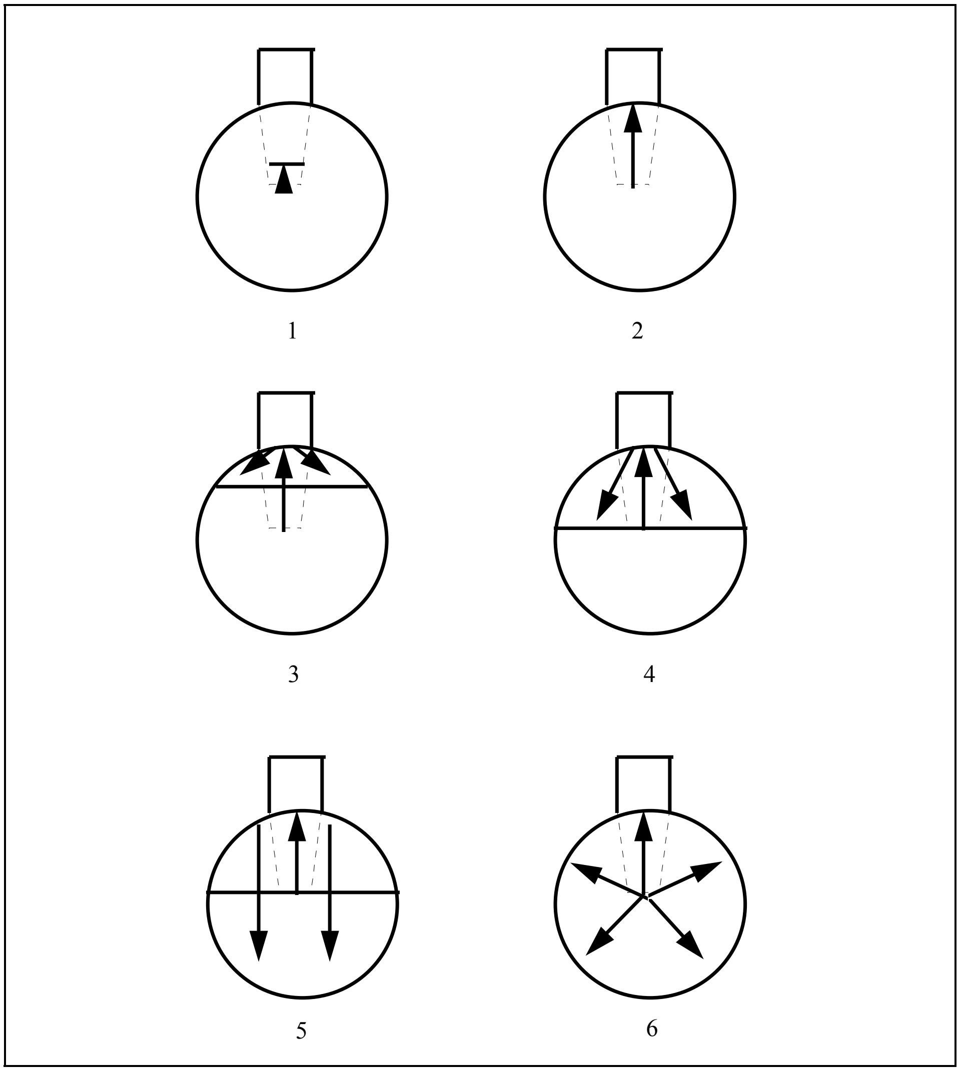

Figure 12. Flammability Classes.

Of the many tests run in the flask, only a few were considered flammable enough to be considered a Class 4 test. No tests were flammable enough to propagate flames below the level of the electrodes or downward immediately after ignition.

4.0 TEST RESULTS

The testing was accomplished in several phases dependent upon the apparatus used. During Phase 1 ignition bench testing, different configurations of DC ignition sources were screened and the energy contained in these ignition sources measured. Phase 2 testing in the explosion sphere measured the flammability limits of both propane and R32 and determined the flammability characteristics of R32 in binary and ternary blends. Ignition parameters, internal starting pressure, air humidity, and refrigerant concentrations were varied. Phase 3 testing in the ASTM E681 flask and insulated chamber involved repeating many of the tests run in the sphere with the added capability of having the mixture heated to 100 °C (212 °F).

4.1. Ignition Testing

4.1.1. Electrode and Spark Gap Evaluation

A series was conducted on the bench simulator to determine the relationship of electrode shape and spark gap separation, as well as other factors, on the ability of the arc to bridge the spark gap. Several other factors, such as the length of the wires between the transformer and the source, were investigated.

Table 4 lists the minimum voltage for each electrode shape and separation for which the spark was generated. At close separations, the voltage required does not vary much and the pointed electrodes require greater voltage. For the other spacings, including the 0.240-inch (6-mm) spacing used in previous NMERI testing, the pointed electrodes require less voltage than the other shapes. Based on these results, the pointed electrodes were used in both the sphere and the ASTM flask for all testing.

TABLE 4. REQUIRED VOLTAGES TO GENERATE ARC (VOLTS).

Electrode Shape	Electrode Separation (in/mm)			
	0.025/0.64	0.240/6.0	0.500/12.7	0.700/17.8
Round	26	103	135	153
Square	23	73	111	151
Pointed	27.5	70	97	145

It was also very apparent that the electrodes required cleaning between tests as after 2 to 3 tests, the arc failed to strike at voltages where it had struck the previous test. When the electrodes were lightly sanded with fine sandpaper, the arc struck again for several tests until the procedure had to be repeated. Therefore, when the low-voltage DC ignition source is used, especially at low voltage levels, the electrodes should be sanded between each test.

One other variable investigated during this series was the length of the wire between the output of the MIPV box and the transformer. The previous NMERI setup involved a long run (over 40 ft (13 m)) of power line. A comparison of this long wire run with a shorter (3 ft [1 m]) wire indicated that the longer run requiring 80-82 volts to strike the arc, while the shorter run required only 73-76 volts. This indicates that the connections between all components should be minimized for optimum results.

4.1.2. Energy Measurement

Prior NMERI testing used an ignition method in which capacitors were charged to a low voltage (less than 200 volts) and then boosted in voltage by the use of a step-up transformer. The energy content in the spark was considered to be the energy contained in the capacitors by the formula $E=\frac{1}{2}FV^2$, less a factor of 0.85 for the transformer. Because of the difficulty in measuring high voltage, the energy content in this spark had never measured, and it became a priority of this program to measure the energy actually in the spark. The technique developed involved the measurement of current and voltage each side of the spark gap.

The energy present in the gap due to the standard NMERI source has been analyzed and plotted (Figure 13). In general, the NMERI DC spark contained a measured energy of between 0.15 and 0.26 J for charging voltages of 125, 168, and 198 volts. These correspond to a stored energy of 27.4, 98.7, and 137.2 J, based on the actual measurement of circuit components, resulting in an efficiency of between 0.2 and 0.5 percent. The major component of this loss is believed to be saturation of the iron core in the transformer, which passes energy effectively over long time periods (such as AC) but which cannot pass a rapid pulse.

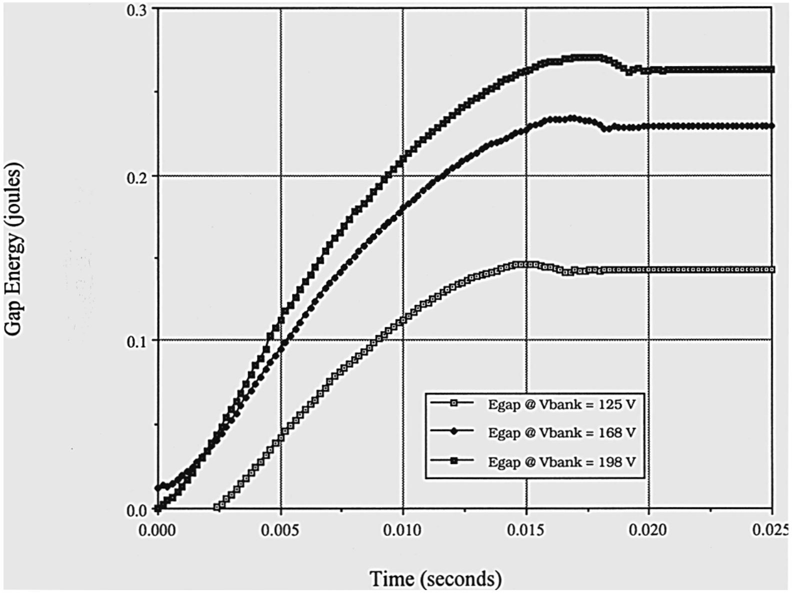

Figure 13. Gap Energy vs. Voltage, Standard NMERI DC Source.

The measured energy in the high voltage source was considerably higher, ranging from 3 J at 7.5 kV to 10 J at 17.5 kV (Figure 14). The stored energy corresponding to these values range from 28J to 153J. Although this energy is still a small percentage of the total stored energy (6.5 - 10 %), it is nonetheless an order of magnitude higher than the NMERI source. These values should give an indication of the energy actually present in the spark gap for ignition sources similar to these, and assist in evaluating the effect of energy on the ignition of refrigerant.

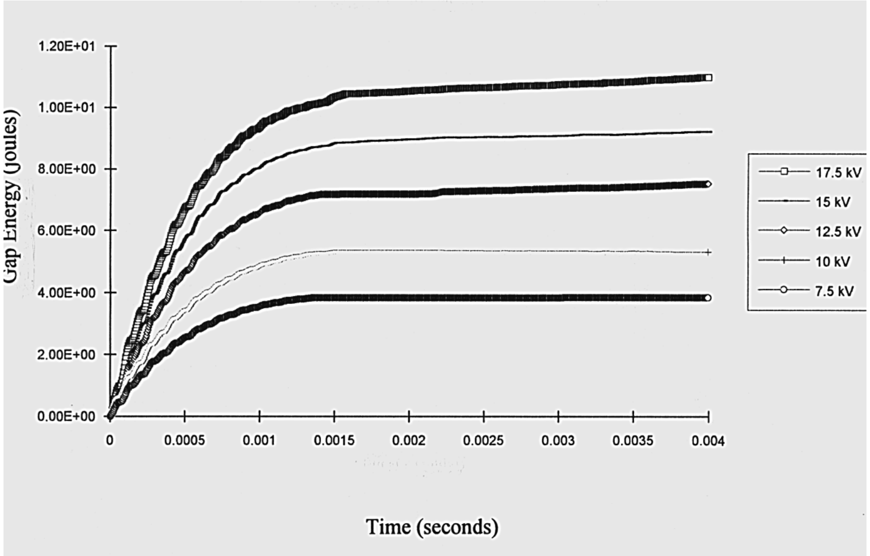

Figure 14. Gap energy vs. Voltage, High Voltage Source.

This high voltage source was used in flask testing on tests 32 through 36. The computer was turned off and all connections (thermocouples and wires) were disconnected for safety reasons, the explanation for why there are no overpressure or temperature data for these tests. Tests were successfully conducted using 7.5 kV, 10 kV, and 12.5 kV. However, when using 15 kV, the spark occurred external to the flask (no arc was seen in the video, although there was a bright flash out of the vision of the camera). The loading transducer experienced some of this high voltage and was damaged. Testing using this source was immediately discontinued at this point. Additional research is required to make the entire system safe for this type of voltage. It is interesting, however, to view the videos of tests 32 through 36 and observe the difference between ignition using this source and others, remembering that this was a lower concentration of R32 (47.5 weight percent).

4.2. Sphere Testing

Two separate test series were conducted in the NMERI explosion sphere. The first, run before the flask was operational, was used to evaluate the lower and upper flammability limits of propane as a comparison with previous results run in the sphere. The upper and lower flammability limits of R32 were also determined to provide a guide to the concentrations required during the flask testing. Tests were also conducted using the various ignition sources to evaluate their effects on the flammability of both propane and R32.

4.2.1. Propane Flammability Limits

The flammability limits of propane were determined for different ignition sources and starting conditions. Test results are reported in Table D-1 in Appendix D. The commonly accepted LFL and UFL for propane are 2.12% and 9.35% respectively.[48] The lower and upper explosion limits of propane were investigated using the NMERI DC spark at 70 joules stored energy, the electrically ignited match, and the heated wire (Table 5). Two criteria for flammability were used: a high value of 1 psi (6.9 kPa), which was used in the past, and a low value, 0.3 psi (2.1 kPa), which corresponds to the visual criteria for flammability using the ASTM E681 flask.

TABLE 5. UPPER AND LOWER EXPLOSIVE LIMITS FOR PROPANE BY IGNITION SOURCE AND STARTING PRESSURE (SPHERE).

Source	LFL (vol %) (high psi criteria)	UFL (vol %) (high psi criteria)	LFL (vol %) (low psi criteria)	UFL (vol %) (low psi criteria)
NMERI Spark (14.7 psia [760 torr])	2.38-2.4	10.4 - 10.5	2.38-2.4	10.5
NMERI Spark (12.0 psia [620 torr])	2.19-2.27	10.18-10.2	2.19-2.27	10.4
Match (14.7 psia [760 torr])	1.6	10.0	1.55	see text
Match (12.0 psia [620 torr])	not tested	9.4-9.7	not tested	9.7
Wire (14.7 psia [760 torr])	2.43-2.51	10.5-10.77	2.43-2.51	10.9
Wire (12.0 psia [620 torr])	2.15-2.2	9.52-9.75	2.1	9.6

The lower flammability limits are almost identical between the two criteria because the transition between non-flammable and flammable occurs within a narrow band of concentrations. The upper flammability limits are slightly higher for the lower overpressure criteria.

a. Spark

The LFL is well defined at about 2.4%, while the UFL is about 10.5%. There is no pressure rise due to the ignition of the spark itself.

b. Match

Ignition of the match in air develops an overpressure in the range of 1.0 psi (6.9 kPa) (tests P35-P36). In a vacuum, which simulates the condition at the UFL whereby the amount of oxygen is limited, the match develops an overpressure of between 0.01 and 0.1 psi (0.069 to 0.69 kPa) (tests 32R21 and 32R22, Table D-2). Therefore, the LFL was determined using the 2.0 psi (13.8 kPa) or 1.3 psi (8.9 kPa) criteria, since 1.0 psi (6.9 kPa) can be attributed to the match alone, and resulted in a value of 1.6% or 1.55%. On the other hand, since the overpressure caused by match ignition at the UFL resulted in an small overpressure, the 1.0 psi (6.9 kPa) or

0.3 psi (2.1 kPa) criteria were used. It is believed that the match, in the absence of oxygen at the propane UFL, initially burns using the oxidizer in the match, but cannot burn once that oxygen is exhausted. Therefore, using a pressure criteria to measure flammability may be incompatible with the use of a match head as an ignition source.

c. Wire

The wire itself generated an overpressure in the range of 0.03 to 0.1 psi (0.207 to 0.690 kPa) (Tests P-56 through -65). This variance is probably due to the length of time the wire heated prior to burning through. If voltage was held on the wire for an extended time, it is believed that this variance would disappear. The LFL is very well defined at 2.4 to 2.5% for both the 1 psi (6.9 kPa) and 0.3 psi (2.1 kPa) criteria, similar to what was seen with the spark. Overpressure at the LFL is similar to that of the spark as well.

Tests were also run with the three ignition sources at the local atmospheric pressure (approximately 12.0 psia [620 torr]) instead of the 14.7 psia (760 torr) used for previous testing. The results indicate lower LFL and UFL for all three ignition sources for the tests with a local atmospheric starting pressure. Note that the number of tests with the wire and match were limited, and may have some affect on the final values. To achieve a local atmospheric starting pressure, the fuel must be loaded in a partial vacuum. The loading transducer, while not designed for measuring negative pressures, was calibrated against a water manometer and found to vary less than 2% from the manometer; thus it is not believed that the equipment contributed to these differences.

Tests were run at a constant propane concentration (10.4%), using starting pressures of 16, 18, and 22 psia (828, 931, and 1137 torr) (Tests p-83 through -85). This necessitated using the overpressure transducer to load the propane, as these pressures are well over the safety limit of the loading transducer. The overpressure transducer has not been precisely calibrated at these pressures, so the final concentrations could vary somewhat. The overpressures were 5.8, 11.08, and 13.7 psi (40, 76, and 94 kPa), for the three starting pressures, as compared to approximately 2.6 psi (18kPa) using a 14.7 psia (760 torr) starting pressure and less than 1 psi (6.9 kPa) using a local atmospheric (12.0 psia [620 torr]) starting pressure.

4.2.2. R32 Flammability Limits

The LFL and UFL of R32 were measured using various starting pressures and the DC spark, wire, match and AC spark ignition sources (Table D-1). The two tests using the wire (32R11 and 32R12) failed to ignite R32 at 15 and 17% by volume, well within the flammability limits, and this source was not used in any further R32 testing. The LFL and UFL were 14.5-15% and 26.5-27%, respectively, for the spark and under 6% and 29% for the match. Note again that the number of samples was low for all tests. The match ignited the R32 at extremely low concentrations, but it is difficult to separate the contribution of the match alone to the pressure rise.

4.2.3. R32 Blend Testing

Following the flask testing, R32 binary and ternary blends were tested in the sphere (Table D-2) as a comparison to the flask results. Both low and high humidity air was used, and several tests were run using starting pressures other than local atmospheric.

4.3. ASTM E681 Flask Testing

A total of 128 tests were run in the ASTM E-681 flask (Table D-3). Of these, 30 involved testing at 100 °C in the heated enclosure.

4.3.1. Interpretation of Video Data

A tape containing videos of 18 separate tests was mailed to researchers in the field of flammable refrigerants to solicit their input on whether tests were considered flammable. All researchers agreed on which tests were and were not flammable, and their comment aided in developing a consistent protocol for determining flammability. Table 6 presents the results of all flask tests sorted by overpressure in ascending order and ranked for flammability by the criteria of Section 3. The two tests using match head ignition cannot be included in the analysis due to the overpressure developed by the match head alone. Test where significant venting occurred are denoted by a "+" after the overpressure, and since most tests over 1 psi (6.9 kPa) resulted in some venting, the overpressures in this range are not precise

4.3.2. Comparison of Video and Overpressure Data

The relationship between visual indicators of flammability and overpressure can be further understood by providing a description of the events seen during an explosion for a specific overpressure level. Two different scenarios may occur, depending whether the explosion occurs near the LFL or the UFL. As the UFL is approached, a very bright, yellow flame appears. Even at concentrations greater than the UFL value, a bright "globe" appears surrounding the ignition point. Although this globe does not rise, it could give a mistaken impression of flammability. The other scenario occurs as the LFL is approached or as the non-flammable refrigerant is increased, approaching the CFR. The flame is blue and not nearly as bright as the previous case. However, it does appear that the behavior of the flame is about the same in both cases, and the description of flame characteristics vs. overpressure will be assumed the same.

The overpressure range has been broken into six very loosely-defined categories. For each category, a general description of the explosion event is provided.

4.3.2.1. Less than 0.3 psi (2.068 kPa). The flame forms around the ignition, and may or may not rise. In no case, however, does the flame extend beyond the imaginary cone and generally dissipates before reaching the neck of the flask or, rarely, in the neck.

4.3.2.2. 0.3 psi (2.068 kPa). The flame fills the cone and begins to spread along the top of the shoulder of the flask. The flame is pale blue and uniform, and begins to become faint

TABLE 6. FLASK TEST RESULTS.

Test Number	Components	Overpressure (psi/kPa)		Flammable?	Flammability Class	Class 1 Height	Comments*
50	32/134a	0.000	0.000		1	75	
80	32/134a/125	0.000	0.000		1	0	
115	32/134a/125	0.007	0.048		1	0	
13	32	0.019	0.131		1	0	
28	32/134a	0.022	0.152		1	0	
41	32/134a	0.038	0.262		1	25	
122	32	0.038	0.262		1	0	
12	32	0.044	0.262		1	0	
69	134a	0.049	0.303		1	0	
114	32/134a/125	0.052	0.338		1	0	
11	32	0.07	0.483		1	0	
31	32/134a	0.071	0.489		2		
10	32	0.072	0.496		1	0	
123	32	0.076	0.524		1	0	
77	32/134a/125	0.085	0.586		1	25	
117	32/125	0.087	0.600		1	50	
76	32/134a/125	0.099	0.683		1	50	
9	32	0.100	0.689		1	25	
75	32/134a/125	0.101	0.696		1	0	
113	32/134a/125	0.105	0.724		1	25	
93	32	0.112	0.772		1	25	
124	32	0.119	0.820		1	0	
125	32	0.119	0.820		1	0	
74	32/134a/125	0.131	0.903		1	0	
8	32	0.141	0.973		1	50	
100	32/134a	0.145	1.000		2		
72	32/125	0.147	1.013		1	75	
68	32/134a	0.153	1.054		1	25	
111	32/134a/125	0.154	1.062		2		
121	32/125	0.154	1.062		1	75	
118	32/125	0.160	1.103		1	25	
1	32	0.164	1.131	x	3		anomaly
83	32/134a/125	0.165	1.138		2		
78	32/134a/125	0.168	1.158		2		
71	32/125	0.173	1.193		1	75	
95	32/134a	0.173	1.193		2		
19	32/134a	0.179	1.234		1	75	
108	32/125	0.184	1.268		2		
101	32	0.186	1.284		2		
103	32/125	0.196	1.351	No video			
40	32/134a	0.197	1.358		2		
49	32/134a	0.218	1.503		1	25	
126	32	0.224	1.544		1	0	

TABLE 6. FLASK TEST RESULTS (CONTINUED).

Test Number	Components	Overpressure (psi/kPa)		Flammable?	Flammability Class	Class 1 Height	Comments
7	32	0.231	1.592		2		match
67	32/134a	0.241	1.661		1	50	
96	32/134a	0.244	1.682		2		
39	32/134a	0.251	1.730		2		
22	32	0.301	2.075		2		
120	32/125	0.301	2.075		2		
70	32/125	0.303	2.089		2		
4	32	0.314	2.165	x	3		
55	32	0.332	2.289		1	0	anomaly
21	32/134a	0.335	2.309	x	3		
63	32/134a	0.347	2.392	x	3		
110	32/134a/125	0.359	2.475	x	3		
66	32/134a	0.36	2.482	x	3		
107	32/125	0.384	2.647	x	3		
98	32/134a	0.386	2.661	x	3		
128	32	0.409	2.820			2	
24	32	0.414	2.854	x	3		
92	32	0.452	3.116	x	3		
112	32/134a/125	0.472	3.254	x	3		
81	32/134a/125	0.49	3.378	x	3		
30	32/134a	0.508	3.502	x	3		
25	32	0.587	4.068	x	3		
102	32/125	0.590	4.069	x	3		
15	32/134a	0.637	4.391	x	3		
99	32/134a	0.637	4.391	x	3		
26	32/134a	0.740	5.102	No video			
6	32	0.778	5.364		1		match
119	32/125	0.781	5.384	x	3		
91	32	0.782	5.391	x	3		
3	32	0.829	5.715	No video			
17	32/134a	0.845	5.825	x	3		
20	32/134a	0.873	6.018	x	3		
43	32/134a	0.921	6.349	x	3		
56	32	0.968	6.673	x	3		
105	32/125	0.985	6.791	x	3		
48	32/134a	0.991	6.832	x	3		
85	32/134a	1.014	6.991	x	3		
51	32/134a	1.02	7.032	x	3		
94	32/134a	1.045	7.204	x	3		
45	32/134a	1.046	7.211	x	3		
44	32/134a	1.096	7.556	x	3		
86	32/134a	1.100	7.583	x	3		
5	32	1.114	7.680	x	3		
29	32/134a	1.133	7.811	x	3		

TABLE 6. FLASK TEST RESULTS (CONCLUDED).

Test Number	Components	Overpressure (psi/kPa))		Flammable?	Flammability Class	Class 1 Height	Comments
47	32/134a	1.146	7.901	x	3		
14	32/134a	1.151	7.935	x	3		
38	32	1.175	8.100	x	3		
65	32/134a	1.199	8.266	x	3		
23	32	1.203	8.293	x	3		
46	32/134a	1.208	8.328	x	3		
16	32/134a	1.215	8.376	x	3		
79	32/134a/125	1.22	8.411	x	3		
90	32/134a	1.766	12.175	x	3		
37	32/134a	2.328	16.049	x	3		
64	32/134a	2.835	19.544	x	4		
54	32	.882+	6.081+	x	3		
57	32	.954+	6.577+	x	3		
127	32	0.994+	6.853+	x	3		
87	32/134a	1.107+	7.632+	x	3		
60	32/134a	1.185+	8.169+	x	3		
109	32/134a/125	1.234+	8.507+	x	3		
58	32/134a	1.285+	8.859+	x	3		
116	32/125	1.286+	8.866+	x	3		outlier
27	32/134a	1.306+	9.004+	x	3		
42	32/134a	1.405+	9.686+	x	3		
97	32/134a	1.453+	10.017+	x	3		
73	32/125	1.608+	11.086+	x	3		
61	32/134a	1.634+	11.265+	x	3		
84	32/134a/125	1.644+	11.334+	x	3		
62	32/134a	1.707+	11.768+	x	3		
106	32/125	1.766+	12.175+	x	3		
18	32/134a	1.793+	12.361+	x	3		
59	32/134a	2.487+	17.145+	x	4		
82	32/134a/125	2.688+	18.531+	x	3		
104	32/125	2.895+	19.958+	x	4		
88	32/134a	3.075+	21.199+	x	3		
89	32/134a	4.111+	28.341+	x	4		
2	32	4.381+	30.203+	x	4		
32	32/134a	High voltage					
33	32/134a	High voltage					
34	32/134a	High voltage					
35	32/134a	High voltage					
36	32/134a	High voltage					
52	32	No test		x	3		
53	32	No test		x	3		

* Anomaly - See text Section 4.3.2; Match - Match ignition used; + - venting during test.

before reaching the top of the flask, It breaks into three segments (each shoulder and the neck) upon reaching the top of the flask and dissipates soon after reaching the top of the flask.

4.3.2.3. 0.5 psi (3.447 kPa). Similar to the 0.3 psi (2.068 kPa) case, the flame expands little until it reaches the neck of the flask, at which time it breaks into the three segments and dies out. More flame is seen in the neck of the flask.

4.3.2.4. 0.7 psi (4.8 kPa). The flame has spread more horizontally as it reaches the neck. The flame is more intense than the previous case but it still breaks up into the three segments at the neck.

4.3.2.5. 1.0 psi (6.9 kPa). The flame has spread well beyond the cone as it reaches the neck. Several “swirls” of different colors can be seen in the flame. It does not break up as it reaches the neck, but fills up the top 1/8 of the flask before dissipating.

4.3.2.6. Above 1.0 psi (6.9 kPa). The flame spreads as in the previous case. At venting, which can be heard on the video, the flame very quickly (in approximately 0.1 seconds) dissipates. In extreme cases, the flame raises and lowers several times in the flask before dissipating. Multiple fluctuations occur in the pressure plot during this time period.

The correlation between visible flammability as defined in Section 3.2 and overpressure lies in the 0.3 psi (2.1 kPa) region. There are two tests which do not fall within these boundaries— Test 1 and Test 55. Test 1 was the first test run in the flask, and the visual indication disagrees with the low overpressure reading. Since it was the first test, there is a possibility that data was not properly recorded. Test 55 was the initial test using the higher humidity air source. A great deal of moisture was seen inside the flask, and the test resulted in a very bright flash at the time of ignition, followed by considerable smoke. There was significant buildup of soot over the inside of the flask, including the electrodes, thermocouples, and stirring bar, as well as the flask itself. That type of behavior was not seen in any other tests, either at elevated temperatures or high humidity. Thus, the results of Test 55 are not considered valid.

4.4. Rate of Pressure Rise

The ability to quantitatively measure overpressure in the flask allows the capability to determine the rate of pressure rise under different conditions. However, while the measurement of the rate is simple, the correlation of the data between tests is not. For example, in comparing the difference in rate of rise generated between two different ignition sources, should the rates be compared at equal volume percent of flammable refrigerants or between tests that created a similar overpressure level? If the goal is to compare the rate of pressure rise between a test conducted in the flask at room temperature and one at 100 °C, should the comparison be made at similar weight concentrations of R32 or at similar overpressures? Higher overpressures are accompanied by greater rates of pressure rise. Therefore, it was chosen, somewhat arbitrarily, to compare the rate of pressure rise only between tests of similar overpressure levels. For test in the flask where venting occurred, the rate of rise is calculated prior to venting.

4.4.1. Ignition Source

Table 7 compares three tests of propane in the sphere at similar concentration at a starting pressure of 14.7 psia (760 torr). All three tests resulted in overpressures over the 30 psi (207 kPa) measuring capability of the overpressure transducer.

TABLE 7. RATE OF PRESSURE RISE BY IGNITION SOURCE, SPHERE, PROPANE.

Test Number	Propane Concentration (Vol %)	Ignition Source	Rate of Pressure Rise (Psi/sec [kPa/sec])	
P-70	2.62	wire	123.7	853
P-33	2.308	match	31.3	213
P-31	2.400	DC	62.4	430

Table 8 illustrates the difference for R32 between AC and DC ignition in the sphere using a starting pressure of 12.0 psia (620 torr). Using the results from this table, the wire ignition creates the highest rate of pressure rise, followed by the DC and AC sources, with the lowest rate of rise created by the match ignition.

TABLE 8. RATE OF PRESSURE RISE BY IGNITION SOURCE, SPHERE, R32.

Test Number	Propane Concentration (Vol %)	Ignition Source	Rate of Pressure Rise (Psi/sec [kPa/sec])	
32R2	15.06	DC	91.7	632
SP2	15.12	AC	57.2	393

4.4.2. Rate of Pressure Rise, Blends

Three tests, each using one of the R32 blends, were chosen to compare the rate of pressure rise between the blends (Table 9). All tests were run in the flask at room temperature with low humidity air and the AC source. The R32/R125 blend created the highest rate of pressure rise, followed by the R32/R34a blend and then by the ternary blend. These trends also follow the weight percent of R32 in the blend.

TABLE 9. RATE OF PRESSURE RISE BY R32 BLEND, FLASK.

Test Number	Components	R32 Weight %	Maximum Overpressure (psi /kPa)		Rate of Pressure Rise (psi/sec [kPa/sec])	
FL72	R32/R125	65	0.147	1.103	0.86	5.939
FL68	R32/R134a	40	0.153	1.055	0.46	3.171
FL74	R32/R134a/R125	30	0.131	0.903	0.27	1.861

5.0 DATA ANALYSIS

One of the major goals of this program was to establish test procedures and conditions to measure the flammability of refrigerants. Section 1 listed potential parameters which were postulated to have an effect on refrigerant flammability. This section analyzes the data presented in the previous section with respect to the parameters from Section 1. The criterion chosen for analysis is the explosion overpressure developed by the ignition of the refrigerant, since it is quantitative and corresponds well to the visual indications of flammability seen in the flask testing. For flammability limits, the overpressure is plotted against the volume percent of refrigerant. For testing of the blends overpressure is plotted against the weight percent of R32 in the refrigerant blend. Unless otherwise noted, the total refrigerant concentration is 20% by volume, and for the ternary R32/R134a/R125 blend, R134a and R125 are blended in a 60:10 weight ratio. Plots are reported in kPa only.

Results are reported as a Non-Flammability Ratio (NFR) using the term NFR rather than the CFR to denote that all concentrations of refrigerants were not tested, normally only 20 %. The NFR is the ratio of the weight percent flammable refrigerant to that of the non-flammable refrigerant, for a 20% total refrigerant concentration, at which the mixture is non-flammable.

5.1. Ignition Source

Four different ignition sources were tested in the two apparatuses. “AC” in the legend of a plot indicates AC boosted through the transformer and gated on for 0.2 seconds. “DC” indicates the NMERI DC source with a nominal 70 J stored energy. “Match” indicates an electrically-ignited match, whereby a nichrome wire is heated and ignites a match head (“Ohio bluetip”), while “Wire” indicates a heated wire alone.

5.1.1. Propane Flammability Limits

All propane tests were conducted in the explosion sphere. The LFL and UFL of propane was determined at 14.7 psia (760 torr) for three different ignition sources - DC, match, and spark. Figures 15 and 16 illustrate the LFL and UFL for the three sources.

A similar comparison for R32 is made in Figures 17, 18, and 19 for the sphere and flask.

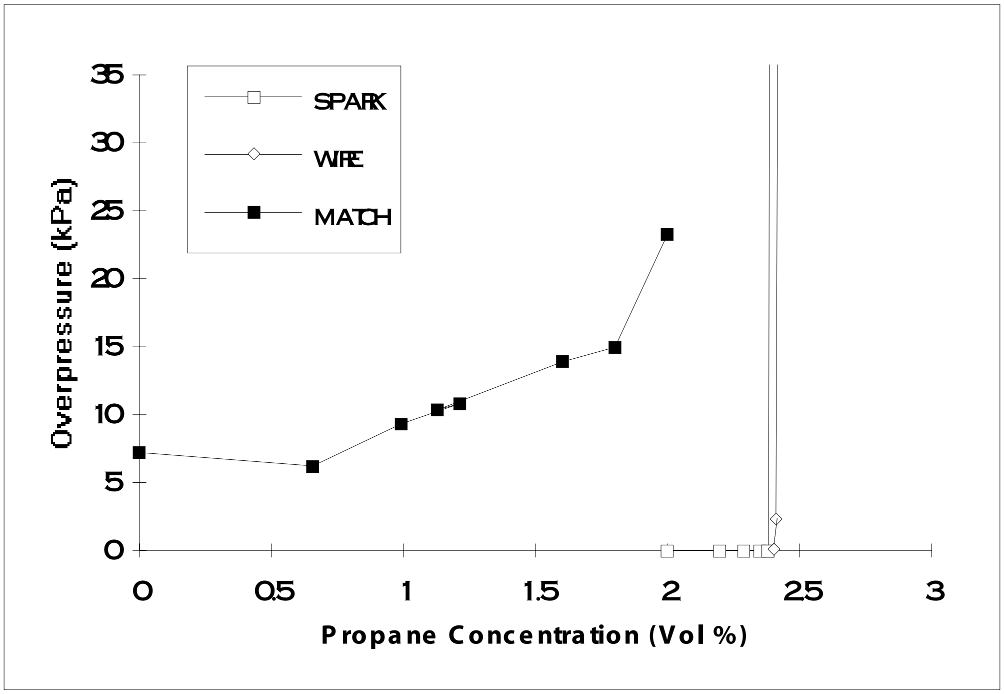

Figure 15. Propane Lower Flammability Limit, Sphere, by Ignition Source.

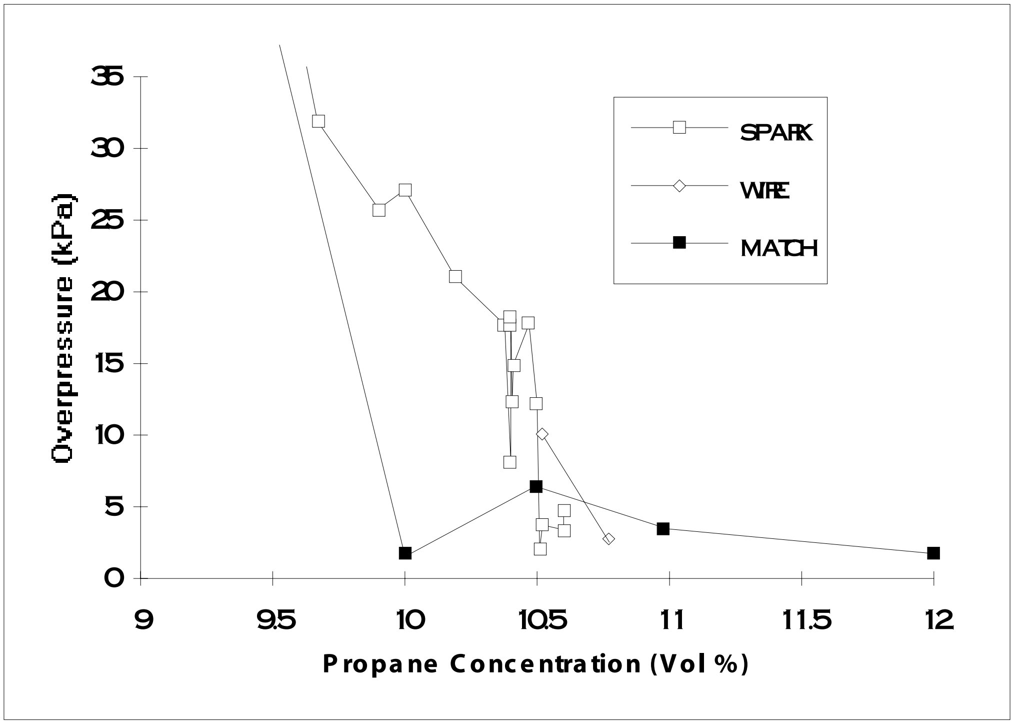

Figure 16. Propane Upper Flammability Limit, Sphere, by Ignition Source.

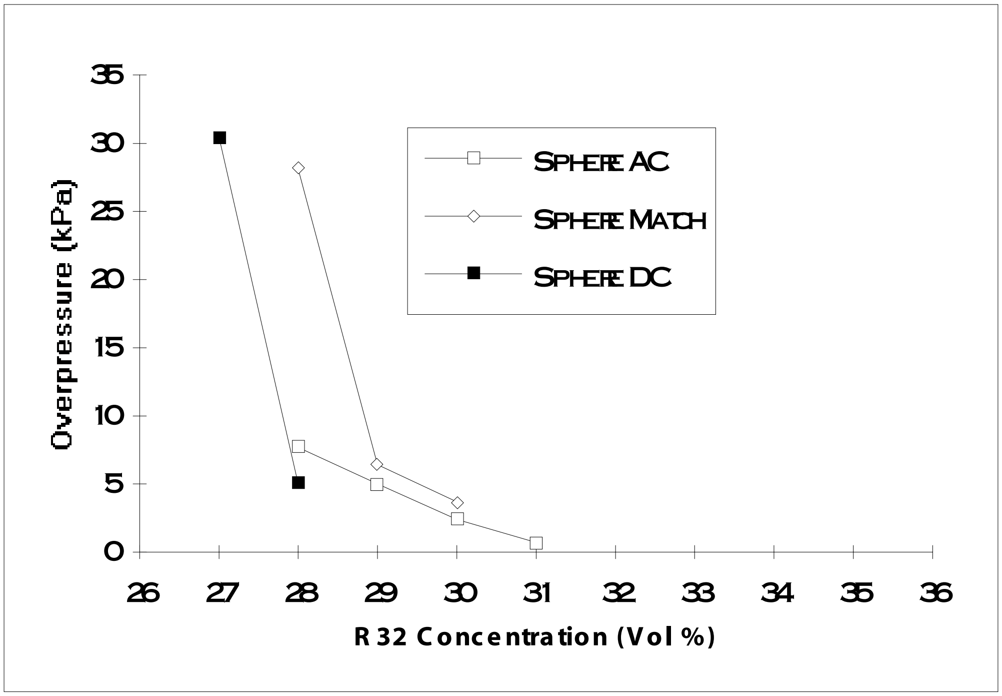

Figure 17. R32 Upper Flammability Limit, Sphere, by Ignition Source.

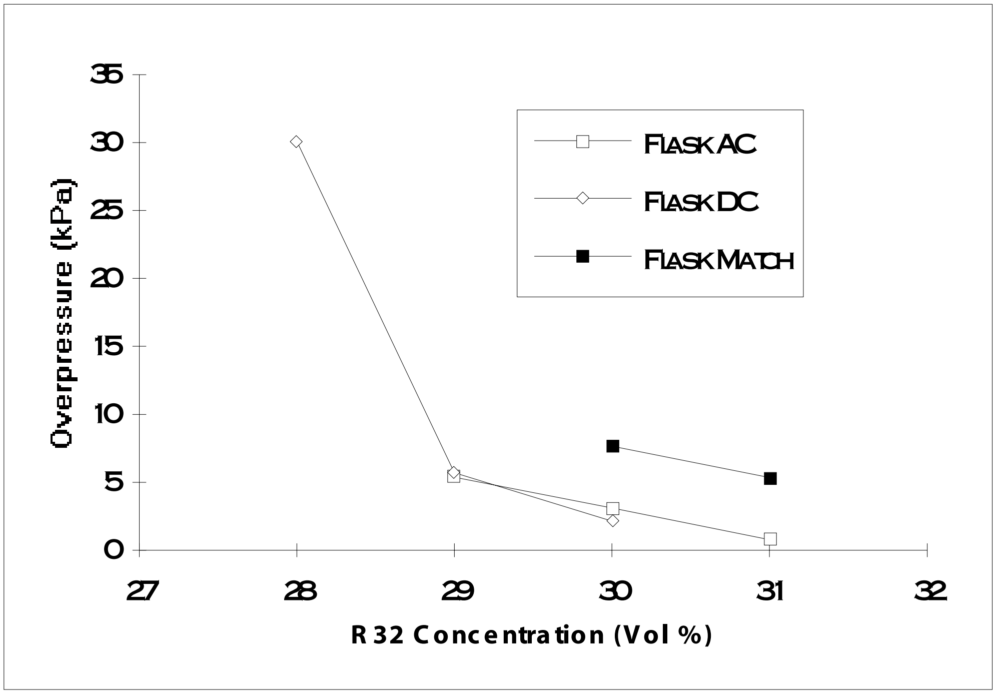

Figure 18. R32 Upper Flammability Limit, Flask, by Ignition Source.

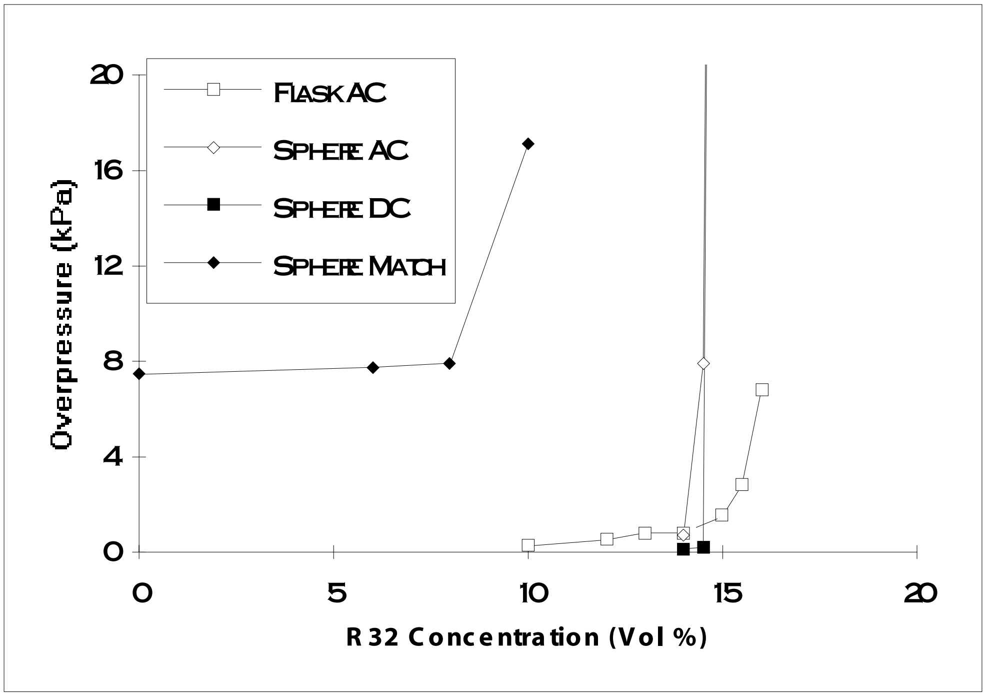

Figure 19. R32 Lower Flammability Limit, Flask, by Ignition Source.

5.1.2. R32/R134a Flammability

The flammability of R32/R134a with respect to the DC and AC ignition sources are compared in Figures 20 and 21 for the sphere and the flask. All tests were run at room temperature using dry air.

In both the sphere and flask, the AC source required a mixture with less R32 to reach a flammable condition.

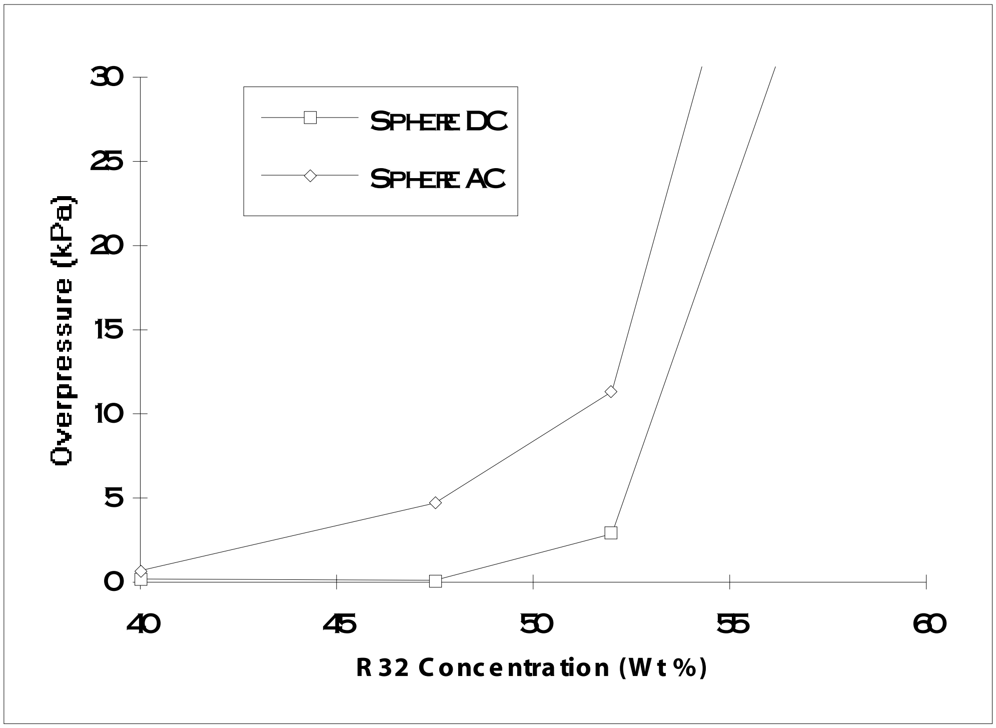

Figure 20. R32/R134a Flammability, Sphere, Room Temperature and Low Humidity.

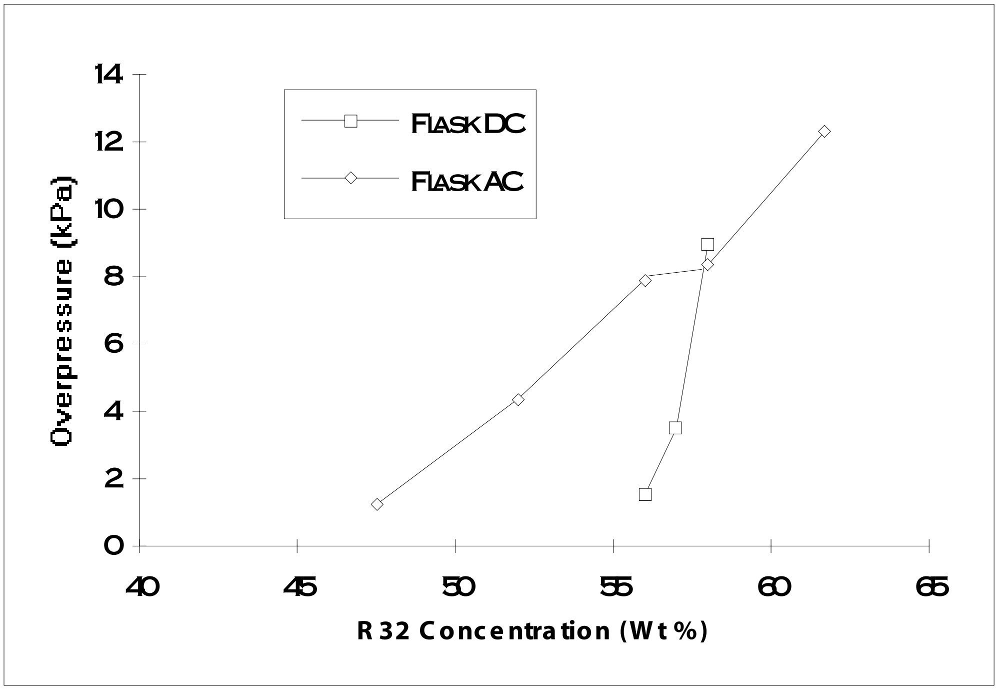

Figure 21. R32\R134a Flammability, Flask, Room Temperature and Low Humidity.

5.2. Temperature of the mixture

The oven enclosure operated very successfully in keeping the flask and contents near 100 °C (212 °F). Temperatures were measured using the thermocouples located inside the flask.

Nearly all of the high temperature tests were run at a temperature of 100 °±2 °C (212 °±4 °F). Two variations on the tests were run—introducing the components into the flask at room temperature and heating the flask to 100 °C (212 °F), and loading the components to the flask at 100 °C (212 °F). Some fluctuations in the internal flask pressure were noticed while loading the cold components into the hot flask, and the values were not as precise as in the case where the flask was loaded at room temperature. However, these fluctuations were small, and, in three sets of tests that compared the two techniques, no significant difference was seen between the techniques. All plotted data obtained at room temperature are noted as "RT" in the plot legend, and 100 °C data are noted as 100C.

5.2.1. R32 Flammability Limits

Figure 22 shows a plot of the upper flammability limit for R32 in the flask for room temperature and 100°C (212°F) using the AC ignition, for both low and high humidity. The effect of higher starting temperature is to raise the UFL by approximately 1 percent. The effect of temperature on the LFL was not examined.

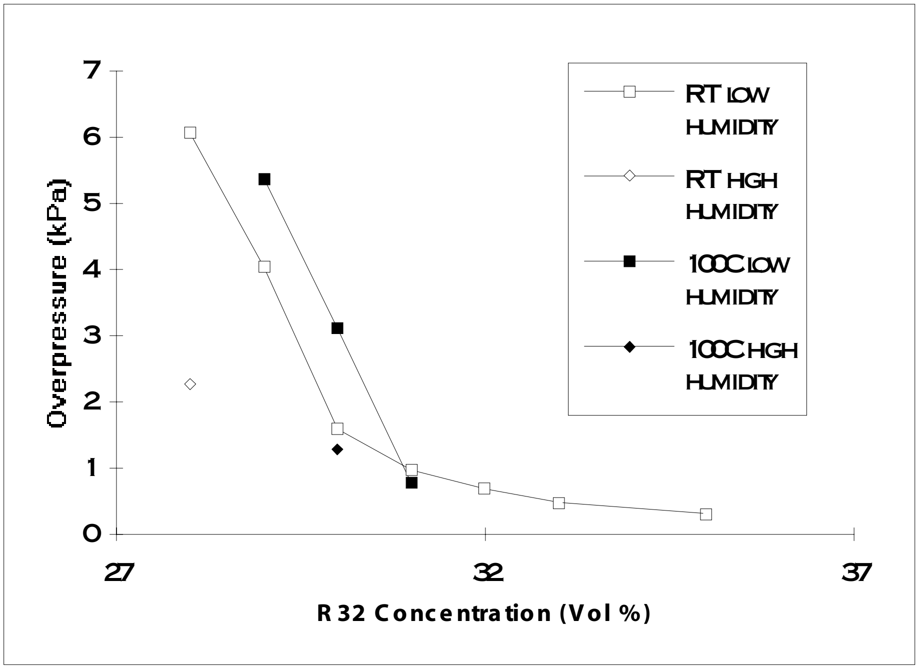

Figure 22. R32 Upper Flammability Limits - Flask.

5.2.2. R32/R134a Non-Flammability Ratio

Figure 23 shows a plot of the overpressure produced in an explosion versus the concentration weight percent of R32 for a 20% by volume total refrigerant concentration for a R32/R134a blend. The AC ignition is used, with room temperature and 100 °C, and the low and high humidity conditions. Using a 0.3 psi (2.070 kPa) criterion for flammability, the weight percent of R32 required for flammability drops from approximately 50% at room temperature to 41% at 100 °C (212 °F).

Expressing these values in percent change of R32 required for flammability, the weight percentage of R32 required to produce a flame decreased by 16-22% of the original value with an increase in temperature from room temperature to 100 °C (212 °F) at constant humidity. The R32 concentration required for flammability decreased by 31-35% of the original value with an increase in relative humidity from 1.7% to 82% at constant temperature.

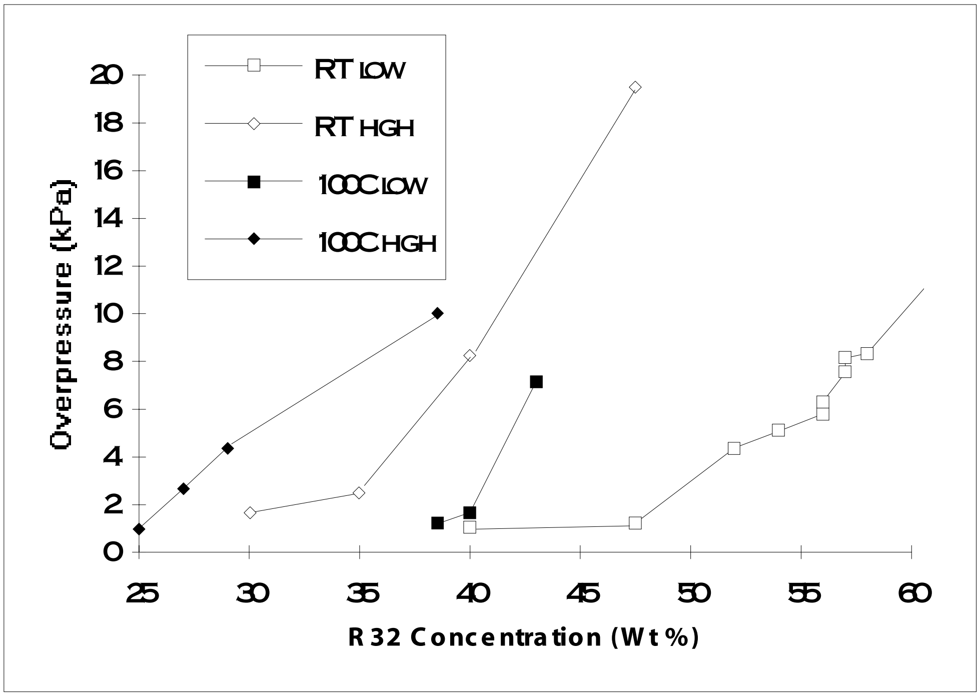

Figure 23. R32/R134a Flammability - Flask.

Figure 24 shows the trend of increasing flammability with higher temperature and humidity with R125 as the non-flammable component.

Figure 25 also illustrates the trend of increasing flammability with higher temperature and humidity using both R134a and R125 (60:10 weight ratio) as the non-flammable refrigerants.

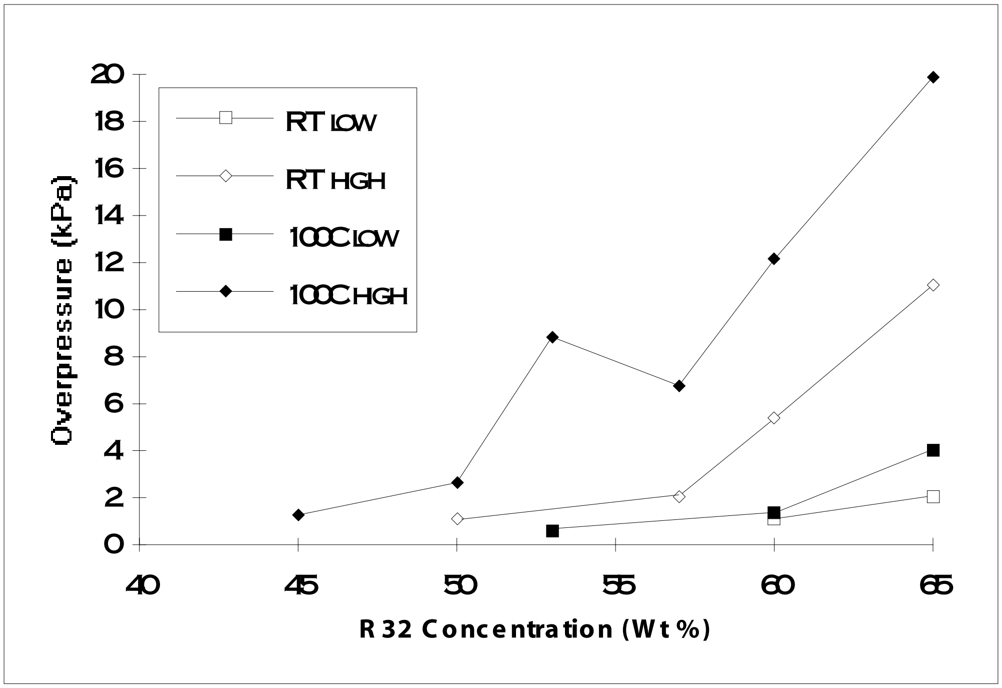

Figure 24. R32/R125 Flammability, Flask.

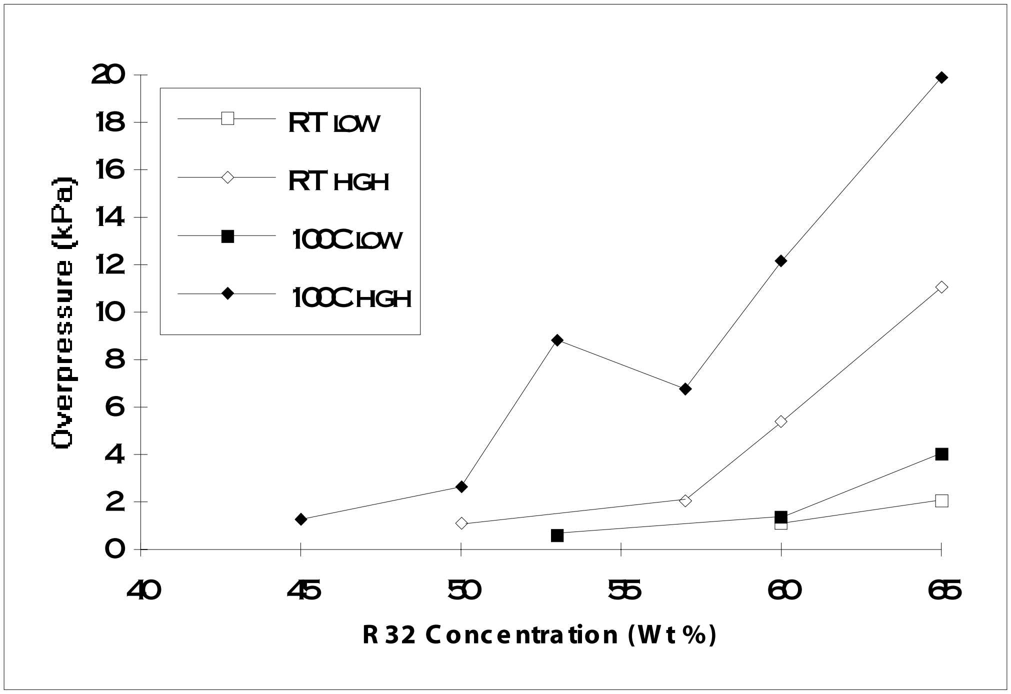

Figure 25. R32/R134a/R125 Flammability, Flask.

5.3. Starting Pressure of the mixture

Although the starting pressure and altitude of test are discussed as two separate parameters, they are related to the same effect, pressure, with the effect of altitude being an unavoidable complication to the test process. The starting test pressure was varied on two occasions in the sphere to investigate the effect on both R32 alone and the CFR of a binary blend. Table 10 illustrates the effect of starting pressure with the DC source and dry air in the sphere. The overpressure drops when the starting pressure is raised from 18 to 22 psia (930-1138 torr), but this may be a factor of ignition variabilities due to the higher pressure.

TABLE 10. EFFECT OF STARTING PRESSURE ON R32.

R32 Concentration (volume %)	Starting Pressure (psia /torr)		Overpressure (psi /kPa)		Percentage of Starting Pressure
10.4	12.062	620	0.255	1.758	2
10.4	14.700	760	2.691	18.552	18
10.4	15.996	827	5.800	40.000	36
10.423	18.000	930	11.076	76.358	61
10.38	22.000	1138	3.700	25.508	16

A second trial was conducted using the sphere, the R32/R134a binary mixture, and the AC source (Table 11):

TABLE 11. EFFECT OF STARTING PRESSURE ON R32/R134A BLEND.

R32 Concentration (weight %)	Starting Pressure (psia /torr)		Overpressure (psi /kPa)		Percentage of Starting Pressure
47.5	8.000	414	0.062	0.427	0.8
47.5	12.058	620	0.685	4.722	6
47.5	14.700	760	1.258	8.673	9
47.5	16.000	827	1.067	7.356	7

Both cases illustrate that higher starting pressures result in higher overpressures up to certain starting pressure. This may be a result of the inability of the ignition to arc across the electrodes at elevated pressures. A fifth test of the 47 weight % binary blend at 30 psia (1551 torr) resulted in an error message from the computer that appeared to indicate that the arc occurred between one electrode and the sphere and not between the electrodes (similar conditions were seen using higher voltage ignition sources). Similarly, on the 22.0 psia (1138 torr) starting pressure test in the propane series, the initial arc did not ignite the mixture and it took a second arc. However, it is not known whether the higher starting pressures impact the ability of the mixture itself to ignite or create ignition problems which then affect the apparent flammability of the mixture.

5.4. Humidity of the Air

The baseline test condition used air from a tank of Matheson "zero gas" air. The relative humidity of this air was measured at approximately 1.7%, although the accuracy of the humidity probe at very low or very high humidities is not guaranteed. Air of high humidity was obtained by bubbling air through a flask via a fritted glass tube (air sparger). This resulted in high relative humidity, measured at approximately 82%. A technique whereby half dry air and half humid air was introduced into the flask was tried, but the results did not significantly differ from the high humidity air. The level of the humidity for all tests is noted on plots by “low” or “high.”

Figures 22 through 25 illustrate the effect of humidity on the R32 UFL and the flammability of the two binary and one ternary blends of R32. The effect of humidity is even greater than that of temperature, with the high humidity and 100 °C condition being most severe.

5.5. Size and Shape of the Test Vessel

Differences between the sphere and the flask results were seen for those cases where the ignition source and conditions were identical. Whether these differences were due to the size or shape differences between the apparatuses, the placement of the electrodes, or some other factor, was not determined.

Figures 26 and 27 illustrate the differences between the two apparatuses for the R32 LFL and UFL.

Figure 28 illustrates the difference in the flammability of R32 in the sphere and the flask in an R32/R134a mixture for room temperature, AC ignition source, and low humidity.

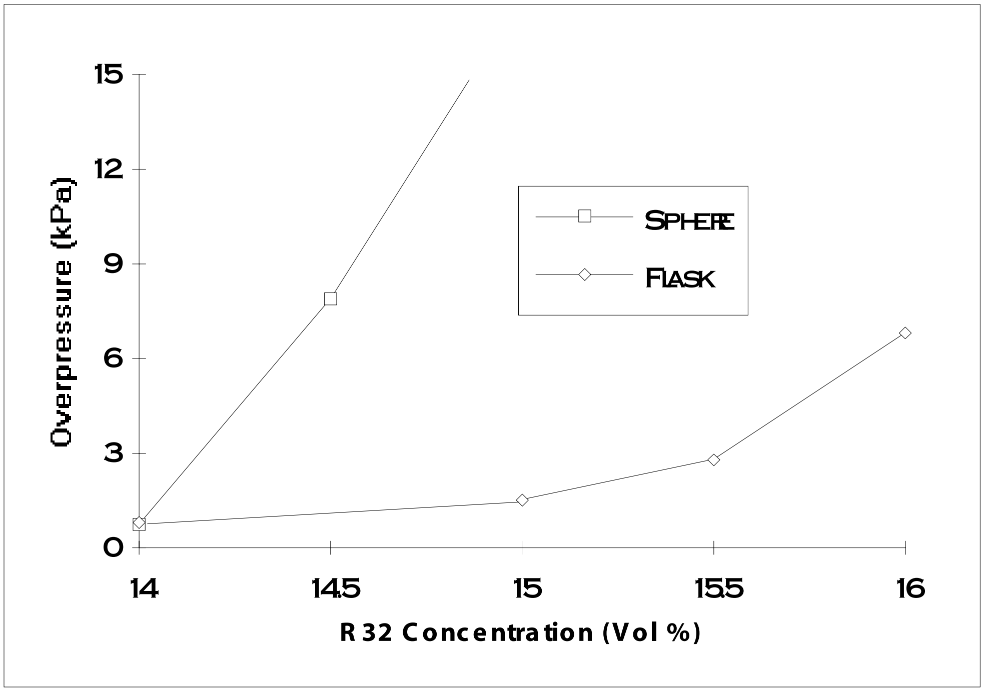

Figure 26. Sphere vs. Flask Comparison, R32 Lower Flammability Limit, Room Temperature and Low Humidity.

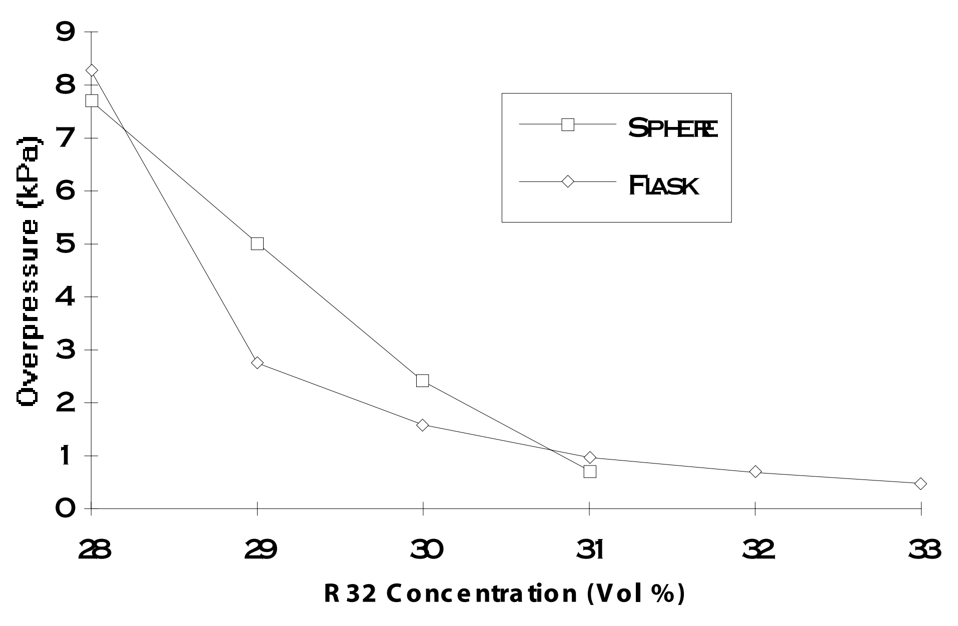

Figure 27. Sphere vs. Flask Comparison, R32 Upper Flammability Limit, Room Temperature and Low Humidity.

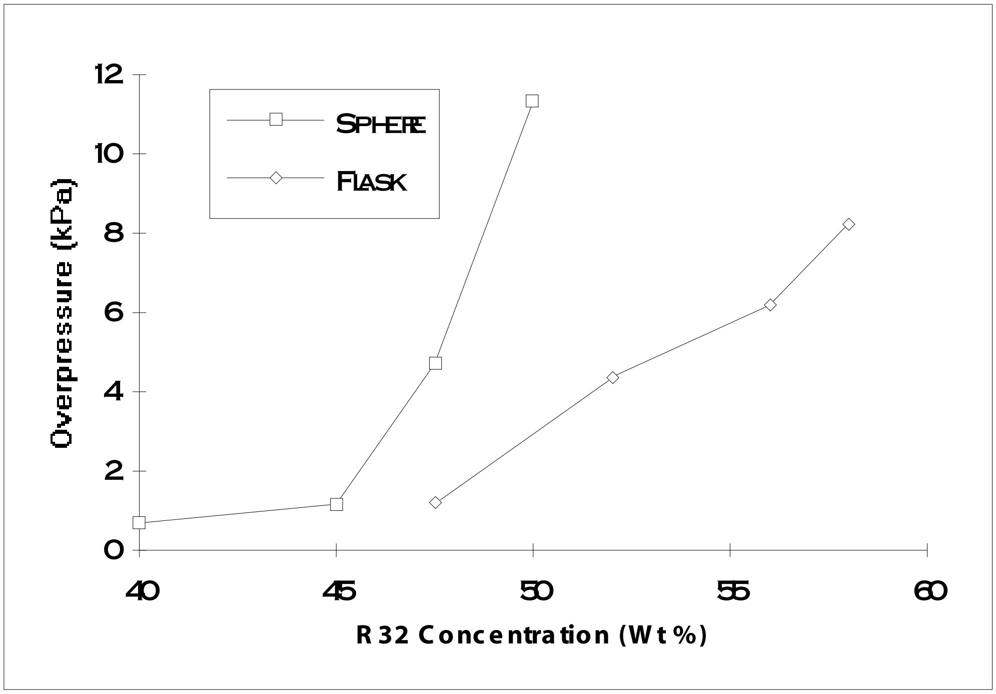

Figure 28. Sphere vs. Flask Comparison, R32/R134a Flammability, Room Temperature and Low Humidity.

5.6. Test Vessel Materials

No data were found to indicate that any differences between results in the explosion sphere and flask were due to the type of material from which they were constructed. Any differences between the flask and the sphere results were more likely due to the size and shape differences than the materials in that the flammability is determined before the flame reaches the vessel wall.

5.7. Turbulence in the Test Vessel

Three tests were run with the stirrer on during testing (Table 12.)

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TABLE 12. EFFECT OF TURBULENCE.

Condition	Overpressure (psi /kPa)		Temp Rise (°C) *
2 minutes mixing (baseline)	0.991/1.020	6.872/7.032	4/1
2 minutes mixing, stirrer moving slowly during test	1.146	7.901	4
2 minutes mixing, stirrer moving rapidly	0.218	1.503	8
2 minutes mixing, stirrer moving rapidly	small	small	7

* Temperatures were measured at top thermocouple.

It is interesting to note that the two tests where the stirrer was moving produced small overpressures, but larger temperature rises. The videos also indicated that the buoyant rise of the spark kernel was interrupted and the blue flames drifted away before they reached the top of the flask.

5.8. Concentration of the Components of the Mixture

The NFR is the minimum ratio, in weight percent, of non-flammable refrigerant that renders the blend non-flammable. The plots displayed in this section show overpressure as a function of the weight percent of R32. The NFR is the R32 amount subtracted from 100%. Table 13 provides the NFRs for several test conditions. The table presents a flammable/nonflammable condition for the R32 and the diluents for both the visual and overpressure criteria for explosion. The final column indicates the range between the 0.3 psi (2.068 kPa) and a 1 psi (6.9 kPa) criterion which is also used for flammability.

TABLE 13. NON-FLAMMABILITY RATIOS FOR R32 BLENDS.

Components	Test Conditions*	NFR Weight % R32/RDiluent Range (Visual Observation)	NFR Weight % R32/RDiluent Range (Pressure)**
R32/R134a	Flask, RT, low RH	48-52/52-48	48-56/52-44
	Flask, RT, high RH	30-35/70-65	30-40/70-60
	Flask, 100°C, low RH	41-33/59-67	43-38/57-62
	Flask, 100°C, high RH	27-29/73-71	38-25/72-75
	Sphere, RT, low RH	45-48/35-52	45-50/55-50
	Sphere, RT, high RH	30-35/70-65	35-40/65-60
R32/R125	Flask, RT, low RH	65-?/35-?	60-65/40-35
	Flask, RT, high RH	55-60/45-40	55-60/45-40
	Flask, 100°C, low RH	60-65/40-35	65-60/35-40
	Flask, 100°C, high RH	45-50/55-50	45-53/55-47
	Sphere, RT, low RH	60-65/40-35	60-65/40/45
	Sphere, RT, high RH	50-60/50-40	50-60/50-40
R32/(R134a/ R125) (60/10 wt%)	Flask, RT, low RH	50-55/50-45	50-60/50-40
	Flask, RT, high RH	30-50/70-50	40-55/60-45
	Flask, 100°C, low RH	>32.4/<67.7	32.4-?/67.7-?
	Flask, 100°C, high RH	23-33/77-67	30-33/70-67
	Sphere, RT, low RH	40-50/60-50	40-50/60-50
	Sphere, RT, high RH	30-40/70-60	30-50/70-50

*AC Spark, high humidity = 82% RH, low humidity = 1.7%, 20 vol% refrigerant in air
**Less than 0.3 psi (2.068 kPa) to greater than 1.0 psi (6.9 kPa)

In many cases, the number of data samples is low, especially for the sphere, so these numbers should not be taken as a definitive NFR. While they should be considered as only as trends, the higher humidity air and higher starting temperatures increase the NFR significantly. The NFR in the sphere also appears to be between 2 and 10 percent higher than in the flask.

5.9. Reactivity of the Components

There was no indication that during the period of testing, reactivity of the refrigerants affected tests.

5.10. Mixing of the Components

Early tests were run with a 5-minute mixing time according to ASTM E681. Other researchers have indicated that 2 minutes is used for mixing. Tests at the same refrigerant concentration were run for 5 minutes stand time after all components were added (with no mixing), no stand time or mixing, and 2 minutes mixing (Table 14). All tests were 20% total refrigerant, 57% R32 by weight, using the AC spark.

TABLE 14. EFFECT OF MIXING

Condition	Overpressure (psi /kPa)	
5 minutes mixing	1.096	7.756
no mixing, 5 minutes wait	1.046	7.211
no mixing, no wait	1.208	8.328
2 minutes mixing, stirrer moving slowly during test	1.146	7.901
2 minutes mixing	0.991/1.020	6.832/7.032

While data are limited, it appears that components diffuse within 5 minutes and that 2 minutes mixing produces results within experimental error.

5.11. Altitude of Testing

Tables 10 and 11 illustrate the effect of testing at local atmospheric at Albuquerque, New Mexico (approximately 12.0 psia [620 torr]) and at sea level (approximately 14.7 psia [760 torr]). The overpressure generated by a 10.4% by volume mixture of R32 and air increased from 0.255 psi (1.758 kPa) at a starting pressure of 12.062 psia (620 torr) to 2.691 psi (18.5 kPa) at 14.7 psia (760 torr) starting pressure. A 47.4% weight mixture of R32 in a 20% by volume R32/R134a blend resulted in a 0.685 (4.722 kPa) overpressure at 12.058 psia (620 torr), and a 1.258 psi (8.673 kPa) overpressure at 14.7 psia (760 torr).

These results were generated in the sphere. Because the flask is open to the atmosphere, it is difficult to test at a higher starting pressure without overpressurizing the flask and creating potential safety problems due to not allowing venting. However, it would be a simple matter for a researcher at sea level to conduct several tests at 12.0 psia (620 torr) to repeat the conditions at Albuquerque.

If the trends seen in these tests are confirmed, it will be critical to investigate further to see how, and to what degree, flammability testing between researchers at different locations varies as a result of the altitude of testing, and to develop methods to normalize results for all test facilities.

6.0 CONCLUSIONS

6.1. Flammability Criteria

Visual indications of flammability, as measured using R32 and R32 blends in this apparatus and determined by the cone method, correspond to an explosion overpressure of slightly over 0.3 psi (2.1 kPa). The overpressure criteria for an explosion, as defined for inertion purposes, is 1 psi (6.9 kPa), although this is also an arbitrary value. Any criteria set for the flammability of refrigerants must be realistic as not to exclude otherwise acceptable refrigerants or refrigerant blends from consideration based solely on the fact that they are “flammable”. The concern for producing a completely nonflammable refrigerant or blend is partially dictated by the public’s perception of flammable being bad. This concern must be weighed against the negative cost and efficiency impacts potentially incurred by not using the most effective and efficient refrigerant. Based on the results of this program, the criteria for flammability must be critically examined to ensure that they are realistic and provide adequate—but not excessive—safety for the users of refrigeration equipment.

6.2. Flammability Parameters

6.2.1. Ignition Sources

Conically shaped electrodes require 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) appears to be adequate for all testing.

The NMERI low-voltage DC spark ignition, when the spark is generated by the discharge of capacitors charged to 70 J, actually delivers only 0.25 J across the gap for an efficiency factor of .025%. The reason for this low efficiency is the saturation of the transformer. The energy content of the spark can be increased by using a transformer with a core material that will not saturate as fast. Depending upon the configuration of the ignition system, the ignition energies used by other researchers may be less than claimed.

The NMERI high-voltage DC spark ignition developed for this program does not use a transformer and is not limited by saturation of the transformer core. The spark generated at a 7.5 kV charging voltage (28 J stored energy in the capacitors) has an energy content of 3 J, an efficiency of 10.7%. At 17.5 kV, the value is 10 J with a stored energy of 153 J. However, as our testing has illustrated, high voltage sources can cause arcs outside the test apparatus and need to be carefully handled.

The AC ignition source developed for this program has been demonstrated to be an effective ignition for refrigerant testing. The duration of discharge was set at 0.2 seconds, and was adequate to ignite refrigerant blends repeatably.

The match ignition resulted in an LFL lower than observed in other sources, but a UFL that was also lower. The match burned differently at the LFL of a refrigerant, where there was sufficient oxygen, than at the UFL, where oxygen was limited.

The heated wire ignition provided enough energy to ignite propane but not R32. It did, however, cause the highest rate of pressure rise of any ignition source in propane. This may have been a result of the greater exposure to a heated source and not the flammable itself.

6.2.2. Temperature

Higher temperature widened the flammability limits and increased the CFR of an R32 refrigerant blend. The effect of higher humidity, however, is greater.

There was no apparent difference between tests conducted at 100 °C (212 °F) when the components were loaded into the cold flask and the temperature of the flask raised, or loaded into the hot sphere. However, because less internal pressure fluctuation were observed when the components were loaded in the cold flask, it is recommended that for final trials, components be loaded into the cold flask where possible.

6.2.3. Starting Pressure

A higher starting pressure at ignition creates an explosion of higher overpressure.

6.2.4. Humidity

The CFR of an R32 blend dramatically increases with increased humidity in the air. Based on limited testing, the UFL decreases slightly with increased humidity in the air.

6.2.5. Test Vessel

The R32 LFL was slightly lower for the sphere than for the flask, and the UFL was about the same. The CFR of the R32/134a blend was higher in the sphere than the flask. It was not determined whether these differences resulted from the size and shape difference between the apparatus or some other factor such as material.

6.2.6. Turbulence

The turbulence of the air affected test results. Tests should only be conducted in still conditions.

6.2.7. Mixing

A five minute mixing time appeared to be adequate for mixing even when the stirrer was not turned on. Tests run with the stirrer on for two minute, rather than the baseline five minutes, did not indicate significant deviations from the baseline.

6.2.8. Reactivity of the Components

No reactivity was observed between any of the tested components.

6.2.9. Altitude of Testing

A significant increase in both the width of the flammability limits and the CFRs were observed as the starting test pressure was increased from 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.

6.3. Test Conditions

The test apparatus and methodology outlined in ASTM E681 have been determined to be marginally adequate for determining the flammability of a refrigerant. The following areas have been identified as requiring additional standardization.

6.3.1. Apparatus

The use of an oven as an enclosure has proven to be very effective in controlling the temperature to within ±2 °C (±4 °F). The use of an enclosure heated by blower-driven hot air may or may not provide adequate temperature control. The enclosure developed in this program used a strip heater for the heat source.

6.3.2. 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.

An ignition system (the high-voltage DC source) was constructed to provide a more energetic ignition energy. However, the high voltage and current combination created high-voltage concerns which prevented significant testing using this source. At a minimum, the pulse width needs to be optimized and the test apparatus and instrumentation should be redesigned to accommodate high voltage.

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.

6.3.3. Humidity of the Air

The humidity of the air used must be specified and controlled. The apparatus must be evacuated and only controlled air be used. 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. Repeatable techniques of generating air containing more humidity should be developed and provided in ASTM E681.

6.3.4. 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.

6.3.5. Mixing

ASTM E681 specifies five minutes stirring before test. This is an adequate amount of time, but it has been demonstrated that two minutes is also adequate. Since many researchers use a two-minute mix time, the length of time for mixing should be re-examined.

6.3.6. Cleaning Between Tests

The procedure suggesting up to three evacuation/flush cycles between tests should be standardized and stated as a requirement. Although ASTM E681 states that final trials shall be made in a clean (no definition of "clean" is provided) vessel, procedures governing the conduct of cleaning between preliminary tests are not provided.

7.0 RECOMMENDATIONS

The results of this project indicate that the method of determining flammability using the procedures outlined in ASTM E681 is marginally adequate but can be improved. An assessment of whether a visual criterion or an overpressure criterion is most appropriate in determining flammability, regardless of the apparatus, was one major goal of this program. Although the visual criterion appears to be repeatable and reliable, it is recommended that a more quantitative criterion be adopted. The rationale is that a quantitative criteria can be adapted to any definition of flammability (for example, 0.3 psi [2.068 kPa] or 1 psi [7 kPa]), but the visual criterion is limited only to that which can be readily agreed to by all observers (for example, the cone method). Moreover, application of the visual criteria requires expertise. It is also recommended that an overpressure gauge capable of reading 0.001 psi (0.007 kPa) be the standard overpressure measuring device.

Both the explosion sphere and the ASTM E681 flask have their own niche in the testing of flammable refrigerants. The explosive sphere, while providing reliable and repeatable data, has shortcomings that limit how it may be used, and the flask also has its own shortcomings relating to the starting pressure of the test.

7.1 Explosion Sphere

The explosive sphere provides repeatable and reliable results, primarily because the loading procedures are more controlled in the sphere and mixing is more thorough. A fan is used for mixing rather than a mixing bar, although in the case of the R32/R134a/R125 mixtures in the flask, no difference was seen between the various mixing times for the techniques used (Section 5.10.) However, the sphere as now designed cannot be heated to 100 °C (212 °F) , the explosion cannot be observed, and the cost of developing and characterizing an explosion sphere is greater than that of the flask. The first two shortfalls may be remedied with modifications of the sphere. The starting pressure for test can be varied above and below the local atmospheric pressure, allowing for greater testing flexibility. The explosive overpressure can be measured for any magnitude of explosion, since the byproducts do not vent as in the flask. Therefore, where precise measurements at room temperature are required, the sphere is recommended as the prime apparatus.

When the sphere is to be used to determine flammability, the following procedures are recommended:

a. Humidity should be controlled to 50%.

b. The internal fan should be running when the components are introduced.

c. The sphere should be opened up after each test and washed with di-ionized water and dried. The electrodes should be lightly sanded with sandpaper.

d. The ignition system should be a 0.2 second AC pulse as defined in section 2.4.2. An alternative method using a paper match to demonstrate the non-flammability of a specific flammability mixture can also be employed.

e. The starting pressure should be controlled to 11.5 psia (595 torr) until such time as procedures are developed to allow starting pressure above local atmospheric pressure to be used in the flask. This value was chosen to accommodate all potential test locations and atmospheric conditions, since testing in the flask can be conducted in a partial vacuum.

7.2 ASTM E681 Flask

The flask is recommended for elevated temperatures and where visual observation of flammability may be critical. The cost of constructing and characterizing the flask is less than that for the sphere, and thus more readily available to more organizations. The major shortfall of this apparatus is that, at this time, refrigerants cannot be tested at starting pressures above the local atmospheric pressure without major modifications. This modification could require pressurizing the oven enclosure or providing a method of positive pressure seal between the stopper and the flask, either of which could prove difficult while still allowing venting to occur. The use of an explosive valve or a blowout disk in the stopper could resolve this problem by allowing a positive pressure inside the flask prior to test while still permitting venting.

When the ASTM E681 method is to be used for determining flammability, the following procedures are recommended:

a. Humidity should be controlled to 50% relative humidity.

b. An oven, rather than the heated enclosure specified by ASTM E-681, should be used for better control of the temperature, and the temperature should be measured inside the flask through the internal thermocouples.

c. All tests should be videotaped, with sound. The sound can indicate whether venting has occurred.

d. A transducer should be used to measure the explosive overpressure in addition to the visual indication.

e. All tests should be run at a starting pressure of 11.5 psia (595 torr) to allow reproducibility between all possible test locations. Procedures should be developed to allow testing to be conducted above local atmospheric pressure.

f. The stirring bar should be turned on for 2 minutes to ensure mixing.

g. In preliminary trials, the flask should be cleaned using two evacuation cycles (evacuate to the limit of the vacuum system and refill), followed by a two minute flush with compressed air, followed by a final evacuation cycle. For final tests, the flask should be removed from the enclosure and thoroughly purged with compressed air.

h. The ignition system should be a 0.2 second AC pulse as defined in section 2.4.2. An alternative method using a paper match to demonstrate the non-flammability of a specific flammability mixture can also be employed.

7.3 General Recommendations

In addition to the specific recommendations for testing, the following areas are recommended for future research:

a. The effects of humidity must be examined more completely. This program has demonstrated that increased humidity increases the amount of non-flammable components required to make the R32 mixture nonflammable, but the humidity level at which changes begin has not been determined. A method of reliably and repeatably producing air of known humidity should be developed. A method of evaluating the reactions occurring in the ignition of the flammable mixture with respect to water vapor content should be also developed.

b. A study should be undertaken to determine what, if any, differences in flammability results are caused by the altitude (variable starting pressure between test locations) of the test apparatus. Procedures to allow testing above atmospheric pressure in the flask should be developed.

c. Work should be conducted to further develop the explosion sphere to allow visual observation or video recording and to allow heating to 100 °C (212 °F).

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APPENDIX A: DATA ACQUISITION SYSTEM INSTRUCTIONS—NMERI EXPLOSION SPHERE AND ASTM E681 FLASK

A. Start-up

1. Turn on power bar to turn on power supplies approximately 30 minutes prior to test to permit warm-up and stabilization of the transducers.
2. Turn on Computer and Monitor. The program manager window will come up. Move arrow to Sphere DAQ icon in center panel and double-click the left mouse button rapidly. The test screen will come up (Figure A-1).

B. Constant Value Entries

1. Enter the desired test name and number in the *File Name* and *Test Name* blocks. The test number will automatically update after a test is saved.
2. Read barometric reading in inches of mercury (should be around 25) on barometer on wall near rear door. Enter the value in inches Hg in the left box in the *Barometric Pressure* box. Hit enter, and the value in psi will appear in right box.
3. Select the target (final) pressure at which you want to test. There are three choices in a drop down box titled *Target Pressure*:

   - 14.7. Sets the value at the sea level atmospheric pressure.
   - Local. Sets the value at the local atmospheric pressure entered in B.2.
   - Other. Allows a different pressure to be entered. Enter the pressure in the box to the right.
4. Select the loading and overpressure transducers in the *Transducers* box. Double-click on the correct transducer. The black light should turn yellow for a second showing the transducer was selected correctly.
5. Select the ignition system variables in the *Charging* box.

   If the current test does not use the NMERI DC spark (from the blue box), select 0 joules. This will open the correct switch to allow testing to occur manually.

   If the system uses the NMERI blue box, enter the correct value for capacitance in the upper right box. Three capacitors equals .006 F. Enter the desired energy level: the voltage will be calculated automatically.
6. Select Components. Select (by double-clicking) the desired test components from the boxes under *Component 1*, *Component 2*, and *Fuel*.

C. Variable Value Entries

1. Update any constant values required in B.
2. Enter Component Percentages. Enter the predetermined percentages (in volume percent) for component 1, component 2, and fuel. If fuel amount is desired to be a stoichiometric fuel-to-air ratio, push the *%* button; otherwise enter the amount in percent. The required amount of each gas, in psi, will be calculated automatically. The air percentage will also be calculated automatically.

D. Add Components

(Note: At this time, the sphere or flask should be ready for test, evacuated, with all valves closed. The valve to the 2.5 psi Druck should be closed. The capacitor charging gate should be closed.)

1. With the apparatus fully evacuated, add air until the overpressure transducer reads a pressure adequate to add all components and still remain below the target pressure. Open loading transducer and allow the pressure to stabilize.
2. Select **Load Components** button on top right of screen. A large popup with four boxes and three selectors underneath will appear.

   The **NEXT** button records the pressure after the addition of a component and moves to the next component.

   The **PREVIOUS** button moves to the component that was last loaded.

   The **Transducer** switch allows selection of the loading or overpressure transducers to measure the pressure. The Druck 2.5 psi (17 kPa) is accurate for ±2.5 psi (17 kPa); and, although it can record up to ± 10 psi (69 kPa), its accuracy decreases outside the ±2.5 psi (17 kPa) range. Whenever the vacuum is below -2.5 psi (17 kPa) or the pressure above +2.5 psi (17 kPa), the overpressure transducer should be used, although its accuracy may not be as great.

   The top left window indicates the amount of a component (in psi) that must be introduced in the sphere (the target amount).

   The top right window indicates the total pressure inside the sphere to which the component must be added.

   The bottom right window indicates the amount of component that has been introduced into the sphere. It should equal the target amount for that component when the correct amount of component has been added.

   The bottom left window indicates the actual pressure within the sphere. It should equal the target pressure for that component when the component is properly loaded.

3. Starting Pressure. The starting pressure is the pressure at which the loading will begin. It should be low enough to allow addition of all components without raising the final pressure above the target pressure. The fan should be turned on in the sphere. Add air from the cylinder to get to a suitable starting pressure. The analog gage, while not precise, can give a good indication of the pressure level. Select the **NEXT** button.
4. Air. Add air from the cylinder to reach either the target amount or target pressure (whichever is easiest to read–both will record the same amount of air). Hit **NEXT** when that pressure is reached. The computer will record that value for calculation of the final percentages.
5. Component 1. The component selected as component 1 will be listed at the top of the window. Add the correct amount to either the target amount or target pressure. Hit **NEXT**.
6. Component 2. Repeat procedure for Component 1.
7. Fuel. Repeat procedure for Component 1. The final pressure after addition of fuel should be the target pressure. Hit **NEXT**. The **Load Component** Popup will disappear and the Load Components button will turn to **Charge**. Turn off fan.
8. Print Screen. Select the print button if you desire to have the screen printed. The screen will be printed on the printer in the mailroom. It may take several minutes to complete the printing.

E. Ignite Mixture

At this point, a small popup saying “close for capacitor charge” may appear as a reminder to close the charge switch.

E.1. NMERI DC Spark

1. Turn on high-voltage power supply and **trig source** switch (located on middle power supply on cabinet next to blue box).
2. Turn on Multimeter which is connected to computer. Set to read 1000 VDC.
3. Turn on charging gate switch by pushing switch (on metal box in from of monitor) toward monitor. Multimeter should read 259 or 260 volts. Adjust power supply if it doesn’t.
4. Click **Charge** button to begin charging process. Multimeter should drop to zero and begin to increase in voltage indicating that the capacitors are being charged. The actual voltage and energy boxes on the test screen will also increase. If the voltage drops to zero and does not increase, turn switch off and on.
5. Monitor the charging using the multimeter. Voltage will also be indicated in the *Actual* box on the screen. Charging should stop when the voltage reaches the value of voltage on the target box in the electrical section of the window. If the voltage reaches 200 or more volts, manually turn off the gate switch.
6. After the capacitor are charged (the **Charging** symbol will flash during charging), the load components button will change to **spark**. To ignite the mixture, select the **spark** button. After two seconds, the capacitors will be discharged and the spark generated across the spark gap.

E.2. AC Source

1. Hit charge button. **Spark** button will appear.
2. Ensure that AC timer switch is in the on position and properly connected.
3. Hit the **Spark** button.
4. After approximately 3 seconds, push the button to ignite mixture.

E.3. Wire or Match Source

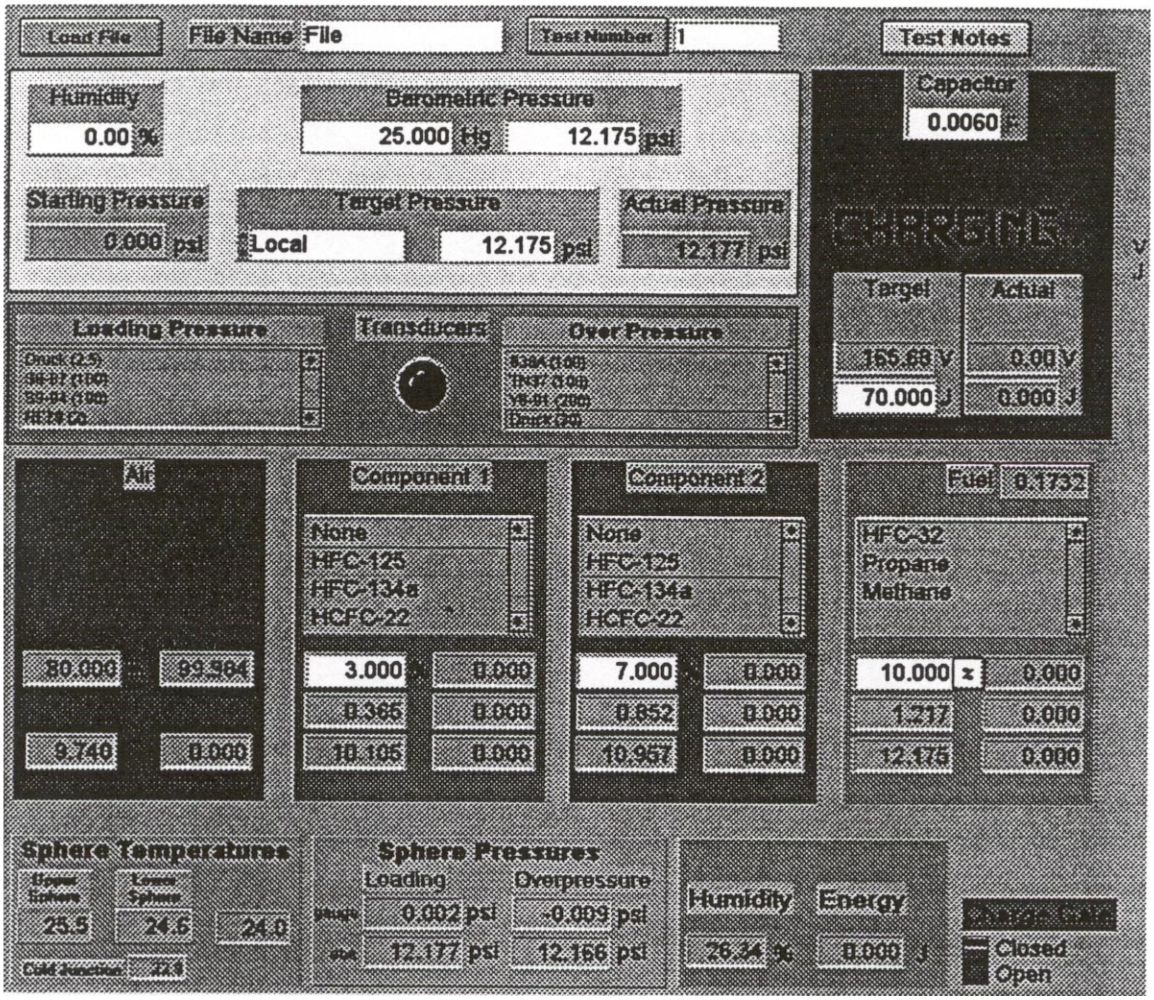
1. Hit charge button. **Spark** button will appear.
2. Connect leads of battery to ends of ignition rods, leaving one connection to battery undone.
3. Hit the **Spark** button.
4. After approximately 3 seconds, connect the second battery wire to ignite mixture.

F. Data Reduction

1. After the initial 2 seconds, data will be recorded for 12 additional seconds (14 seconds in all), stored in a temporary file, and plotted. After several minutes, a screen with four plots (three temperature and one overpressure versus time) will appear (Figure A-2). The maximum value for each of the plots will also appear.
2. Print the screen by selecting the **print** button. (Note: Printing takes several minutes.)
3. Save the data by pushing the **save** button. The test number will increment. Check to see if it is one number higher than the previous tests. If you do not desire to save data, go to next step.
4. Clear the data screen and bring up the test screen (after saving) by pushing the **discard** button.

G. **End of Day**

1. Hit **Exit Program** button. The Program Manager window will again appear. Double-Click on File Manager in Main and select C:\Sphere\Data directory. The test files will be stored in that directory. Choose the directory on the server (normally F:\) where files are stored and “drag and drop” the files from the data acquisition C drive to the server. Delete the transferred files on the C drive.
2. Exit Windows and turn computer off.



File Name: File
Test Number: 1

Humidity: 0.00 %
Barometric Pressure: 25.000 Hg 12.175 psi
Capacitor: 0.0060 F
Starting Pressure: 0.000 psi
Target Pressure: Local 12.175 psi
Actual Pressure: 12.177 psi

Target: 165.68 V 70.000 J
Actual: 0.00 V 0.000 J

Air: 80.000 99.984 9.740 0.000
Component 1: 3.000 % 0.000 0.365 0.000 10.105 0.000
Component 2: 7.000 % 0.000 0.852 0.000 10.957 0.000
Fuel: 0.1732 10.000 z 0.000 1.217 0.000 12.175 0.000

Sphere Temperatures: Upper Sphere 25.5 Lower Sphere 24.6 24.0
Cold Junction 22.9

Sphere Pressures: Loading Overpressure
gauge 0.002 psi -0.009 psi
abs 12.177 psi 12.166 psi

Humidity 26.34 %
Energy 0.000 J

Figure A-1. Data Entry Screen

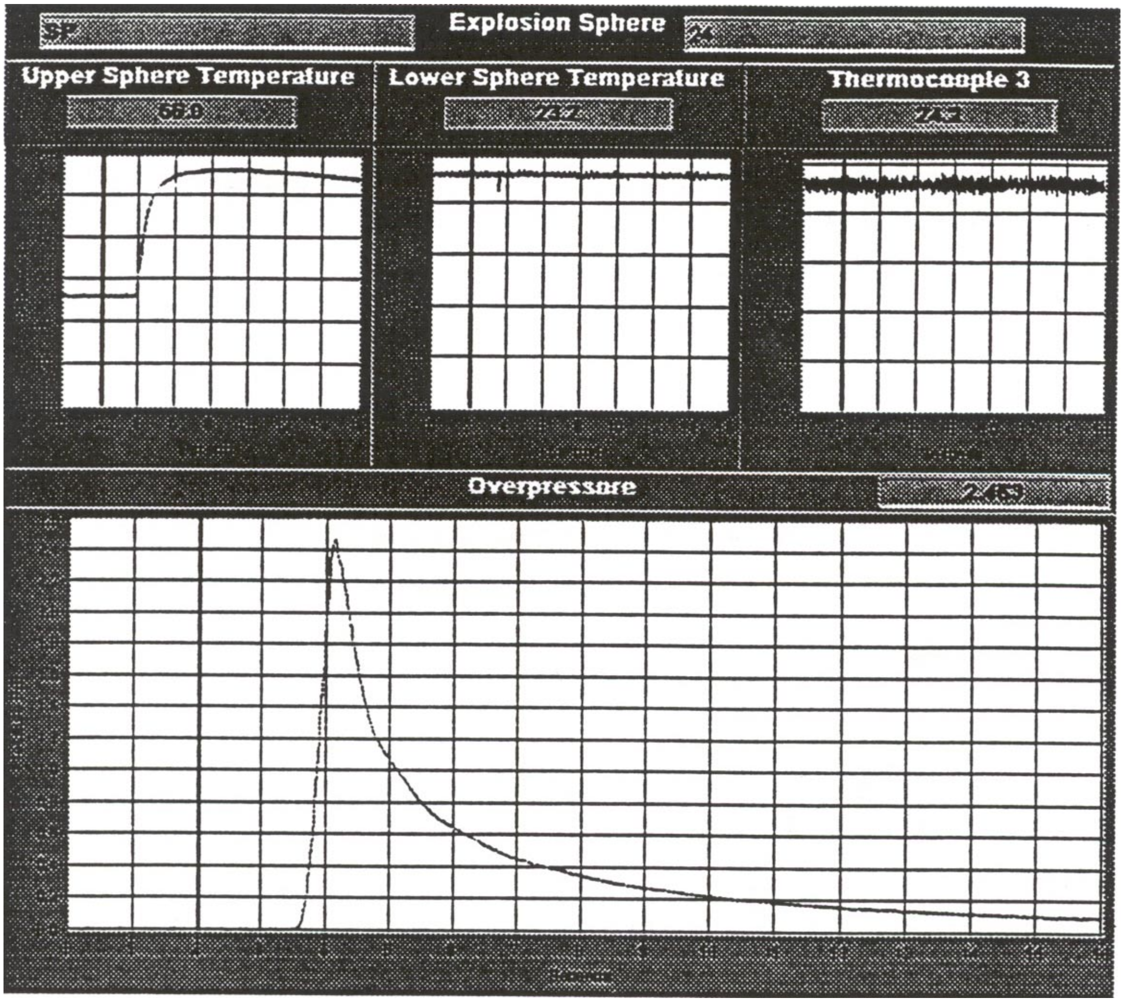


Figure A-2. Data Screen

APPENDIX B: OPERATING INSTRUCTIONS—NMERI EXPLOSION SPHERE

(Note: Operations having a safety impact are denoted by *italics*)

A. Startup

1. Open valves to air cylinder, vacuum, all component and fuel cylinders. *Bleed all lines until pure fuel or agent comes out (use your judgment, but it is better to be conservative and bleed a little more than to have air in the line and compromise one or more tests). Ensure that hood is at proper height.* Blow out inside of sphere with air to remove traces of components.
2. *If using the NMERI DC spark, check the system by charging the capacitors using the manual charge button and discharging by the manual discharge button and observing the spark in the sphere. Use the multimeter for monitoring the voltage. Check operation of the AC spark by pushing button.* ***Stay clear of apparatus during this test****!*
3. *Add liquid nitrogen to cryotrap. Use gloves and eyeshield.*
4. Turn on the fan and check operation. Turn off.

B. Operation

1. With top removed from sphere, open the valve to the loading transducer and zero by turning the screw inside the top of the transducer casing. Close the valve.
2. Prepare ignition source, if using other than NMERI spark ignition.
3. Close sphere and evacuate using vacuum pump under the fume hood. Monitor the vacuum on analog gauge and overpressure transducer. *Tighten bolts*.
4. Turn on fan.
5. Add sufficient air from cylinder to raise internal pressure to starting pressure, leaving enough "room" to add fuel and all components and still be below target pressure.
6. Open loading transducer valve if above -2.5 psig (17 kPa). If not, use overpressure transducer to initially load components to that level.
7. Add air, fuel, and components according to data acquisition procedures.
8. Turn off fan and close loading transducer valve.
9. *Close hood cover*.
10. *Charge capacitors using Data Acquisition System Instructions, turn on AC source, or prepare to ignite match or wire*.
11. *Ignite mixture*.
12. *Exhaust mixture through house vacuum system. Let set at maximum vacuum for 1 minute and open air inlet for 1 minute; repeat twice, then flush with fresh air for 2 minutes*.

13. If sphere is to be cleaned between tests, pull vacuum and unscrew bolts. Release vacuum and remove cover. Open and close loading transducer valve to release pressure.
14. If sphere is not cleaned between tests, prepare for next test.
15. Otherwise, wipe out sphere with distilled water, and lightly sand ends of electrodes with fine sandpaper, or remove any wire or match heads. Dry inside of sphere with compressed air.
16. Check fan operation and close sphere. Repeat test sequence.

C. End-of Day

1. *Close all fuel, component, air, and vacuum lines, shut off vacuum lines*.
2. *Bleed all gas lines*. *Ensure that hood door is at proper height.*
3. Clean sphere and leave open.
4. Turn off all power supplies.

APPENDIX C: OPERATING INSTRUCTIONS—ASTM E681 FLASK

(Note: Operations having a safety impact are denoted by *italics*)

A. Startup

1. Open valves to air cylinder, vacuum, all component and fuel cylinders. *Bleed all lines until pure fuel or agent comes out (use your judgment, but it is better to be conservative and bleed a little more than to have air in the line and compromise one or more tests). Ensure that hood door is at proper height.* Introduce air through manifold to remove traces of components
2. Empty flask of water and rinse with distilled water (flask will be stored filled with tap water). Rinse with several ounces of acetone and dry with compressed air. Lightly grease top of flask with vacuum grease and place mixing bar in flask.
3. Prepare ignition source, if using other than NMERI spark ignition.
4. Pull out oven shelf. Raise top assemble and rotate top backward so that the flask can be slipped over the electrodes and thermocouples and into hole on top of oven. Raise flask and install shelf. *Close oven door.*
5. *If using the NMERI DC spark, check the system by charging the capacitors and discharging and observing the spark in the sphere. Check operation of the AC spark by pressing button.*
6. *Add liquid nitrogen to cryotrap. Use gloves and eyeshield.*
7. Set up video camera. Attach to camera tripod and align and focus.

B. Operation

1. Make test sign for video.
2. Evacuate flask using vacuum pump under the fume hood. Monitor the vacuum on analog gauge and overpressure transducer.
3. Add sufficient air from cylinder to raise internal pressure to starting pressure, leaving enough “room” to add fuel and all components and still be below target pressure.
4. Open loading transducer valve if above -2.5 psig (17 kPa). If not, use overpressure transducer to initially load components to that level.
5. Add air, fuel, and components according to data acquisition procedures.
6. Close loading transducer valve.
7. Turn on stirrer for five minutes.
8. *Close fume hood.*
9. *Charge capacitors using Data Acquisition Instructions, turn on AC source, or prepare to ignite match or wire.*

10. Turn on video camera. Turn off lights 5 seconds after camera is turned on.
11. *Ignite mixture*.
12. Turn on lights. Turn off camera 5 seconds after lights are on. Observe video of test and advance 5 seconds beyond end of recording for separation between tests.
13. *Exhaust mixture through house vacuum system. Let set at maximum vacuum for 1 minute and open air inlet for 1 minute; repeat twice, then flush with fresh air for 2 minutes.*
14. Repeat for next test. *Remove flask if required for cleaning or replacing ignition source (match or wire) reversing order in A.2 being careful not to breathe air near mouth of flask should any by-products remain*.

C. End of Day

1. Exhaust mixture and flush with fresh air as at the end of a regular test. *Remove flask being careful not to breathe air near mouth of flask should any by-products remain*. Remove mixing bar. Wash flask and fill with water.
2. Disassemble camera.

APPENDIX D: TEST RESULTS

The following tables present the data from the test series. Table D-1 presents results from the flammability limit testing of propane and R32 in the explosion sphere, Table D-2 presents the flammability testing of R32 blends in the sphere, and Table D-3 presents the results from the ASTM E681 flask testing.

TABLE D-1. TEST RESULTS, FLAMMABILITY LIMITS, EXPLOSION SPHERE.

Test Number	Refrigerant Vol %	Ignition Type	Starting Pressure (psia /torr)		Overpressure (psi (kPa))	
P1	2.410	DC	14.7	760	0.000	0.000
P2	2.595	DC	14.7	760	0.000	0.000
P3	2.815	DC	14.7	760	30.000	207
P4	no test					
P5	2.587	DC	14.7	760	30.000	207
P6	2.400	DC	14.7	760	30.000	207
P7	2.200	DC	14.7	760	0.000	0.000
P8	2.377	DC	14.7	760	0.000	0.000
P9	2.529	DC	14.7	760	30.000	207
P10	2.382	DC	14.7	760	0.000	0.000
P11	2.741	DC	14.7	760	30.000	207
P12	2.449	DC	14.7	760	0.000	0.000
P13	2.415	DC	14.7	760	0.000	0.000
P14	no test					
P15	9.414	DC	14.7	760	8.700	60.000
P16	9.496	DC	14.7	760	7.000	48.258
P17	9.670	DC	14.7	760	4.700	32.402
P18	9.900	DC	14.7	760	3.800	26.197
P19	9.990	DC	14.7	760	4.000	27.576
P20	10.190	DC	14.7	760	3.100	21.371
P21	10.410	DC	14.7	760	2.200	15.176
P22	10.600	DC	14.7	760	0.700	4.826
P23	10.510	DC	14.7	760	0.300	2.068
P24	10.600	DC	14.7	760	0.500	3.447
P25	10.500	DC	14.7	760	1.800	12.409
P26	10.400	DC	14.7	760	1.200	8.273
P27	10.400	DC	14.7	760	2.600	17.924
P28	10.520	DC	14.7	760	0.550	3.792
P29	2.350	DC	14.7	760	0.000	0.000
P30	2.000	DC	14.7	760	0.000	0.000
P31	2.400	DC	14.7	760	30.000	207
P32	10.382	DC	14.7	760	1.560	10.755
P33	2.308	match	14.7	760	30.000	207

P34	2.000	match	14.7	760	3.392	23.384
P35	0.000	match	14.7	760	1.095	7.549
P36	0.000	match	14.7	760	0.952	6.563
P37	0.000	match	14.7	760	1.072	7.390
P38	1.800	match	14.7	760	2.177	15.008
P39	1.603	match	14.7	760	2.620	18.062
P40	1.427	match	14.7	760	1.529	10.541
P41	1.214	match	14.7	760	1.590	10.961
P42	0.990	match	14.7	760	1.375	9.478
P43	2.408	wire	14.7	760	30.000	207
P44	0.657	match	14.7	760	0.907	6.235
P45	11.995	match	14.7	760	0.256	1.765
P46	10.984	match	14.7	760	0.515	3.550
P47	10.000	match	14.7	760	0.220	1.517
P48	10.017	match	14.7	760	0.254	1.751
P49	0.000	match	14.7	760	1.085	7.480
P50	9.025	match	14.7	760	10.110	68.700
P51	9.500	match	14.7	760	13.400	92.380
P52	9.990	match	14.7	760	7.610	52.463
P53	10.505	match	14.7	760	0.945	6.515
P54	10.524	wire	14.7	760	1.478	10.189
P55	11.500	wire	14.7	760	0.088	0.607
P56	0.000	wire	14.7	760	0.170	1.172
P57	0.000	wire	14.7	760	0.074	0.510
P58	0.000	wire	14.7	760	0.073	0.503
P59	0.000	wire	14.7	760	0.048	0.331
P60	0.000	wire	14.7	760	0.062	0.427
P61	0.000	wire	14.7	760	0.067	0.462
P62	0.000	wire	14.7	760	0.103	0.710
P63	0.000	wire	14.7	760	0.049	0.338
P64	0.000	wire	14.7	760	0.052	0.358
P65	0.000	wire	14.7	760	0.031	0.214
P66	11.009	wire	14.7	760	0.090	0.620
P67	10.768	wire	14.7	760	0.402	2.771
P68	0.000	wire	14.7	760	0.048	0.331
P69	2.428	wire	14.7	760	0.344	2.372
P70	2.620	wire	14.7	760	30.000	207
P71	2.512	wire	14.7	760	30.000	207
P72	2.455	wire	14.7	760	30.000	207
P73	2.390	wire	14.7	760	0.000	0.000
P74	10.410	DC	14.7	760	1.817	12.506
P75	no test					
P76	10.400	DC	14.7	760	2.691	18.552

P77	10.380	DC	14.7	760	2.609	17.986
P78	10.470	DC	14.7	760	2.620	18.062
P79	10.410	DC	14.7	760	1.881	12.682
P80	10.395	DC	14.7	760	3.000	20.682
P81	10.433	DC	14.7	760	2.516	17.345
P82	10.415	DC	12	620	0.000	0.000
P83	10.423	DC	18	930	11.086	76.427
P84	10.400	DC	16	827	5.800	39.985
P85	10.380	DC	22	1137	3.700	25.508
P86	10.400	DC	12	620	0.255	1.758
P87	9.976	DC	12	620	0.906	6.246
P88	10.015	DC	12	620	2.332	16.104
P89	10.022	DC	12	620	1.470	10.134
P90	10.011	DC	12	620	1.547	10.665
P91	10.013	DC	12	620	1.688	11.637
P92	9.998	DC	12	620	1.385	9.548
P93	10.200	DC	12	620	0.726	5.005
P94	10.204	DC	12	620	0.637	4.391
P95	10.100	DC	12	620	1.154	7.956
P96	10.182	DC	12	620	3.751	25.859
P97	2.414	DC	12	620	30.000	207
P98	2.352	DC	12	620	30.000	207
P99	2.291	DC	12	620	30.000	207
P100	2.271	DC	12	620	30.000	207
P101	2.188	DC	12	620	0.025	0.172
P102	2.086	DC	12	620	0.025	0.172
P103	2.133	DC	12	620	0.014	0.097
P104	2.100	wire	12	620	0.359	2.475
P105	2.150	wire	12	620	27.850	192
P106	2.200	wire	12	620	30.000	207
P107	9.516	wire	12	620	5.310	36.607
P108	9.760	wire	12	620	0.080	0.552
P109	9.750	wire	12	620	0.056	0.386
P110	9.765	match	12	620	0.203	1.399
P111	9.493	match	12	620	2.492	17.180

Test Number	Refrigerant Vol %	Ignition Type	Starting Pressure (psia/torr)		Overpressure (psi /kPa)	
32R1	14.000	DC	12	620	0.020	0.138
32R2	15.060	DC	12	620	30.000	207
32R3	14.500	DC	12	620	0.030	0.207
32R4	14.490	DC	12	620	0.103	0.710
32R5	27.970	DC	12	620	0.734	5.060
32R6	26.000	DC	12	620	13.230	91.200
32R7	27.000	DC	12	620	0.008	0.055
32R8	27.020	DC	12	620	0.005	0.034
32R9	26.000	DC	12	620	18.386	126.753
32R10	26.492	DC	12	620	4.431	30.547
32R11	15.000	wire	12	620	0.063	0.434
32R12	17.000	wire	12	620	0.050	0.345
32R13	15.000	match	12	620	30.000	207
32R13a	13.070	match	12	620	30.000	207
32R14	11.000	match	12	620	2.998	20.670
32R15	10.000	match	12	620	2.492	17.180
32R16	8.000	match	12	620	1.533	10.569
32R17	6.000	match	12	620	1.128	7.776
32R18	26.020	match	12	620	11.919	82.170
32R19	27.980	match	12	620	4.095	28.231
32R20	30.000	match	12	620	0.538	3.709
32R21	0.000	match	0	0	0.010	0.069
32R22	0.000	match	0	0	0.102	0.703
32R23	27.010	match	12	620	0.936	6.453
SP1	13.9	AC	12	620	0.110	0.758
SP2	15.120	AC	12	620	30.000	207
SP3	14.500	AC	12	620	1.149	7.921
SP4	28.000	AC	12	620	1.120	7.721
SP5	29.000	AC	12	620	0.730	5.033
SP6	30.000	AC	12	620	0.350	2.413
SP7	31.000	AC	12	620	0.105	0.724

TABLE D-2. TEST RESULTS, R32 BLENDS, EXPLOSION SPHERE.

Test Number	Components	Refrigerant Volume %	32 Weight %	Ignition Type	Starting Pressure (psia/torr)		Humidity	Overpressure (psi /kPa)	
SP8	32/134a	20%	40	AC	12	620	low	0.100	0.689
SP9	32/134a	20%	47.5	AC	12	620	low	0.685	4.722
SP10	32/134a	20%	50	AC	12	620	low	1.647	11.354
SP11	32/134a	20%	45	AC	12	620	low	0.170	1.172
SP12	32/134a	20%	30	AC	12	620	high	0.085	0.586
SP13	32/134a	20%	35	AC	12	620	high	0.380	2.620
SP14	32/134a	20%	40	AC	12	620	high	1.122	7.735
SP15	32/134a	20%	35	AC	12	620	high	0.330	2.275
SP16	32/125	20%	50	AC	12	620	high	0.215	1.482
SP17	32/125	20%	60	AC	12	620	high	1.373	9.465
SP18	No test								
SP19	32/125	20%	60	AC	12	620	low	0.259	1.786
SP20	32/125	20%	65	AC	12	620	low	0.823	5.674
SP21	32/125	20%	65	AC	12	620	high	3.271	22.550
SP22	32/134a/125	20%	30	AC	12	620	high	0.074	0.510
SP23	32/134a/125	20%	40	AC	12	620	high	0.485	3.344
SP24	32/134a/125	20%	50	AC	12	620	high	2.463	16.980
SP25	32/134a/125	20%	40	AC	12	620	low	0.094	0.648
SP26	32/134a	20%	40	DC	12	620	low	0.027	0.186
SP27	32/134a	20%	47.5	DC	12	620	low	0.016	0.110
SP28	32/134a	20%	52	DC	12	620	low	0.419	2.889
SP29	32/134a	20%	57	DC	12	620	low	5.274	36.359
SP30	32/134a	20%	57	AC	12	620	low	7.783	53.656
SP31	32/134a/125	20%	50	AC	12	620	low	0.801	5.522
SP32	32/134a/125	20%	60	AC	12	620	low	11.778	81198
SP33	32/134a/125	20%	40	AC	12	620	low	0.074	0.510
SP34	32/134a	20%	47.5	AC	8	465	low	0.062	0.427
SP35	32/134a	20%	47.5	AC	14.7	760	low	1.258	8.673
SP36	No test								
SP37	32/134a	20%	47.5	AC	12	620	low	1.067	7.356

TABLE D-3. TEST RESULTS, ASTM E681 FLASK.

Test Number	Components	Refrigerant Volume %	32 Weight %	Ignition Type	Starting Temp	Humidity	Overpressure (psi /kPa)		Flammable?
1	32	30	100	DC	RT	low	0.164	1.131	x
2	32	28	100	DC	RT	low	4.381+	30.203+	x
3	32	29	100	DC	RT	low	0.829	5.715	no video
4	32	30	100	DC	RT	low	0.314	2.165	x
5	32	30	100	Match	RT	low	1.114	7.680	x
6	32	31	100	Match	RT	low	0.778	5.364	
7	32	30	100	AC	RT	low	0.231	1.592	
8	32	31	100	AC	RT	low	0.141	0.972	
9	32	32	100	AC	RT	low	0.100	0.689	
10	32	33	100	AC	RT	low	0.072	0.496	
11	32	33	100	AC	RT	low	0.070	0.483	
12	32	35	100	AC	RT	low	0.044	0.303	
13	32	32	100	DC	RT	low	0.019	0.131	
14	32/134a	20	56	AC	RT	low	1.151	7.935	x
15	32/134a	20	52	AC	RT	low	0.637	4.391	x
16	32/134a	20	58	AC	RT	low	1.215	8.376	x
17	32/134a	20	56	AC	RT	low	0.845	5.825	x
18	32/134a	20	61.75	AC	RT	low	1.793+	12.361+	x
19	32/134a	20	47.5	AC	RT	low	0.179	1.234	
20	32/134a	18	56	AC	RT	low	0.873	6.018	x
21	32/134a	22	56	AC	RT	low	0.335	2.309	x
22	32	29	100	AC	RT	low	0.301	2.075	
23	32	28	100	AC	RT	low	1.203	8.293	x
24	32	29	100	AC	RT	low	0.414	2.854	x
25	32	29	100	AC	RT	low	0.587	4.086	x

TABLE D-3. TEST RESULTS, ASTM E681 FLASK (CONTINUED).

Test Number	Components	Refrigerant Volume %	32 Weight %	Ignition Type	Starting Temp	Humidity	Overpressure (psi /kPa)		Flammable?
26	32/134a	20	54	AC	RT	low	0.740	5.102	no video
27	32/134a	20	58	DC	RT	low	1.306+	9.004	x
28	32/134a	20	56	DC	RT	low	0.022	0.152	
29	32/134a	20	58	DC	RT	low	1.133	7.811	x
30	32/134a	20	57	DC	RT	low	0.508	3.502	x
31	32/134a	18	56	DC	RT	low	0.071	.0489	
32	32/134a	20	47.5	7.5 kV	RT	low	X	X	
33	32/134a	20	47.5	10 kV	RT	low	X	X	
34	32/134a	20	47.5	12.5 kV	RT	low	X	X	
35	32/134a	20	47.5	15 kV	RT	low	X	X	
36	32/134a	20	47.5	15 kV	RT	low	X	X	
37	32/134a	20	58	DC	RT	low	2.328	16.029	x
38	32	29.1	100	DC	RT	low	1.175	8.100	x
39	32/134a	18	58	DC	RT	low	0.251	1.730	x
40	32/134a	22	58	DC	RT	low	0.197	1.358	
41	32/134a	20	56	DC	RT	low	0.038	0.262	
42	32/134a	20	58	AC	RT	low	1.405+	7.204+	x
43	32/134a	20	56	AC	RT	low	0.921	6.349	x
44	32/134a	20	57	AC	RT	low	1.096	7.556	x
45[1]	32/134a	20	57	AC	RT	low	1.046	7.211	x
46[2]	32/134a	20	57	AC	RT	low	1.208	8.328	x
47[3]	32/134a	20	57	AC	RT	low	1.146	7.961	x
48[4]	32/134a	20	57	AC	RT	low	0.991	6.832	x
49[5]	32/134a	20	57	AC	RT	low	0.218	1.503	
50[5]	32/134a	20	57	AC	RT	low	0.000	0.000	
51[4]	32/134a	20	57	AC	RT	low	1.020	2.032	x

TABLE D-3. TEST RESULTS, ASTM E681 FLASK (CONTINUED).

Test Number	Components	Refrigerant Volume %	32 Weight %	Ignition Type	Starting Temp	Humidity	Overpressure (psi /kPa)		Flammable?
52	No test						X	X	x
53	No test						X	X	x
54	32	28	100	AC	RT	low	.882+	6.081+	x
55	32	28	100	AC	RT	high	0.332	2.289	
56	32	28	100	AC	RT	mid	0.968	6.673	x
57	32	28	100	AC	RT	low	.954+	6.577+	x
58	32/134a	20	57	AC	RT	high	1.285+	8.859+	x
59	32/134a	20	57	AC	RT	high	2.487+	17.145+	x
60	32/134a	20	57	AC	RT	low	1.185+	8.169+	x
61	32/134a	20	52	AC	RT	high	1.634+	11.265+	x
62	32/134a	20	52	AC	RT	mid	1.707+	11.768+	x
63	32/134a	20	52	AC	RT	low	0.347	2.392	x
64	32/134a	20	47.5	AC	RT	high	2.835	19.544	x
65	32/134a	20	40	AC	RT	high	1.199	8.266	x
66	32/134a	20	35	AC	RT	high	0.360	2.482	x
67	32/134a	20	30	AC	RT	high	0.241	1.661	
68	32/134a	20	40	AC	RT	low	0.153	1.054	
69	134a	12	100	AC	RT	high	0.049	0.338	
70	32/125	20	65	AC	RT	low	0.303	2.089	
71	32/125	18	65	AC	RT	low	0.173	1.193	
72	32/125	22	65	AC	RT	low	0.147	1.013	
73	32/125	20	65	AC	RT	high	1.608+	11.086+	x
74	32/134a/125	20	30	AC	RT	low	0.131	0.903	
75	32/134a/125	22	30	AC	RT	low	0.101	0.696	
76	32/134a/125	20	30	AC	RT	high	0.099	0.683	
77	32/134a/125	20	40	AC	RT	low	0.085	0.586	

TABLE D-3. TEST RESULTS, ASTM E681 FLASK (CONTINUED).

Test Number	Components	Refrigerant Volume %	32 Weight %	Ignition Type	Starting Temp	Humidity	Overpressure (psi /kPa)		Flammable?
78	32/134a/125	20	50	AC	RT	low	0.168	1.158	
79	32/134a/125	20	60	AC	RT	low	1.220	8.411	x
80	32/134a/125	20	55	AC	RT	high	0.000	0.000	
81	32/134a/125	20	55	AC	RT	low	0.490	0.338	x
82	32/134a/125	20	55	AC	RT	high	2.688+	18.531+	x
83	32/134a/125	20	40	AC	RT	high	0.165	1.138	
84	32/134a/125	20	50	AC	RT	high	1.644+	11.334+	x
85[6]	32/134a	20	57	AC	RT	low	1.014	6.991	x
86[7]	32/134a	20	57	AC	RT	low	1.100	7.583	x
87[8]	32/134a	20	57	AC	RT	low	1.107+	7.632+	x
88	32/134a	20	57	AC	100C[9]	low	3.075+	21.199+	x
89	32/134a	20	57	AC	100C	low	4.111+	28.341+	x
90	32/134a	20	43.3	AC	100C	low	1.766	12.175	x
91	32	29	100	AC	100C	low	0.782	5.391	x
92	32	30	100	AC	100C	low	0.452	3.116	x
93	32	31	100	AC	100C	low	0.112	0.772	
94	32/134a	20	43.3	AC	100C[9]	low	1.045	7.204	x
95	32/134a	20	38.4	AC	100C	low	0.173	1.193	
96	32/134a	20	40.8	AC	100C	low	0.244	1.544	
97	32/134a	20	38.4	AC	100C	high	1.453+	10.017+	x
98	32/134a	20	26.9	AC	100C	high	0.386	2.661	
99	32/134a	20	29.1	AC	100C[9]	high	0.637	4.391	x
100	32/134a	20	25.3	AC	100C	high	0.145	1.000	
101	32	30	100	AC	100C	high	0.186	1.282	
102	32/125	20	65	AC	100C	low	0.590	4.067	x
103	32/125	20	59.8	AC	100C	low	0.196	1.351	no video

TABLE D-3. TEST RESULTS, ASTM E681 FLASK (CONTINUED).

Test Number	Components	Refrigerant Volume %	32 Weight %	Ignition Type	Starting Temp	Humidity	Overpressure (psi /kPa)		Flammable?
104	32/125	20	64	AC	100C	high	2.895+	19.952+	x
105	32/125	20	56.6	AC	100C	high	0.985	6.791	x
106	32/125	20	60	AC	100C	high	1.766+	12.175+	x
107	32/125	20	50	AC	100C	high	0.384	2.647	x
108	32/125	20	45	AC	100C	high	0.184	1.268	
109	32/134a/125	20	39.6	AC	100C	high	1.234+	8.507+	x
110	32/134a/125	20	29.7	AC	100C[9]	high	0.359	2.475	x
111	32/134a/125	20	30	AC	100C	high	0.154	1.062	
112	32/134a/125	20	33.2	AC	100C	high	0.472	3.254	x
113	32/134a/125	20	32.4	AC	100C	low	0.105	0.724	
114	32/134a/125	20	23	AC	100C[9]	low	0.052	.0358	
115	32/134a/125	20	23	AC	100C	high	0.007	0.048	
116	32/125	20	53	AC	100C	high	1.286+	8.866+	x
117	32/125	20	53	AC	100C	low	0.087	0.600	
118	32/125	20	60	AC	RT	low	0.160	1.103	
119	32/125	20	60	AC	RT	high	0.781	5.384	x
120	32/125	20	55	AC	RT	high	0.301	2.075	
121	32/125	20	50	AC	RT	high	0.154	1.062	
122	32	10	100	AC	RT	low	0.038	.0262	
123	32	12	100	AC	RT	low	0.076	0.524	
124	32	13	100	AC	RT	low	0.119	0.820	
125	32	14	100	AC	RT	low	0.119	0.820	

TABLE D-3. TEST RESULTS, ASTM E681 FLASK (CONCLUDED).

Test Number	Components	Refrigerant Volume %	32 Weight %	Ignition Type	Starting Temp	Humidity	Overpressure (psi /kPa)		Flammable?
126	32	15	100	AC	RT	low	0.224	1.544	
127	32	16	100	AC	RT	low	0.994+	6.853	x
128	32	15.5	100	AC	RT	low	0.409	2.820	

[1] No mixing - five minute wait
[2] No mixing - no waiting time
[3] Two minute mixing - stirrer moving slowly
[4] Two minute mixing stirrer off
[5] Two minute mixing - stirrer on
[6] 7-mm electrode separation
[7] 5-mm electrode separation
[8] 1-cm electrode separation
[9] Flask loaded cold and raised to 100°C