

**EFFECT OF CARBON DIOXIDE (CO₂) SUPPRESSION ON EXPLOSIVE
LIMITS OF METHANE**

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BORANG PENGESAHAN STATUS TESIS

JUDUL : **EFFECT OF CARBON DIOXIDE (CO₂) SUPPRESSION ON
EXPLOSIVE LIMITS OF METHANE**

SESI PENGAJIAN: **2009/2010**

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A report submitted in partial fulfillment of the
requirements for the award of degree of
Bachelor of Chemical Engineering.

Faculty of Chemical Engineering & Natural Sources
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April 2010

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“I declare that this thesis entitled “Effect of Carbon Dioxide (CO₂) Suppression on Explosive Limits of Methane” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not currently submitted in candidature of any other degree.”

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Date : 13 APRIL 2010

DEDICATION

*To my beloved
My Dad Md Jais Bin Basim,
Mum Ramlah Binti Chik,
My brother Kamarul, Firdaus, Faiz
my sister Rogayah.
And my friends.....*

ACKNOWLEDGEMENTS

First I would like to express my grateful to ALLAH S.W.T as for his blessing I can finish my project.

In preparing this thesis, I was in contact with many people and academicians. They have contributed towards my understanding and thoughts and also guidance. In particular, I wish to express my sincere appreciation to my main thesis supervisor, Mr. Izirwan Bin Izhab, for his valuable guidance, advice and continuous encouragement, constructive criticisms and suggestion throughout this project. Without his continued support and interest, this thesis would not have been the same as presented here.

My sincere appreciation also extends to all my colleagues and others who have provided assistance at various occasions. Their views and tips are useful indeed in helping me to achieve doing this project. Unfortunately, it is not possible to list all of them in this limited space. I am grateful to all my family members for supporting strength and encouragement.

ABSTRACT

Knowledge of the explosion hazards of gaseous fuels is of importance to ensure the safety in industrial and domestic applications that produce or use flammable mixtures. Thus, the experiment is conducted to determine the upper and lower flammability limits of methane based on volume percent of inert gas (CO_2). The purpose of the experiment to reduce the concentration of oxygen below the limiting oxygen concentration (LOC) for the purpose of lowering the likelihood of explosion. The inert gas is usually nitrogen or carbon dioxide, although sometimes steam may be used. But in this experiment, gas CO_2 was used. The experiments were performed in a 20 L closed explosion vessel. The mixtures were ignited by using spark permanent wire that placed at the centre of the vessel. In this study, the result shows the lower explosive limit (LEL) and upper explosive limit (UEL) for CH_4 start from 5 vol % to 19 vol % and have revealed that the addition of suppression agent as 5 vol % and 10 vol % of CO_2 in CH_4 extends the LEL and UEL initially 6 vol % to 12 vol % and 3 vol % to 6 vol %.

ABSTRAK

Pengetahuan tentang bahaya letupan bahan api gas adalah penting untuk menjamin keselamatan dalam industri dan aplikasi dalam negeri yang menghasilkan atau menggunakan campuran mudah terbakar. Dengan demikian, percubaan dilakukan untuk menentukan batas atas dan bawah dari metana mudah terbakar berdasarkan peratus kelantangan gas inert (CO_2). Tujuan percubaan untuk mengurangkan kepekatan oksigen di bawah kepekatan oksigen menyekat (LOC) untuk tujuan menurunkan kemungkinan letupan. Gas inert ini biasanya nitrogen atau karbon dioksida, walaupun kadang-kadang stim boleh digunakan. Namun dalam percubaan ini, gas karbon dioksida (CO_2) digunakan. Percubaan dilakukan dalam 20-L tertutup kapal letupan. Campuran yang dinyalakan dengan menggunakan percikan kawat kekal yang ditempatkan di tengah-tengah kapal. Dalam kajian ini, hasilnya menunjukkan batas letupan rendah (LEL) dan batas letupan atas (UEL) untuk CH_4 adalah dari 5 % vol hingga 19 % vol dan telah mendedahkan bahawa penambahan agen penindasan sebagai 5 % vol dan 10 % vol CO_2 di CH_4 memperluaskan LEL dan UEL awalnya 6 % vol hingga 12 % vol dan 3 % vol ke 6 % vol.

TABLE OF CONTENTS

CHAPTER	TITLE	PAGE
	SUPERVISOR DECLARATION	iii
	STUDENT DECLARATION	v
	DEDICATION	vi
	ACKNOWLEDGEMENT	vii
	ABSTRACT	viii
	ABSTRAK	ix
	TABLE OF CONTENT	x
	LIST OF TABLES	xiv
	LIST OF FIGURES	xv
	LIST OF ABBREVIATIONS	xvi
	LIST OF APPENDICES	xvii
1	INTRODUCTION	
	1.1 Introduction	1
	1.2 Scope of Study	3
	1.3 Objectives Research	4
	1.4 Problem Statement	5

2	LITERATURE REVIEW	
2.1	Carbon Dioxide	6
2.2	Methane	8
2.3	20-Liter Spherical Explosion Vessel	10
2.4	Maximum Explosion Overpressure (P _{max}), Maximum Rate of Explosion Pressure Rise (dP/dt) _{max} , and Gas or Vapor Deflagration Index (K _g)	11
2.4.1	Minimum Oxygen Concentration (MOC)	11
2.5	Explosion Limits	12
2.5.1	Lower Explosive Limit (LEL)	12
2.5.2	Upper Explosive Limit (UEL)	13
2.5.3	Detonation	13
2.6	Gas and vapor cloud Explosion	14
2.6.1	Gas and Vapor Cloud Formation	15
2.7	Ignition	16
2.8	Explosion Efficiency	16
2.9	Explosion Suppression	17
2.9.1	Carbon Dioxide as Suppression Agent	18

3	RESEARCH METHODOLOGY	
3.1	Experimental Apparatus	19
3.1.1	20-L-Apparatus	20
3.1.2	Measurement and Control System KSEP 332	21
3.2	Experimental conditions	21
3.2.1	Pressure and temperature	22
3.2.2	Ignition	22
3.3	Experimental procedure	23
4	RESULTS AND DISCUSSIONS	
4.1	Methane (CH ₄) Explosion without the Presence of Suppressants	26
4.2	Methane (CH ₄) with the Presence of 5 vol% Carbon Dioxide (CO ₂)	28
4.3	Methane (CH ₄) with the Presence of 10 vol% Carbon Dioxide (CO ₂)	31
4.4	Comparison of the Data	33

5	CONCLUSION AND RECCOMENDATION	
	5.1 Conclusion	35
	5.2 Recommendation	36
	REFERENCES	37
	APPENDICES	40

LIST OF TABLES

TABLE NO.	TITLE	PAGE
2.1	Properties of CO ₂	7
2.2	Properties of CH ₄	9
3.1	Test condition of experiment	22
4.1 (b)	Summary P _{max} of CH ₄ Explosion	28
4.2 (b)	Summary P _{max} of CH ₄ Explosion with Presence of 5 vol% CO ₂	30
4.3 (b)	Summary P _{max} of CH ₄ Explosion with Presence of 10 vol% CO ₂	32
4.4	Comparison data of LEL, UEL, P _{max} and dp/dt between different vol % of suppression agent (CO ₂)	34

LIST OF FIGURES

FIGURE NO.	TITLE	PAGE
3.1	Schematic diagram of 20-L-Apparatus	19
3.2	Schematic diagram of experimental set up	20
3.3	20-L Apparatus	23
3.4	Diagram of igniter between the electrode rods	23
3.5	KSEPT 6.0	24
3.6	Experimental work flows	25
4.1 (a)	Graph Pressure Maximum (Pmax) vs vol% CH ₄	27
4.2 (a)	Graph Maximum Pressure (Pmax) vs 5% CO ₂ + CH ₄	29
4.3 (a)	Graph Maximum Pressure (Pmax) vs 10% CO ₂ + CH ₄	31

LIST OF ABBREVIATIONS

CH ₄	- Methane
CO ₂	- Carbon Dioxide
Vol %	- Volume Percent
LEL	- Lower Explosion Limits
UEL	- Upper Explosion Limits
P max	- Maximum Pressure
dp/dt	- Maximum Rate of Explosion Pressure Rise
K _g	- Gas Vapor Deflagration Index

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
A	Manual of 20-L-Apparatus	40
B	Technical Data of 20-L-Sphere	45
C	Data from computer	46

CHAPTER 1

INTRODUCTION

1.1 Introduction

Knowledge of the explosion hazards of gaseous fuels is of importance to ensure the safety in industrial and domestic applications that produce or use flammable mixtures. In accordance with generally accepted usage, the flammability limits are known as those regions of fuel–air ratio within which flame propagation can be possible and beyond which flame cannot propagate. And there are two distinct separate flammability limits for the fuel–air mixture, namely, the leanest fuel-limit up to which the flame can propagate is termed as lower flammability limit (LFL), and the richest limit is called as upper flammability limit (UFL).

It is now acknowledged that flammability limits are physical–chemical parameters of flammable gases and vapors of flammable liquids, which are related to many factors including the heat losses from the flame by conduction, convection and radiation to the apparatus walls, instabilities in the flame front resulting from buoyant convection, selective diffusion and flame stretch, as well as radical loss or their generation on apparatus walls. Thus, more attentions have been given to the study the effects of environmental parameters, such as the vessel size, initial temperature and pressure, on this fundamental characteristic. The standardized measurements of flammability limits are usually conducted in the flammability tubes or closed vessels.

Generally, large size of combustion chamber can minimize the wall effects and potentially allow to the use of strong igniters to ensure the absence of ignition limitation, so most of the flammability measurements were conducted in the closed chambers recently. In combustion vessel test, spark igniter is commonly adopted. It is known that the minimum ignition energy is a strong function of the compositions near the flammability limits.

There are several criteria to determine the flammability limits in experimental measurements. A successful attempt can be determined by one or a combination of the following two criteria: (1) visualization inspection of the flame kernel development produced by the spark, namely visual criterion; (2) measurements of the pressure and/or temperature histories in the vessel, where an appropriate pressure or temperature rise criteria can be used to designate flammability rather than the purely visual observation of flame generation.

There exist large array of experimental data on flammability limits for gaseous mixtures of fuel–air–inerts, and the inert gases considered herein were nitrogen gas, carbon dioxide or their mixture, which are different from the real residual gaseous in combustion chamber, and most of previous studies were conducted at atmospheric conditions. For engineering application, the fundamental research on flammability characteristics of fuel–air mixtures with combustion bomb meter, especially at high temperature and pressure like those of combustion is worthwhile. Practically, due to the time consumed in measurements, it is desirable to choose some reliable criteria for quick estimation of flammability limits.

1.2 Scope of study

Thus, for the purpose of prediction of effect CO_2 of flammability limits in CH_4 /air structure, this is the contents of the scope of study:

For this experiment, equipment that can be used is a bomb explosion unit. This unit usually has five streams which one will indicate the air/oxygen (O_2) and the other stream are fuel and inert stream.

This experiment is conducted to determine the upper and lower flammability limit of CH_4 based on volume percent of inert gas (CO_2).

A type of fuel hydrocarbon that is selected to be examined out is CH_4 and mixed with CO_2 . For the air stream, normally source of air is directly taken from the surroundings.

1.3 Objectives Research

To predict the upper/lower explosive limit of a mixture composed of hydrocarbon and inert CO₂. Different methods have been proposed to predict the explosive limits, especially the lower explosive limit (LEL), for pure flammable gases.

To determine the effect of CO₂ suppressing on flammability limits of premixed CH₄-air mixture in a combustion bomb at atmospheric pressure and ambient temperature.

To reduce the concentration of oxygen below the limiting oxygen concentration (LOC) for the purpose of lowering the likelihood of explosion. The inert gas is usually N₂ or CO₂, although sometimes steam may be used. But in this experiment, CO₂ was used.

1.4 Problem Statement

An early detection of an explosion is very important especially in the process industries as a prevention and explosion protection. A disadvantage this method is potential of the suppression agent to mitigate the explosion limits especially when dealing with CH_4 as a fuel source. This problem can be solved by added different vol % of suppression agent, CO_2 with the one which are more effective and can be rely on.

CHAPTER 2

LITERATURE RIVIEW

2.1 Carbon Dioxide (CO₂)

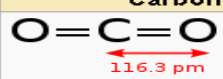
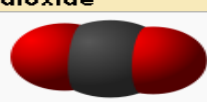
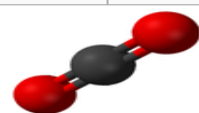
Carbon dioxide (chemical formula CO₂) is a chemical compound composed of two oxygen atoms covalently bonded to a single carbon atom. It is a gas at standard temperature and pressure and exists in earth's atmosphere in this state. CO₂ is a trace gas being only 0.038 % of the atmosphere.

Carbon dioxide is used by plants during photosynthesis to make sugars, which may either be consumed in respiration or used as the raw material to produce other organic compounds needed for plant growth and development. It is produced during respiration by plants, and by all animals, fungi and microorganisms that depend either directly or indirectly on plants for food. It is thus a major component of the carbon cycle. Carbon dioxide is generated as a by-product of the combustion of fossil fuels or the burning of vegetable matter, among other chemical processes. Small amounts of carbon dioxide are emitted from volcanoes and other geothermal processes such as hot springs and geysers and by the dissolution of carbonates in crustal rocks.

Carbon dioxide has no liquid state at pressures below 5.1 atmospheres. At 1 atmosphere (near mean sea level pressure), the gas deposits directly to a solid at temperatures below $-78\text{ }^{\circ}\text{C}$ ($-108.4\text{ }^{\circ}\text{F}$; $195.1\text{ }^{\circ}\text{K}$) and the solid sublimates directly to a gas above $-78\text{ }^{\circ}\text{C}$. In its solid state, carbon dioxide is commonly called dry ice.

Carbon dioxide is used in enhancing oil recovery where it is injected into or adjacent to producing oil wells, usually under supercritical conditions. It acts as both a pressurizing agent and, when dissolved into the underground crude oil, significantly reduces its viscosity, enabling the oil to flow more rapidly through the earth to the removal well. In mature oil fields, extensive pipe networks are used to carry the carbon dioxide to the injection points.

Table 2.1: Properties of Carbon Dioxide (CO_2)

Carbon dioxide	
	
	
Properties	
Molecular formula	CO_2
Molar mass	44.010 g/mol
Appearance	colorless, odorless gas
Density	1.562 g/mL (solid at 1 atm and $-78.5\text{ }^{\circ}\text{C}$) 0.770 g/mL (liquid at 56 atm and $20\text{ }^{\circ}\text{C}$) 1.977 g/L (gas at 1 atm and $0\text{ }^{\circ}\text{C}$) 849.6 g/L (supercritical fluid at 150 atm and $30\text{ }^{\circ}\text{C}$)
Melting point	$-78\text{ }^{\circ}\text{C}$, 194.7 K, $-109\text{ }^{\circ}\text{F}$ (<i>subl.</i>)
Boiling point	$-57\text{ }^{\circ}\text{C}$, 216.6 K, $-70\text{ }^{\circ}\text{F}$ (at 5.185 bar)
Solubility in water	1.45 g/L at $25\text{ }^{\circ}\text{C}$, 100 kPa
Acidity ($\text{p}K_{\text{a}}$)	6.35, 10.33
Refractive index (n_{D})	1.1120
Viscosity	0.07 cP at $-78\text{ }^{\circ}\text{C}$
Dipole moment	zero

2.2 Methane (CH₄)

Methane is a chemical compound with the chemical formula CH₄. It is the simplest alkane, and the principal component of natural gas. Methane's bond angles are 109.5 °C. Burning methane in the sufficient presence of oxygen produces carbon dioxide and water. The relative abundance of methane makes it an attractive fuel. However, because it is a gas at normal temperature and pressure, methane is difficult to transport from its source. In its natural gas form, it is generally transported in bulk by pipeline or LNG carriers; few countries transport it by truck.

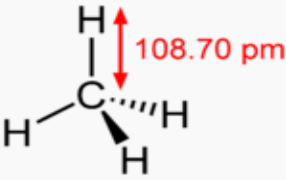


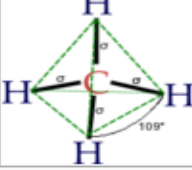
Methane is a relatively potent greenhouse gas with a high global warming potential of 72 (averaged over 20 years) or 25 (averaged over 100 years). Methane in the atmosphere is eventually oxidized, producing carbon dioxide and water. As a result, methane in the atmosphere has a half life of seven years.

Methane can trap about 20 times the heat of CO₂. In the same time period, CO₂ increased from 278 to 365 parts per million. The radiative forcing effect due to this increase in methane abundance is about one-third of that of the CO₂ increase. In addition, there is a large, but unknown, amount of methane in methane clathrates in the ocean floors. The earth's crust contains huge amounts of methane. Large amounts of methane are produced anaerobically by methanogenesis. Other sources include mud volcanoes, which are connected with deep geological faults, landfill and livestock (primarily ruminants) from enteric fermentation.

Methane is used in industrial chemical processes and may be transported as a refrigerated liquid (liquefied natural gas, or LNG). While leaks from a refrigerated liquid container are initially heavier than air due to the increased density of the cold gas, the gas at ambient temperature is lighter than air. Gas pipelines distribute large amounts of natural gas, of which methane is the principal component.

In the chemical industry, methane is the feedstock of choice for the production of hydrogen, methanol, acetic acid, and acetic anhydride. When used to produce any of these chemicals, methane is first converted to synthesis gas, a mixture of carbon monoxide and hydrogen, by steam reforming. In this process, methane and steam react on a nickel catalyst at high temperatures (700–1100 °C).

Table 2.2: Properties of Methane (CH₄)

Methane	
	
	
other names [hide] Methyl hydride, Marsh gas, firedamp	
Identifiers	
CAS number	74-82-8 ✓
PubChem	297
ChemSpider	291
SMILES	[show]
InChI	[show]
Properties	
Molecular formula	CH ₄
Molar mass	16.042 g/mol
Appearance	Colorless gas
Density	0.717 kg/m ³ (gas, 0 °C) 415 kg/m ³ (liquid)
Melting point	-182.5 °C, 91 K, -297 °F
Boiling point	-161.6 °C, 112 K, -259 °F
Solubility in water	35 mg/L (17 °C)

2.3 20-Liter Spherical Explosion Vessel

The experimental 20-L-Apparatus (or 20 Liter Spherical Explosion Vessel) was obtained from Adolf Kühner AG. The test chamber is a stainless steel hollow sphere with a personal computer interface. The top of the cover contains holes for the lead wires to the ignition system. The opening provides for ignition by a condenser discharging with an auxiliary spark gap (Siwek, 1996; Operating Instructions for the 20-L-Apparatus, 1996), which is controlled by the KSEP 310 unit of the 20-L-Apparatus. The KSEP 332 unit uses piezoelectric pressure sensors to measure the pressure as function of time (ASTM, 1991; Operating Instructions for the 20-L-Apparatus, 1996). A comprehensive software package KSEP 5.0 is available, which allows safe operation of the test equipment and an optimum evaluation of the explosion test results.

In recent years, international standard use has been made of the more convenient and less expensive 20-L-Apparatus as the standard equipment. The explosion behavior of combustible materials (combustible dusts, flammable gases, or solvent vapors) must be investigated in accordance with internationally recognized test procedures. For the determination of combustible gases or vapors, the test is generally accomplished in a quiescent state (ignition delay time, $t_v = 0$ sec) (Operating Instructions for the 20-L-Apparatus, 1996).

In the 20-L-Apparatus, important explosion characteristics of gases and vapors, such as flammability limits, maximum explosion overpressure (P_{max}), gas or vapor deflagration index (K_g), and minimum oxygen concentration (MOC), can be determined with adherence to standardized test procedures.

2.4 Maximum Explosion Overpressure (Pmax), Maximum Rate of Explosion Pressure Rise (dP/dt)max, and Gas or Vapor Deflagration Index (Kg)

The peak values that accompany the explosion of a combustible vapor are the maximum pressure (Pmax) and the maximum rate of explosion pressure rise (dP/dt)max. Experimentally, the peak values can be obtained from tests over a wide range of concentrations using one chemical igniters (Crowl & Louvar, 1990). The explosion indices, Pmax and (dP/dt) max, are defined as the mean values of the maximum values of all three series. Subsequently, the gas or vapor deflagration index (Kg) is calculated from (dP/dt) max by means of the Cubic Law (NFPA, 1992)

$$V^{1/3} (dP/dt)_{max} = K_g$$

Where Kg and V are the maximum gas explosion constant specific to the gas and the volume of test apparatus. As there are many gas products and industrial practices, it is appropriate to assign this maximum constant to one of several explosion classes, and to use these as a basis for sizing explosive relief.

2.4.1 Minimum Oxygen Concentration (MOC)

The LEL is based on fuel in air. However, oxygen is the key ingredient, and MOC is required to propagate a flame. When oxygen concentration is less than the MOC, the reaction cannot generate enough energy to heat the entire gas mixtures (including the inerts) to the extent required for the self-propagation of the flame. MOC is an especially useful parameter, because explosions and fires are preventable by reducing the oxygen concentration regardless of the concentration of the fuel.

This concept is the basis for the common procedure called inerting. The MOC has units of percent oxygen in air plus fuel. If experimental data are not available, the MOC is estimated by using the stoichiometry from the combustion reaction and the LEL. This procedure works quite well for many hydrocarbons (Siwek, 1996; Crowl & Louvar, 1990).

2.5 Explosion Limits

The explosive limits of a gas or a vapour, is the limiting concentration (in air) that is needed for the gas to ignite and explode. There are two explosive limits for any gas or vapor, the lower explosive limit (LEL) and the upper explosive limit (UEL). At concentrations in air below the LEL there is not enough fuel to continue an explosion; at concentrations above the UEL the fuel has displaced so much air that there is not enough oxygen to begin a reaction.

Controlling gas and vapor concentrations outside the explosive limits is a major consideration in occupational safety. Methods used to control the concentration of a potentially explosive gas or vapor include use of sweep gas, an inert gas such as nitrogen or argon to dilute the explosive gas before coming in contact with air. Uses of scrubbers or adsorption resins to remove explosive gases before release are also common. Gases can also be maintained safely at concentrations above the UEL, although a breach in the storage container can lead to explosive conditions.

2.5.1 Lower Explosive Limit

Lower Explosive Limit (LEL): The explosive limits of a gas or a vapor are the limiting concentration (in air) that is needed for the gas to ignite and explode. The lowest concentration of a gas or a vapor in air capable of producing a flash of fire in presence of an ignition source (arch, flame, heat). At concentration in air below the LEL there is not fuel to continue an explosion.

Concentrations lower than LEL are "too lean" to burn. For example methane gas has a LEL of 4.4 % (at 138 °C) by volume, meaning 4.4 % of the total volume of the air consists of methane. At 20 °C the LEL is 5.1 %.

If the atmosphere has less than 5.1 % methane, an explosion cannot occur even if a source of ignition is present. When methane (CH₄) concentration reaches 5 % an explosion can occur if there is an ignition source. Each combustible gas has its own LEL concentration. These percentages should not be confused with LEL instrumentation readings. Instruments designed and calibrated to read LEL also read as percent values. (Jones, 1938; Crowl & Louvar, 1990).

2.5.2 Upper Explosive Limit

Upper Explosive Limit (UEL): Highest concentration (percentage) of a gas or a vapor in air capable of producing a flash of fire in presence of an ignition source (arch, flame, heat). Concentration higher than UEL are "too rich" to burn. Also called upper flammability limit (UFL).

2.5.3 Detonation

Detonation involves an exothermic front accelerating through a medium that eventually drives a shock front propagating directly in front of it. They are observed in both conventional solid and liquid explosives as well as in reactive gases. The velocity of detonations in solid and liquid explosives is much higher than that in gaseous ones, which allows far clearer resolution of the wave system in the latter.

Gaseous detonations normally occur in confined systems but are occasionally observed in large vapor clouds. Again, they are often associated with a gaseous mixture of fuel and oxidant of a composition, somewhat below conventional flammability limits.

There is an extraordinary variety of fuels that may be present as gases, as droplet fogs and as dust suspensions. Other materials, such as acetylene, ozone and hydrogen peroxide are detonable in the absence of oxygen; fuller lists are given by both. Oxidants include halogens, ozone, hydrogen peroxide and oxides of nitrogen and chlorine.

In terms of external damage, it is important to distinguish between detonations and deflagrations where the exothermic wave is subsonic and maximum pressures are at most a quarter [citation needed] of those generated by the former. Processes involved in the transition between deflagration and detonation are covered thoroughly by Nettleton.

2.6 Gas and Vapor Cloud Explosion

Vapor cloud explosion is the term used to describe the explosive combustion of the gas or vapor cloud formed by the release of flammable material such as fuel, into the atmosphere. The vapor cloud can result from both gas phase releases and the evaporation of liquid fuels.

Because liquefied vapors have a much higher density than compressed gases, the formation of clouds containing tones of vapor is much likely than the formation of clouds containing tones of gases. In the following sections those factors which effect explosion severity are described.

However, it is important to know that blast damage will not always result from a release of flammable material. For an example, a vapor cloud open, unobstructed terrain is likely to produce damaging overpressure in any but most exceptional circumstance (a gas which substantially more reactive than typical hydrocarbon fuels or there is a very high energy ignition). The following sections give methods for estimating the worst case overpressure should an explosion occur.

2.6.1 Gas and Vapor Cloud Formation

The rate of energy release which is proportional to the rate of combustion depends upon the mixture composition and is a maximum at a point close to the air fuel ratio. The explosion overpressure will therefore also be highest under these conditions.

There are lower limits to the concentration of fuel and air in the vapor below which the vapor will not burn. In some instances, a detail consideration of how the

fuel dispersed may discount the possibility of a vapor cloud explosion by showing that a flammable mixture cannot form. Also, at mixture concentrations above the upper limits of flammability the rate of burning is controlled by the rate of mixing with air and therefore it is not likely that destructive blast will be generated by rich mixtures.

Factors that must be considered to predict the dispersion of gas are the release rate and direction, atmospheric conditions, the presence of obstruction and also whether the gas is lighter or heavier than air. When there is any doubt about the fuel air concentration in a vapor cloud, conservative prediction of overpressure can be obtained by assuming that the fuel and air have mixed in stoichiometric proportion (Abbort, 1990)

2.7 Ignition

Every effort should be made to eliminate potential sources. For an example by the use of explosion protected electrical equipment, area classification and permit to work procedure. However, even with such precautions it is not wise to assume that a possible ignition sources does not exist.

At certain point, the ignition sources can be automatically generated because of the rise in temperature and hence causes explosion to occurring.

The process of slow oxidation with accompanying evolution of heat, sometimes lead auto-ignition if the energy is not removed from the system. Liquids with relatively low volatility are particularly susceptible to this problem.

2.8 Explosion Efficiency

The magnitude of the blast wave is determined principally by the rate of combustion and as the density of the unburnt mixture decreases by a factor of its burns (at constant pressure), this can be related to the rate at which the products expand, or equivalently the rate at which an increasing volume of products gas is generated.

As the product expand and because of the inertia of the surrounding atmosphere, higher blast pressures are generated for higher combustion rates. This in turn increases the efficiency with which the chemical energy of the vapor is converted to the mechanical energy of the blast wave, thus one of the parameter to determine the efficiency of the explosion is the maximum flame speed.

2.9 Explosion Suppression

To mitigate against the threat of industrial explosions, explosion protection products and services to must be provides, include the explosion testing, explosion venting systems, explosion suppression systems, and explosion isolation systems.

Explosion suppression systems use pressure sensor detectors to detect and act upon the developing explosion. The control panel receives the detection signal from the pressure sensors and then sends a release signal to the container containing fire suppressant. Fire suppressant containers hold fire suppressing agents, and release the agent at the command of the control panel. Suppressant agents typically include CO₂ and water mist.

Optionally, an explosion suppression system may contain either a mechanical or chemical isolation system to prevent flame or pressure from traveling through connected ducts or piping into other process equipment.

2.9.1 Carbon Dioxide as Suppression Agent

Carbon dioxide (CO₂) gas has a high ratio of expansion which facilitates rapid discharge and allows for three dimensional penetration of the entire hazard area quickly. CO₂ extinguishes a fire by reducing the oxygen content of the protected area below the point where it can support combustion.

Due to the extreme density of the carbon dioxide, it quickly and effectively permeates the protected hazard area and suppresses the fire. Rapid expansion of the 4-6 inches of carbon dioxide snow to gas reduces the ambient temperature in the protected hazard area which aids in the extinguishing process and retards re-ignition.

CO₂ Is Non-Damaging

When installed properly, carbon dioxide fire suppression systems will not normally damage sensitive electronic equipment. Carbon dioxide has no residual clean-up associated with its use as a fire suppressing agent. When it is properly ventilated, the gas escapes to atmosphere after the fire has been extinguished.

CHAPTER 3

METHODOLOGY

3.1 Experimental Apparatus

The experimental 20-L-Apparatus (or 20 litre Spherical Explosion Vessel) was obtained from Adolf Kühner AG and is shown in Fig. 3.1. The test chamber is a stainless steel hollow sphere with a personal computer (PC) interface.

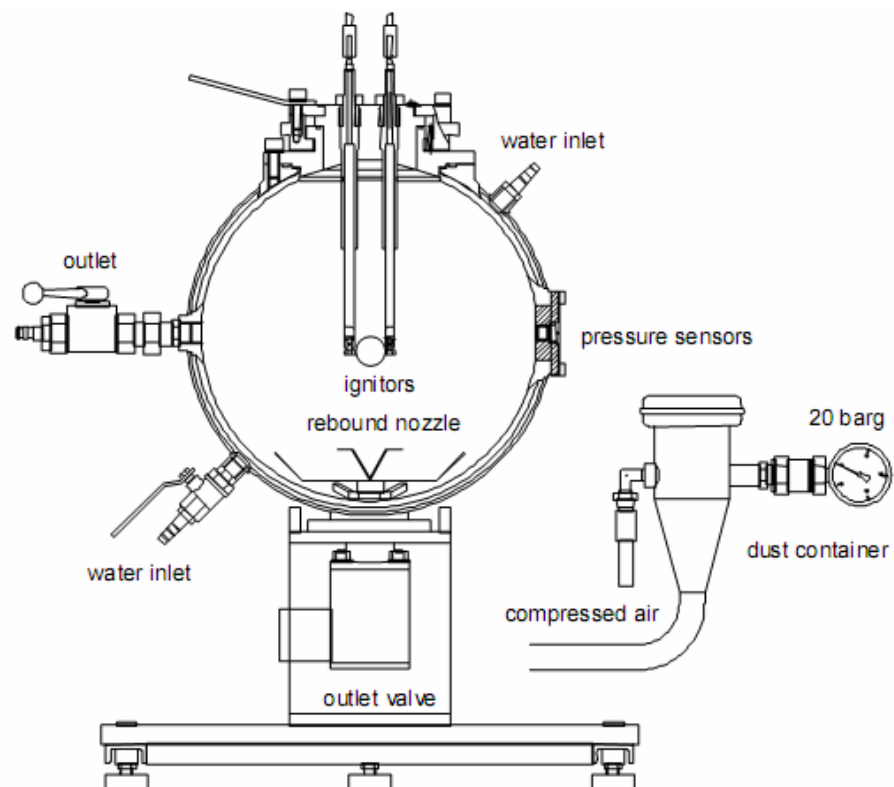


Figure 3.1: Schematic diagram of 20-L-Apparatus

3.1.1 20-L-Apparatus

The top of the cover contains holes for the lead wires to the ignition system. The opening provides for ignition which controlled by the KSEP 320 unit of 20-L-Apparatus. The KSEP 332 unit uses piezoelectric pressure sensor's to measure the pressure as a function of time. A comprehensive software package KSEP 6.0 is used to allow safe operation of the test equipment and an optimum evaluation of the explosion test results.

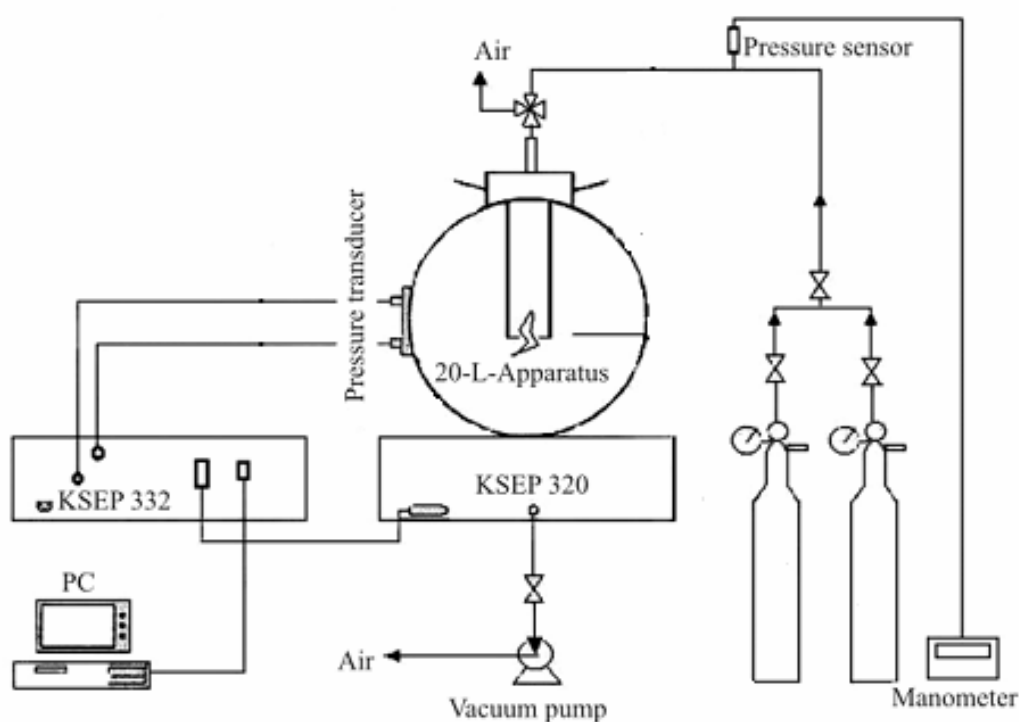


Figure 3.2 Schematic diagram of experimental set up

3.1.2 Measurement and Control System KSEP 332

The KSEP 332 unit uses piezoelectric pressure sensor's to measure the pressure as a function of time and controls the valves as well as the ignition system of the 20-L-apparatus. The measured values to be processed by a personal computer are digitized at high resolution. The use of two completely independent measuring channels gives good security against erroneous measurements and allows for self checking.

For the determination of combustible gases or vapors, the test accomplished in a quiescent state which the ignition delay time, $t_v = 0$ s. (Operating Instructions for the 20-L-Apparatus, 1996)

3.2 Experimental Conditions

The explosive limits of CH₄/air mixtures were investigated at CH₄ concentration from 3 vol% to 19 vol% and also used suppression agent (CO₂) at concentration 5 vol% and 10 vol%. In this study, CH₄ with 99.5 % purity which indicates the natural gas and carbon dioxide with 99.9 % purity are used.

3.2.1 Pressure and Temperature

The initial pressure in the 20-L-sphere is regulated to 1 bar absolute. A water jacket to dissipate the heat of explosions or to maintain thermostatically controlled test temperatures. It is necessary to keep the operating temperature at approximately 20 °C by means of water cooling, whereby the operating temperature would correspond to room temperature. Therefore, there is always some flow of water and so that the outlet temperature of the cooling medium never exceeds 25 °C.

3.2.2 Ignition

Ignition is achieved by permanent spark which placed in the centre of the vessel. Since experiment was conducted under quiescent conditions, ignition energy (IE) is 10 J and the ignition delay is 0 s. The igniter releases 10 J independently of pressure or temperature. The pressure evolution after ignition is measured with a Kistler 701A piezoelectric pressure transducer connected to a charge amplifier (Kistler 5041B).

Table 3.1 Test condition of experiment

Pressure	1 bar absolute
Temperature	Ambient temperature
Mixture state	Quiescent state
Ignition energy	10J
Ignition delay time	0s

3.3 Experimental Procedure

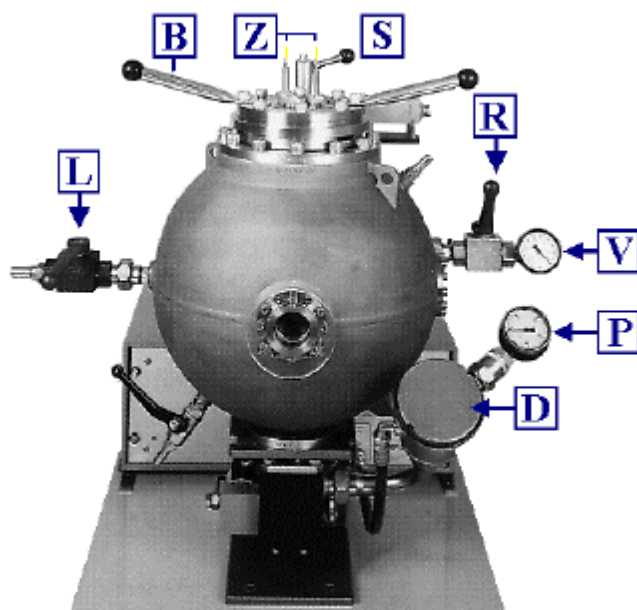


Figure 3.3: 20-L Apparatus

- 1) The apparatus and computer were switched on.
- 2) The safety switch (S) was opened and the bayonet-ring (B) was turned over anti-clock wise to open the cover of the explosion vessel.
- 3) The cover vessel was lifted off in order to place the igniter at the centre of the vessel by fitting the igniter in parallel to the electrode rods that connected to the cover of the vessel as shown in figure 3.1.

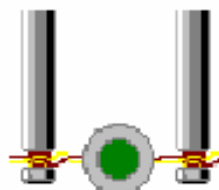


Figure 3.4: Diagram of igniter between the electrode rods

- 4) Then the cover is placed back to vessel and the ignition lines (Z) is connected.
- 5) The ball valve (L) and ball valve (R) are opened. The vacuum pump is started. The vessel was evacuated to 0.1 bar absolute. Then the ball valve (R) is closed.
- 6) Software KSEPT 6.0 is opened in the computer. The test condition is entered into the computer as shown in figure 3.5.



Figure 3.5: KSEPT 6.0

- 7) The CH_4 and CO_2 fuel from cylinder storage and air from surrounding are fed in to the vessel via the outlet valve and nozzle. The required composition $\text{CH}_4/\text{CO}_2/\text{air}$ mixture is produced readily with the partial pressure procedure. Then the ball valve (L) valves at pressure gauge are closed.
- 8) Then, the gas mixture is ignited. After ignition the pressure evolution is measured by pressure transducer as pressure versus time by the software.
- 9) After each test, the ball valve (L) is opened to release exhaust gas and pressure in the vessel.
- 10) The ignition line (Z) is disconnected and the safety switch (S) is opened. The bayonet-ring (B) is turned over anti-clock wise to open the cover sphere. The cover vessel is lifted off in order to clean up inside the explosion. The vessel is cleaned up using gas vacuum cleaner.

- 11) Then, the burn-out igniter is removed and the electrode rods were cleaned. The burn-out igniter was placed with a new igniter. The cover vessel was closed and the bayonet-ring (B) was turned in the final position.
- 12) The steps (4) to (11) was repeated for other fuel gas composition.

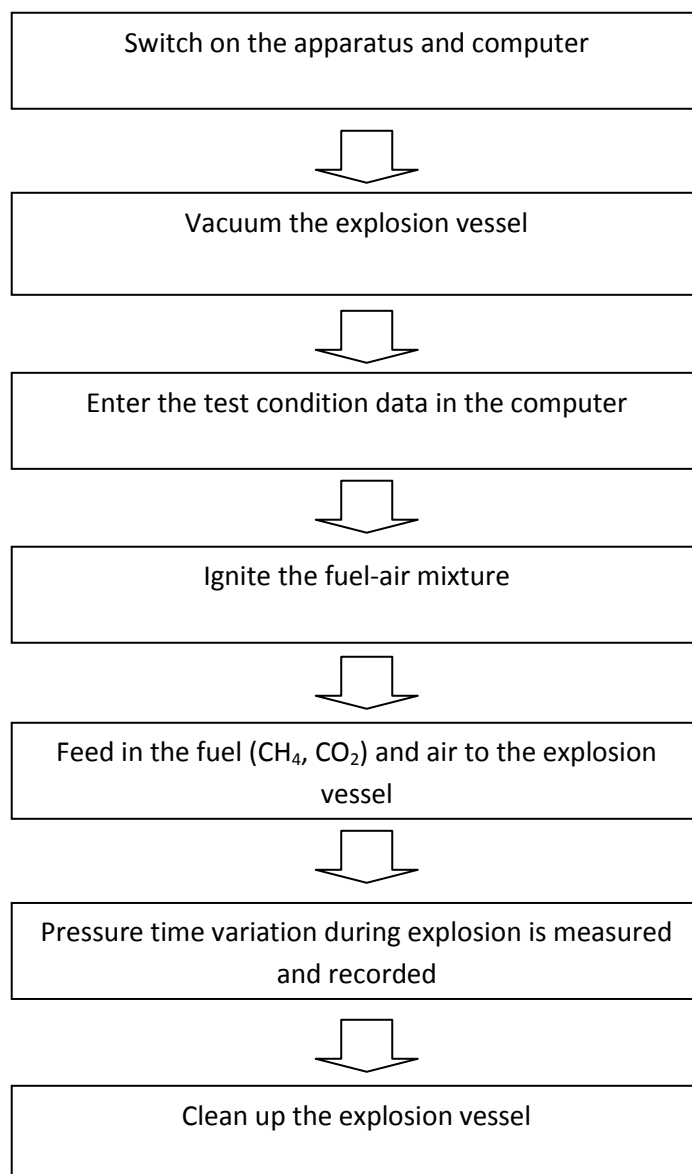


Figure 3.5: Experimental work flows

CHAPTER 4

RESULT AND DISCUSSION

4.1 Methane (CH₄) Explosion without the Presence of Suppressants

The experiment carried out to determine the air/fuel ratio in order for the explosion occurs. Through this experiment, the maximum pressure (P max) can be obtained. Each maximum pressure is different based on their characteristics of either lower explosive limit (LEL) or upper explosive limit (UEL).

In order to stop and mitigate the explosion activity, it is very important to determine the characteristics of the gas explosion at the initial stage to the final stage. One of the methods to determine the characteristics of the explosion is through determining its maximum pressure.

The maximum pressure can be obtained based on either through lower explosive limit (LEL) or upper explosive limit (UEL).

Figure 4.1 (a) and table 4.1 (b) shows the result obtained from CH₄ explosion. From the experiments, the (LEL) start from 5 vol % to 19 vol % of (UEL) and maximum pressure is 5.5 bar at 9 vol % of CH₄ were analyzed. From the data obtained, it shows that the P_{max}, LEL and UEL that can be achieved by CH₄ explosion phenomenon by the usage of fire explosion unit.

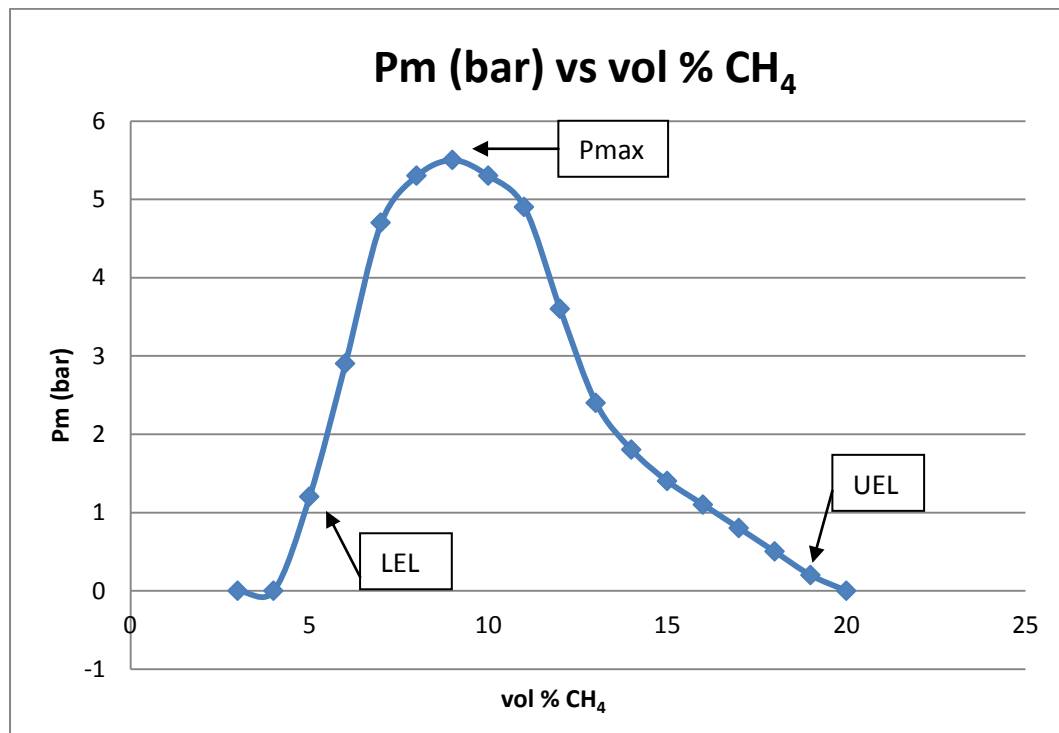


Figure 4.1 (a): Graph Pressure Maximum (Pmax) vs vol % CH₄

Table 4.1 (b): Summary Pmax of CH₄ Explosion

No	CH ₄ composition (air / fuel ratio %)	O ₂ (air) composition (air/fuel ratio %)	Maximum Pressure (Pmax)(bar)	Maximum rate of Pressure Rise (dp/dt)(bar/s)
1	3	97	0.0	0
2	4	96	0.0	0
3	5	95	1.2	0
4	6	94	2.9	16
5	7	93	4.7	88
6	8	92	5.3	118
7	9	91	5.5	154
8	10	90	5.3	114
9	11	89	4.9	62
10	12	88	3.6	37
11	13	87	2.4	5
12	14	86	1.8	5
13	15	85	1.4	5
14	16	84	1.1	5
15	17	83	0.8	5
16	18	82	0.5	0
17	19	81	0.2	0
18	20	80	0.0	0

4.2 Methane (CH₄) with the Presence of 5 vol % Carbon Dioxide (CO₂)

The purpose of the experiment is to determine the effect of 5 vol % CO₂ to the CH₄ explosion based on the maximum pressure (Pmax)

Based on the result obtained, it shows that CO_2 does effect the CH_4 explosion. In this phenomena, CO_2 act as a suppression agent where it reduces the amount of oxygen present in the air thus mitigating the explosion to the minimum pressure.

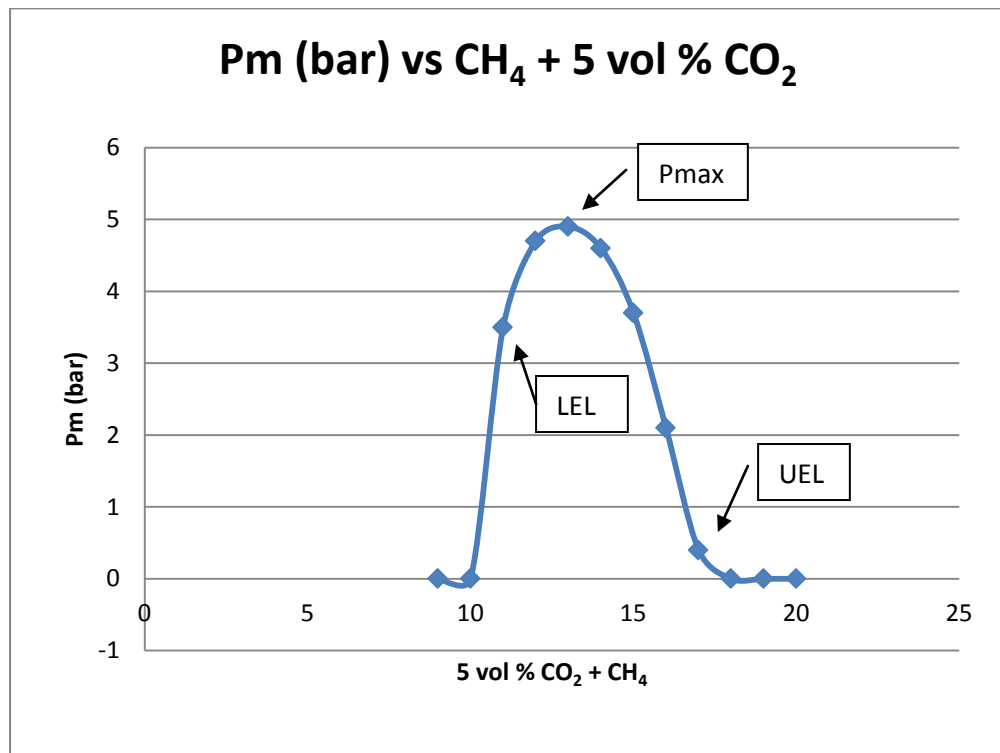


Figure 4.2 (a): Graph Maximum Pressure (Pmax) vs 5 vol % $\text{CO}_2 + \text{CH}_4$

Table 4.2 (b): Summary Pmax of CH₄ Explosion with Presence of 5 vol % CO₂

No	CH ₄ composition (air / fuel ratio %)	Suppression ratio, CO ₂ (5 vol %)	O ₂ (air) composition (air/fuel ratio %)	Maximum Pressure (Pmax)(bar)	Maximum rate of Pressure Rise (dp/dt)(bar/s)
1	4	5	91	0.0	0
2	5	5	90	3.2	10
3	6	5	89	3.5	15
4	7	5	88	4.7	60
5	8	5	87	4.9	112
6	9	5	86	4.6	78
7	10	5	85	3.7	27
8	11	5	84	2.1	6
9	12	5	83	0.4	5
10	13	5	82	0.0	4
11	14	5	81	0.0	2
12	15	5	80	0.0	0

From figured 4.2 (a) and table 4.2 (b) shows the summary of the result for CH₄ explosion with the presence of CO₂ which is commonly used in the suppression system. From these results the Pmax of CH₄ explosion with the presence of CO₂ can be determine.

From the data obtained, it shows that, CO₂ does effect the CH₄ explosion pressure curve and from the table 4.2 (b) the LEL and UEL can be determine which start from 5 vol % (LEL) to 12 vol % (UEL) CH₄. It reduces the amount of oxygen present in the surrounding thus reduces the pressure blast cause by the CH₄ explosion.

4.3 Methane (CH₄) with the Presence of 10 vol % Carbon Dioxide (CO₂)

The purpose of the experiment is to determine the effect of 10 vol % CO₂ to the CH₄ explosion based on the maximum pressure (P_{max})

Based on the result obtained, it shows that CO₂ does effect the CH₄ explosion. In this experiment, we are used 10 vol % of CO₂ act as a suppression agent where it can be more reduces the amount of oxygen compare with 5 vol % of CO₂ present in the air thus mitigating the explosion to the minimum pressure.

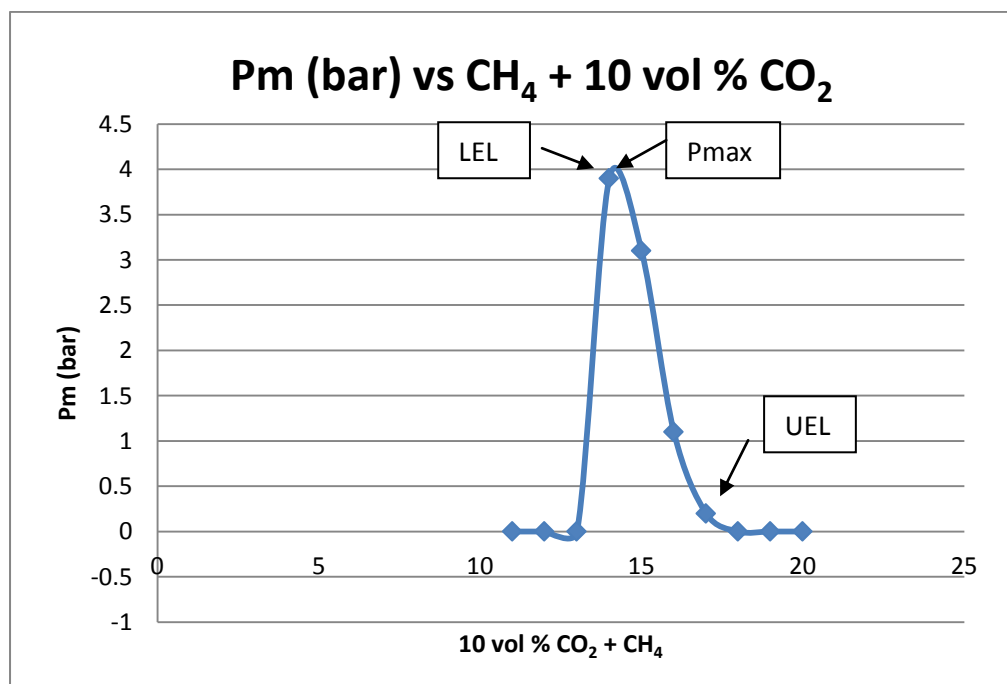


Figure 4.3 (a): Graph Maximum Pressure (P_{max}) vs 10 vol % CO₂ + CH₄

Table 4.3 (b): Summary Pmax of CH₄ Explosion with Presence of 10 vol % CO₂

No	CH ₄ composition (air / fuel ratio %)	Suppression ratio, CO ₂ (10 vol %)	O ₂ (air) composition (air/fuel ratio %)	Maximum Pressure (Pmax)(bar)	Maximum rate of Pressure Rise (dp/dt)(bar/s)
1	1	10	89	0.0	0
2	2	10	88	0.0	0
3	3	10	87	0.0	0
4	4	10	86	3.9	17
5	5	10	85	3.1	7
6	6	10	84	2.5	7
7	7	10	83	0.2	7
8	8	10	82	0.0	0
9	9	10	81	0.0	0

From the data obtained, it shows that, CO₂ does effect the CH₄ explosion pressure curve and from the table 4.3 (b) the LEL and UEL can be determine which start from 4 vol % (LEL) to 7 vol % (UEL) of CH₄. It extinguishes a fire by reducing the oxygen content of the surrounding below the point where it can support combustion. Besides CO₂ also reduces the ambient temperature and retards re-ignition.

4.4 Comparison of the Data

Based on the results obtained, the comparison between the effects of vol % CO₂ to the CH₄ explosion can be done. The summary shown in table 4.4.

From the data obtained, it shows that the two vol % of suppression agents used which is 5 vol % and 10 vol % of CO₂ does affect the explosion pressure curve for CH₄. Table 4.7 shows the comparison of the overall data.

From the comparison of the data, it was proven that 10 vol % of CO₂ suppresses the gas explosion more effectively compared to the 5 vol % of CO₂ which the UEL reduces from 12 vol % to 7 vol %, LEL from 5 vol % to 4 vol % and Pmax from 4.9 bar to 3.9 bar.

CO₂ is effective in the suppression system due to its properties that can suppress the explosion development chemically and physically. Through chemical and physical reaction, CO₂ is heavier than oxygen so these suppress work by displacing or taking away oxygen from the surrounding area.

CO₂ is also very cold so it also works by cooling the fuel. While they are environmentally safe and leave no residue they are not recommended for normally occupied areas because of the asphyxiating effect on people.

Table 4.4: Comparison data of LEL, UEL, Pmax and dp/dt between different vol % of suppression agents

No	Fuel Type	Suppression Agent	LEL (vol%)	UEL (vol%)	Pmax (bar)	dp/dt (bar/s)
1	(CH ₄)	Without (CO ₂)	5	19	5.5	154
2		5 vol % (CO ₂)	5	12	4.9	112
3		10 vol % (CO ₂)	4	7	3.9	17

From table 4.4 shows the summary of the result for CH₄ explosion with the presence of CO₂ which is commonly used in the suppression system. From these results the LEL, UEL, Pmax and dp/dt of CH₄ explosion with the presence of CO₂ can be determine.

From the data obtained, it shows that, vol % CO₂ does effect the CH₄ explosion pressure curve and from the table 4.4 the LEL and UEL for without CO₂ can be determine which start from 5 vol % (LEL) to 19 vol % (UEL) CH₄ whether for 5 vol % and 10 vol % of CO₂ its start from 5 vol % (LEL) to 12 vol % (UEL) and 4 vol % (LEL) to 7 vol %. (UEL). Pmax in 10 vol % CO₂ is 3.9 bar which it's lower than 4.9 bar (5 vol % CO₂) and 5.5 bar (without CO₂).

Due to the extreme density of the carbon dioxide, it quickly and effectively permeates the protected hazard area and suppresses the fire by reducing the oxygen content of the protected area below the point where it can support combustion.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Based on the results of this study, it can be concluded that, knowledge of physical and chemistry of fire essential to determine the behavior of fire. By understanding limits of flammability and fire triangle, any explosion or fire can be avoided. Suppression system is one of the method of preventing explosion from occurring in its early stage. However, sometimes the suppression agents used failed and have a drawback in causing the explosion to be worsen.

That is why in this research, the volume percent, vol% of suppression agent is to be determined so that the suppression system used can be much more effective in preventing explosion from occurring. The suppression agent used in the experiment to determine which one is the best and much more effective in preventing explosion when dealing with CH₄ which is easily cause explosion.

From the experiment and data collected, it is proven that 10 vol % of CO₂ has high potential to mitigate and prevent explosion from occurring. It is shown that the maximum pressure (P_{max}) generated by the methane (CH₄) explosion is lower compare to the 5 vol % of CO₂.

It is proven that CO₂ does mitigate the explosion by chemical and physical reaction. Through chemical reaction, CO₂ pushing the oxygen required for the chemical reaction out of the way, surrounding it with CO₂ and quickly smothering the fire.

5.2 Recommendation

Fire and explosion should be prevented and mitigated from its early stage in order to prevent catastrophe. It is important however to use the best suppression agents that can suppress the gas explosion so that this system could be reliable to be apply in the industries.

Therefore, this research done to prove that while CO₂ is commonly used in the prevention of fire and explosion. It is shows the best potential to mitigate explosion more effectively and also environmental friendly.

As in the future plan, it is hope that this research can be continued in order to overcome the problems cause by the suppression agents so that the improvement can be done. Research to identify other powder form of suppression agents besides CO₂ such as sodium bicarbonate and ammonium phosphate and many more must also be carried out so that the ability of other chemical suppression agents can also be identified.

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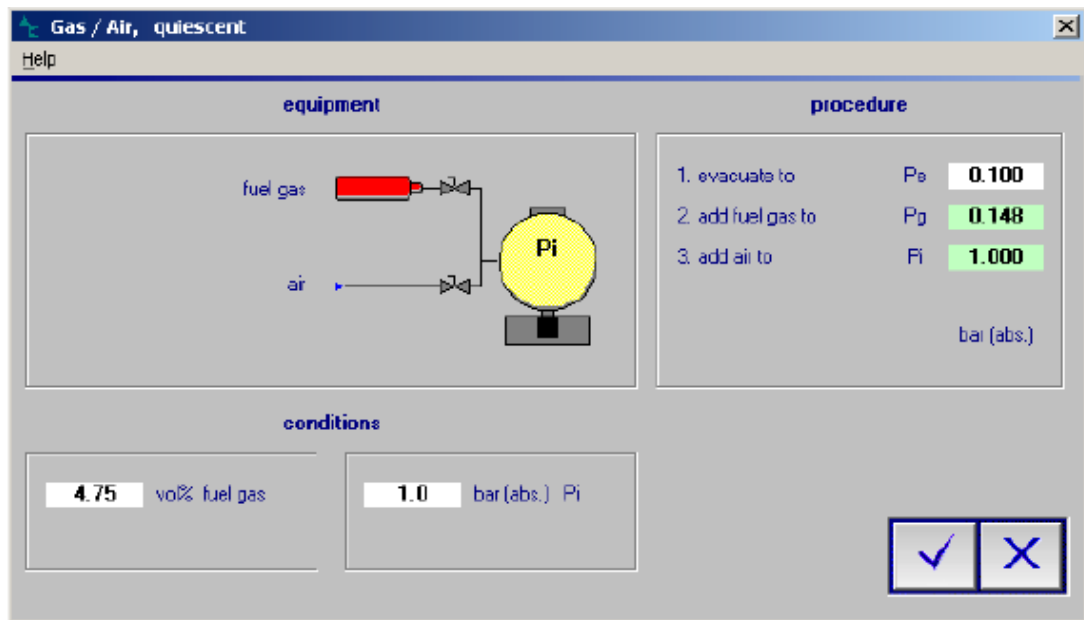
APPENDICES

APPENDIX A

MANUAL OF 20-L APPARATUS

1.1 General Rules for Gas and Solvent Vapors (Quiescent)

1. It is customary to determine the explosion indices for gas and solvent vapors, if possible, at room temperature and normal pressure, using as an ignition source a permanent spark with an ignition energy of approximation $E = 10 \text{ J}$.
2. The explosion characteristics can be determined either in a quiescent state or under turbulent condition for the gas (vapor)/air mixtures. In the following the condition for the gas/air mixtures will be quiescent and therefore the dust dispersion device has to be removed and the connection sealed.
3. The required gas-air mixtures can be produced easily with the partial - pressure - procedure. Prior to the tests it is recommended to check the composition of these gas/air mixtures using suitable instruments.



4. For investigations under quiescent conditions input $t_v = 0$. The admixture of the fuels takes place directly in the 20-L-sphere and not through the storage container. Therefore no expansion pressure of the storage container can be measured ($P_d = 0$ bar). The ignition signal occurs immediately after starting the test. The evaluation of P_d and t_d will be suppressed.

1.2 Gas Lower Explosion Limit (LEL) and Gas Upper Explosion Limit (UEL)

1.2.1 Test Conditions

Function = Gas, Solvent

Ignition source = Permanent Spark

Ignition energy $IE = 10$ J

Ignition delay time $t_v = 0$ ms

Dispersion pressure $P_z = 0$ bar (1 bar absolute, no pre-evacuation)

1.2.2 Test Method

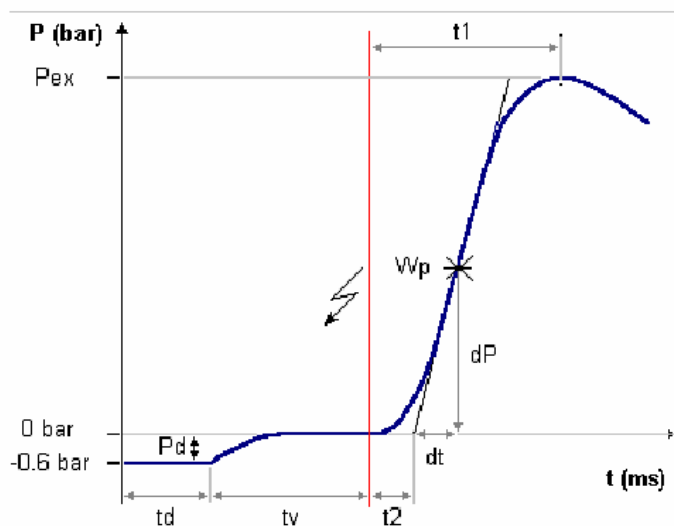
The 20-L-sphere has to be cleaned thoroughly before each test. A test series is initiated, starting with gas concentrations of an integral multiple of 0.25 vol % for example 2 or 3 vol %. The series is continued with a systematic increase of the gas concentration until ignition of the gas/air mixture is observed. Repeat the test with a gas concentration 0.25 vol % lower, and continue to reduce the concentration in further tests until a concentration is reached at which no ignition of the gas/air mixture is observed in three successive tests. For the determination of the UEL the LEL-procedure can be used accordingly. To obtain accurate values for the LEL and UEL, 3 negative tests for each concentration must be observed.

1.2.3 Test Evaluation (IE = 10 J)

P _{ex} [bar]	P _m [bar]	Decision:
< 0.1	< 0.1	no ignition
≥ 0.1	≥ 0.1	ignition

The lower explosive limit LEL as well as the upper explosive limit UEL are reported as those concentrations at which a gas explosion is just not possible in 3 successive tests. If only one test series is carried out (1 negative test), the value for LEL or UEL has to be reported as approximately vol %.

1.3 Pressure Course: Definitions



Pressure/time-diagram of a fuel explosion

P_{ex} - Explosion overpressure: the difference between the pressure at ignition time (normal pressure) and the pressure at the culmination point is the maximum explosion overpressure P_{ex} measured in the 20-L-apparatus at nominal fuel concentration.

P_m - Corrected explosion overpressure: Due to cooling and pressure effects caused by the chemical igniters in the 20-L-apparatus, the measured explosion overpressure P_{ex} has to be corrected.

P_{max} - Maximum explosion overpressure: Maximum value of P_m determined by tests over a wide range of fuel concentrations. $(dP/dt)_m$ Rate of pressure rise with time at nominal fuel concentration. It is defined as the maximum slope of a tangent through the point of inflexion (W_p) in the rising portion of the pressure vs. time curve.

$(dP/dt)_{max}$ - Maximum rate of pressure with time: Maximum value of (dP/dt) m determined by tests over a wide range of fuel concentrations.

K_{max} - Product specific constant = $0.27144 \times (dP/dt)_{max}$.

t_1 - Duration of combustion: time difference between the activation of the ignition and the culmination point.

t_2 - Induction time: time difference between the activation of the ignition

APPENDIX B**TECHNICAL DATA OF 20-L-SPHERE**

Material of construction No.:	1.4435
Wall thickness of inner jacket:	min. 4 mm
Wall thickness of outer jacket:	min. 2 mm
Volume of sphere:	20 l
Volume of water jacket:	1.5 l
Design pressure of sphere:	30 bar
Design pressure of jacket:	10 bar
Test pressure of sphere:	39 bar
Test pressure of jacket:	14.3 bar
Design temperature:	60 °C
Bayonet ring aperture:	96 mm diameter
Cleaning aperture:	140 mm diameter
Sight glass:	30 mm diameter
Measuring flange:	3 tapped bores M14 x 1.25
Venting connection:	hose ID. 12 mm
Vacuum connection:	Serto, 1/4" G
Water connections:	hose ID. 10 mm
Overall dimensions	(w,h,d): 650 x 875 x 820 mm
Weight:	5 kg

APPENDIX C 1

DATA FROM COMPUTER

Fkksa Ump

Sample: **CH₄ + Air 5/3/2010**

Customer: Mohd Hafizul
Reason: PSM 2 Final Project.
Data to sample origin: BKG KC07069
Preparation of sample: Hafiz
Median value: CH₄ + Air

Apparatus: 20-L-sphere
Ignition source: chemical igniters
Dust-dispersion system: rebound nozzle

Explosion Indices

Max. explosion pressure: Pmax = 5.5 bar
Max. rate of pressure rise: (dP/dt)max = 154 bar / s
Product specific constant: Kmax = 25 m·bar / s

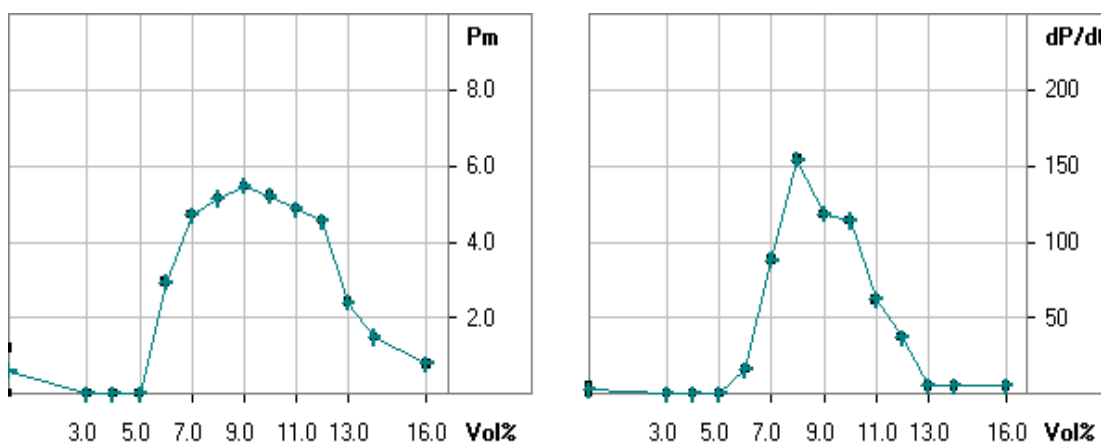


Figure 5: Graph pm vs vol % and dP/dt vs vol % of CH₄ + air

CH₄ + Air

(series) Test	Conc. [vol%]	Pm [bar]	dP/dt [bar/s]	t1 [ms]	tv eff [ms]
(1) 1	3.0	.0	0	0	0
(2) 2	4.0	.0	0	0	0
(3) 3	5.0	1.2	0	0	0
(4) 4	6.0	2.9	16	352	0
(5) 5	7.0	4.7	88	129	0
(6) 7	8.0	5.3	118	114	0
(7) 8	9.0	5.5	154	101	0
(8) 9	10.0	5.3	114	116	0
(9) 10	11.0	4.9	62	180	0
(10) 11	12.0	3.6	37	244	0
(11) 12	13.0	2.4	5	935	0
(12) 13	14.0	1.8	5	954	0
(13) 14	15.0	1.4	5	908	0
(14) 15	16.0	1.1	5	816	0
(15) 16	17.0	.8	5	977	0
(16) 17	18.0	.5	0	0	0
(17) 18	19.0	.2	0	0	0
(18) 19	20.0	.0	0	0	0

APPENDIX C 2

Fkksa Ump

Sample: **5 vol % CO₂ + CH₄ 09/03/2010**

Customer: Mohd Hafizul Bin Md. Jais
 Reason: PSM 2
 Data to sample origin: Gas Eng. Lab
 Preparation of sample: Hafiz
 Median value: CO₂ & CH₄

Apparatus: 20-L-sphere
 Ignition source: chemical igniters
 Dust-dispersion system: rebound nozzle

Explosion Indices

Max. explosion pressure: P_{max} = 4.9 bar
 Max. rate of pressure rise: (dP/dt)_{max} = 112 bar / s
 Product specific constant: K_{max} = 30 m·bar / s

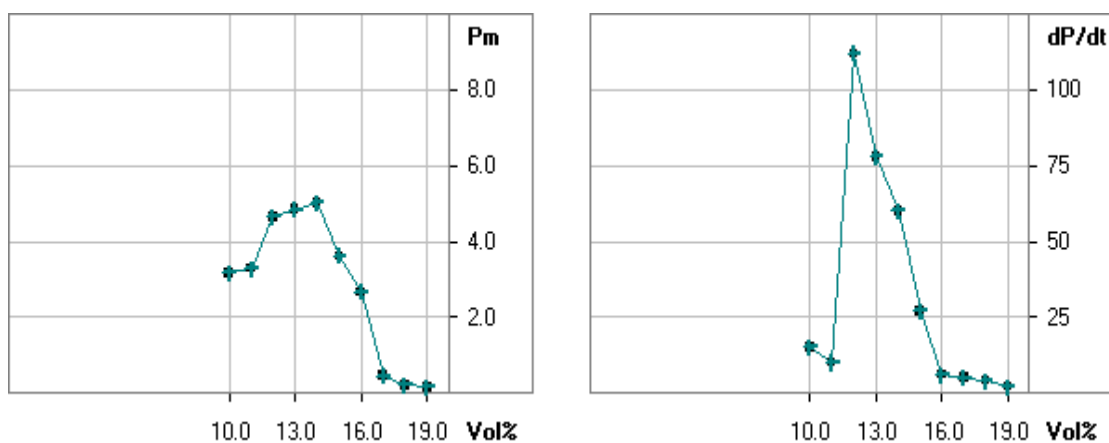


Figure 6: Graph pm vs vol % and dP/dt vs vol % of CH₄ + air + 5 vol % CO₂

5 vol % CO₂ + CH₄ + Air

(series) Test	Conc. [vol%]	Pm [bar]	dP/dt [bar/s]	t1 [ms]	tv eff [ms]
(1) 1	10.0	3.2	10	501	0
(1) 2	11.0	3.5	15	446	0
(1) 3	12.0	4.7	60	156	0
(1) 4	13.0	4.9	112	162	0
(1) 5	14.0	4.6	78	176	0
(1) 6	15.0	3.7	27	305	0
(1) 7	16.0	2.1	6	812	0
(1) 8	17.0	.0	5	1082	0
(1) 10	18.0	.0	4	493	0
(1) 11	19.0	.0	2	260	0

APPENDIX C 3

Fkksa Ump

Sample: **10 vol % CO₂ + CH₄ 13/04/2010**

Customer: Hafizul Bin Md Jais
 Reason: PSM 2 Final Project
 Data to sample origin: Gas Eng. Lab.
 Preparation of sample: Hafiz
 Median value: CO₂ & CH₄

Apparatus: 20-L-sphere
 Ignition source: chemical igniters
 Dust-dispersion system: rebound nozzle

Explosion Indices

Max. explosion pressure: P_{max} = 3.9 bar
 Max. rate of pressure rise: (dP/dt)_{max} = 17 bar / s
 Product specific constant: K_{max} = 5 m·bar / s

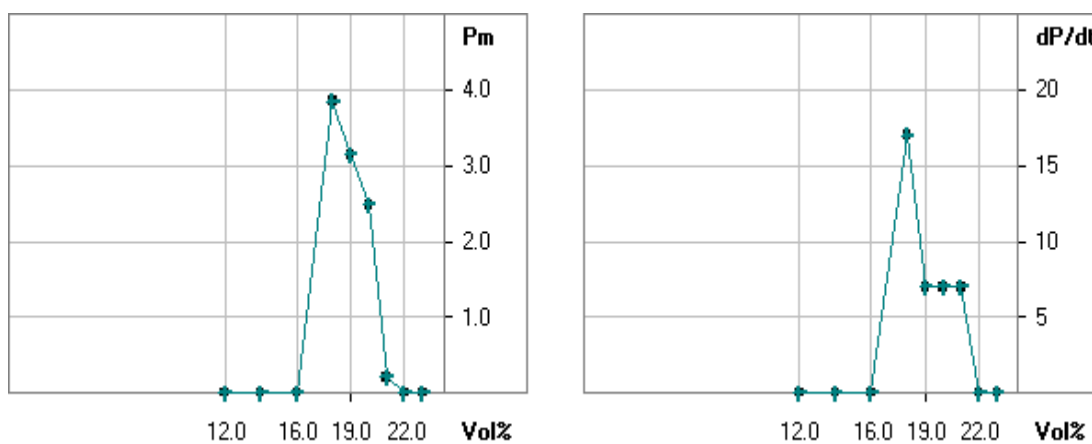


Figure 7: Graph pm vs vol % and dP/dt vs vol % of CH₄ + air + 10 vol % CO₂

10 vol % CO₂ + CH₄ + Air

(series) Test	Conc. [vol%]	Pm [bar]	dP/dt [bar/s]	t1 [ms]	tv eff [ms]
(1) 1	12.0	.0	0	0	0
(1) 2	14.0	.0	0	0	0
(1) 3	16.0	.0	0	0	0
(1) 4	18.0	3.9	17	356	0
(1) 6	19.0	3.1	7	685	0
(1) 7	20.0	2.5	7	704	0
(1) 8	21.0	.2	7	408	0
(1) 9	22.0	.0	0	0	0
(1) 10	23.0	.0	0	0	0
