A DEVISED DETECTOR TUBE FOR DETERMINATION OF XYLENE IN WORKING AREA

BUNCHA SRIVIWATTANAKUL

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Buncha Sriviwattanakul

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A DEVISED DETECTOR TUBE FOR DETERMINATION OF XYLENE IN WORKING AREA.

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ABSTRACT

The purpose of this study was to construct a detector tube for the determination of xylene in working areas. The indicating reagent was prepared by coating potassium iodate solution onto the silica gel. The coated silica gel was packed into the blank glass tube and hermetically sealed at both ends of the tube by the liquid petroleum gas burner. The equilibrium reaction between xylene and the indicating reagent produced the red-brown color of iodine. The stainlength on the tube was indicated of xylene concentration.

Experiment was conducted to test the xylene detector tube for overall accuracy, detection limit, specificity, storage stability and to compare it with commercial xylene detector tubes in the field.

Results showed that the xylene detector tube could be used for the analysis of xylene in the air at the concentrations of 10 to 100 ppm with the detection limit of 10 ppm. The overall system accuracy for analysis of xylene in the air at 0.5 and 1.0 TLV was 21.24 and 21.96%, respectively. Toluene was an interference of the experimental xylene detector tube because it masked the xylene when they were mixed together. Regarding storage stability, the tube stored in refrigerators could be stable for more than 2 weeks. The field concentrations of xylene analyzed by the experimental and commercial xylene detector tubes were not significantly different.

The results revealed that the studied xylene detector tube could be an effective and low cost detector tube for the analysis of xylene in working areas.

KEY WORDS : XYLENE / DETECTOR TUBE /
POTASSIUM IODATE SOLUTION / GAS CHROMATOGRAPH

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การสร้างหลอดตรวจวัดสารไซลีนในพื้นที่ปฏิบัติงาน (A DEVISED DETECTOR TUBE FOR DETERMINATION OF XYLENE IN WORKING AREA)

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บทคัดย่อ

การศึกษาครั้งนี้มีวัตถุประสงค์เพื่อสร้างหลอดตรวจวัดสำหรับตรวจวัดไอระเหยของสารไซลีนในพื้นที่ปฏิบัติงาน สารเคมีตัวระบุปฏิกิริยาถูกเตรียมจากเจลที่เคลือบด้วยสารละลายโปแตสเซียมไอโอเดตซึ่งจะบรรจุลงไปในหลอดแก้วเปล่าแล้วเชื่อมปิดหัวท้ายด้วยหัวเผาแก๊ส ปฏิกิริยาระหว่างสารไซลีนและสารเคมีตัวระบุปฏิกิริยาจะให้สีน้ำตาลแดงของไอโอดีน ความยาวของสีจะระบุถึงความเข้มข้นของสารไซลีน

การทดลองเพื่อทดสอบหลอดตรวจวัดสารไซลีน ได้แก่ ความถูกต้องรวม ความเข้มข้นต่ำสุดที่ตรวจวัดได้ ความจำเพาะ อายุการใช้งานของหลอดและทดสอบตรวจวัดไซลีนในสถานที่ปฏิบัติงานเปรียบเทียบกับหลอดตรวจวัดไซลีนที่มีขายทั่วไป

ผลการศึกษาพบว่าหลอดตรวจวัดไซลีนนี้สามารถใช้วิเคราะห์สารไซลีนที่ระดับความเข้มข้น 10 ถึง 100 ppm ซึ่งมีค่าความเข้มข้นต่ำสุดที่ตรวจวัดได้ 10 ppm ,ความถูกต้องรวมที่ 0.5 และ 1.0 ppm มีค่าเท่ากับ 21.24 % และ 21.96% ตามลำดับ,สารโทลูอีนถือเป็นสารตัวกวนเพราะไม่สามารถอ่านค่าไซลีนได้เมื่อบรรยากาศมีสารโทลูอีนผสมอยู่, หลอดตรวจวัดไซลีนสามารถให้ผลการตรวจวัดที่คงเดิมเมื่อเก็บที่อุณหภูมิในตู้เย็นมากกว่า 2 อาทิตย์ การตรวจวัดความเข้มข้นของไซลีนด้วยหลอดตรวจวัดไซลีนกับชนิดที่มีขายทั่วไปพบว่าไม่แตกต่างกันอย่างมีนัยสำคัญทางสถิติ

ผลการศึกษาครั้งนี้ชี้ให้เห็นว่า หลอดตรวจวัดไซลีนมีความเป็นไปได้ที่จะใช้เป็นหลอดตรวจวัดไซลีนที่มีประสิทธิภาพดีและราคาถูก สำหรับตรวจวัดสารไซลีนที่ปนเปื้อนในพื้นที่การปฏิบัติงาน

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CONTENTS

		Page
ACKNOWLEDGEMENTS		iii
ABSTRACT		iv
LIST OF TABLES		viii
LIST OF FIGURES		x
CHAPTER I	**INTRODUCTION**	
	Background and rationale	1
	Objectives	2
	Hypothesis	3
	Scope of study	3
	Variables	3
	Glossary of terms and definitions	4
CHAPTER II	**LITERATURE REVIEW**	
	The development of detector tube	6
	Recommendation for detector tube	8
	NIOSH quality standards for detector tubes	9
	Xylene	10
	Potassium iodate	13
	Sulfuric acid	14
	Silica gel	15
	Gas Chromatography	16
	Literature cite	20
CHAPTER III	**MATETIALS AND METHODS**	
	Apparatuses and chemical reagents	26
	Preparation	27
	Methods	34

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CONTENTS (CONT.)

		Page
	Statistical analysis	37
CHAPTER IV	**RESULTS**	
	Result of preparation of studied xylene detector tube.....	39
	Preparation of xylene concentration by static system.....…	40
	Calibration scale for studied xylene detector tube .....……	42
	The efficiency test of studied xylene detector tube ……	43
CHAPTER V	**DISCUSSION**	
	The studied xylene detector tube preparation……	49
	The preparation of xylene concentration by static system…	51
	The efficiency of the studied xylene detector tube …….	51
CHAPTER VI	**CONCLUSION AND RECOMMENDATIONS**	
	Conclusion…….	54
	Recommendation for further study……	54
REFERENCES……....		56
APPENDIX		
	APPENDIX A: The calculation of xylene concentration Method…….	58
	APPENDIX B: The calculation of accuracy precision Method…….	62
	APPENDIX C: The analytical results of studied xylene detector tube…….	64
	APPENDIX D: The detail of cost per tube…….	73
	APPENDIX E: Figures……	75
BIOGRAPHY……...		77

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LIST OF TABLES

Tables		Page
2-1	Physical and chemical properties of xylene……	11
2-2	Single inhalation exposure to xylene in human……	12
2-3	Physical and chemical properties of potassium iodate……...	13
2-4	Physical and chemical properties of sulfuric acid…..……...	14
2-5	Several carrier gases used in gas chromatography……	17
3-1	The proportion of reagent for coating of silica gel……..	29
3-2	Analytical gas chromatographic condition……..……	32
4-1	The proportion of reagent for coating of silica gel and result of color development…….	39
4-2	Xylene concentration and peak area……	41
4-3	The mean and the standard deviation of stain length corresponding to different xylene concentrations……	42
4-4	The mean and the standard deviation of stain length corresponding to different xylene concentrations……	43
4-5	Overall system accuracy test……	44
4-6	Detection limit of studied xylene detector tube…….	45
4-7	Specicifation of studied xylene detector tube…….	45
4-8	Storage stability of studied xylene detector tube……..…	46
4-9	Field test for studied xylene detector tube and Draeger tube	47

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LIST OF TABLES (CONT.)

Tables		Page
B-1	Mean and standard deviation of precision test ..……...	63
B-2	Overall system accuracy test.……..……...	63
C-1	The analytical results of calibration scale for studied xylene detector tube.……..	65
C-2	The accuracy and precision of studied xylene detector tube ……...	66
C-3	Detection limit of studied xylene detector tube. …….	67
C-4	Specification of studied xylene detector tube …….	68
C-5	The shelf life test at 0 week ……	68
C-6	The shelf life test for 0.1 TLV ……	69
C-7	The shelf life test for 1.0 TLV …….	70
C-8	The shelf life test for 0.1 TLV…….	71
C-9	The shelf life test for 1.0 TLV …….	72
D-1	The detail of cost per tube ……	74

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LIST OF FIGURES

Figures		Page
3-1	The feature of the studied xylene detector tube……	30
3-2	Schematic diagram of zero air generation……..…….	31
3-3	The calibration scale of studied xylene detector tube.....	35
4-1	The standard curve of xylene concentration….……	41
4-2	The calibration scale of studied xylene detector tube…….…….	43
E-1	The Field test for thestudied xylene detector tube and Draeger tube	76
E-2	The studied xylene detector tube was sealed by LPG flame	76

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CHAPTER I
INTRODUCTION

1.1 Background and rationale:

Rapid development of industrialization in Thailand has created air quality problem in working area. The chemical agents such as the organic solvents and raw material were imported for wide use in several industries while the health hazard control still lack of emission, monitoring and need a lot of improvement.

Xylene is widely used chemical in several industries. It has the excellent characteristics as a solvent. It is also commercially important chemical produced in enormous quantities throughout the world (1).

Xylene may have an acute impaired effect on the sensory-motor and information-processing function of the central nervous system (CNS)(1). It can also cause headache, dizziness, respiratory tract irritation and unconsciousness. These effects depend on the amount of chemical intake and duration of exposure. Therefore, accurate determination level of xylene concentration will lead to choosing methods that provide adequate control to ensure that the worker will not expose to excessive concentration while in the working area.

One type of recommended method for analysis of xylene in air is NIOSH method # 1500 and 1501 which have been most frequently used (1,2). The methods describe how to sampling xylene by solid sorbent tube and measure the concentration by gas chromatography. The methods have been used widely but it is rather difficult and time consuming method. The instrument is very expensive and the analysis has to be done in the laboratory room by the professional.

One classical measurement technique of gas analysis is detector tube. In 1919, the first detector tubes patent appeared in U.S.A. Two Americans, A.B. Lamb and C.R. Hoover, impregnated pumice with a mixture of iodine pentoxide and sulphuric acid. This preparation, which they put in the vital, became the first chemical sensor for measuring or detecting carbon monoxide in coal mining (3). Then, in 1947 gas detector tubes were studied. The early detector tubes were made for carbon

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monoxide, hydrogen sulfide, and benzene (4). Since the development of the first detector tube, more than 80 years ago, there has been a great expansion in the development and use of these tubes. The commercial company ,Draeger-Tubes, has expanded the product line to more than 400 Draeger-Tubes capable of measuring more than 400 different gases and vapours (4). They are used widely by industrial hygienists, safety officer and others for measuring concentrations of toxic gas and vapor contaminants in the working area and process line.

The commercial xylene detector tubes are available not only Draeger tubes (Germany) but also Kitagawa tubes(Japan), Gastec tubes(USA) ,Sensidyne tubes (USA) and MSA tubes(USA). Those tubes are imported to Thailand. The tubes are produced in laboratory and used in working environments having many different environmental conditions such as humidity, temperature and pressure. These factors are important for the interaction of reaction in tubes to gain high accuracy of chemical concentration. Moreover, the cost of this commercial tubes is very expensive. It is approximately 150-350 bath / tube.(3)

The objective of this study is to construct an appropriate xylene detector tube . The potassium iodate solution will be used as a coating material on silica gel and the silica gel will be carefully packed in a glass tube. The chemical reaction of studied xylene detector tube is reduction of xylene by potassium iodate solution which are coated on silica gel to iodine. The equilibrium reaction will produce the red-brown color of iodine. The stain length on the tube is an indicator of xylene concentration. The efficiency of the studied xylene detector tube should not differ from the available commercial tube.

1.2 Objective:

1.2.1 The general objective

The general objective is to construct the studied xylene detector tube for sampling and analysis of xylene vapor in working environment and to test its efficiency of the studied xylene detector tube.

1.2.2 The specific objectives :

1.2.2.1 To determine the suitable concentration of potassium iodate solution used for coating on silica gel.

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1.2.2.2 To study the accuracy and precision of the studied xylene detector tube.

1.2.2.3 To study the specificity of the studied xylene detector tube.

1.2.2.4 To study the detection limit of the studied xylene detector tube.

1.2.2.5 To compare the stability of the studied xylene detector tubes when stored in room temperature with those stored in refrigerated temperature.

1.2.2.6 To compare the the studied xylene detector tube with commercial xylene detector tube from Dreager company for sampling and analysis of xylene vapors in the field.

1.3 Hypothesis:

1.3.1 The accuracy of studied xylene detector tube has met with the NIOSH recommended criteria.

1.3.2 The field concentrations of xylene analyzed by studied xylene detector tube and commercial xylene detector tube from Dreager company are not different.

1.4 Scope of this study :

1.4.1 The method used for preparation of the xylene detector tube was applied from H.M. Factory Inspectorate, Minitry of Labour, England.

1.4.2 The experimental conditions in this study were set at temperature, pressure, and relative humidity in laboratory conditions.

1.4.3 The standard concentrations of xylene at 0.1, 0.5, 1.0 TLV were tested in this study.

1.4.4 The concentrations of xylene generated from the static system were reassured by gas chromatography.

1.5 Variables:

1.5.1 Independent variables:

1.5.1.1 The concentration of potassium iodate solution coated on silica gel.

1.5.1.2 The concentration of xylene.

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1.5.2 Dependent variables:

1.5.2.1 The color.

1.5.2.2 The length of stain.

1.5.2.3 The accuracy and precision of the detector tube for xylene.

1.5.3 Control variable:

1.5.3.1 Room temperature of 24-30°C and refrigerated temperature of 10°C

1.5.3.2 Pressure at 760 mmHg

1.5.3.3 Relative humidity (75-76%)

1.5.3.4 Volume of air sample (1,000 ml)

1.6 Glossary of terms and definitions :

1.6.1 National Institute for Occupational Safety and Health(NIOSH): A federal agency conducts research testing, equipment certification, and training programs related to occupational health and safety.

1.6.2 The American Industrial Hygiene Association (AIHA): An association works , studies and produce manual publication, in the field of industrial hygiene .

1.6.3 Threshold limit value(TLV): Airborne concentrations of substances published annually by the American Conference of Governmental Industrial Hygienists (ACGIH) representing condition under which the ACGIH believes the majority of people can routinely work day after day without adverse effects.

1.6.4 Studied xylene detector tube : A glass tube containing a colorimetric reactive compound adsorbed onto fine grain silicas. The glass tube size is 12-cm long with 5-mm outer diameter and 3-mm inner diameter. The reagents which was coated by potassium iodate plus fuming sulfuric acid are sensitive to the xylene gas or vapor and reacted quantitatively to provide red brown color of iodine .

1.6.5 Indicating layer: The colorimetric reactive portion of the detector tube. The silica gel, which was coated by potassium iodate solution, is the indicating layer.

1.6.6 Length of stain: The length of color change in a detector tube. The length of stain is proportional to the concentration of the contaminant in the sampled atmosphere

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1.6.7 Accuracy: The measure of the correctness of data, as given by the difference between the measured value and the true or specified value. Ideal accuracy is zero difference between measured and true value.

1.6.8 Precision: A measure of the agreement among individual measurements of the same variable under similar conditions that is, a measure of the reproducibility of a measured value.

1.6.9 Percent recovery: An estimation of the bias of an analytical method. The percent recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Predicted concentration} \times 100}{\text{True concentration}}$$

1.6.9 Limit of detection (LOD): The lower limit of the range of measurement

1.6.10 Specificity of detection: The tendency of detector tubes to respond to more than one compound or other compounds

1.6.11 Calibration: The comparison of a measuring device with a standard of known quantity and any subsequent marking or correcting of its measurement scale (18). In this study, the concentration of xylene which was read from the studied xylene detector tube was analyzed by the Gas Chromatography.

1.6.12 Shelf life: The period and condition in which detector tubes remain stable when stored in accordance with a manufacturer's specifications. In this study, the studied xylene detector tube will be stored in room temperature compared with that in the refrigerated temperature. The tube stability criterion was recommended by NIOSH (21)

1.6.13 Reagent grade: A chemical reagent that meets standard for purity set by the American Chemical Society. These reagents are produced for laboratory used.

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CHAPTER II
LITERATURE REVIEW

2.1 Development of detector tube

Three types of direct-reading, colorimeteric indicators have been in use for the determination of contaminant concentrations in air: liquid reagent, chemically treated papers and glass detector tube.(5)

2.1.1 Liquid reagents

The liquid reagents are supplied in sealed ampoules or tube, frequently in concentrated or even solid forms that are diluted or dissolved for use. The commercial tube for this type is available for tetraethyl lead and tetramethyl lead. The method of measurement is performed by drawing an air sample into a 50- or 100-ml glass syrings containing a measured quantity of absorbing liquid reagent to produce the color change. These methods are somewhat inconvenient to perform in the field and the methods require skilled person.

2.1.2 Chemically treated papers

Chemically treated papers have been employed to detect and determine gases concentration because of their convenience and compactness. An early example of them is the Gutzeit method in which arsine blacken a paper strip previously impregnated with mercuric bromide. Such papers may be freshly prepared and used wet, or stored and used in the dry state. Semi-quantitative determinations may be made by hanging the paper in the contaminated air. The accuracy of such procedures is limited by the fact that the volume of the air sample is rather indefinite and the degree of color change in the paper is influenced by air currents and temperature. More quantitative results may be obtained by employing a sampling device capable of passing a measured volume of air over or through a definite area of paper at a controlled rate as is done in a commercial device for hydrogen fluoride. The determination was done by addition of liquid reagents to the sample on a filter paper. Evaluation of the stains on the paper may be made visually by comparison with color

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charts or by photoelectric instruments. Accuracy of these methods requires uniform sensitivity of the paper, stability of all chemicals employed and careful calibration.

2.1.3 Glass detector tubes

Detector tubes are glass tubes containing a colorimetric reactive compound adsorbed onto a medium such as fine grain silica gel, activated alumina, silica glass or silica sand. The purpose of most detector tubes is to measure a specific contaminant concentration, although some only identify qualitatively the presence of chemical classes of materials. The measuring range generally includes the range of toxic contaminant concentrations important in the field of industrial hygiene. The glass tubes have constant inner diameters to make measurements precise. The reagent systems are kept stable by hermetically sealing the tubes and not opening them until use. The reagents are sensitive to particular gases, vapors and most of them react quantitatively to provide a colorimetric length-of-stain indication or depth of color comparison. The reaction between the reactant and the contaminant must be rapid and complete.

Now, there are many uses for indicator tubes; they are convenient for evaluation of toxic hazards. They are also useful for air pollution studies. Indicator tube may be used for detection of explosive hazards, as well as for process control of gas composition. Indicator tube may be used for law enforcement purpose, such as determining alcohol in the breath, or gasoline in soil in cases of suspected incendiarism.

The factor of error in detector tube such as the rigid control of the purity of reagents, grain size of gel, method of tubes packing , moisture content in the gel, uniformity of tube diameter, and proper storage precautions is required for the optimal performance of detector tube. It was found during a visit to a detector tube manufacturing facility that there was a lapse of many hours between the packing and sealing of the tubes. Indeed, such a practice permits the entry of not only moisture but also other contaminating vaporous substances which might produce a deleterious effect on the reagent's future response to the analyzed substance.

The tube diameter is another critical parameter that this situation leads to calibration errors as high as 50% due to a varying volume of air sample per unit cross sectional area as compared with those provided under standard test conditions.

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The variations in the grain size of the adsorbed medium such as silica gel, the purity of the reagents, tubes sealing technique and the cleanliness of the air in the manufacturing facility can affect the properties of different batches of the indicating gel markedly.

The shelf life of detector tube, the period in which detector tubes remain stable when stored in accordance with a manufacturer's specifications, will be affected by method of storage. Deterioration of the tubes increases greatly at elevated temperatures and storage under refrigeration is recommended by both the manufacturer and the user .

2.2 Recommendation for detector tube:

In 1971, a joint committee of the American Conference of Governmental Industrial Hygienists (ACGIH) - American Industrial Hygiene Association (AIHA) made the following recommendations.(6)

1. Manufacturers should supply a calibration chart (ppm) for each batch of tubes.

2. Length- of-stain tubes are preferable to those exhibiting change in hue or intensity of color.

3. Tests of calibrations should be made at 0.5, 1.0, 2.0, and 5.0 times the ACGIH Threshold Limit Value (TLV).

4. The manufacturer should specify the methods of tests. Values should be checked by two independent methods.

5. Calibration at each test point should be accurate within ± 25% (95% confidence limit)

6. Allowable ranges and corrections should be listed for temperature, pressure, and relative humidity.

7. Each batch of tubes should be labeled with a number and an expiration date. Instructions for proper storage should be given.

8. Tolerable concentrations of interferences should be listed.

9. Pumping volumes should be accurate within ± 5%, and flow rates should be indicated.

10. Special calibrations should be provided for extended sampling for low concentrations, and flow rates should be specified.

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2.3 NIOSH quality standards for detector tubes: (7,8,18)

In 1978, NIOSH was appointed by the US government as the sole responsible authority for execution of the certification programme for gas detector tubes.

1. The range of measurement of the detector tube concerned should cover the following concentration 0.5, 1.0,2.0,5.0 x limit value

2. At the TLV concentration, either the stain length must be 15 mm or greater.

3. The deviation from the theoretical indication at 0.5 X limit value should not exceed ± 35% while at 1 x limit value, 2 x limit value and 5 x limit value, the maximum admissible deviation is ± 25%. These data relate to the 95% confidence interval.

4. The relative standard deviation of the readings of the same tube by three or more independent readers must be less than 10%

$$\%CV = SD / \bar{X} \leq 10\%$$

CV is the coefficient of variation.

SD is the standard deviation of the tube readings.

$\bar{X}$ is the mean reading for a given tube.

5. The maximum variation of stain length around the circumference of the tube at the interface between stained and unstained reagent is expressed as:

$$\Delta L/M \leq 0.20$$

where : $\Delta L = L_2 - L_1$

L_2 = longest indication.

L_1 = shortest indication.

M = the median value of L_2 and L_1

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2.4 Xylene (1)

Xylene is an aromatic hydrocarbon which exists in three isomeric forms: ortho, meta and para. The technical grade xylene contains a mixture of the three isomers and some ethylbenzene.

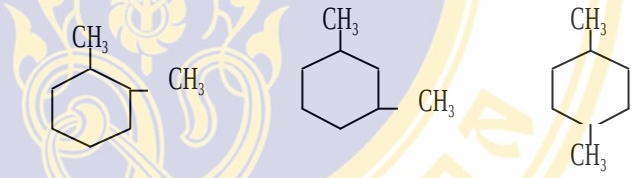
It is a colourless liquid at room temperature with an aromatic odour. The vapour pressure lies between 0.66 and 0.88 kPa for the three isomers that is volatile, flammable, and explosive in air. Approximately 92% of mixed xylenes is blended into petrol. It is also used in a variety of solvent applications, particularly in the paint and printing ink industries.

2.4.1 Identity

Chemical formula

C_8H_{10}	C_8H_{10}	C_8H_{10}

Chemical structure



Chemical name

ortho-xylene	*meta-xylene*	*para-xylene*

Synonyms

1,2-dimethyl-benzene	1,3-dimethyl-benzene	1,4-dimethyl-benzene
o-methyltoluene	m-methyltoluene	p-methyltoluene
1,2-xylene	1,3-xylene	1,4-xylene
o-xylol	m-xylol	p-xylol

Relative molecular mass

106.16	106.16	106.16

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CAS registry number

95-47-6 108-38-3 106-42-3

RTECS registry number

ZE 2450000 ZE 22750000 ZE 26250000

2.4.2 Physical and chemical properties

Some physical and chemical properties are given in Table 2-1

Table 2-1 Physical and chemical properties of xylene

Description	*o*-xylene	*m*-xylene	*p*-xylene
Physical state(20 °C;101.3 kPa)	Liquid	Liquid	Liquid
Colour	Colourless	Colourless	Colourless
Boiling point(°C; 101.3 kPa)	144.4	139.1	138.3
Melting point(°C;101.3 kPa)	-25.2	-47.9	13.3
Relative density (25°C /4 °C)	0.876	0.860	0.857
Vapour pressure(kPa at 20°C)	0.66	0.79	0.86
Flash point (°C) (closed cup)	30	25	25
Explosion limits(vol-% in air)	1.0 – 6	1.1 – 7	1.1 - 9
Autoignition temp (°C)	465	525	525
Solubility in water (mg/litre)	142	146	185

2.4.3 Exposure limits

OSHA : OHSA permissible exposure limits (PELs) are 100 ppm (375 mg/m^3) for an 8 –hour time weighted average (TWA) concentration, and 150 ppm (560 mg/m^3) for a 15- minute short-term exposure limit (STEL)

NIOSH: NIOSH recommended exposure limit (REL) are 100 ppm for TWA of an 8 hour workday and a 40-hour workweek, and 200 ppm (750 mg/ m^3) as a 10-minute ceiling limit.

ACGIH: ACGIH threshold limit values (TLVs) are 100 ppm as a TWA for a normal 8-hour workday and a 40-hour workweek.

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2.4.4 Toxicity

Human are exposed to xylene mainly by inhalation. This compound does not accumulate significantly in the human body. Xylene appears not be a mutagen or a carcinogen

Acute effects: The acute exposure to high concentration can result in central nervous system (CNS) effects in human. A summary of acute effects from inhalation exposure is presented in table 2-2

Table 2-2 Single inhalation exposure to xylene in human

Exposure concentration (mg/m^3)	Time	Effect
3,000	1h	Dizziness, Irritation
3,000	15 min	Dizziness
1,300	2 h	Performance decrement
900	4 h	Prolonged reaction times
900	4 h	Impairment of vestibular and visual function and prolonged reaction time
900	4 h	Minor effect on EEG
600	4 h	No effect on reaction time
450	4 h	Prolonged reaction time
300	4 h	No effects in psychophysiological test

Chronic effects: The long-term exposure to xylene is suspected to affect the nervous system adversely because chronic toxic encephalopathy and milder functional disturbances of the brain have sometime been found among exposed painters and other workers. Likewise, slight changes in kidney tubular function may occur. Apparently irreversible effects on the CNS were found 4 months after a 3-month inhalation exposure (24h per day) of Mongolian gerbils to xylene at concentration of 696 or 1,392 mg/m^3

No adequate studies of reproduction and development toxicity in human exposed to xylene alone have been published.

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In serveral short-term and long term animal studies, effects on the activities of various metabolic enzymes in different organs have been observed.

2.5 Potassium iodate

2.5.1 Identity

Chemical formula

KIO_3 ; I 59.305%, K 18.27%, O 22.43%

Chemical name

Potassium iodate

Synonyms

Iodic acid, Potassium salt

Formula wt

214.00

CAS registry number

07758-05-6

2.5.2 Physical and chemical properties

Some physical and chemical properties are given in Table 2-3

Table 2-3 Physical and chemical properties of Potassium Iodate

Description	Potassium Iodate
Physical state(20 °C;101.3 kPa)	Solid
Colour	White crystals
Boiling point(°C;)	N/A
Melting point(°C;)	560
Vapor density	N/A
Specific gravity	3.89
Solubility in water	> 10%

Potassium Iodate is a strong oxidizer, when contacts with other materials may cause fire then, its contact with clothing and other combustible materials should be avoid. Potassium iodate should not store near combustible materials. The contact of this chemical with eyes, skin, clothing must be avoided. It should be kept in tightly

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closed container. The worker should wash thoroughly after handling. In case of fire, this chemical should be soaked with water. In case of spill, this chemical should be swept up and removed and the spill area should be flushed.

2.6 Sulfuric acid:

2.6.1 Identity

Chemical formula

H_2SO_4

Chemical name

Sulfuric acid

Synonyms

Oil of vitriol

Formula wt

98.08

CAS registry number

7664-93-9

2.6.2 Physical and chemical properties

Some physical and chemical properties are given in Table 2-4

Table 2-4 Physical and chemical properties of sulfuric acid

Description	Sulfuric acid
Physical state(20 °C;101.3 kPa)	Liquid
Colour	Clear, Colourless to light yellow, oily odorless liquid
Boiling point(°C;)	327 °C
Melting point(°C;)	-2 °C
Vapor density	3.4
Specific gravity	1.84
Solubility in water	complete

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Sulfuric acid has precautionary label statements such as poison danger, harmful if inhaled, cause severe burns, may be fatal if swallowed and reacts violently with water. The warning of this chemical is as follows: do not get in eyes, on skin, on clothing and do not breathe its vapor. This chemical should be kept in tightly closed container, loosen closure cautiously. This chemical has to be used with adequate ventilation and washed thoroughly after handling. In case of spill ,this chemical has to neutralize with soda ash or lime and place in dry container.

2.7 Silica gel: (3,10)

Silica gel was developed just prior to World War I (WWI) and was used in Allied gas masks; then, in WWII it was used extensively to keep penicillin dry. It remains one of the highest capacity adsorbents available today. Silica gel is a partially form of polymeric colloidal silicic acid. The chemical composition can be expressed as SiO_2 .nH2O. The water content, which is presented mainly in the form of chemically bound hydroxyl groups, amounts typically to about 5 wt.%.

The internal structure of each small silica gel granule is composed of a vast network of inter-connecting microscopic pores. The silica gel has the hydroxyl groups imparts a degree of polarity to the surface so that molecules such as water, alcohols, phenols, and amines (which can form hydrogen bonds) and unsaturated hydrocarbons (which can form π-complexes) are adsorbed in preference to non-polar molecules such as saturated hydrocarbons.

Because of its selectivity for aromatics, silica gel was used as the adsorbent in the Arosorb process for separation of aromatics from paraffins and naphthenes but by far the most important current application is as a desiccant which attracts and hold moisture by a phenomena known as physical adsorption and capillary condensation.

There are two types of silica gel for use purpose:

- One has a built-in color indicator, bright blue crystals of cobalt chloride which act as moisture indicators. As the silica gel begins to absorb moisture from the environment , the cobalt chloride crystals will indicate this by turning to light blue and then pink. When the silica gel has absorbed up to 40 % of its own weight in moisture, it will no longer be effective in removing moisture from anything.

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- The other type of crystal is white. This type is prepared for gas chromatography and laboratory experiment.

There are three types of silica gel for density purpose:

Regular-density silica gel: very small particles with high surface area approximately 750 – 800 m^2/g . with average pore diameter of 2.2 – 2.6 nm.

Intermediate-density silica gel: low surface area approximately 300 – 350 m^2/g . with average pore diameter of 12 – 16 nm.

Low-density silica gel: lower surface area approximately 100 – 200 m^2/g. with average pore size of 18 – 22 nm.

2.8 Gas Chromatography (8,19,20)

Chromatography is a collective term for separations of mixtures based on the partition of substances between two immiscible heterogeneous phases, one of which is a stationary or fixed phase with a large surface area, and the other a moving or mobile phase which flows over the first phase. Gas chromatography is the most recent branch of chromatograph and includes all the chromatographic processes in which the substance to be analyzed occurs in the gaseous or vapor state or can be converted into such a state.

In gas chromatography, the sample is vaporized and injected onto the head of a chromatographic column. Elution is brought about by the flow of an inert gaseous mobile phase. The mobile phase does not interact with molecules of the analyte; its only function is to transport the analyte through the column. Two types of gas; chromatography are encountered, gas-solid chromatography (GSC) and gas-liquid chromatography (GLC). Gas-liquid chromatography finds widespread use in all fields of science, where its name is usually shortened to gas chromatography (GC).

Gas-solid chromatography (GSC) is based upon a solid stationary phase in which retention of analytes is the consequence of physical adsorption. The stationary phase is an active solid such as alumina, charcoal, silica gel, molecular sieves, or the newer plastic granules.

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Gas-liquid chromatography (GLC) is based upon the partition of the analyte between a geseous mobile phase and a liquid phase immobilized on the surface of an inert solid.

The basic components of an instrument for gas chromatograph consist of : (1) carrier gas supply and controls, (2) sample injection system, (3) chromatographic column and oven, (4) detection systems, and (5) integrator and chart recorder.

2.8.1 Carrier gas supply

The carrier gas acts as the mobile phase and transports the sample component through the column to the detector, retardation occurring due to interaction with the stationary phase. Carrier gases, which must be chemically inert, include helium, introgen, and hydrogen. The type of detector used often dictates the choice of gases. Associated with the gas supply are pressure regulators, gauges, and flow meters. In addition, the carrier gas system often contains a molecular sieve to remove water or other impurities. The applications of carrier gases are shown in Table 2-5

Table 2-5 Several carrier gases used in gas chromatography

Carrier gas	Application	Comments
Helium	General carrier gas or make up gas.	Excellent but can be expensive cannot use with ECD.
Nitrogen	General carrier gas or make up gas.	Cheap, not good for chemicals with long retention time.
Hydrogen	Carrier gas for capillary column and combustion gases for FID, NPD, FPD	Highly flammable.
Argon	Carrier gas for TCD.	Quantitative determination of other gases.

Impurities in the carrier gas such as air, water vapor and trace gaseous hydrocarbons can cause sample reactions, column deterioration and affect detector performance. Carrier gas purity should be better than 99.99% and 99.999%. In

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addition, small variations in the carrier gas flow-rate will affect column performance and retention times. Therefore, to achieve reproducible separations, it is necessary to maintain a constant flow rate.

2.8.2 Sample injection system.

The sample injector located ahead of the column is designed to allow the introduction of a sample rapidly and in a reproducible manner into the column. The most common method of sample injection involves the use of a micro-syring to inject a liquid or gaseous sample through a self-sealing, silicone-rubber diapharagm or septum into a flash vaporizer port located at the head of the column.

The sample should be introduced onto the beginning of the column with the minimum dispersion in the mobile phase. The quantity of liquid sample ranges from 1 to 10 microliters and gas sample varies from 0.05 to 50 milliliters. Most samples are introduced by means of a small, calibrated syringe or a micro-syringes.

2.8.3 Chromatographic columns

The column is the “heart” of the chromatograph. Provided that the equipment is good and operating conditions are suitably chosen, the analysis will be as good as the performance of the column selected because it is here that separation of the sample components is affected. Two general types of column, are the packed and capillary column. To date, the vast majority of gas chromatography has been carried out on packed columns.

2.8.3.1 The packed column

The packed column consists of a length of tubing 1-6 millimeters I.D. and usually 0.5 to 6 meters in length, which is normally coiled or looped to be accommodated in the space provided in the instrument. This tubing is packed with a finely divided inert solid support, which is coated with a thin layer of a non-vaporizable liquid, referred to as the liquid phase.

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2.8.3.2 The capillary column

The capillary column is an empty tube, the inner walls of which serve as the support or adsorbent. This type of column has an I.D. of 0.1 – 1.0 millimeter and usually is 30-100 meters in length.

Column temperature is an important variable that must be controlled to a few tenths of a degree of precised work. Thus, the column is ordinarily housed in a thermostated oven. The optimum column temperature depends upon the boiling point of the sample and the degree of separation required. Roughly, a temperature equals to or slightly above the average boiling point of a sample results in a reasonable elution time (2 to 3 min).

2.8.4 Detection system

The detector is located at the exit of the column for analyzing the concentration of components, which appear in the carrier gas as it emerges from the column, by means of change in a physical or chemical property of the effluent gas stream. The detector provides information on the distribution of individual peak within a chromatogram as well as their relative quantity.

There are numerous detectors used for GC, only three widely use of these are described: flame ionization detector, thermal conductivity, detector and electron capture detector.

Flame Ionization Detector (FID)

The flame ionization detector is the most widely used and generally applicable detector for gas chromatograph. The effluent from the column is mixed with hydrogen and air and then ignited electrically. Most organic compounds, when vaporized at the temperature of a hydrogen/air flame, produce ions and electrons that can conduct electricity through the flame.

A potential of a few hundred volts is applied across the burner tip and collector electrode located above the flame. The resulting current ($\approx 10^{-12}$ A) is then directed into a high-impedance operational amplifier for measurement. The ionization of carbon compounds in a flame is a poorly understood process, although is observed that the number of ions produced is roughly proportional to the number of reduced carbon

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atoms in the flame. The flame ionization detector responds to the number of carbon atoms entering the detector per unit of time.

Thermal Conductivity Detector (TCD)

Thermal conductivity is a measure of the ability of substance to conduct heat. TCD still finds wide application, is based upon changes in the thermal conductivity of the gas stream brought about by the presence of analyte molecules. There are two pairs of electrically heated wires in the ECD, one pair being located in the flow of the effluent from the column and the other in the gas stream ahead of the sample injection chamber.

The resistance of the wire or thermistor gives a measure of the thermal conductivity of the gas; in contrast to the wire detector, the thermistor has a negative temperature coefficient. The change in resistance is monitored and changed into electrical signal. The advantage of the thermal conductivity detector is its simplicity, its large linear dynamic range (~10^5), its general response to both organic and inorganic species, and its nondestructive character, which permits collection of solutes after detection.

Electron-Capture Detector (ECD)

Electron-capture detector is widely used for environmental samples because this detector selectivity detects halogen-containing compounds, such as persticides and polychlorinated biphenyls. The effluent from the column is passed over a B-emitter, usually nickel-63. An electron from the emitter causes ionization of the carrier gas (often nitrogen) and the production of a brust of electrons. In the absence of organic species, a constant standing current between a pair of electrodes results from this inization process.

2.9 Literature cited:

In 1960-1970,(3) Draeger (Germany) developed the xylene detector tube. The xylene 10/a was used for measuring xylene concentration from 10 to 400 ppm with 5 pump strokes of air (measuring period: approx. 1 minute) The reaction method was $C_6H_4(CH_3)_2$+ HCHO + H_2SO_4 ⟶ chinoid reaction products and it produced

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reddish brown color. Temperature used between 0-40°C and maximum humidity used was 3 - 15 mgH_2O/L. The standard deviation of xylene conc. was ± 20-30%. The chemicals causing cross sensitivity were styrene, vinyl acetate , toluene, ethyl benzene and acetaldehyde, however, with differing sensitivity.

In 1966,(9) Ministry of Labour, England developed the methods to determine the concentration of toluene and xylene quickly and easily. Principle of the method was that toluene and xylene reacted with an acid solution of potassium iodate to produce a yellow color. The intensity of this color which was proportional to the toluene concentrations was read by comparison with a series of Lovibond permanent glass color standards. The limitation of this method was potassium iodate solution was stable for 1 week after that it must be discarded and this disc contained three standards which corresponded to 100, 200, and 300 ppm of toluene and/or xylene when a 120-ml sample of air is drawn through the bubbler at 25ml/minute. Comparing the colour with the standards in the disc, 2 to 4 minute after the completion of sampling, and reading off the toluene/xylene concentration from the indicator window in the comparator are recommended. When toluene and xylene were presented together, the total concentration found should be quoted as xylene. The interferences of this method were styrene and ethyl benzene reacted like toluene and xylene, benzene also reacted but at 25 ppm or less and the color produced was insignificant. ortho and para Xylene and some commercial xylenes gave results which might be 10% low.

In 1976, Frederick K. Colen (11) studied the interchange ability of the length-of-stain gas detector tubes and sampling pumps of different manufactures and the relationships such as pressure-time, flow rate-time, and actual interchange data were evaluated. Interchanging gas detector tubes and sampling pumps of different manufactures are not recommended.

The results showed that the interchange of various manufacturers' gas detector tubes with various manufacturers' sampling pumps could introduce systematic errors. Each manufacturers' CO gas detector tube calibrated for a fixed sampling volume and air flow rate dictated by its sampling pump. To connect its CO gas detector tube to other manufacturer's sampling pump would yield purely qualitative results. These systematic errors added to current errors already inherent in each sampling pump and detector tube would increase the total error to the point that

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the quantitative result would be reduced to a qualitative level. This modification is not in the best interests of the user. Also, NIOSH will be certifying a manufacturer's gas detector tube and sampling pump to be used as a unit, and therefore, to use each system most effectively, the gas detector tube and sampling of a specific manufacturer should not be interchanged with those of another manufacturer unless qualitative results are desired.

In 1977, Kurt Leichnitz (12) used detector tube under extreme condition (humidity, pressure, temperature) for studying the effects of environmental conditions to detector tubes. Detector tubes are mainly used in a temperature range from 0°C to 40°C. By utilizing simple warming-up methods or cooling devices, the temperature of the gas to be tested can be adjusted to a temperature which is suitable for detector tubes. Humidity often influences chemical reagents. In detector tubes, this interfering effect can be eliminated by special drying preparations. The reading of most of the detector tubes is directly proportional to the absolute air pressure. Pressure correction can be done easily by the gas law.

Influence of humidity on the indication behavior of detector tubes can be eliminated by the tube containing a preliminary layer as a special filter, in which the humidity of the air sample is quantitatively adsorbed. Thus the sample air coming into contact with the reagent of the indicating layer is always dry.

Elimination of effect of temperature, cold ambient air can be eliminated by grasping the detector tube in the hand. Hot ambient air can be eliminated by a cooling probe. The probes described here are successfully used for air investigations of flue gases. CO, CO_2, SO_2, and nitrogen oxide.

In 1981,D.Jentzch and D.A. Fraser (14) evaluated long term detector tubes for analysis of vapors in the laboratory using a sensitive dynamic system capable of generating a relative humidity from 10 to 100% and vapor concentrations from 5 to 2000 ppm. The detector tubes were exposed to known concentrations under specified test conditions. Charcoal tubes were also tested simultaneously under the same conditions. The results indicated that the color development of the long term detector tubes was more affected by humidity and sampling volume than that of the short term tubes. They found that the trend showed accuracy decreased as humidity increases and the detector tubes indicated a lower concentration of solvent than was actually

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presented. Different approval criteria and test methods may be required for these tubes.

The long term detector tubes may well compete with charcoal tubes if certain improvements can be made by the manufacturers to increase reliability. The effects of humidity on indicating behavior should be tested. The response time of the indicating layer should be tested The precision and accuracy of long term detector tubes may encourage the practicing industrial hygienist to use these devices for monitoring workplace environment.

In 1982, Charles S. McCammon, et. (13) studied the effect of extreme humidity and temperature on gas detector tube performance. U.S. Bureau of Mines informed that the detector tube certification program did not use in adverse environmental condition of coal mines. In order to respond to the Bureau of Mines requested, NIOSH planned a testing program to determine exactly how detector tubes were affected by adverse environmental extremes.

The five replicate samples of each of two brands of detector tubes were tested at two concentrations (at the current OSHA standard and 2 times the standard) under four different environmental conditions of temperature and humidity for carbon monoxide, carbon dioxide, nitrogen dioxide, nitric oxide, hydrogen sulfide and formaldehyde. Each tube was tested by three different observers. An analysis of variance was used to statistically determine the magnitude of biases due to humidity and temperature. Both humidity and temperature caused increases in the coefficient of variation of certain tubes from different manufacturers.

The conclusions showed that in general, humidity and temperature affect the readings of both brands of gas detector tubes with a severity which is dependent upon the concentration being observed. Of the twelve certified tubes tested, eight would have failed the certification testing if the conditions of high and low temperature and humidity used in this experiment were required in the certification program.

In 1982, David H. Carlson, et.(15) studied a laboratory method to determine the accuracy of techniques for measurement of airborne pollutant concentrations in diluted diesel exhaust. The method involves dilution of the exhaust from a diesel engine operated at various controlled speeds and loads and sampling the exhaust-air mixture in a manner designed to simulate sampling in air surrounding

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diesel equipment operated in confined spaces such as in and underground mine. The method developed was applied to detector tubes for CO, CO_2, NO, NO_2 and SO_2. This included re-calibration of the detector tubes with certified gases checked along side of NBS Standard Reference Gases. Diesel exhaust measurements by detector tube were compared with simultaneous measurements by portable instruments which earlier proof specific for the pollutant of concern. The differences between detector tube and instrument readings were assumed to be made up of detector tube calibration and interference errors. The calibration errors were subtracted from the total error to obtain the interference error. All of the detector tubes tested were found to have significant diesel exhaust interference errors.

The conclusions showed that a procedure has been developed by which the existence of significant detector tube measurement errors resulting from interfering substances in diesel exhaust can be identified. Application of the procedure developed for assessing the effects of diesel exhaust interferences in small samples of detector tubes which measured the concentrations of CO, CO_2, NO, NO_2 and SO_2 in the TLV range revealed observer bias, calibration errors and interference error. The observer bias in determining the position of the end-of-stain was a relatively small source of error which increased with decreasing clearity of the end of stain. The calibration errors were much larger than those due to bias and were outside NIOSH limits for several certified tubes. The interference errors were greater than 35% of the correct reading for significant percentages of measurements with each of the detector tube types tested.

In 1996, Phuthaporn Suwanajarus (16) studied the comparison of test paper and detector tube to measure the concentration of sulfur dioxide in air. The lowest volume of air sampling of sulfur dioxide was determined by test paper and detector tube were 100 ml and 500 ml, respectively. The lowest concentration of sulfur dioxide was determined by test paper and the highest reliability was 1 ppm but the lowest concentration of sulfur dioxide was determined by detector tube was 5 ppm and the highest reliability were 5, 10 and 20 ppm respectively. The conclusion showed that test paper had higher sensitivity but lower reliability than the detector tube. Many factors affected to the efficiency of detector tube, therefore; the controlling factors and the special technique was required to produce the appropriate detector tube.

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In 1998, Apiradee sriopas. produced a toluene detector tube model and compared with the commercial one and tested the reproducibility under different storage conditions. The indication gel packed into the tube was prepared by coating silica gel with potassium iodate solution (KIO_3) . This indicating reagent become brown color when reacted with toluene. The research study showed that the efficiency of the above toluene tube model could be equivalent to the commercial one. The intensity and the stain length could be read clearly and easily. Therefore, the method used to produce toluene detector tube should be appropriate for commercial production in Thailand in order to reduce the costs of industrial hygiene spent for toluene monitoring in workplace.

In 2001 Haag W.R., studied interchangeability of gas detection tubes and hand pumps. Users of gas detection tubes occasionally seek the convenience of using a single hand pump with different brands of tube, to avoid the need to carry more than one pump. Several professional organizations recommend against such interchangability. However, these recommendations appear to be based on a single study of pump designs that mostly are no longer use. The present study was undertaken to determine if current hand pumps were interchangeable. Both piston type and bellow type hand pump were evaluated by comparing pump flow profiles and test gas measurements with a variety of tubes. The results demonstrated that three piston hand pump in common use (Sensidyne/Gastec GV/100, RAE Systems LP1200 and Matheson-Kitagawa 8104-400A) were fully interchangeable. Two bellows pump (Draeger Accuro and MSA Kwik-Draw) also were interchangeable with each other. Mixing of bellows and piston system is often possible, but there is enough exception to conclude that such practice should be discouraged because it can give inaccurate readings. It is recommended that technical standards be adopted, such as total volume and an initial pump vacuum or a pump flow curve, to assess hand pump interchangeability. When two manufacturers' pumps meet the same standard and routine leak tests are conducted, interchangeable is scientifically valid and poses no risk to the end user while offering greater convenience.

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CHAPTER III
MATERIALS AND METHODS

The study is an experimental research design to construct the xylene detector tube, which is suitable for sampling and analysis of xylene vapour. There will be the following steps for constructing the xylene detecter tube:

1. Preparation of studied xylene detector tube.
2. Preparation of known concentration of xylene.
3. Testing efficiency of studied xylene detector tube by the following steps:
   3.1 The accuracy and precision of studied xylene detector tube.
   3.2 The detection limit of studied xylene detector tube.
   3.3 Comparison of the efficiency of studied xylene detector tube when stored at room temperature with those stored at refrigerated temperature.
   3.4 Comparison of the studied xylene detector tube for sampling and analysis of xylene vapors in the field with the commercial detector tube from Dreager company.
4. The statistical analysis

3.1 Apparatuses and Chemical Reagents

3.1.1 Apparatuses

1. Glass tube(pyrex) with 5-mm O.D. and 3-mm I.D.
2. Glass wool low in lead, (AJAX, Australia)
3. Aluminum piece
4. Gas burner
5. 15 Kg of LPG
6. Beaker
7. Pipet, 1.0 and 5.0-ml
8. Electronic analytical balance, (LIBROR AEL-160 SERIES,Shimadzu,Japan)
9. Desiccator

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10. Oven, (Model-25, Memmer, Germany)
11. U-tube manoteter
12. Vibration shaker
13. Gas chromatograph (G.C. 14B,Shimadzu, Japan), equipped with flame ionization 14. detector, an integrator (Shimadzu C-R7A,Japan)
15. Air compressor (Model CS-51,Cheg Yuan industrail,Taiwan)
16. Soap bubble meter
17. Stop watch
18. 5,25-liter Tedlar air bag (SKC,USA)
19. 2.0 ml gastight syringe (Hamilton)
20. 10 yl micro syringe (Hamilton)
21. Hydrogen gas (Ultra high purity grade, TIG,Thailand)
22. Helium gas (High purity grade, TIG,Thailand)
23. Zero air (High purity grade, TIG,Thailand)
24. Aspirating pump model AP-1(Kitagawa, Japan)
25. Draeger hand pump (Draeger ,Germany)
26. Draeger xylen 10/a tube (Draeger ,Germany)
27. SKC personal air sampling pump (SKC. Eighty Four,Pa,USA)

3.1.2 Chemical reagents

1. Xylene (AR grade, Merck)
2. Silica gel 60, 35-70 mesh ASTM (MERCK, Germany)
3. Potassium iodate (AJAX, Australia)
4. 95-97% Sulfuric acid (MERCK, Germany)
5. 65% Nitric acid (MERCK, Germany)
6. Distilled water

3.2 Preparations

3.2.1 Preparation of solutions

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3.2.1.1 Nitric acid solution. Conc. Nitric of 50-ml was diluted in 50-ml of water.

3.2.1.2 Potassium iodate solution. Potassium iodate solution was prepared at three different concentrations: one mg of potassium iodate was dissolved in 8, 9 and 10 ml of conc. sulfuric acid, respectively.

3.2.2 Preparation of studied xylene detector tube

3.2.2.1 The blank glass tube preparation.

The glass tubes (5-mm OD, 3-mm ID) were cut for 120-mm long, open ends. They were soaked in nitric acid solution for 30 minutes to remove possible contaminants and cleaned by a lot of distilled water and dried at 110^{o} C for 1 hours. Then, they were cooled down for a few minutes by leaving them in the oven after turning it off. After that, they were hermetically sealed at one of the open ends of tube by the liquid petroleum gas (LPG) flame. They were cleaned and dried again and kept them in a desicator.

3.2.2.2 The appropriate condition for coating of silica gel with potassium iodate solution

The silica gel was dried at 110^{o} C for 1 hour to remove the moisture and let it cool down in the desiccator. The silica gel was coated with potassium iodate solution by mixing 0.75-gm silica gel with different volume of potassium iodate solution (section 3.2.1.2) in the test tube following the proportion of reagents in Table 3-1.

Table 3-1 The proportion of reagent for coating of silica gel

Potassium iodate solution		Coating of silica gel	
Potassium iodate (gm)	Sulfuric acid (ml)	Silica gel (gm)	Potassium iodate solution (ml)
1	8	0.75	0.75

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Table 3-1 The proportion of reagent for coating of silica gel (contanuous)

Potassium iodate solution		Coating of silica gel	
Potassium iodate (gm)	Sulfuric acid (ml)	Silica gel (gm)	Potassium iodate solution (ml)
1	8	0.75	0.40
		0.75	0.25
		0.75	0.20
1	9	0.75	0.25
		0.75	0.20
1	10	0.75	0.25
		0.75	0.20

The silica gel was coated with different proportion of potassium iodate solution and the coated silica gel was packed into the blank glass tube simultaneously. The packed silica gel tube will be tested with 100 ppm of xylene to select the appropriate potassium iodate solution and the appropriate volume of potassium iodate solution to be coated on 0.75-gm silica gel. The appropriate potassium iodate solution and the right quantity of it will be used for coating of silica gel for the rest of experiment.

3.2.2.3 Packing of the studied silica gel tube.

The 0.5 cm. long of glass wool was inserted into the open end of the blank glass tube (section 3.2.2.1) and pushed it to the end of tube (Close end). Each type of coated silica gel was poured into the blank glass tube using a small funnel to obtain 7-cm long of coated silica gel. The 0.5-cm long of glass wool was put into the tube above the coated silica gel. The glass tube were hermetically sealed at the open end of tube by LPG flame. The prepared silica gel tube was called as studied xylene detector tube. The feature of studied xylene detector tube is shown in Figure 3-1

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Figure 3-1 The feature of the studied xylene detector tube.

3.2.2.4 Testing of pressure drop of the studied xylene detector tube.

The 10% of studied xylene tube was sampling for testing of pressure drop. The studied xylene detector tube were cut at the both ends of tube. The tube was connected to the U-tube manometer and connected to the personal air sampling pump. The pump was run at 1 LPM and the pressure drop (inch of water) was recorded.

3.2.3 Preparation of standard curve of xylene

3.2.3.1. The zero air generator development

The zero air cylinder ,which was equipped to a pressure regulator and a pre-cleaning column (packed with activated carbon) , was connected to a rotaneter. The float level of rotameter was calibrated by soap bubble meter and marked at position of 2 LPM. The 2 LPM of air flow rate was controlled at the marked position.

3.2.3.2 The air sampling bag preparation.

The 5 and 25- liter Tedlar air bag were cleaned by flushing with zero air for several times to ensure that the bags were cleaned and possible

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contaminants adsorbed on the internal surface was removed. Then , a bag was evacuated completely. The schematic diagram of the system is shown in Figure 3-2

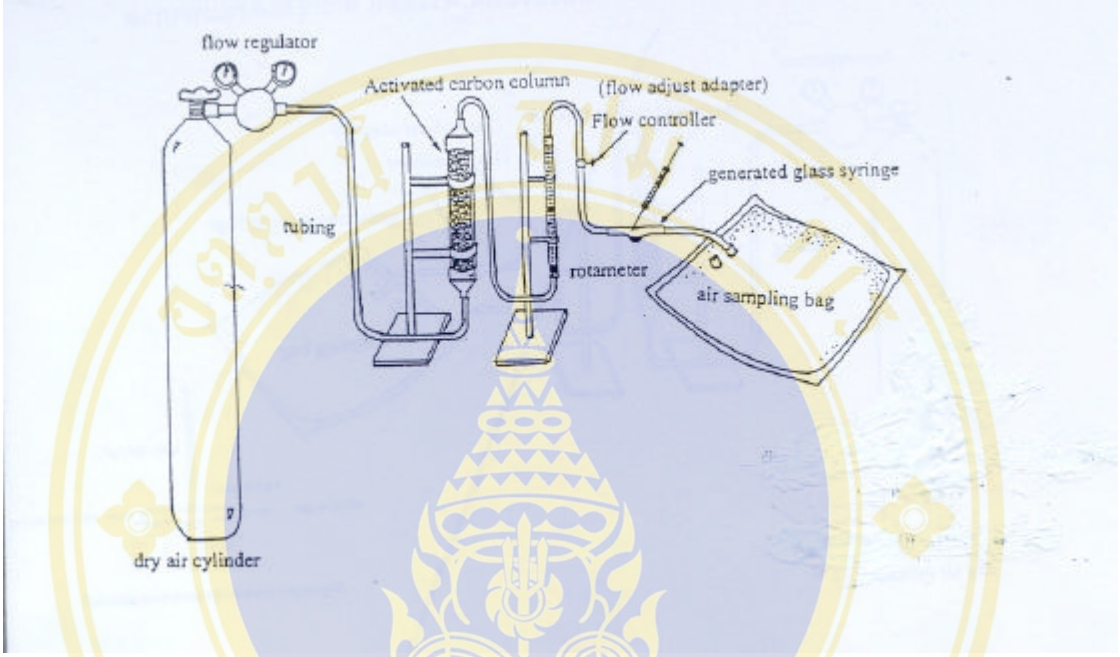

Figure 3-2 Schematic diagram of zero air generation.

3.2.3.3 The preparation of standard xylene concentration in air

The standard xylene concentration in air was prepared by mixing the amount of liquid xylene with air in the Tedlar bag. The volume of liquid xylene was calculated by the following formula :

m	=	**$4.1 \times 10^{-8} \times M \times V \times C$**
m	=	mass of liquid required in grams (g) at 25°C, 1 atm
M	=	molecular weight in atomic mass units (amu)
V	=	volume of dilution vessel in L
C	=	concentration in ppm
Volume	=	$\frac{m}{d}$
d	=	density of xylene (0.868 g/ml)

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The standard xylene concentration in air at 10 , 50 , 100 and 150 ppm was prepared by injecting 0.2 , 1.0 , 2.0 and 3.0 µl of liquid xylene in each 4-litre of air in Tedlar bags, respectively. The detailed calculation was shown in appendix A . The concentration of xylene in the bag was analyzed by the gas chromatographic system.

3.2.3.4 The standard curve of xylene concentration in air by GC.

The condition of gas chromatograph was set at the condition in Table 3-2

Table 3-2 Analytical gas chromatographic condition (G.C. Condition)

Gas chromatograph	Condition
Detector	Flame ionization detector (FID)
Column	DB^{TM}-1 capillary colume (30 mX0.53 mm dia)
Temperature	70 °C (column) 150 °C (injector) 180 °C (detector)
Carrier gas	Helium (10 ml/min.)
Hydrogen	Flow rate of 20ml/min.
Injection size	2 ml (Vapor)

The standard curve of xylene in air was prepared at the concentration of 10, 50, 100 and 150 ppm. The 2 ml of vapour of different xylene concentrations were drawn from each bag by gas-tight syringes. The air sample was injected through an injection port located at the head of the gas chromatographic column. The air sample was analyzed by gas chromatography. The peak area of xylene concentrations was recorded in mVS. The standard curve of xylene was plotted between xylene concentration (ppm) in air and peak area (mVS).

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3.2.4 Preparation of known concentration of xylene and toluene in air.

3.2.4.1 The preparation of known xylene concentration for detection limit testing

The volume of liquid xylene was calculated following the section 3.2.3.3. The known xylene concentration at 5 ppm and 10 ppm were prepared by injecting 0.2 µl of liquid xylene in 8 liter of air in tedlar bags (25-liter) and in 4 liter of air in tedlar bags (5-liter) respectively . The concentration of xylene in the bag was analyzed by the gas chromatographic system following the section 3.2.3.4. The concentration of xylene was read from the standard curve of xylene in air.

3.2.4.2 The preparation of known toluene concentration for specificity testing

The volume of liquid toluene was calculated following section 3.2.3.3 and injected this a mount of toluene into the tedlar bags. The known toluene concentration at 50 ppm were prepared by injecting 4.4 µl of liquid toluene in 4 liter of air in tedlar bags(5-liter) . The concentration of toluene in the bag was analyzed by the gas chromatographic system following the section 3.2.3.4. The concentration of toluene was read from the standard curve of toluene in air.

3.2.4.3 The preparation of mixing known toluene and xylene concentration for specificity testing

The volume of liquid toluene and xylene were calculated following the section 3.2.3.3 and injected those mounts of toluene and xylene into the same tedlar bags. The known xylene concentration at 50 ppm toluene and 50 ppm xylene were prepared by injecting 4.4 µl of liquid toluene and 1.0 µl of xylene into the 8 liter of air in the same tedlar bag (25-liter) respectively . The concentration of toluene and xylene in the bag were analyzed by the gas chromatographic system following the section 3.2.3.4. The

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concentration of toluene and xylene was calculated from the respective standard curve of those two chemicals.

3.3 Methods

3.3.1 Testing of studied xylene detector tube.

The hand pump was checked for leakage. Before sampling, the studied xylene detector tube was cut at both ends of the tube. One end of the tube was connected to the pump following the direction specified on the tube. The handle of the pump was pulled at a full stroke of 100 ml. The air sample was drawn to the tube for 10 strokes per tube (1,000 ml). The studied xylene detector tube was removed from the pump inlet. The xylene concentration was read visually or measured from concentration scale as the stain length of red brown color on the tube.

3.3.2 The appropriate studied xylene detector tube

All types of studied xylene detector tubes were prepared in section 3.2.2.. The studied xylene detector tubes were packed with different conc. of potasium iodate solution and different proportion of chemical coating on silica gel. These tubes were tested with 100 ppm of xylene according to the method in section 3.3.1. The studied xylene detector tube was read visually and the length of stain and the color intensity were recorded. The appropriate studied xylene detector tube was selected from the tube showing strong red brown color. The appropriate xylene detector tube will be used for the rest of experiment.

3.3.3 The calibration scale for studied xylene detector tube.

The calibration scales were used for determination of xylene concentration from the studied xylene detector tube. The studied xylene detector tube was tested with xylene concentrations of 0.1(10 ppm) , 0.5 (50 ppm)and 1.0 TLV (100 ppm) following the method in section 3.3.1 and the experiments were performed for 5 replications. The length of stain was measured and recorded. The xylene

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concentration was determined by the corresponding length of stains from the standard curve. The calibration scale of xylene concentration was made in the form of sticker posted on the tube studied xylene detector tube. The calibration scale of studied xylene detector tube is shown in Figure 3-3

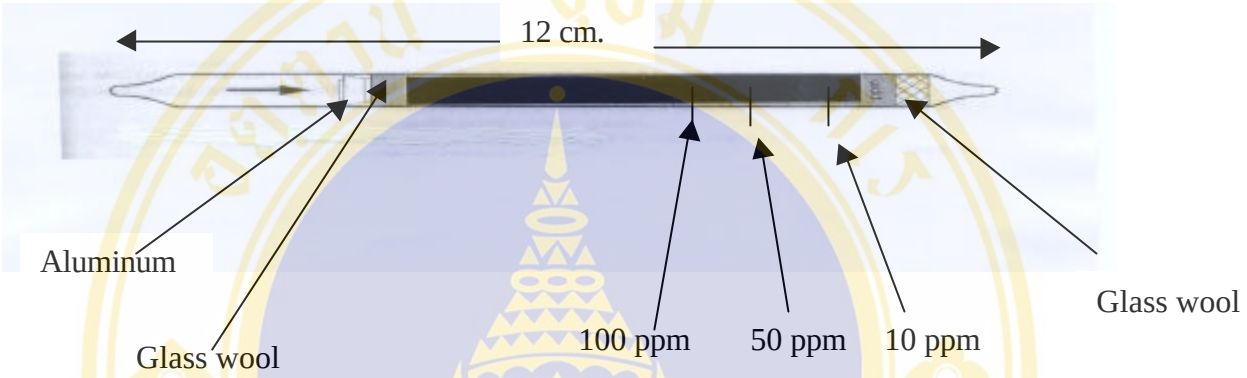

Figure 3-3 The calibration scale of the studied xylene detector tube.

3.3.4 The accuracy and precision of studied xylene detector tube.

The accuracy and precision must be considered when the detector tube was used. The studied xylene detector tube was tested with the concentrations of xylene at 0.1(10 ppm) , 0.5 (50 ppm)and 1.0 TLV (100 ppm) following the protocol in section 3.3.1 and the experiments were performed for 10 replications. The length of stain and the xylene concentrations were recorded.

3.3.5 The sensitivity of xylene detector tube.

The studied xylene detector tube was tested with 5 and 10 pm of xylene concentrations following the preparation in section 3.2.4.1 and method in section 3.3.1. The experiment was repeated for 5 times. The length of stain was recorded and xylene concentrations were read from the caribration scale on the tube.

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3.3.6 Specificity of xylene detector tube.

The concentration of 50 ppm toluene was prepared by static system following the preparation in section 3.2.4.1 and tested method in section 3.3.1. The studied xylene detector tube was tested with 50 ppm of toluene concentration in air following the method in section 3.3.1 and the experiment was repeated for 5 times. The length of stain , color and color intensity were recorded. The mixture of 50 ppm xylene and 50 ppm toluene was prepared in the Tedlar bag following the preparation in section 3.2.4.2 and tested with the studied xylene detector tubes following the method in section 3.3.1. The length of stain , color and color intensity were recorded again. The concentration of xylene detector tube was read from the calibration scale on the tube.

3.3.7 Comparison of the efficiency of xylene detector tube when stored at room temperature with those stored at refrigerated temperature.

The tube stability was tested to study the appropriate condition for tube storage. The studied xylene detector tube was comparatively tested between the immediate preparation tube and the tube kept in refrigerated and room temperature for 1 and 2 weeks. After that, those tubes were tested with 10 and 100 ppm of xylene in air for five replications following the protocol in section 3.3.1. The length of stain was recorded and recovery of xylene was calculated.

3.3.8 Comparison of the studied and commercial xylene detector tube for sampling and analysis of xylene vapors in the field

The field test was conducted to study the efficiency of the tube to analyze the concentrations of xylene in working environment. The 20 pairs of the studied xylene detector tube and commercial xylene detector tube from Dreager company were tested for xylene concentration in the field at the same point source and at same time. For studied xylene detector tube , the length of stain was recorded. The xylene concentration was calculated from the xylene calibration scale on the tube. For Draeger tube, the xylene concentration was read from the scale provided.

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3.4 Statistical analysis

3.4.1 The mean and the standard deviation were calculated from:

$$\bar{X} = \frac{\sum_{i=1}^{n} Xi}{N}$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (Xi - \bar{X})^2}{N-1}}$$

$\bar{X}$ = sample mean

Xi = data

N = the number of value in the sample

SD = standard deviation

3.4.2 The precision was calculated in terms of the coefficient of variation (CV):

$$\%CV = \frac{SD}{\bar{X}} \times 100$$

3.4.3 The accuracy was calculated from NOISH method (Appendix B) and suggested guideline by AIHA (18):

Overall Accuracy = 2CV + Absolute Bias

Where

CV = coefficient of variation

Overall Accuracy = 25% for the concentration of 1,2 and 5 TLV

= 35% for the concentration of 0.5TLV

Bias = 100(predicted conc. – true conc.)/true conc.)

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3.4.4 The linear regression and the coefficient of determination

The linear regression and the coefficient of determination were calculated between the concentration of xylene (ppm) and peak area (mVS) from the standard curve of xylene for gas chromatography, and the concentration of xylene and the stain length to construct a calibration scale of the studied xylene detector tube.

3.4.5 The Paired t-test

The Paired t-test was used to calculate the difference of the concentrations of xylene in the field between the studied xylene detector tube and commercial xylene detector tube from Draeger.

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CHAPTER IV
RESULTS

4.1 Results of preparation of studied xylene detector tube.

4.1.1 The appropriate condition for coating of silica gel with potassium iodate solution.

The silica gel was coated with potassium iodate solution by mixing 0.75-mg silica gel with different volume of potassium iodate solution. The result is shown in Table 4-1. The chemical reaction of xylene with potassium iodate could be described below:

$$5[C_6H_4(CH_3)_2] + 12IO_3^- + 12H^+ \rightarrow 5C_8H_6O_4 + 6I_2 + 16H_2O$$

The potassium iodate solution reacted with xylene and produced the acid , iodine and water.

Table 4 -1 The proportion of reagent for coating of silica gel and result of color development.

Potassium iodate solution		Coating of silica gel		Result
KIO_3 (gm)	H_2SO_4 (ml)	Silica gel (gm)	Potassium iodate solution (ml)	
1	8	0.75	0.75	The silica gel was dissolved partly in the potassium iodate solution.
		0.75	0.40	
		0.75	0.25	The intensity of red-brown color developed was light.
		0.75	0.20	
1	9	0.75	0.25	The intensity of red-brown color developed was clear and strong.

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Table 4 -1 The proportion of reagent for coating of silica gel and result of color development. (continue)

Solution ratio		Silica gel + Solution ratio		Result
KIO_3 (gm)	H_2SO_4 (ml)	Silica gel (gm)	Solution (ml)	
1	9	0.75	0.20	The intensity of brown color developed was not so strong.
1	10	0.75	0.25	The intensity of brown color developed was not so strong.
		0.75	0.20	

The result showed that one gram of potassium iodate mixed with 9 ml sulfuric acid gave the best result and the proportion of reagent for coating of silica gel was 0.75-gm of silica gel for 0.25 ml potassium iodate solution. The color developed was clear and intense red-brown color, while other proportion of reagent for coating of silica gel resulted in less intense red-brown color . This set of reagent will be used for the rest of experiment. The coated silica gel was packed easily into the tube. After that, the packed tube was hermetically sealed by LPG flame within 45-60 second per tube. The 0.5-cm. of coated silica gel near the sealed end of the tube was changed from white to light pink color but the stain color disappeared after 24 hours.

4.1.2 Pressure drop testing

The 10 % of the studied xylene detector tube were checked for the pressure drop. The range of pressure drop was 6.4 – 7.0 inches of H_2O. The mean pressure drop was 6.73 inches of water and standard deviation was 0.22 inches of water. The packing density of each tube was calculated from 0.42 gm of silica gel per 0.49 cm^3 of tube volume. The packing density was 0.85 gm/cm^3 .

4.2 Preparation of xylene concentration by static system

4.2.1 The standard curve of xylene in air.

The relationship of xylene concentration and the peak area of xylene from gas chromatography was investigated. The xylene concentration at 10, 50, 100 and 150 ppm gave the peak area ranging from 3,9875.5 to90,280 mVS in Table 4-2.

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Table 4-2 xylene concentration and peak area

Xylene concentration (ppm)	Average peak area (mVS) (n = 3)
10	3,978.5
50	29,510
100	59,162.5
150	90,280

The plot of xylene concentrations from 10 to 150 ppm and peak area showed linear relationship (Fig. 4-1). It illustrated the relationship between the known xylene concentration and peak area by GC/FID. The coefficient of the standard curve of xylene in air was 0.9998.

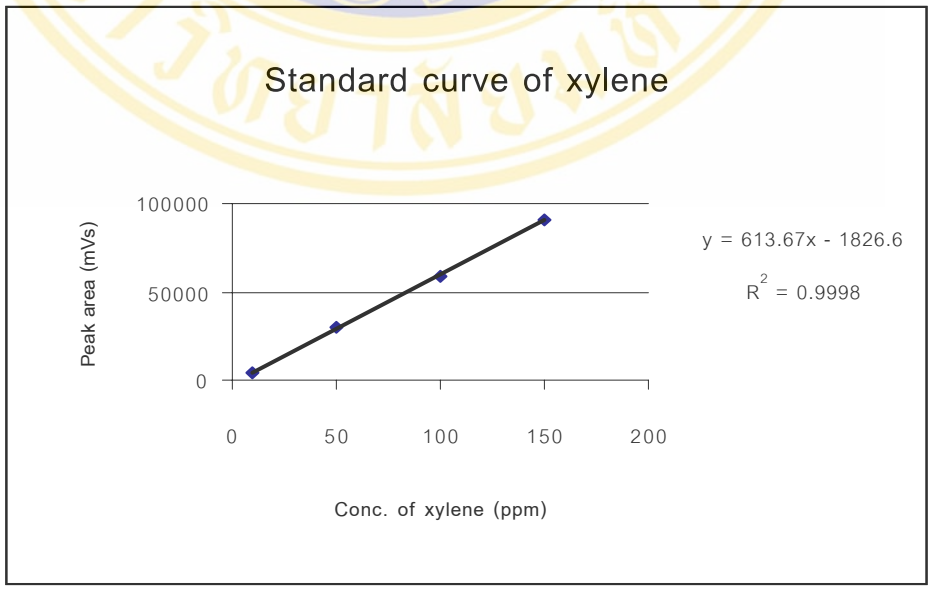

Figure 4-1 The standard curve of xylene concentrations.

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4.3 Calibration scales for studied xylene detector tube

This calibration scales were used for determination of xylene concentration from the tube. The concentration of xylene in air was generated by a static system and gas chromatograph was used to reassure the exact concentration of xylene in air at 9.48,15.88,52.28 and 99.58 ppm. The studied xylene detector tube was used to sampling these xylene concentrations in air. The results of the mean and standard deviation of stain length at different xylene concentrations are shown in Table 4-3

Table 4-3 The mean and the standard deviation of stain length corresponding to different xylene concentrations

True xylene concentration (GC.) (ppm)	Stain length (cm.)
9.48 ± 0.24	0.58 ± 0.04
15.88 ± 0.16	1.0 ± 0
52.28 ± 0.69	2.0 ± 0
99.58 ± 1.10	2.9 ± 0.1

Then, the standard curve between concentration of xylene (ppm) and stain length (cm) was plotted and shown in Figure 4-2

For Figure 4-2 , The graph shows the calibration scale curve. It illustrated the relationship between the concentration of xylene (ppm) and stain length (cm). The coefficients of determination of this curve was 0.9836 and The relationship of the length of stain and the concentration of xylene could be presented by the equation below.

$$Y = 5.6561e^{1.0249x}$$

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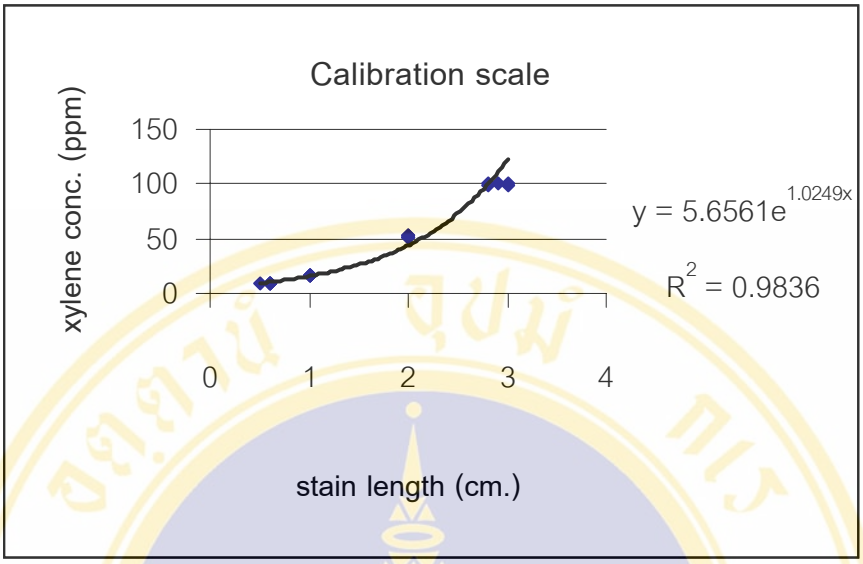

Figure 4-2 The calibration scale of studied xylene detector tube

4.4 The efficiency test of studied xylene detector tube

4.4.1 The accuracy and precision of studied xylene detector tube

The studied xylene detector tube was tested at 10, 50 and 100 ppm of xylene in air for 10 replications. Then, the concentrations were read from the calibration scale of the studied xylene detector tube. The results of the value and standard deviation at different xylene concentrations are shown in Table 4-4

Table 4-4 The mean and the standard deviation of stain length corresponding to different xylene concentrations

True conc. (ppm)	Stain length (cm)	Predicted conc. (ppm)	%CV	Average recovery,%
9.57 ± 0.186	0.61 ± 0.056	10.58 ± 0.61	5.76	110.55
48.94 ± 0.384	1.89 ± 0.056	39.29 ± 2.28	5.80	80.28
95.32 ± 0.407	2.79 ± 0.073	98.96 ± 7.52	7.59	103.81

The overall accuracy of detector tube was described by the American Industrial Hygiene Association (AIHA). The overall accuracy is equal to 2CV + Absolute Bias. The accuracy, precision and bias must be considered when interpreting results of detector tube measurements. (18)

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The studied xylene detector tube was tested with the concentration of xylene at 0.1, 0.5, and 1.0 TLV, respectively . The true concentration from GC, percent coefficient of variations of 10 replicated sample and overall accuracy from the atudied xylene detector tube were determined and shown in Table 4-5. The detailed results are presented in Appendix C

Table 4-5 Overall system accuracy test

Target Conc. (ppm)	True Conc. (ppm)	Predicted conc. (ppm)	%Error (%B_I)	%CV (%S_{rt})	S_{rt}	B_i	Overall accuracy (%)
0.1 TLV	9.57	10.58	1.01	5.76	0.0576	0.0101	12.53%
0.5 TLV	48.94	39.29	- 9.64	5.80	0.0580	-0.0964	21.24%
1.0 TLV	95.32	98.96	6.78	7.59	0.0759	0.0678	21.96%

The results showed that the coefficient of variations of studied xylene detector tube were 5.84%, 5.80%, 7.59 % at the xylene concentrations of 10.58, 39.29 and 98.96 ppm, respectively. The systematic error or bias (B_i) of xylene concentration were presented from the difference of xylene concentration from the studied xylene detector tube (found conc.) and the xylene concentration from GC (true concentration). The bias were ranged from –9.64 to 6.78 %. The percent coefficient of variations were in the range of 5.80 to 7.59 %. The percent overall system accuracy of the studied xylene detector tube were 12.69% , 21.24% and 21.96% at xylene concentration of 0.1, 0.5 and 1.0 TLV ppm respectively.

4.4.2 The detection limit of studied xylene detector tube.

The detection limit of studied xylene detector tube was carried out by testing xylene concentration at approximately 10 and 5 ppm for 5 replications. The results are shown in Table 4-6

For 5.82 ppm xylene, the indicating reagent changed from white to red brown colour. The length of stain was 0.5 – 0.7 cm which was similar to 9.48 ppm but the color intensity of 5.82 ppm level was less strong than that of the 9.48 ppm. The coefficient of variations for 5.82 ppm and 9.48 ppm were 5.54% and 8.66% , respectively. Therefore, the detection limit of studied xylene detector tube was 10 ppm xylene.

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Table 4-6 Detection limit of studied xylene detector tube.

Target Conc. (ppm)	True conc. (ppm) Mean ± SD	Stain length (cm) Mean ± SD	Predicted conc. (ppm) Mean ± SD	%CV	Recovery,%
0.1 TLV	9.48 ± 0.24	0.56 ± 0.05	10.05 ± 0.55	5.54	106.01
0.05 TLV	5.82 ± 0	0.58 ± 0.083	10.27 ± 0.89	8.66	176.46

4.4.3 Specificity of studied xylene detector tube

The studied xylene detector tube was tested with approximately 50 ppm toluene concentration in air for 5 replications. The color and the stain lengths were recorded. The detailed results are presented in Appendix C

The mean and standard deviation of this study are shown in Table 4-7

Table 4-7 Specification of studied xylene detector tube.

Tested chemicals	True conc. (ppm)	Stain length (cm)	Predicted xylene conc. (ppm)	%CV	Average Recovery.%
Toluene	50.03± 0.28	2.49 ± 0.08	0	0	0
Xylene	49.21± 0.36	1.88 ±0.083	38.95 ± 3.38	8.67	79.15
Mixture of toluene and xylene	50.05± 0 48.35± 0	3.42 ±0.21	0	0	0

For the result, the indication reagent which was coated by potasium iodate reacted with 50.03 ppm toluene vapor and changed from white to black brown colour. The length of stain was 2.3 - 2.5 cm. The concentration of xylene obtained was non detectable because the black brown color obtained was different from red brown color developed for xylene.

When, the studied xylene detector tube was tested with mixture of 50.05 ppm xylene and 48.35 ppm toluene. The indicating reagent was changed from white to strong brown colour and light brown colour. The length of stain for strong

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brown colour was presented at 2.3 – 2.4 cm long after that, the light brown color ranged from 3.1 – 3.6 cm. Again, the concentration of xylene was non detectable. The results clearly showed that toluene was an interference of studied xylene detector tube.

4.4.4 Comparison of studied xylene detector tube stored in room temperature and refrigerated temperature.

The studied xylene detector tubes were tested immediately after preparation, 1 and 2 weeks after stored at room temperature and at refrigerated temperature. The tubes were tested with 10 and 100 ppm xylene for 5 replications. The length of stain were recorded and xylene concentration were calculated for % recovery.

Table 4-8 Storage stability of studied xylene detector tube

Xylen conc. (ppm)	Duration of tube storage (Average recovery ,%) n = 5				
	0 week	1 week		2 weeks	
	Room + Ref. T.	Room T.	Ref. T.	Room T.	Ref. T.
10	106.21	118.83	108.31	185.74	107.13
100	102.40	112.11	107.44	86.09	102.38

Note Room T = room temperature, Ref. T = refrigerated temperature.

The percent recovery of xylene after storage the tube at room temperature for 2 weeks, the recovery of 10 and 100 ppm changed from 106.21 to 185.74 % and 102.40 to 86.09 % respectively. The percent recovery after storage at refrigerated temperature changed from 106.21 to 107.13 % at 10 ppm xylene and changed from 102.40 to 102.38 % at 100 ppm xylene.

The result clearly showed that the studied xylene detector tube was stable for at least 2 weeks at refrigerated temperature.

4.4.5 Comparison of xylene concentrations determined by the studied xylene detector tube and commercial xylene detector tube from Draeger company in the field.

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Air sampling was collected from the two areas of working environment of a petrochemical industry namely, Xylene unit and Parex unit. The xylene unit prepares the feed (mixed xylene) to the Parex unit. The Parex unit separates the paraxylene from the mixed xylene using adsorbent technique. The studied xylene detector tube and Draeger tube were used to measure xylene concentration at two process areas.. The sampling was performed for 20 pairs of that tubes.

At the xylene unit, the concentrations of xylene were detected by studied xylene detector tube ranged from 11.59 ppm to 110.49 ppm. Meanwhile, for the Draeger tube, the xylene concentrations ranged from 10 ppm to 100 ppm.

At the parex unit, the concentration of xylene detected by studied xylene detector tube ranged from 11.59 ppm to 43.92 ppm. Meanwhile, for the Draeger tube, the xylene concentration ranged from 10 ppm to 50 ppm.

The results are shown in Table 4-9

Table 4-9 Field test for studied xylene detector tube and Draeger tube

Xylene Unit

Test no.	Studied xylene detector tube (ppm)	Draeger tube (ppm)
1	43.92	40.00
2	110.49	100.00
3	39.64	50.00
4	11.59	10.00
5	39.64	40.00
6	43.92	50.00
7	73.33	70.00
8	99.72	100.00

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Test no.	Studied xylene detector tube (ppm)	Draeger tube (ppm)
9	12.84	10.00
10	12.84	10.00

Parex Unit

Test no.	Studied xylene detector tube (ppm)	Draeger tube (ppm)
1	11.59	10.00
2	12.84	10.00
3	43.92	50.00
4	12.84	10.00
5	43.92	50.00
6	15.76	20.00
7	10.46	10.00
8	43.92	50.00
9	43.92	50.00
10	12.84	10.00

Regarding the comparison of xylene concentration for the xylene detector tube and Draeger tube by Paired t-test , the result showed that xylene concentrations were not significant different between the studied xylene detector tube and Draeger tube (p-value > 0.25 at xylene unit and >0.10 at Parex unit).

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CHAPTER V
DISCUSSION

Xylene was an aromatic hydrocarbon which was widely used in industries. The xylene was released into the environment and dispersed into the atmosphere directly. The worker mainly exposed by inhalation. For xylene monitoring, there are verious types of sampling and analysis of xylene in air. NIOSH presented a method for measuring xylene concentrations in air. Xylene was adsorbed to coconut shell charcoal, extracted with carbon disulfide and determined using gas chromatograph with a flame-ionization detector (FID)(2). There were commercially available badges based on passive sampling of xylene in air . After extraction with carbon disulfide, the xylene can be detected by gas chromatography. Xylene can also be detected with the detector tube via hand pump. Earlier methods for determination of xylene concentration had been presented by H.M. Factory Inspectorate, Ministry of Labour , England (9)

The objective of this study is to construct a studied xylene detector tube. The method and chemical in this study were applied from the study and recommendation of H.M. Factory Inspectorate, Ministry of Labour , England. This method required using potassium iodate solution for sampling of xylene and read the color intensity. The recommended chemical solution was modified to coat onto the silica gel and packed them into the glass blank tube in this study. The color changed at tube could be visually read and estimated the concentration from the length of stain. The studied xylene tube was tested step by step following the standard guideline of NIOSH and AIHA. The efficiency of the studied xylene detector tube should not differ from the commercial tube.

5.1 The studied xylene detector tube preparation.

This study modified the method to measure the xylene concentration in the workplace by applying the method from H.M. Factory Inspectorate, Ministry of

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Labour, England. The principle of this method was that toluene and xylene reacted with an acid solution of potassium iodate to produce a yellow color. In this study, the acid solution of potassium iodate was applied to coat on the adsorbent. The adsorbent was filled into the blank glass tube. The contaminated air was collected into the tube by a hand pump. The concentration of xylene was read from the length of stain and the color intensity.

The adsorbent chosen for this study should not react with the acid solution of potassium iodate because the potassium iodate reacts with organic materials, such as strong reducing agent and aluminum(3). Therefore , silica gel was used as the adsorbent. Moreover, the silica gel was very small particles which was easy to fill through the glass tube and silica gel had high surface area approximately 750-800 m^2/g.(3,10)

Other factors should be considered, such as the size of the glass tube. The selected one was glass tube with 5-mm O.D. and 3-mm I.D. This size was easy to pack silica gel and could be sealed by an LPG flame faster than other size. This method could reduce the heat of exposure affecting the indicating reagent. In addition, The packing density of this silica gel tube was tested in terms of pressure drop. The result showed the consistence of packing of coated silica gel in the tube.

The method which was used to close the end of the tube was hermetically sealed by LPG flame. The heat affected the end of the indicating reagent which was close to the sealing point . The color of indicating reagent at that area changed from white to light pink and disappeared after 24 hours. Although, the affected area was approximate 0.5 cm. but this area was not the target indicating reagent area. So, this type of seal could be applied to use in this study.

The indicating reagent was prepared using one mg potassium iodate and 9 ml conc. sulfuric acid and coated on silica gel by mixing 0.75 gm silica gel with 0.25 ml potassium iodate solution. The chemical could react with the xylene concentration and gave the red brown color at the indicating layer. This color was quite clear. The relationship of the length of stain and the concentration of

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xylene could be presented by the equation below. Y was the length of stain and X was concentration of xylene.

$$Y = 5.6561e^{1.0249x}$$

5.2 The preparation of xylene concentration by static system.

5.2.1 The standard curve of xylene concentration.

This standard xylene concentration was prepared by injecting the amount of liquid xylene into the 5-liter Tedlar bag for producing known concentration of xylene. The mixing duration of xylene in the plastic bag was controlled and fixed about 3 minutes each bag. After that, the xylene gas was drawn from the bag and immediately injected to the gas chromatograph. The Peak area of xylene was integrated from three xylene isomers: para , meta and ortho-xylene .

The relationship between peak area and the xylene concentration was calculated as coefficients of determination of 0.9998. This value showed high reliability of this standard curve.

5.2.2 The calibration scales preparation for studied xylene detector tube.

The calibration scales of studied xylene detector tube was presented as $Y = 5.6561e^{1.0249x}$. This equation could be used to predict the xylene concentration deviated from 10, 20, 50 or 100 ppm. The calibration scale of studied xylene detector tube was shown as follow; 0.6 cm for 9.48 ppm, 1.0 cm for 15.88 ppm, 2.0 cm for 52.28 ppm and 2.9 cm for 99.58 ppm.

5.3 The efficiency of the studied xylene detector tube model.

5.3.1 Accuracy and precision of the studied xylene detector tube.

The percent overall system accuracy was carried out to determine the accuracy and precision of the studied xylene detector tube. AIHA suggested guideline as follows : Overall accuracy = 2CV + absolute bias. The overall accuracy

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should not exceeded 35 % for 0.5 TLV level and 25% for 1,2, and 3 X TLV level. In this study, overall accuracy for 0.5 and 1.0 TLV xylene was 21.24% and 21.96% ,respectively. In addition, the 0.1TLV xylene still presented the accuracy within 25 % . The result indicated that the accuracy and precision of the method were satisfactory.

5.3.2 Detection limit of studied xylene detector tube.

The detection limit of the studied xylene detector tube was studied by sampling 5.82 ppm and 9.48 ppm xylene concentration in air. All results were less than 10 percent coefficient of variation (%CV). The color stain of 9.48 ppm xylene was quite clear and more intense than that of 5.82 ppm. Therefore, detection limit of the studied xylene detector tube was 9.48 ppm.

5.3.3 Specificity of the studied xylene detector tube.

The studied xylene detector tube tested with toluene produced black brown color which was different from the red brown color of xylene. The length of stain of black brown color was longer than the length of stain of red brown color of xylene. The length of stain of toluene was longer than that of xylene at the same concentration. When testing for mixed toluene and xylene vapor at the same concentration in the same bag, the result showed the effect of toluene to interfere with xylene concentration . The length of stain and color development of mixed vapors was different from pure xylene vapor and could not read the xylene concentration at that tube.

Therefore, toluene was an interference for studied xylene detector tube because the color developed for toluene was different from the xylene and masked the color of xylene.

5.3.4 The comparison of tube storage at room temperature and refrigerated temperature.

Regarding the NIOSH recommendation for assessing the sample stability by storing them under defined condition and analyzed at specified time periods, the

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percent recovery of all tubes stored at refrigerated temperature were reduced from immediately analyzed tube less than 10% after 1 and 2 weeks . The percent recovery of all tubes stored at room temperature were changed from immediately analyzed tube more than 10% at 1 or 2 weeks. Therefore, the result of tube stored at refrigerated temperature met with storage stability criterion of NIOSH.

5.3.5 The comparison of the studied xylene detector tube and commercial xylene tube from Draeger company in the field.

This studied used potassium iodate solution for coating on silica gel to prepare indicating gel. The Draeger tube used formadehyle (HCHO) solution. When compared the measurement of xylene concentrations on both tubes, the result of xylene concentration was not significantly different between these two tubes.. So, the studied xylene detector tube was an alternative detector tube to use for determination of xylene in working area.

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CHAPTER VI
CONCLUSION AND RECOMMENDATION

6.1 Conclusion

This modified detector tube was studied using an indicating gel. The solution ratio was prepared by mixing one mg of potassium iodate and 9 ml of sulfuric acid. This solution was coated on the silica gel using 0.25 ml of solution and 0.75 mg silica gel. This indicating gel was packed into the blank gas tube. When tested with xylene , the reaction between the xylene and indicating gel produced the red brown color. The reliability of studied xylene detector tube was tested for accuracy, precision, detection limit , specificity and storage stability of the tube.

The percent overall system accuracy of studied xylene detector tube were 12.69%, 21.24% and 21.96% at 0.1 TLV,0.5TLV and 1.0 TLV ,respectively. Those result met NIOSH accuracy criterion. The detection limit of xylene detector tube was 9.48 ppm for xylene concentration in air. Toluene was an interference of xylene detector tube.

The studied xylene detector tube could be stored and used after storage in the refrigerated temperature for more than 2 weeks. When compared between studied and commercial xylene detector tube for analysis of xylene in the field , the results were not significantly different. Therefore, studied xylene detector tube could be used for monitoring xylene concentration in working area.

For the cost comparison , the studied xylene detector tube was cheaper than the xylene detector tube from Draeger tube (The detailed cost per tube is shown in Appendix D).

6.2 Recommendation for further study

1) An effect of heat from an LPG flame should be investigated to reassure that it does not affect the color development of indicating layer.

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2) The study of interferent chemical affecting the studied xylene detector tube.

3) The detection limit of this detector tube should be improved to detect efficiently low concentration of xylene.

4) This study may be used as guideline to construct detector tube for other chemicals.

5) The comparison of the measurement of xylene concentration between the studied xylene detector tube and the Draeger tube in the laboratory condition should be tested.

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15. พุทธพร สุวรรณจรัส : การสร้างอุปกรณ์ชนิดอ่านค่าได้ทันทีเพื่อตรวจสอบความเข้มข้นหาก๊าซ ซัลเฟอร์ไดออกไซด์ในบรรยากาศ (วิทยานิพนธ์) ปริญญาวิทยาศาสตร์มหาบัณฑิต สาขา สุขศาสตร์อุตสาหกรรม และความปลอดภัย กรุงเทพมหานคร บัณฑิตวิทยาลัย มหาวิทยาลัยมหิดล ; 1987

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APPENDIX A

THE CALCULATION OF XYLENE CONCENTRATINS METHOD

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Preparation of xylene concentration.

The calculated volume of xylene which was injected into the gas bag, using the formula below;

m = $4.1 \times 10^{-8} \times M \times V \times C$

m = mass of liquid required in grams (g) at 25°C, 1 atm

M = molecular weight in atomic mass units (amu ,xylene = 106.16)

V = volume of dilution vessel in Liter (4 Liters)

C = concentration in ppm

Volume = $\frac{m}{d}$

d = density of xylene (0.868 g/ml)

1. 10 ppm of xylene concentration was filled into 5 liters bag (4 liters of air dilution)

m = $4.1 \times 10^{-8} \times 106.16 \times 4$ liter $\times$ 10 ppm

m = 1.74×10^{-04} ml

Volume = $\frac{1.74 \times 10^{-04}}{0.868}$ ml

Volume = 2.0×10^{-04} ml

Volume = 0.2 µL

The volume of xylene injected into the 5-lit gas bag to produce the xylene conc. at 10 ppm was 0.2 µL

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2. 50 ppm of xylene concentration was filled into 5-lit bag (4 liters of air dilution)

m = $4.1 \times 10^{-8} \times 106.16 \times 4$ liter $\times 50$ ppm

m = 8.705×10^{-04} ml

Volume = $\frac{8.705 \times 10^{-04}}{0.868}$ ml

Volume = 1.0×10^{-03} ml

Volume = 1.0 µL

The volume of xylene injected into the gas bag to produce the xylene conc. at 50 ppm was 1.0 µL

3. 100 ppm of xylene concentration was filled into 5-lit bag (4 liters of air dilution)

m = $4.1 \times 10^{-8} \times 106.16 \times 4$ liter $\times 10$ ppm

m = 1.74×10^{-03} ml

Volume = $\frac{1.74 \times 10^{-03}}{0.868}$ ml

Volume = 2.0×10^{-03} ml

Volume = 2.0 µL

The volume of xylene injected into the gas bag to produce the xylene conc. at 100 ppm was 2.0 µL.

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4. 150 ppm of xylene concentration was filled into 5-lit bag (4 liters of air dilution)

m = $4.1 \times 10^{-8} \times 106.16 \times 4$ liter $\times$ 150 ppm

m = 2.61×10^{-03} ml

Volume = $\frac{2.61 \times 10^{-03}}{0.868}$ ml

Volume = 3.0×10^{-03} ml

Volume = 3.0 µL

The volume of xylene injected into the gas bag to produce the xylene conc. at 150 ppm was 3.0 µL

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APPENDIX B

THE CALCULATION OF ACCURACY AND PRECISION METHOD

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Example of the calculation of accuracy and precision of the method

Table B-1 Mean and standard deviation of precision test

100 ppm				
Rep No.	GC conc. (ppm)	Stain length (cm)	Tube Conc. (ppm)	% Error
1	95.52	2.8	99.72	6.24
2	95.52	2.7	90.01	-5.51
3	95.52	2.8	99.72	4.2
4	95.41	2.8	99.72	4.31
5	95.41	2.8	99.72	4.31
Mean	95.476	2.78	99.77	4.91
SD	0.060	0.044	4.34	0.91
%CV			4.35	

Table B-2 Overall system accuracy test

Target Conc. (ppm)	True Conc. (ppm)	Predicted conc. From tube (ppm)	%Error ($\%B_I$)	%CV (S_{rt})	S_{rt}	B_i	Accuracy
1.0 TLV	95.476	99.77	4.91	4.35	0.0435	0.0491	13.61%

Overall accuracy = 2CV + absolute bias

= 2 x 0.0435 + 0.0491

= 0.1347

= 13.61%

Note : The test requirements are 25% accuracy in Laboratory studied for concentration of 1,2 an 5 TLV and 35% accuracy for concentration of 0.5 TLV

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APPENDIX C

THE ANALYTICAL RESULTS OF STUDIED XYLENE DETECTOR TUBE

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C-1 The analytical results of calibration scale for studied xylene detector tube

Xylene conc.	Xylene concentration (ppm)	Stain length (cm)
10 ppm	9.22	0.6
	9.22	0.5
	9.66	0.6
	9.66	0.6
	9.66	0.6
20 ppm	16.07	1.0
	16.07	1.0
	15.76	1.0
	15.76	1.0
	15.76	1.0
50 ppm	51.78	2.0
	51.78	2.0
	51.78	2.0
	53.04	2.0
	53.04	2.0
100 ppm	100.39	2.9
	100.39	2,8
	100.39	3.0
	98.37	3.0
	98.37	2.8

Note TLV (Threshold limit value) of xylene is 100 ppm

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Table C-2 The accuracy and precision of studied xylene detector tube

Rep. No.	Concentration level (TLV)								
	10ppm(0.1 TLV)			50 ppm(0.5 TLV)			100 ppm(1.0 TLV)		
	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)
1	9.41	0.6	10.46	49.48	1.90	39.64	95.52	2.8	99.72
2	9.41	0.5	09.44	49.48	2.00	43.92	95.52	2.7	90.01
3	9.81	0.6	10.46	49.48	1.80	35.78	95.52	2.8	99.72
4	9.81	0.6	10.46	48.81	1.90	39.64	95.41	2.8	99.72
5	9.81	0.6	10.46	48.81	1.80	35.78	95.41	2.8	99.72
6	9.58	0.6	10.46	48.81	1.90	39.64	95.41	2.9	110.49
7	9.58	0.6	10.46	48.81	1.90	39.64	95.84	2.9	110.49
8	9.58	0.6	10.46	48.58	1.90	39.64	95.84	2.7	90.01
9	9.36	0.7	11.59	48.58	1.90	39.64	94.67	2.8	99.72
10	9.36	0.7	11.59	48.58	1.90	39.64	94.67	2.7	90.01
Mean	9.57	0.61	10.58	48.94	1.89	39.29	95.32	2.79	98.96
SD	0.186	0.056	0.61	0.384	0.056	2.28	0.407	0.073	7.52
%CV	1.94	9.18	5.76	0.78	2.96	5.80	0.42	2.61	7.59

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Table C-3 Detection limit of studied xylene detector tube.

Rep. No.	Concentration of xylene (ppm)					
	10			5		
	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)
1	9.22	0.6	10.46	5.28	0.5	9.44
2	9.22	0.5	9.44	5.28	0.6	10.46
3	9.66	0.6	10.46	5.28	0.7	11.59
4	9.66	0.6	10.46	5.28	0.5	9.44
5	9.66	0.5	9.44	5.28	0.6	10.46
Mean	9.48	0.56	10.05	5.28	0.58	10.27
SD	0.24	0.05	0.55	0	0.083	0.89
%CV	2.53	8.92	5.54	0	14.31	8.66

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Table C-4 Specification of studied xylene detector tube .

Concentration level (TLV)						
50 ppm of toluene		50 ppm of xylene			50 ppm of toluene 50 ppm of xylene	
GC conc. (ppm)	Stain length(cm)	GC conc. (ppm)	Stain length (cm)	Predicted conc. (ppm)	GC conc. (ppm)	Stain length(cm)
49.88	2.4	49.48	1.90	39.64	50.05 48.35	3.6
49.88	2.5	49.48	2.00	43.92	50.05 48.35	3.5
49.88	2.3	49.48	1.80	35.78	50.05 48.35	3.6
50.26	2.5	48.81	1.90	39.64	50.05 48.35	3.1
50.26	2.5	48.81	1.80	35.78	50.05 48.35	3.3

Table C-5 The shelf life test at 0 week

Rep. No.	0 week					
	01. TLV			1.0 TLV		
	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)
1	9.41	0.6	10.46	95.52	2.8	99.72
2	9.41	0.5	9.44	95.52	2.7	90.01
3	9.81	0.6	10.46	95.52	2.8	99.72
4	9.81	0.6	10.46	95.41	2.8	99.72
5	9.81	0.6	10.46	95.41	2.8	99.72
Mean	9.65	0.58	10.25	95.47	2.78	97.77
SD	0.219	0.044	0.45	0.060	0.044	4.34
%CV			4.39			4.43

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Table C-6 The shelf life test for 0.1 TLV

Rep. No.	1 week					
	Room temperature			Refrigerator temperature		
	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)
1	9.77	0.6	10.46	9.86	0.6	10.46
2	9.77	0.7	11.59	9.86	0.7	11.59
3	9.77	0.7	11.59	9.86	0.6	10.46
4	9.77	0.7	11.59	9.86	0.6	10.46
5	9.77	0.8	12.84	9.86	0.6	10.46
Mean	9.77	0.7	11.61	9.86	0.62	10.68
SD	0	0.07	0.84	0	0.044	0.50
%CV			7.23			4.68

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Table C-7 The shelf life test for 1.0 TLV

Rep. No.	1 week					
	Room temperature			Refrigerator temperature		
	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)
1	95.10	2.9	110.49	95.01	2.8	99.72
2	95.10	2.8	99.72	95.01	2.7	90.01
3	95.10	3.0	122.41	95.01	2.9	110.49
4	95.10	2.7	90.01	95.01	2.9	110.49
5	95.10	2.9	110.49	95.01	2.8	99.72
Mean	95.10	2.86	106.62	95.01	2.82	102.08
SD	0	0.114	12.27		0.083	8.63
%CV			11.50			8.45

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Table C-8. The shelf life test for 0.1 TLV

Rep. No.	2 week					
	Room temperature			Refrigerator temperature		
	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)
1	10.66	1.1	17.46	10.65	0.6	10.46
2	10.66	1.2	19.34	10.65	0.6	10.46
3	10.66	1.2	19.34	10.65	0.8	12.84
4	10.66	1.3	21.43	10.65	0.6	10.46
5	10.66	1.3	21.43	10.65	0.8	12.84
Mean	10.66	1.22	19.8	10.65	0.68	11.41
SD	0	0.083	1.67	0	0.109	1.30
%CV			8.43			11.39

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Table C-9 The shelf life test for 1.0 TLV

Rep. No.	2 week					
	Room temperature			Refrigerator temperature		
	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)	GC conc. (ppm)	Stain length (cm)	Predicted Conc. (ppm)
1	107.02	2.8	99.72	108.37	3.0	122.41
2	107.02	2.8	99.72	108.37	3.0	122.41
3	107.02	2.7	90.01	108.37	2.9	110.49
4	107.02	2.6	81.24	108.37	2.8	99.72
5	107.02	2.7	90.01	108.37	2.8	99.72
Mean	107.02	2.72	92.14	108.37	2.9	110.95
SD	0	0.083	7.79	0	0.1	11.34
%CV			8.45			10.22

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APPENDIX D

The detail of cost per tube

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D-1 The detail of cost per tube

No.	Materials	Cost (Baht)
1.	Silica gel 60, 35-70 mesh ASTM (MERCK, Germany) 1Kg	2,247
2.	LPG burner	1,950
3.	Glass tube	430
4.	Others	208
		Total 4,835/300 tube (Approximately 16 Baht per tube)

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APPENDIX E

Figures

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Figure E- 1 The Field test for the studied xylene detector tube and Draeger tube

Figure E- 2 The studied xylene detector tube was sealed by LPG flame

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