

UL 9540A Test Method for Evaluating Thermal Runaway Fire Propagation in Battery Energy Storage Systems

Cell Level Test Report Model Model 6LH3L8

Prepared by UL LLC for Contemporary Amperex Technology Co., Limited Issued: May 31, 2019

Project Number 4788679452

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### **Summary of UL 9540A Test Results**



### UL 9540A Test Report for Contemporary Amperex Technology Co., Limited

#### **Battery Energy Storage Description**

#### **Battery Energy Storage System Configuration**

Table 1 – Product details

Cell		
Manufacturer	Contemporary Amperex Technology Co Limited	
Model Number	6LH3L8	
Chemistry	Lithium Iron Phospate/Graphite (LFP)	
Electrical Ratings	3.2 V and 271 Ah	
Dimensions	71.57 ±1mm x 173.9 ±1mm x 207.3 ±1mm	
Construction Description	Prismatic	
Tested by UL to UL 1642?	Yes (MH63050)	
Tested by UL to UL 1973?	No	

#### Figure 1 – Photo of cell

### **Cell Level Test Description**

#### **Sample Preparation**

Five cells were prepared according to the requirements specified in UL 9540A Section 6.1.

#### Cell Conditioning and Stabilization (Section 8.1.4)

The cells were subject to a cycle of pre-charge, discharge, charge, discharge, and then final charge. The cells were charged to 100% state of charge (SOC) per the manufacturer's instructions, summarized in Table 2. Charging profiles are shown in Figures 2a – 2e.

#### Table 2 – Conditioning parameters

Charging		Discharging	
Rated Charge Current	271 A	Rated Discharge	271 A
		Current	
Maximum Charge	3.65 V	Rated Voltage	3.2 V
Voltage			
Cutoff Current	13.55 A	Cutoff Voltage	2.5 V

The ambient temperature of the space during cell conditioning was maintained within  $25\pm5^{\circ}C$  (77 $\pm9^{\circ}F$ ) and  $50\pm25\%$  RH.

Final charge capacity for each cell was presented in Table 3. Individual charge/discharge profiles are included in the figures below.

Cell Number	Test Number	Final Charge (Ah)
1	N/A	N/A
2	1	271.22
3	N/A	N/A
4	2	269.53
5	3	267.79
6	5	267.94
7	4	270.15

Table 3 – Final cell charge	capacities
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Figure 2a – Plot of cell charge data Cell 2



Figure 3b – Plot of cell charge data Cell 4



Figure 4c – Plot of cell charge data Cell 5



Figure 5d – Plot of cell charge data Cell 6



Figure 6e – Plot of cell charge data Cell 7

A plot of open circuit voltage (OCV) and state of charge (SOC) was not developed to determine cell state of charge. Instead, cell state of charge was verified by charge capacity at the end of the final charge cycle.

More than one hour elapsed after completing cell charging and before testing each cell, as shown in Table 4.

Cell Test #	Cell Number	Charge Completion Time	Cell Test Time
1	2	01/24/19 01:52 PM	01/28/19 02:35 PM
2	4	01/28/19 05:42 PM	01/29/19 10:20 AM
3	5	01/29/19 07:30 AM	01/29/19 02:33 PM
4	7	01/30/19 10:37 AM	01/30/19 12:27 PM
5	6	01/30/19 12:05 PM	01/30/19 05:06 PM

Table 4 – Charge completion and cell test initiation times

#### **Cell Level Test Instrumentation**

The initiating cell was instrumented with a total of five Type K thermocouples. One on the side of the cell body, two under the heater, one above the vent, and one on the plastic terminal base. Flexible film heaters were applied to both large faces of the cell. See Figure 3 for thermocouple and heater setup.



Figure 3 – Thermocouple and Heater setup

To represent the as-deployed configuration, all cell tests were conducted using an aluminum jig to mechanically constrain the heaters in close proximity to the cell. The jig consisted of two 6.35 mm thick aluminium plates. Figure 4 shows a completely assembled cell test assembly positioned on a noncombustible test surface. The bolts were fastened so as to prevent outward movement of the jig plates, but without applying initial pressure to the cell.



Figure 4 – Completed cell test setup

#### **Cell Level Test Procedure**

The test was initiated by supplying voltage to the heater adhered to the surface of the cell. Voltage was manually increased at a rate sufficient to achieve a 4°C/min to 7°C/min heating rate of the initiating cell until thermal runaway was observed. When thermal runaway was observed, the heater was immediately de-energized. From this point, the performance of the cell was observed.

#### **Cell Level Test Results**

#### Test 1 – Demonstration of Thermal Runaway Propensity by Heating Externally with Flexible Film Heaters

Thermal Runaway Results		
Test Date	01/28/2019	
Test Start Time	02:35 PM	
Venting Time	03:08:24 PM	
Venting Temperature	149.34°C	
Thermal Runaway Time	03:23:42 PM	
Thermal Runaway Temperature	236.34°C	

Figure 7 shows the surface temperatures measured during the test, in which cell venting and thermal runaway were observed. Physical behavior of the cell is documented in Figure 8.



Figure 7 – Surface temperatures measured on cell



Figure 8 – Highlights of Cell Test 1

Figure 9 shows a photograph of the cell after the test.



Figure 9 – Photograph of cell after test

#### Test 2 – Repeat #1 of Thermal Runaway Methodology

Table 6 – Thermal runaway results

Thermal Runaway Results	
Test Date	01/29/2019
Test Start Time	10:20 AM
Venting Time	10:57:29 AM
*Venting Temperature	152°C
Thermal Runaway Time	11:08:26 AM
*Thermal Runaway Temperature	215°C

\*See rationale below for temperature rationale based on TC5.

Figure 9 shows the surface temperatures measured during the test, in which cell venting and thermal runaway were observed. Visual observations of cell venting and thermal runaway behavior were consistent with Cell Test 1 and are well represented by Figure 8. During the test the sides of the battery buldged enoughed and severed the connection of thermocouple TC 1 located on the side cell body. Based

Test 1 - 4, it can be seen that TC2 and TC3 are similar in temperature through the test. Based on Test 3 and 4, TC1 and TC5 are similar in temperature up until the thermal runaway event. Therefore we can infer that the Venting and Thermal Runaway temperatures can be determined for Test 2 based on the temperature measured on TC5.



Figure 9 – Surface temperatures measured on cell

Figure 10 and 11 shows photographs of the cell after the test. Figure 11 shows TC 1 that fell off from its location during testing.



Figure 10 -11 – Photographs of cell after test

#### Test 3 - Repeat #2 of Thermal Runaway Methodology

Table 7 – Thermal runaway results

Thermal Runaway Results		
Test Date	01/29/2019	
Test Start Time	02:33 PM	
Venting Time	03:08:40 PM	
Venting Temperature	149.79°C	
Thermal Runaway Time	03:21:14 PM	
Thermal Runaway Temperature	235.02°C	

Figure 12 shows the surface temperatures measured during the test, in which cell venting and thermal runaway were observed. Visual observations of cell venting and thermal runaway behavior were consistent with Cell Test 1 and are well represented by Figure 8.



Figure 12 – Surface temperatures measured on cell

Figure 13 shows a photograph of the cell after the test.



Figure 13 – Photograph of cell after test

#### Test 4 - Repeat #3 of Thermal Runaway Methodology

Table 8 – Thermal runaway results

Thermal Runaway Results	
Test Date	01/30/2019
Test Start Time	12:27PM
Venting Time	01:03:29 PM
Venting Temperature	160.93°C
Thermal Runaway Time	01:13:32 PM
Thermal Runaway Temperature	222.48°C

Figure 14 shows the surface temperatures measured during the test, in which cell venting and thermal runaway were observed. Visual observations of cell venting and thermal runaway behavior were consistent with Cell Test 1 and are well represented by Figure 8.



Figure 14 – Surface temperatures measured on cell

Figure 15 shows a photograph of the cell after the test.



Figure 15 – Photograph of cell after test

#### Test 5 – Gas Composition

The gas composition test was conducted with the battery test jig inserted into the battery gas composition test chamber and the chamber was sealed. The battery gas composition test chamber is an 82 L pressure vessel and is shown in Figure 16.



Figure 16 – Battery gas composition test chamber

Prior to initiating thermal runaway, the chamber's atmosphere was purged until a condition of less than 1% oxygen by volume, as shown in Figure 17. Following the purge of the chamber atmosphere, the chamber pressure was relieved to 0 psig (Figure 18).







Figure 18 – Pressure inside the battery gas composition test chamber before energizing the heaters

Thermal Runaway Results*		
Test Date	01/30/2019	
Test Start Time	05:06 PM	
Venting Time	05:43:47 PM	
Venting Temperature	109°C	
Thermal Runaway Time	06:17:53 PM	
Thermal Runaway Temperature	222.34°C	
*These results are not a direct comparison with the results from Cell Tests 1-4, as this test was		
conducted inside a pressure vessel with an inert gas atmosphere.		

Figure 19 shows the surface temperatures measured during the test, in which cell venting and thermal runaway were observed.



Figure 19 – Surface temperatures measured on cell

After the cell experienced thermal runaway, gasses collected inside the test chamber were transferred to a clean sample gas cylinder. The contents of the sample cylinder were analyzed via gas chromatography to identify and quantify the composition of the gases generated.

Gas Composition Test		
Volume of Gas Generated (STP Conditions)	153.5 L	

Gas that vented from the cell in Test 5 was collected and analyzed using gas chromatography. Table 11 summarizes the results of the types and volume fractions of gases identified. The complete analysis report is included in the Appendix.

Gas		Measured %
Hydrogen	H <sub>2</sub>	50.73
Carbon Monoxide	CO	11.17
Carbon Dioxide	CO <sub>2</sub>	24.86
Methane	CH <sub>4</sub>	6.60
Ethylene	$C_2H_4$	3.06
Ethane	$C_2H_6$	1.19
Propene	$C_3H_6$	1.01
Propane	C <sub>3</sub> H <sub>8</sub>	0.40
-	C <sub>4</sub> (Total)	0.88
-	C₅ (Total)	0.10
Total	-	100

Table 11 – Components measured in vented cell gas

Analysis of lower flammability limit (LFL), maximum pressure rise ( $P_{max}$ ), burning velocity ( $S_u$ ) of the cell thermal runaway gases was conducted using the methodologies specified in UL 9540A. Please refer to Appendix A for testing details. The results are as follows:

- LFL: 5.9%
- Pmax: 91 psi
- S<sub>u</sub>: 150 cm/sec

### **Summary of Cell Test Results**

#### **Cell Vent and Thermal Runaway Results**

A summary of cell venting times and temperatures, and thermal runaway time and temperatures are presented in Table 12.

Test	Venting Time (mm:ss)	Venting Temperature (°C)	Thermal Runway Time (mm:ss)	Thermal Runway Temperature (°C)
1	33:08	149	48:26	236
2	36:53	152	47:50	215
3	34:59	149	47:50	235
4	36:35	160	46:23	222
5 <sup>1</sup>	37:47	109	1:11:53	222

Table 12 – Summary of measurements collected in Cell Tests 1-5

The average vent temperature based upon tests 1, 2, 3, and 4 is 153 degrees C. The average thermal runaway temperature is 227 degrees C.

#### **Cell Thermal Runaway Gas Analysis**

The total amount of gas collected from the cell after thermal runaway was 153.5 L at STP. Flammability properties were determined empirically:

- LFL: 5.9%
- Pmax: 91 psi
- S<sub>u</sub>: 150 cm/sec

<sup>&</sup>lt;sup>1</sup> Cell test 5 is not included in the average venting temperature or thermal runaway temperature, as the test environment inside the gas composition chamber is not the same as free air.

## Appendix

Gas composition, LFL, burning velocity, and Pmax reports are provided below:



## Gas Composition Analysis of Project # 4788679452

ClientULContactMr. Thomas SkoweraReport issue dateMarch 29, 2019Report numberUL26328RP Gas Analysis





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#### 1. Project Details and Test Work Approval Statement

Quotation Number	31655
Job Number	26328
DEKRA Insight Facility	DEKRA Process Safety, 113 Campus Drive, Princeton, NJ 08540.
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	Michael F. Carolan
Report Reviewed by	Jena Dorrin, Test Engineer
	Jundfute
Date of Experimental Work	3/11/19-3/29/19

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#### 2. INTRODUCTION

This report summarizes the results of the testing to identify and quantify the components in gas samples supplied by UL.

This work is in response to quotation number 31655. A formal hazard assessment of the process / plant has not been conducted by DEKRA Process Safety and the consequences of specific process deviations have not been examined.

Study initiation date\*: 3/11/19 Start date of the experimental work: 3/29/19

\* Sample, purchase order or last information receipt date, whichever is the latter. The materials used in this assessment were supplied by UL.

Name of client: UL

Sample ID	UL. Sample ID
20190061-01	4788679452



Off-gases from a battery degradation experiment were submitted in a pressurized cylinder and characterized by multiple analytical techniques to identify and quantify the volatile reaction species. GC-TCD and GC-MS (HP5890A, Shimadzu GC 2010SE) were used to identify and quantify the components in the submitted sample. The gas sample cylinder was heated to about 30°C by wrapping the metal container with heating tape. A portion of the gas from the warmed cylinder was introduced into a Tedlar gas sampling bag and then injected into the GC. All major peaks were identified and quantified (Figure 1-3). Major peaks were identified and quantified (Figures 1-3 and Table 1) using a one-point calibration and mixed gas standards (Restek Refinery Gas Standard #1, Scott Gas 23442). Tables 1 and 2 show the measured concentrations.

Table 1. Gas Quantification		
Project # 4788679452		
Gas	Measured %	
H <sub>2</sub>	30.7	
СО	6.76	
CO <sub>2</sub>	15.04	
CH <sub>4</sub> (Methane)	3.99	
C <sub>2</sub> H <sub>4</sub> (Ethylene)	1.85	
C <sub>2</sub> H <sub>6</sub> (Ethane)	0.72	
C <sub>3</sub> H <sub>6</sub> (Propylene)	0.61	
C <sub>3</sub> H <sub>8</sub> (Propane)	0.24	
C <sub>3</sub> H <sub>4</sub> (Propadiene)	<0.02	
C4' *	0.53	
$n-C_5H_{12}$ (Pentane)	0.06	
C <sub>6</sub> H <sub>14</sub> (Hexane)	<0.02	

\* total C4 including (i-C4 (isobutane), t-C4 (trans-2-butene), n-C4 (n-butane), cis-C4 (cis-2-butene), 1-Butene, isobutene, and 1,3-butadiene)

Approximately 60.5% of the gas mass is accounted for in the Project # 4788679452 cell sample based on the hydrocarbon standards used. Most of the remainder of the gas are air components ( $N_2$ ,  $O_2$ ), with minor amounts of unquantified hydrocarbon species. Figure 4 shows the order of elution for the column used. The peaks on the left side of Figure 3 correspond to  $N_2$  and  $O_2$ . Table 2 shows the concentrations re-normalized, excluding  $O_2$ ,  $N_2$  and the unquantified species.



Table 2. Gas Quantification, excluding  $\mathsf{O}_2$  and  $\mathsf{N}_2$ 

	Project # 4788679452
Gas	Measured %
H <sub>2</sub>	50.74
СО	11.17
CO <sub>2</sub>	24.86
CH <sub>4</sub> (Methane)	6.60
C <sub>2</sub> H <sub>4</sub> (Ethylene)	3.06
C <sub>2</sub> H <sub>6</sub> (Ethane)	1.19
C <sub>3</sub> H <sub>6</sub> (Propylene)	1.01
C <sub>3</sub> H <sub>8</sub> (Propane)	0.40
C <sub>3</sub> H <sub>4</sub> (Propadiene)	-
C4' *	0.88
$n-C_5H_{12}$ (Pentane)	0.10
C <sub>6</sub> H <sub>14</sub> (Hexane)	-

\* total C4 including (i-C4 (isobutane), t-C4 (trans-2-butene), n-C4 (n-butane), cis-C4 (cis-2-butene), 1-Butene, isobutene, and 1,3-butadiene)





Figure 1: GC-TCD Hydrogen Assay Project # 4788679452 sample (top) vs H<sub>2</sub> Standard Mix (bottom)



Figure 2: GC-TCD CO-CO<sub>2</sub> Assay – Project # 4788679452 sample (top) vs CO-CO<sub>2</sub> Standard Mix (bottom)







Figure 3. GC-MS Hydrocarbon Assay – Project # 4788679452 sample (top) vs. Standard Mix (bottom)

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Figure 4. Chromatogram showing the order of elution for the column used to produce the chromatogram in Figure 2.





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(c) For additional terms and conditions, which apply with respect to the provision of this report, see the Quotation provided by the Company and executed by Customer. If any terms set forth in the Quotation conflict with the terms set forth herein, the terms set forth herein shall apply.



## Flammability Characteristics Test Results Mixture Part # X10HY50C15AC001, Cylinder # CC316979

Client	Underwriters Laboratory LLC
<b>Client location</b>	333 Pfingsten Road
	Northbrook, IL 60062
Contact	Mr. Michael Fanta
	Research engineer II
Report issue date	May 31, 2019
Report number	2019042-1913



# **DEKRA**

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#### 1. PROJECT DETAILS AND TEST WORK APPROVAL STATEMENT

Quotation Number	3919
Project Number	1913
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Project Manager/ Author	Abdollah Kashani Senior Process Safety Specialist
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#### 2. INTRODUCTION

This report contains gas mixture flammability test data for Underwriters Laboratories LLC. Specifically, the following tests have been performed:

- Lower Flammable Limits (LFL)
- Explosion Severity Test (Kg)
- Flame Linear Speed / Flame Burning Velocity

This work is in response to UL. LLC's Purchase Order # 7202114572. The material used in this assessment was supplied by AirGas and identified as:

#### • Gas Mixture Part # X10HY50C15AC001 - Cylinder # CC316979

The Certificate of Analysis (COA) for the above gas mixture is shown in Section 4, below.

The purpose of the experiments is to determine the Lower Flammability Limit (LFL), Explosion Severity (Kg), flame speed and burning velocity characteristics of the above gas mixture. The test results will assist the client in providing a basis for a relative comparative flammability risk assessment to refrigerant R-32 (difluoromethane).

#### 3. EXECUTIVE SUMMARY OF TEST RESULTS

The Executive Summary of Test Results are presented in Table 1.

#### Table 1. Executive Summary of Flammability Test Results

#### **Material Tested:** Gas Mixture Part # **X10HY50C15AC001**, Cylinder # **CC316979 Reference Material Tested:** *R-32* (difluoromethane)

Parameter Tested/Analyzed	Results
Concentration Limits of Flammability (volume %)	
Lower Concentration Limits of Flammability (Measured, Vol.%, ASMT E681)	5.9
Lower Concentration Limits of Flammability (prediction, Estimated)	NA
Upper Concentration Limits of Flammability (prediction, Estimated)	NA
Explosion Severity Output	
Maximum Pressure (psig)	91
Maximum Pressure Rise Rate (psi/sec)	7162
Max. Temperature recorded 1" from the chamber wall(°C)	144.
Deflagration Index; Kg (bar·m/sec)	85
Burning Velocity	
Gas Mixture Linear Flame Speed (cm/sec)	410
Gas Mixture Burning Velocity (cm/sec)	150
R-32 Linear Flame Speed (cm/sec)	25.0
R-32 Burning Velocity (cm/sec)	6.67



All testing described in this report was conducted to measure and ascertain the properties of the materials tested in response to heat under controlled laboratory conditions. It should not be used to describe or appraise the fire hazards or fire risk of materials, products or assemblies under actual fire conditions. However, results of the tests may be used as elements of a fire risk assessment that take into account all of the factors that are pertinent to an assessment of the fire hazard of the particular end use.

#### 4. MATERIAL PREPARATION

R-32 (difluoromethane) vapor phase and the Battery Gas Mixture Gas Mixture Part # X10HY50C15AC001 - Cylinder # CC316979 was tested in the "as received" state.

The Battery Gas Mixture part # Part # X10HY50C15AC001 was formulated by AirGas and provided with the certificate of gas analysis, below.

Airgas USA, LLC 24075 US Hwy 6 Stryker, OH 43557 Airgas.com

#### **CERTIFICATE OF ANALYSIS** Grade of Product: CERTIFIED HYDROCARBON

Part Number:         X10HY50C15AC001           Cylinder Number:         CC316979           Laboratory:         124 - Stryker (SAP) - OH           Analysis Date:         May 06, 2019           Lot Number:         141-401483134-1	Reference Number: Cylinder Volume: Cylinder Pressure: Valve Outlet: Expiration Date:	141-401483134-1 171.2 CF 2000 PSIG 350 May 06. 2022	
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Traceability Statement: Hydrocarbon Process standards are NIST traceable either directly by weight or by comparison to Airgas laboratory standards that are directly NIST traceable by weight.

CERTIFIED CONCENTRATIONS				
	Requested	Reported		
Component	Concentration	Mole %	Accuracy	
N PENTANE	0.1000 %	0.0999 %	+/- 2%	
PROPANE	0.4000 %	0.3999 %	+/- 2%	
N BUTANE	0.8800 %	0.8798 %	+/- 2%	
PROPYLENE	1.000 %	1.001 %	+/- 2%	
ETHANE	1,200 %	1.204 %	+/- 2%	
ETHYLENE	3.000 %	3.005 %	+/- 2%	
METHANE	6.600 %	6.604 %	+/- 2%	
CARBON MONOXIDE	11.00 %	10.99 %	+/- 2%	
CARBON DIOXIDE	25.00 %	24.98 %	+/- 2%	
HYDROGEN	50.82 %	50.8364 %	+/- 2%	

Permanent Notes:UL LLC

PN# UL 4788679452

Notes: LABELED WITH 133433-P65-2

A tatis Approved for Release

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#### 5. LOWER AND UPPER LIMITS OF FLAMMABILITY TESTS ASTM E681

The testing was conducted at atmospheric pressure using ASTME 681-15, "Standard Test Method for Concentration Limits of Flammability of Chemicals." The material was tested to determine both Lower Flammable Limits (LFL) and Upper Flammable Limits (UFL). The Lower concentration Limits of Flammability tests were conducted at 25±2°C temperature and at atmospheric pressure.

#### <u>Test Setup</u>

The test apparatus consisted of a 5-liter combustion glass chamber (flask), a heating oven, a vacuum pump, a stirrer/propeller with drive motor, a high voltage power supply (for the ignition source) and a supply of controlled relative humidity air. The combustion chamber was a one-necked, round bottom glass flask having a total volume of 5,300 ml. The electrical oven was equipped with mechanical air convection to heat the flask uniformly. For gaseous sample a precision digital vacuum gauge of 0-800 mmHg range (Ashcroft Model 2074) was used to measure vacuum. The sample was introduced by measured volume or by partial pressure. Test setup is shown in Figure 1.

The ignition system (electrical spark) consisted of a high voltage luminous tube transformer (15,000 V, 30 mA) equipped with a timer relay and a set of Tungsten electrodes. The gap between the electrodes was spaced to be 6.3 mm ( $\frac{1}{4}$  in.).

#### <u>Procedure</u>

For each trial, a measured amount (by volume) of the material was injected via a syringe into the prevacuumed glass flask. The stirrer/propeller combination was turned on. flask was then brought back to atmospheric pressure with air.

A stirrer/impeller combination was left on for 3 minutes to obtain complete (gas/air) mixing and attainment of thermal equilibrium. After the mixing time, the stirrer was turned off. The ignition source, the high voltage luminous tube transformer (15,000 V, 30 mA) equipped with a timer (0.4 sec) was turned on after 15-20sec to arming mode. An electric spark between the two tungsten electrodes was then generated to ignite the gas mixture inside the flask. Observations were made to determine if the material gas/air mixture inside the flask ignited.

The flask was then purged with compressed air to clean out the combustion residue. The amount of the sample was varied between trials until the concentrations that could just sustain propagation, or non-propagation, of flame was determined.





Figure 1. Flammability Chamber setup for LFL/UFL determinations.



#### Flame Propagation Criterion

In a 5-liter glass combustion chamber, propagation of flame is defined as an upward and outward movement of the flame front from the ignition source to the vessel walls or at least to within 13 mm (1/2 in.) of the wall. The upward and outward propagation of the flame away from the ignition source is rated by visual observation.

#### LFL/UFL Calculations

Lower and Upper Flammability Limits (LFL and UFL) in terms of volume percent were calculated as follows:

$$LFL = \frac{L_m}{V}$$
 with  $L_m = \frac{L_l + L_2}{2}$ 

$$UFL = \frac{U_m}{V} \quad with \quad U_m = \frac{U_1 + U_2}{2}$$

Where:

 $L_1$  = Lowest sample amount of material which gives flame propagation

- $L_2$  = Highest sample amount (just below  $L_1$ ) of material which does not give flame propagation
- $U_1$  = Highest sample amount of material which gives flame propagation
- $U_2$  = Lowest sample amount (mg) (just above  $U_1$ ) of material which does not give flame propagation
- V = Total volume of flask used (in liters)



#### 5.1 Test Results for Lower Concentration Limit of Flammability

The lower concentration limit of flammability test results using a 5-liter flask are summarized in Table 2.

## Table 2 Lower Flammable Limit (LFL) and Upper Flammable Limit (UFL) Test Results

Material Tested: **Battery Gas Mixture** *Part # X10HY50C15AC001, Cylinder # CC316979* Standard Test Method used: ASTM E681-09 (Reapproved 2015) Volume of flask used: 5.25 Liter round bottom glass flask Environment Tested: Room air Test Temperature: 25±3°C

Test Pressure: Ambient atmospheric (750-760 mmHg)

Test	C			
Temperature (°C)	Flame Propagation	No-Flame Lower Flammable propagation Limit		Observations/ Remarks
25±2	6.1	5.7	5.9	Light Blue Flame

#### 5.2 Test Setup – Explosion Severity and Lower Flammability Limit

The test apparatus consisted of a 5-liter combustion test chamber, a vacuum pump, heating jacket (coils), an ignition trigger system, and a chart recorder and/or computer-based data acquisition system. The test chamber is of spherical shape with an inside diameter of 8.3 inches and made of a two semi-symmetrical stainless steel cylindrical blocks (Figures 1a and 1b). Sample liquid/gas ports are machined tangential to the chamber wall, providing turbulent motion (mixing) when gases/vapors are introduced. Three other ports are machined and threaded radially to support a rupture disk assembly, the ignition source and instrumentation (pressure transducer and temperature probe) assembly. The test chamber, with fitting, has a total volume of 5000-ml (5000-gram water). The chamber volume complies with ASTM E 2070-07, "Standard Test Methods for Limiting Oxygen (Oxidant) Concentration in Gases and Vapors<sup>17</sup> and NFPA 68" Standard on Explosion Protection by Deflagrating Venting" (Recent Edition).

The two-block chamber with seal (high-temperature material O-rings) was tightened firmly together with bolts (see Figures 1a and 1b). It is enclosed in a cylindrical-shaped metal jacket supporting a ceramic-beaded heating coil. The rupture disk assembly and the ignition source are connected to a Champion N-12YC spark plug and the instrumentation assembly was mounted on the chamber. One thermocouple was placed at the outer wall of the chamber and another one inside the chamber to control and monitor the temperature of the system. A pressure transducer and digital pressure gauge were connected to the chamber. The digital gauge was used to help in the loading of the test mixture(s) components. The pressure transducer (and digital gauge) monitors the internal pressure



during a test reaction. The chamber was also equipped with a motor driven mixing propeller assembly.

The completed apparatus was insulated with fiberglass materials. The test setup is shown in Figure 2 (insulation not shown). The test chamber was connected via metal pipe to a fume hood behind a barricaded wall with the rupture disk contained and directed away from the operators.

Measuring and monitoring equipment consisted of microprocessor-based temperature controllers for controlling and monitoring the temperature of the heating coils, and a piezoelectric pressure transducer for measuring the explosion pressure.



Figure 1a. Upper Hemisphere Part of 5-Liter Chamber



Figure 1b. Bottom Hemisphere of 5-Liter Chamber

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#### **5.3 Chamber Leak Test and Pressure and Temperature Calibration**

The 5-liter test chamber was designed to withstand 5,000-psig internal pressure. It was equipped with a 1,000-psig rupture disc for safety. Prior to calibration, the chamber was heated to the desired test temperature. A leak test was performed by pressurizing the combustion chamber to 300-psig nitrogen (or air) and then isolating it. The chamber pressure was monitored. A drop in the chamber pressure (if any) would indicate a leak. The chamber also was tested under vacuum conditions.

Upon completion of a satisfactory leak test using a digital pressure gauge (0-1000 psig), the chamber was then pressurized at the desired increments. The output of the pressure transducer for each pressure step change was recorded on the computer and cross-referenced with the digital pressure gauge.

#### 5.4 Test Procedure

The 5-liter chamber was heated to the desired test temperature (250± 5°C). The required gas cylinders of nitrogen and air were conditioned at room temperature. For each test, the condition of the chamber is inspected for residue from the prior test being released into the fume hood. If soot formation was noticed, the chamber was cleaned by a fine metal rotating brush and purged several times with nitrogen or air. Then the igniter was secured on the extended spark plug electrodes. The used silicone high-temperature O-ring of the spark plug was replaced by a new one. The spark plug (with igniter) was mounted on the chamber and tightened to seal the chamber. The release (vent) gas port valve was opened, and the chamber was purged with air (or nitrogen) gas.

The chamber was then evacuated to approximately minus 13-14 psig. The chamber pressure was then brought back with gas (air or gas mixture depending on the test program) to about 15-25 psig above atmospheric pressure. Once again, the chamber was evacuated. The gauge reading was recorded.

A measured amount of liquid sample (by weight) was inserted into the small sample port. The propeller mixer was then turned on.

Enough time (about 30-60 sec) was allowed for sample evaporation and temperature equilibrium. After the gas/vapor in the insulated chamber reached the desired test temperature, air was added to bring the chamber to the atmospheric pressure level. About two-minutes soak/mixing time was allowed for temperature to equilibrate and mixing of the vapor and air gas mixture.

The power supply leads were connected to electrodes and the electrodes shunt wire was removed. The data recording acquisition system was turned on. From a remote area behind a barricaded wall, the igniter was activated, and the pressure of the chamber was recorded for further analysis.

The pressure trace was analyzed for the development of the maximum reaction pressure, pressure rise rate output. After each test, the combustion chamber was vented. Then the spark plug assembly was removed and the chamber purged with a copious amount of compressed air to clean out any remaining combustion residues as noted earlier.



Successive tests were conducted at various concentrations to determine the maximum pressure output. Additional tests were conducted at one level below and one level above the sample concentration for maximum pressure output to confirm the results. Ignition/reactions of the mixtures were indicated by a sharp increase in pressure and/or temperature. The general criterion for flame propagation/reaction is stated below. Pressure rise due to igniter (if any) was subtracted from mixture reaction pressure.

#### 6. EXPLOSION SEVERITY TEST AND LOWER LIMIT OF FLAMMABILITY ASTM E918

This test method is designed to determine the deflagration parameters of a combustible vapor/gasair mixture within a spherical closed vessel of 5-liter or greater volume. The pressure/time curve is recorded on a suitable piece of equipment. The parameters measured are maximum pressure and maximum rate of pressure rise.

 $\mathbf{P}_{max}$  -is the maximum pressure reached during the course of a deflagration for the optimum concentration of the vapor/gas tested.

(dP/dt)<sub>max</sub> -is the maximum rate of pressure rise during the course of a single deflagration test. It is measured at the optimum vapor/gas concentration.

**Kg** -is known as deflagration index and is the maximum dP/dt normalized to a 1.0 m<sup>3</sup> volume.

$$Kg = (dP/dt)_{max} \times V^{1/3}$$

Where:

V= volume of vessel (m<sup>3</sup>) (dP/dt)<sub>max</sub> has units in bar/sec Kg has units in bar·m/sec



#### 6.1 Ignition system/Igniter

The ignition system consisted of the following:

Chemical Ignitor and Electrical Chemical Match. Squib (fireworks brand) Ignitor approximate energy output is about 10-15 Joules. The igniter was attached to modified extended spark plug electrode and secured at the center of the chamber. A 12 or 24-volt DC power source was used to activate the Chemical Match igniter.



Figure 2. 5-Liter stainless steel combustion chamber (insulation and some other parts are not shown).



#### 6.2 Criterion for Flame Propagation/ignition per ASTM E918

Using the definition from ASTM E918-83 (Reapproved 2005) "Standard Practice for Determining Limits of Flammability of Chemicals at Elevated Temperature and Pressure" and ASTM E 2070-07, "Standard Test Methods for Limiting Oxygen (Oxidant) Concentration in Gases and Vapors<sup>1</sup>," a propagation of flame is said to occur when a combustion reaction produces at least a 7% rise of the initial absolute pressure. This pressure increase amount to a 1 psia per atmosphere increase from the "initial" test pressure. Increase in temperature was also used as a secondary indication of reaction. The mixture in the chamber was considered flammable when the absolute pressure response of the reaction was greater than 1.07 times initial pressure, or if the pressure and/or temperature response shapes strongly suggest that a reaction took place. Therefore, the 7% rise criteria as used in these methods can be expressed as:

 $\frac{\Pr(\text{absolute reaction pressure})}{\Pr(\text{absolute initial pressure})} \ge 1.07$ 

#### 6.3 Test Results for Explosion Severity Test Result

The Explosion Severity test result is listed in Table 3. For comparison of results, reference NFPA 68 *"Standard on Explosion Protection by Deflagrating Venting"*, which has a list of explosion severity (Kg) for other gaseous materials.

## Table 3Explosion Severity Test Results

Material Tested: **Battery Gas Mixture** *Part # X10HY50C15AC001, Cylinder # CC316979* Combustion Vessel: 5-liter Stainless Steel Spherical Chamber Test Temperature: 23°C

Initial test Pressure: Atmospheric

Ignition Source: Electrical Chemical Match

		Explosion Severity Output					
Trial #	Sample Concentration (Volume%)	Reaction Pressure (psig)	Pressure Rise rate (psi /sec)	Temperature in the chamber (1" from wall) (°C)	Deflagration Index; Kg (bar·m/sec)		
1	9.8	33	188	95	2		
2	11.8	51	412	112	5		
3	13.8	62	1008	119	12		
4	15.7	70	2236	118	27		
5	17.7	77	3738	128	44		
6	19.7	84	6023	141	71		
7	20.6	88	5509	134	65		
8	21.6	88	6706	144	80		
9	21.6	87	5973	136	71		
10	21.6	87	4883	130	58		
11	21.6	88	6071	141	72		
12	22.6	90	7162	129	85		
13	22.6	90	5902	101	70		
14	23.6	89	6475	131	77		
15	23.6	90	6122	134	73		
16	25.6	88	5661	120	67		
17	27.5	85	4942	123	59		
<u>Overall</u>	Overall Test Results:						
<u>Explos</u>	sion Severity Outp	<u>out</u>	<u>Results</u>				
Maxin	num Pressure (psig	g)	91				
Maximum Pressure Rise Rate (psi/sec)		7,162					
in the	chamber (1" from	wall) (°C)	144				
Deflagration Index; Kg (bar·m/sec)			85				



#### 7. BURNING VELOCITY (BV) MEASUREMENT

Maximum burning velocity can be used to rank the flammability of flammable vapors and gases. Two methods which can be used to measure the burning velocity of fuels are the vertical tube and cylindrical vessel. The vertical tube method was used in this project.

The Burning Velocity (BV) is the velocity at which the flame propagates in a normal direction relative to the unburned gas ahead of it. The heat released during this exothermal reaction and the BV are related, and the higher the heat release, the higher the temperature rise and the faster the burning rate. The reaction rate introduces the time during which the reaction occurs and is expressed by the BV, so the BV is an essential parameter, measuring how fast a given substance will react when it is ignited.

The flammability hazard can be related to the BV, especially when a relative scale is established. Since burning behavior of an alternative refrigerant mixture in a tube is considered as one of the parameters for refrigerant classification in ANSI/ASHRAE Standard 34-2016 as well as other engineering fire risk assessments.

#### 7.1 Test Setup for Burning Velocity (BV)

The experimental apparatus consisted of two glass tubes 150 cm long with internal diameter of 40 mm. The two-tube setup allowed for running two tests on the same concentration almost simultaneously. The tubes were mounted vertically. The tungsten electrodes were fitted in the bottom part of the tubes. The electrodes were spaced ¼" apart and were attached to a 15kV 30 mA power supply. The top and bottom of the tubes was fitted with a system of valves that allowed vacuuming of the tubes and then filling them with a premixed vapor phase of refrigerant with air in a desired concentration at atmospheric pressure. The top valve was then closed, bottom valve was opened and the mixture was ignited with a power supply activated for duration of 0.4 sec.

The experimental procedure was similar to the one outlined by Jabbour<sup>1</sup> for the measurement of burning velocity.

If a flame was observed it was digitally recorded and analyzed. The digital image allowed for the calculation of the flame surface area and measurement of flame linear speed. The tube was then flushed with air and set for new run.

The R32 refrigerant vapor phase or Gas Mixture in desired concentrations was prepared and loaded in a pre-evacuated cylinder by partial pressure with dry air. For each trial, enough mixture with air was prepared to ensure the complete flush of the pre-evacuated test tubes to reach atmospheric pressure and bubble the excess through water. Two tests at each concentration were performed.

<sup>1</sup> <u>References</u>

Jabbour, T.; *Flammable Refrigerant Classification Based on the Burning Velocity;* Doctoral Thesis; Ecole Des Mines De Paris; 2004.



#### 7.2 Calculation of Linear Flame Speed and Burning Velocity

A photo of flame propagation inside the tube was taken for each trial using a digital camera. The flame front shapes are acquired with an average rate of 30 frames/second, or (60 frames/second), then images were transferred to a PC where they are saved for further processing (flame propagation speed measurement and flame front area calculation). With weakly luminous flames, a compromise was arranged between the flame front precision brought by a larger quantity of light admitted to the camera (longer exposure time) and the precision of the flame front position (displacement of the flame front during the exposure time). The camera field was adjusted to the appropriate position where the flame movement inside the tube is known to be uniform. A set of adjustments and different operating modes, including exposure time, number of frames during record, background light, and parameters for better images output, were made as appropriate.

The linear flame propagation speed is obtained from the direct measurement of the flame front displacement brought by two successive images in a known time interval that is given by the camera acquisition frequency. The flammable gas-air mixture, when ignited at the lower end of a vertical tube and propagated up to the upper end, the flame front propagation is at a constant speed; the shape and the size of the flame are also constant. As the temperature and pressure of the unburned mixture ahead of the flame are constant, the volume of burned gas can be calculated. The expression for the BV, (Su) is obtained from the equation of mass conservation for the unburned gas. The volume of burned gas per second and per unit area, or the BV, is obtained by dividing the mixture volume consumed per second (at the test temperature and pressure) by the flame surface area, (A). The volume consumption of the mixture per second is the volume swept by a cross-sectional area of the flame base, (a) with a speed equal to the propagation speed of the flame, (Ss).

This relation can be written as follows:

#### $Su = Ss \times a / A$

At a given temperature and pressure, the BV is only a function of the flammable substance, its concentration with the oxidant, and, to a limited extent, of the experimental apparatus. Therefore, the equation above implies the knowledge of three parameters:

- 1. The flame propagation speed, Ss
- 2. The flame-base cross section, a
- 3. The flame front area, A





Figure 3. Typical picture of upward flame propagation in 40-mm ID glass tube (flame shape for R32)

The flame front area was determined by marking the flame front profile with a fitting line (points), dividing it into two or more sections, and for each section an equation of appropriate area for that shape was used. The area of each section is then calculated separately and added together. The area of each elementary section is calculated based on the assumption of a revolution shape.

#### 7.3 Burning Velocity Apparatus Validation

A test run was performed to verify if the setup produces correct numbers for flame speed. Pure R32 (difluoromethane) was used as a reference material (per ASHRAE Std 34). Tests were run at ambient temperature using dry air to prepare test samples.

The results are shown in the Table 4 below and graphed as shown in Figure 4.



#### 7.4 Test Results for Burning Velocity of R-32

#### Table 4

#### Flame Speed and Burning Velocity Test Results (Reference Material)

Reference Material Tested: R32 (difluoromethane) Test Method: See Reference below Environment: Dry Air Ignition Source: Electrical Spark Glass Tube: 40 mm, ID 85mm OD. 1500mm long (closed top end during ignition) Glass Tube Temperature: ambient (20±3°C) Test Pressure: Atmospheric (760±10 mmHg)

Trial #	Concentration Tested (Vol.%)	Linear Flame Speed Average of Two Trials (cm/sec)	Linear Flame Speed Maximum (cm/sec)	Flame Area/Cross Section	Burning Velocity (cm/sec)
1	17	24.01	24.01	3.93	6.11
2	18	24.25	24.48	3.70	6.62
3	19	24.74	25.00	3.75	6.67
4	20	25.00	25.00	4.05	6.17
5	21	24.01	24.01	3.70	6.49
6	22	24.25	24.01	3.87	6.20



Figure 4. Verification Test Results for R-32

The value for R32 burning velocity of 6.6-6-.7 cm/s is in agreement with value of 6.7 cm/s as reported in ASHARE Standard P-34.



#### 7.5 Test Results for Burning Velocity of Battery Gas Mixture

The same apparatus consisting of two 40-mm ID, 1500-mm long glass tubes as described earlier was used for experimental test with the Gas Mixture (X08CM36C33A0000). The linear flame speed of the gas mixture was 6.3 times faster the linear flame speed of R-32 reference refrigerant gas. Due to high flame speed of gas mixture, the distortion of the flame front propagation was noted. The flame front area was estimated based on the best assumption of flame shape recorded. The results for the Gas Mixture tested are shown in Table 5 below and graphed as shown in Figure 5. Flame Speed and Burning Velocity Test Results compared to Reference Material is shown in Table 6.

## Table 5Flame Speed and Burning Velocity Test Results

#### Material Tested: **Battery Gas Mixture Part # X10HY50C15AC001, Cylinder # CC316979** Environment: Dry Air Ignition Source: Electrical Spark Glass Tube: 40-mm, ID 50-mm OD. 1500mm long (closed top end during ignition) Glass Tube Temperature: ambient (20±3°C)

*Test Pressure: Atmospheric (760±10 mmHg)* 

		Tube 1			Tube 2			
Trial		Linear			Linear			
#	Concentration	Flame	Flame	Burning	Flame	Flame	Burning	
"	Tested	Speed	Area/Cross	Velocity	Speed	Area/Cross	Velocity	
	(Vol.%)	(cm/sec)	Section	(cm/sec)	(cm/sec)	Section	(cm/sec)	
1	14	142.9	NA		133.3	2.17	61.4	
2	15	150.4	2.57	58.5	157.9	3.01	52.5	
3	16	150.4	2.58	58.3	150.4	2.47	60.9	
4	17	114.8	NA		109.3	NA		
5	18	253.0	2.88	87.9	265.1	NA		
6	19	265.1	2.47	107.3	288.0	3.08	93.5	
7	20	306.0	2.66	115.0	253.0	2.85	88.8	
8	21	328.4	2.76	119.0	291.0	2.85	102.1	
9	22	410.0	2.77	148.0	350.0	2.33	150.2	
10	23	313.4	2.85	110.0	328.4	2.81	116.9	
11	24	270.1	2.55	105.9	276.1	2.6	106.2	
12	25	358.2	2.89	123.9	340.0	2.7	125.9	
13	26	335.8	2.46	136.5	313.4	3.65	85.9	
14	27	306.0	2.9	105.5	313.4	2.74	114.4	
15	28	283.6	2.81	100.9	283.6	2.46	115.3	
16	29	241.0	2.89	83.4	234.9	1.82	129.1	
17	30	241.0	2.54	94.9	234.9	2.97	79.1	
18	31	140.0	2.76	50.7	146.6	3.15	46.5	

NA: Not calculate due to excessive distortion of the flame front of the gas mixture tested.





Figure 5. Burning Velocity vs Concentration for Gas Mixture

Table 6
Flame Speed and Burning Velocity Test Results Compared to Reference Material

Glass Tube Temperature: ambient (20±3°C) Test Pressure: Atmospheric (760±10 mmHg)

Test Name	Description	Test Results
Burning Speed/Velocity		
(Battery Gas Mixture)	Linear Flame Speed (cm/sec)	410
Part # X10HY50C15AC001,	Burning Velocity (cm/sec)	150
Cylinder # CC316979		
Burning Speed/Velocity	Linear Flame Speed (cm/sec)	25.0
(Reference R32)	Burning Velocity (cm/sec)	6.67



#### 8. CONCLUSIONS AND RECOMMENDATIONS

The summary of experimental test results is presented earlier in Table 1. Due to the distortion of the flame front of the gas mixture tested, for the fire risk analysis, a proper safety margin (e.g. +10% or more) should be added to the values obtained in this report.

The hazardous properties of the substances, when determined carefully under desired process conditions and applied properly, will provide information on the reaction behavior of fire and explosion hazards of the specific substance under specific conditions.

The results of the above tests, conducted under controlled laboratory conditions, should not be considered sufficient to fully characterize the overall fire hazard or fire risk of the material (and assemblies) under actual fire conditions. However, the results may be used as data for fire risk potential, which takes into account all of the factors that are pertinent to an assessment of the fire hazard of a particular end use.

Knowledge of the explosion hazards of flammable vapor and gaseous fuels is of importance to ensure the safety in industrial and domestic applications that produce or use flammable mixtures. Engineering measures to control or eliminate the flammability conditions and ignition sources should adopted.

To future characterize the flammability of the above materials DEKRA Process Safety recommends the following thermal stability and flammability testing and analysis be considered for further study:

- Upper Concentration Limits of Flammability (UFL)
- Autoignition Temperature (AIT) Test
- Minimum Ignition Energy (MIE)
- Limiting Oxidant Concentration (LOC) Test
- Combustion By-Product Analysis
- Process Hazard Analysis (PSM, PHA, HZOP, HA, FMEA, etc.).

Electrostatic discharge from personal and equipment is sufficient to ignite the gaseous materials in air. Engineering control of ignition sources shall be implemented where these gas mixtures are processed. Information on and control of static electricity and other protection/prevention are referred to NFPA 77, 70, 68 and 69.

If you have any questions regarding the contents of this report, feel free to contact us at any time.

Sincerely, DEKRA Process Safety

A-Kashani

Abdollah Kashani Senior Process Safety Specialist



#### 9. APPENDIX

#### A) Legal Disclaimer and Liability

- Limitation of Liability. The test procedures and/or consulting services conducted by DEKRA (a) Process Safety (the "Company") were performed under controlled laboratory conditions, which the Company considers reliable. Although the Company performed its testing services pursuant to reliable and generally accepted testing procedures in the industry, the Company does not guarantee or provide any representations or warranties with respect to Client's use, interpretation or application of the test results and/or consulting services provided by the Moreover, the results of the testing procedures are based upon certain Company. assumptions, information, documents, and procedures provided by the Customer. AS SUCH, IN NO EVENT AND UNDER NO CIRCUMSTANCE SHALL THE COMPANY BE LIABLE FOR SPECIAL, INDIRECT, PUNITIVE OR CONSEQUENTIAL DAMAGES OF ANY NATURE WHATSOEVER, INCLUDING WITHOUT LIMITATION, ANY LOST REVENUE OR PROFITS OF THE CUSTOMER OR ITS CUSTOMERS, AGENTS AND DISTRIBUTORS, RESULTING FROM, ARISING OUT OF OR IN CONNECTION WITH, THE SERVICES PROVIDED BY THE COMPANY OR THE RESULTS OF ANY TESTS PERFORMED BY THE COMPANY. The Customer agrees that the Company shall have no liability for damages, which may result from Client's use, interpretation or application of the test results and/or consulting services provided by the Company.
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