EFFECTS OF TEMPERATURE IN REACTIVE DISTILLATION OF VAPOR FROM CATALYTIC PENTAERYTHRITOL TETRADODECANOATE (PETD) REACTOR

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JUDUL: **EFFECTS OF TEMPERATURE IN REACTIVE DISTILLATION OF VAPOR FROM CATALYTIC PENTAERYTHRITOL TETRADODECANOATE (PETD) REACTOR**

SESI PENGAJIAN: **2006/2007**

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Date : 20 November 2006

DEDICATION

To my beloved father, mother,
brothers and sisters……

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Bismillahirrahmanirrahim

First of all praise and gratitude to Allah S.W.T for giving me strength went through many difficulties and successful finished my project industrial. After all the hardwork and difficulty, I personally would like to acknowledge the many wonderful people that have guided me and support me in completing this thesis.

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ABSTRACT

Pentaerythritol-tetradodecanoate (PETD) was used as an essential varnish additive in magnetic wire production, It recently been conducted in many foreign countries, but in Malaysia it’s still not yet localized by any local company. The objective of this research is to study effects of temperature in catalytic PETD reactor. Method was applied by using reactive distillation as the main unit operation involving the mixture of lauric acid and pentaerythritol substrates occurred at the range 180°C to 230°C. The product from the experiment is analyzed with Gas Chromatography (GC). The result from analysis shows that the optimum temperature is at 200°C. From observation, 200°C is an optimum temperature, where it is suitable to obtain maximum reaction and higher production of PETD. From GC analysis also shows that high production of PETD when catalyst was used if compare to experiment without catalyst. Outcome from this research can be considered as pioneer process in localizing the technology, scale up and commercialization of PETD production especially in Malaysia.

ABSTRAK

Pentaerythritol Tetradodecanoate (PETD) adalah ester baru yang digunakan dalam pembuatan wayar magnetic. Di negara-negara luar, penghasilan PETD telah dijalankan namun di Malaysia, teknologi penghasilan PETD masih belum diterokai oleh mana-mana syarikat di Malaysia. Objektif kajian ini adalah untuk mengkaji kesan suhu terhadap penghasilan PETD dengan penggunaan mangkin. Eksperimen dijalankan menggunakan penyulingan reaktif sebagai unit operasi melibatkan campuran asid laurik dan pentaerythritol dengan enam suhu yang berbeza dari 180°C hingga 230°C. Hasil produk dari eksperimen ini kemudiannya dianalisis dengan menggunakan Kromatografi gas. Keputusan analisis menunjukkan suhu optimum adalah 200°C. Daripada pemerhatian yang dilakukan, suhu 200°C adalah suhu yang sesuai untuk mendapatkan kadar penghasilan PETD yang tingggi. Selain itu dengan penggunaan pemangkin ia juga dapat menambahkan kadar penghasilan PETD Hasil dari kajian ini boleh dianggap sebagai langkah awal dalam mengkomersialkan penghasilan PETD ini.

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NOMENCLATURE

PETD = Pentaerythritol Tetradodecanoate
RD = Reactive Distillation
GC = Gas Chromatography
w/w % = weight per weight percent

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CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

Ester is essentially applied in a variety of areas such as solvents, plasticizers, pharmaceuticals and intermediates. Pentaerythritol-Tetradodecanoate (PETD) also known as Pentaerythritol-Tetralaurate is one of the ester was used as varnish additive in the magnetic wire coating [4]. PETD is produced from esterification process by using the mixture pentaerythritol and lauric acid as reactant.

Esterification is the general name for a chemical reaction in which two chemicals (typically an alcohol and an acid) form an ester as the reaction product. Different approaches have obviously been employed to prepare esters. Esters are obtained by refluxing the parent carboxylic acid with the appropriate alcohol with an acid catalyst. The equilibrium can be driven to completion by using an excess of either the alcohol or the carboxylic acid, or by removing the water as it forms.

A common method of operating equilibrium-limited reactions is to use one excessive reactant in order to increase the conversion of the limiting reactant. In reactive distillation, the continual separation of products from reactants forces the reaction to surpass the equilibrium conditions.[1] Reactive distillation is a process where separation of the components of a reaction system is accompanied by a chemical reaction in a column. The combination of a reversible reaction and distillation is a widely used technique to increase the conversion of the reactants to levels above equilibrium conversion. The reactive distillation is becoming more and more popular in chemical industries.

Catalysts provide an alternative pathway of lower activation energy, for a reaction to proceed whilst remaining chemically unchanged them. A catalyst is a substance that accelerates the rate (speed) of a chemical reaction. Chemical catalysts participate in reactions but are neither chemical reactants nor chemical products.

1.2 PROBLEM STATEMENT

Malaysia is gearing towards achieving industrialized and developed nation. PETD has been used as a varnish additive in production of magnetic wire for a few years ago. The product of PETD as a varnish additive is recently produced and supplied directly from Japan by KANEKA. The esterification of PETD has been produced in many foreign countries, but the process technology is still not yet localized by local company to support manufacturing needs especially in reducing the product price. Local companies has still not known the technology to produce the fine chemical at an optimum output in terms of purity, production rate, energy consumption and process minimization.

This study is considering the production of PETD using esterification. Esterification is a reversible reaction, so the suitable instrument was reactive distillation because the application of Le Chateliers' principle. In reactive distillation, the reaction occurs and at the same time separates the product. Meanwhile, temperature is affected in the term of reaction in reactive distillation.

From the problem arise; it is essential to study in the process of PETD production. The hypothesis of the study is the temperature of reaction and the production of PETD is increased proportionally until the optimum temperature that occurs at 200 °C.

1.3 OBJECTIVE

The aim of this study is to study the effect of temperature on the performance of catalytic reaction in PETD production.

1.4 SCOPE RESEARCH

To achieve the objective of this research, there are three scopes that have been identified:

a) This study was on the effects of difference temperature to the reaction. Six sample were tested at temperature was range between 180^{o}C to 230^{o}C. at 1 atm, 16 hours and same rig. The range is used because in the past literature review, the optimize temperature is 190^{o}C. From experiment, the conversion in PETD production is the main idea to make a conclusion by observing the relation of temperature and PETD production.

b) Identify the temperature for optimum reaction. The optimum is the point which the amount of PETD is the most favorable. The optimum temperature is decided when the sample is analyze. The sample is analyzed with Gas Chromatography (GC). The optimum temperature is the temperature that enhanced the conversion to its maximum. The analysis of the sample can give the idea to predict the outcome of result.

c) Next scope is effect of catalyst in PETD reaction. Homogeneous catalyst is use in the experiment. The effect of catalyst is compared with the control test to see the difference. Catalysts reduce the activation of the energy and accelerate speed of reaction. So, the time of production is reduced to minimum as possible.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Magnet wire is an insulated electrical conductor, usually copper or aluminum that when wound into a coil and energized creates a useful electrical field. Electricity is essentially useless without magnet wire. 90% of all electrical energy requires modification through the use of magnet to be of use. It plays an important role in the machine function. Enameled wires are used to make electrical coils for the conversion (interchange between electrical & magnetic energy) of electrical energy to electromagnetic energy and this is why enameled wire is also called magnet wire.

At the moment Kaneka Chemical has patent the PETD as one of its product. The product of PETD as a varnish additive is recently produced and supplied directly from Japan.

2.2 Pentaerythritol Tetradodecanoate

PETD is an ester that was produced by esterification reaction in many foreign countries; the production of PETD used pentaerythritol and lauric acid as the raw material. It involves the mixture of lauric acid and pentaerythritol substrates occurred at the range 180^{o}C to 230^{o}C.

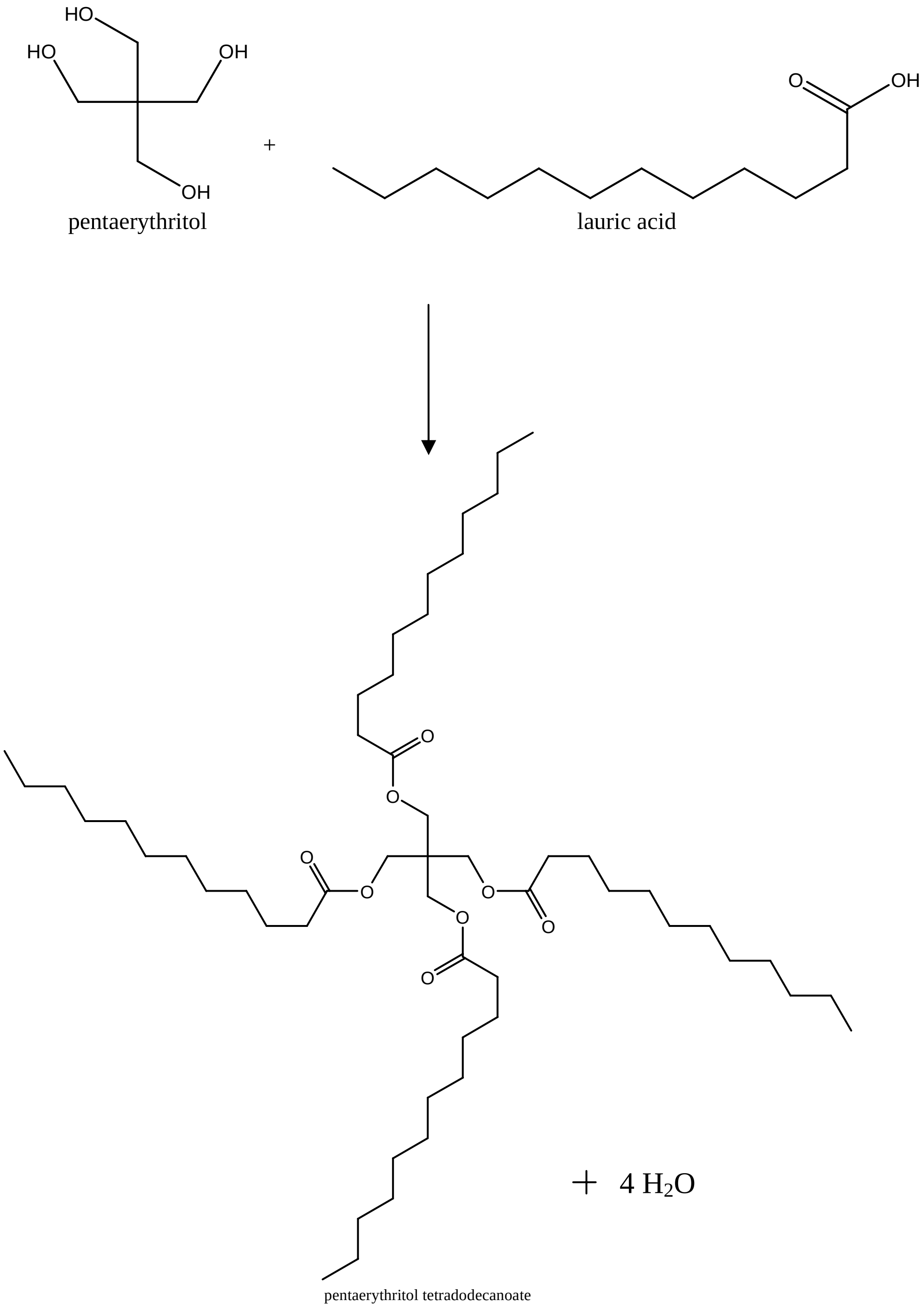


Figure 2.1: The general reaction of PETD with Pentaerythritol and Lauric acid as raw material.

2.3 Esterification

Esterification is the general name for a chemical reaction in which two chemicals (typically an alcohol and an acid) form an ester as the reaction product. These are commonly used as fragrance or flavour agents.

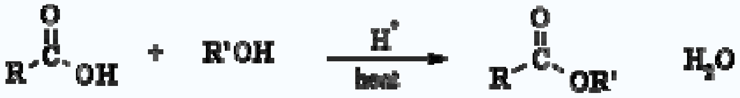


Figure 2.2: General equations of esterification

2.3.1 Acid-Catalyzed Esterifications

Acid-catalyst esterifications are called Fischer esterifications. They proceed very slowly in the absence of strong acids, but they reach equilibrium within a matter of a few hours when an acid and an alcohol are refluxed with a small amount of concentrated sulfuric acid or hydrogen chloride. Since the equilibrium controls the amount of the ester formed, the use of an excess of either the carboxylic acid or the alcohol increases the yield based on the limiting reagent. Just which components we choose to use in excess will depend on its availability and cost. The yield of an esterification reaction can also be increased by removing water from the reaction mixture as it is formed.

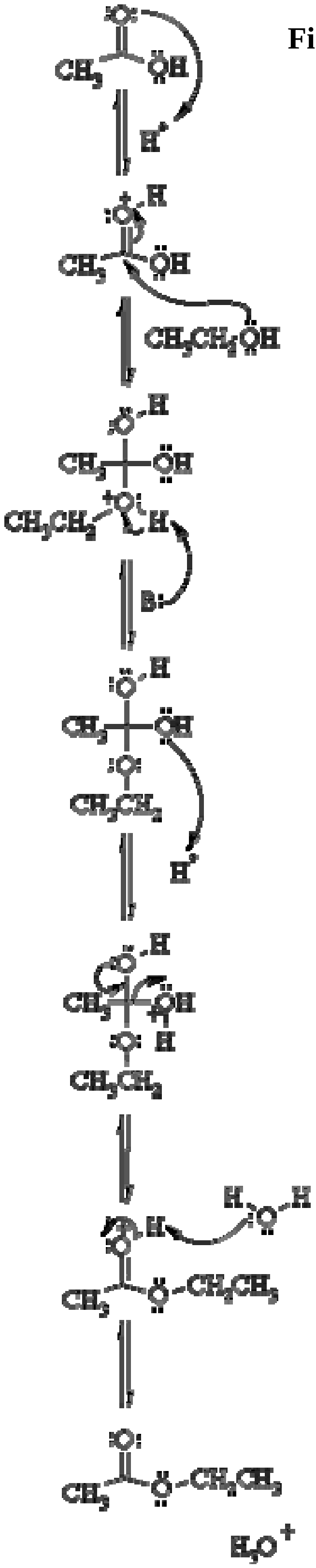


Figure 2.3: Reaction type: Nucleophilic Acyl Substitution

Step 1:
An acid/base reaction. Protonation of the carbonyl makes it more electrophilic.

Step 2:
The alcohol O functions as the nucleophile attacking the electrophilic C in the C=O, with the electrons moving towards the oxonium ion, creating the tetrahedral intermediate.

Step 3:
An acid/base reaction deprotonate the alcoholic oxygen.

Step 4:
An acid/base reaction. Need to make an -OH leave, it doesn't matter which one, so convert it into a good leaving group by protonation.

Step 5:
Use the electrons of adjacent oxygen to help "push out" the leaving group, a neutral water molecule.

Step 6:
An acid/base reaction. Deprotonation of the oxonium ion reveals the carbonyl in the ester product.

2.4 Catalyst

Estimates are that 60% of all commercially produced chemical products involve catalysts at some stage in the process of their manufacture. [3] A **catalyst** is a substance that accelerates the rate (speed) of a chemical reaction. Chemical catalysts participate in reactions but are neither chemical reactants nor chemical products. More generally, one may sometimes call anything which accelerates a reaction without itself being consumed or transformed a catalyst.

Catalysts provide an alternative pathway of lower activation energy, for a reaction to proceed whilst remaining chemically unchanged them. This can be observed on a Boltzmann distribution and energy profile diagram. This means that catalysts reduce the amount of energy needed to start a chemical reaction. Molecules that would not have had the energy to react or that have such low energies that they probably would have taken a long time to react are able to react in the presence of a catalyst. Thus, more molecules that need to gain less energy to react will go through the chemical reaction.

Catalysts cannot make energetically unfavorable reactions possible; they have no effect on the chemical equilibrium of a reaction because the rate of both the forward and the reverse reaction are equally affected.

A catalyst is not destroyed or changed during a reaction, so it can be used again. This is the ideal situation. In reality, catalyst will also involve in reaction and hard to be recovered. The catalyst will disturb the reaction by form the complex medium. The traditional homogenous catalysed reactions are less favoured owing to the attendant problems of separation and reuse. [5] There for many other catalysts that used for esterifications such as acidic ion exchange resin.

Catalysts can be either heterogeneous or homogeneous. Heterogeneous catalysts are present in different phases from the reactants (e.g. a solid catalyst in a liquid reaction mixture), whereas homogeneous catalysts are in the same phase (e.g. a dissolved catalyst in a liquid reaction mixture). A simple model for heterogeneous catalysis involves the catalyst providing a surface on which the reactants (or substrates) temporarily become adsorbed. Bonds in the substrate become weakened sufficiently for new bonds to be created. The bonds between the products and the catalyst are weaker, so the products are released. Homogeneous catalysts generally react with one or more reactants to form a chemical intermediate that subsequently reacts to form the final reaction product, in the process regenerating the catalyst. This experiment use homogeneous type of catalyst.

In research of esterifcation of Acetic Acid with Ethanol Catalysed by an Acidic Ion-Exchange Resin by S Ismail Kirbaslar, Z. Baris Baykal and Umur Dramur [1], they observed that percentage of equilibrium between acetic acid and ethanol is increasing due to increasing the amount catalyst. The graph below shown increasing of percentage of equilibrium by temperature profiles.

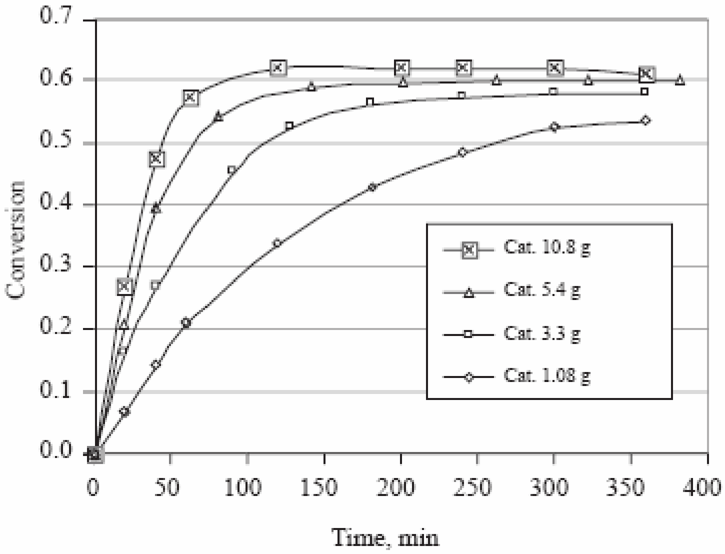


Figure 2.4: Effect of changing catalyst concentrations on the conversion of example (molar ratio of acetic acid and ethanol (M= 1/1) and temperature 353 K)

2.5 Reactive Distillation

Reactive distillation comprises the processes of catalytic reaction and multistage distillation carried out simultaneously in a single vessel.[9] A reactive distillation column replaces the reactor and a series of distillation columns, thereby reducing the number of process vessels and materials transfer and control equipment required.

Benefits of reactive distillation include:

- Increased speed and improved efficiency
- Lower costs – reduced equipment use, energy use and handling
- Less waste and fewer byproducts
- Improved product quality – chemicals are exposed to heat only once, reducing opportunity for degradation

Reactive distillation is a complex technology that is not right for every project. Generally, normal operating conditions for distillation and reaction, such as temperature and pressure conditions, should overlap, and other specific requirements must be met.

Reactive distillation can be used with a wide variety of chemistries, including the following:

- Acetylation
- Aldol condensation
- Alkylation
- Amination
- Dehydration
- Esterification
- Etherification
- Hydrolysis
- Isomerization
- Oligomerization
- Transesterification

2.5.1 Concept of Reactive Distillation

Reactive distillation is used with reversible, liquid phase reactions. Suppose a reversible reaction had the following chemical equation:

$$A + B \rightleftharpoons C + D$$

For many reversible reactions the equilibrium point lies far to the left and little product is formed:

$$A + B \rightleftharpoons C + D$$

However, if one or more of the products are removed more of the product will be formed because of Le Chatlier's Principle:

$$A + B \rightleftharpoons C + D\uparrow$$

Removing one or more of the products is one of the principles behind reactive distillation. The reaction mixture is heated and the product(s) are boiled off. However, caution must be taken that the reactants won't boil off before the products.

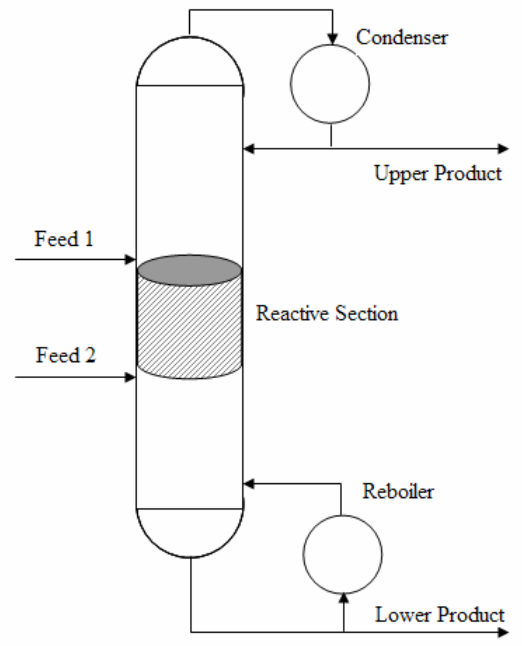


Figure 2.5: Typical Reactive Distillation Column

2.6 Le Chatelier's Principle

In chemistry, Le Chatelier's principle can be used to predict the effect of a change in conditions on a chemical equilibrium.

Chemical equilibrium is the state in which a chemical reaction proceeds at the same rate as its reverse reaction; the rates of the forward and reverse reactions are equal, and the concentration of the reactants and products stop having net change. When this condition is met, there is no change in the proportion; i.e., concentrations of the various compounds involved, and it appears that the reaction ceases to progress. However the forward and the reverse reactions continue to occur at the same rates. This process is known as dynamic equilibrium [2].

A statement of Le Chatelier's Principle

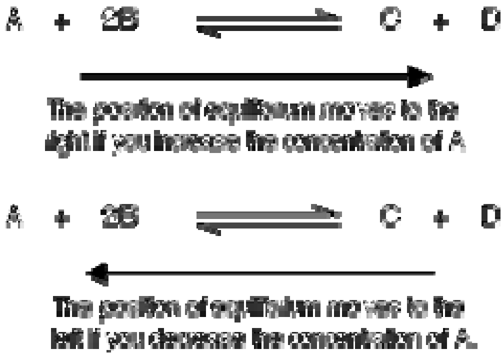
- If a dynamic equilibrium is disturbed by changing the conditions, the position of equilibrium moves to counteract the change.

Le Chatelier's Principle can be summarized, if a chemical system at equilibrium experiences a change in concentration, temperature or total pressure the equilibrium will shift in order to minimize that change.

2.7.1 Using Le Chatelier's Principle with a change of concentration

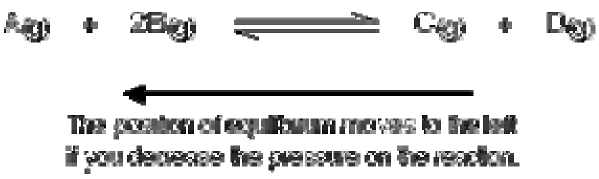
Suppose you have an equilibrium established between four substances A, B, C and D.

$$A + 2B \rightleftharpoons C + D$$



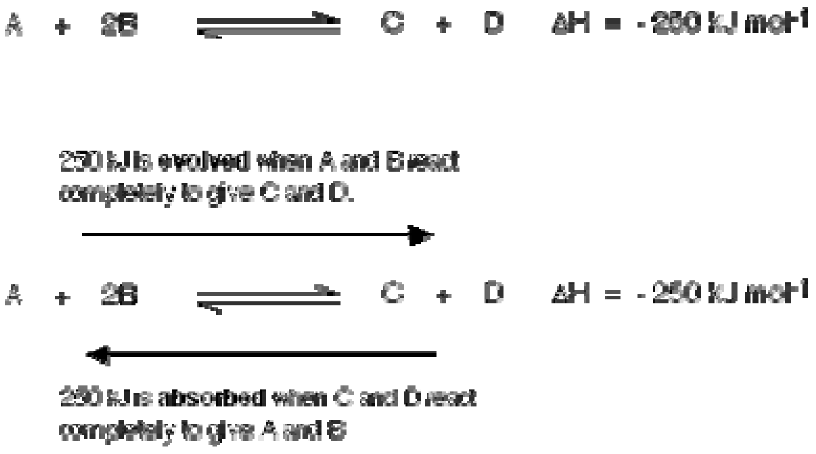
2.7.2 Using Le Chatelier's Principle with a change of pressure

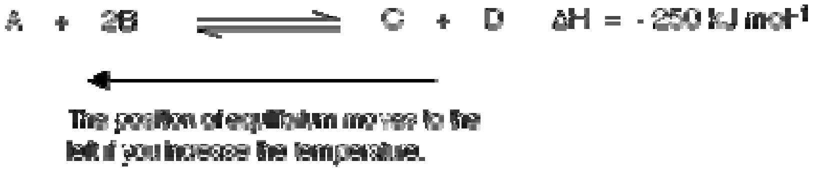
This only applies to reactions involving gases:

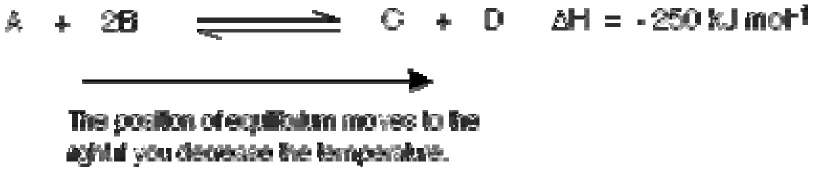


2.7.3 Using Le Chatelier's Principle with a change of temperature

For this, you need to know whether heat is given out or absorbed during the reaction. Assume that our forward reaction is exothermic (heat is evolved):







In this study, Le Chatelier’s Principle with change of concentration and temperature was applied in each experiment to make sure it’s work. First, by increasing the temperature, it will drive the reaction to forward. Second, by remove the water, the reaction will always drive forward to archive the dynamic equilibrium.

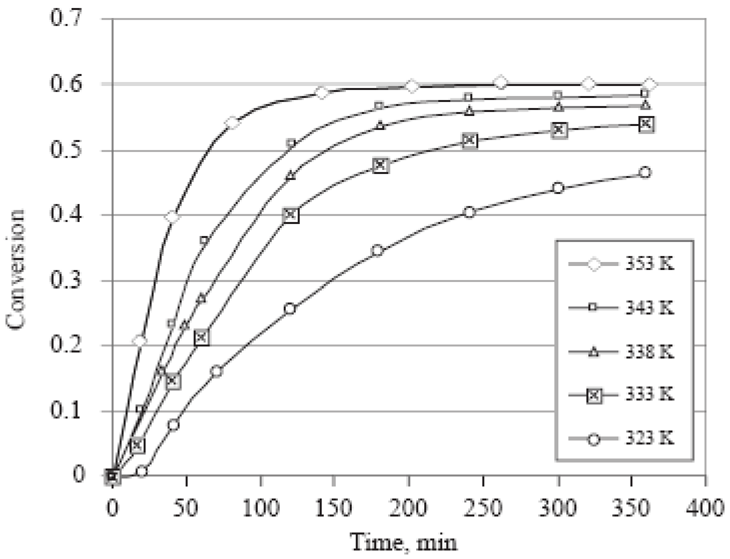


Figure 2.6: Effect of temperature on the conversion of acetic acid; Example (M=1/1; catalyst loading 5.4 g).

In research of esterifcation of Acetic Acid with Ethanol Catalysed by an Acidic Ion-Exchange Resin by S Ismail Kirbaslar, Z. Baris Baykal and Umur Dramur [1], they observed that percentage of equilibrium between acetic acid and ethanol is increasing due to temperature rise. The graph above shown increasing of percentage of equilibrium by temperature profiles.

CHAPTER 3

METHODOLOGY

3.1 Introduction

The objective of this research is to study the effect of temperature on the performance of catalytic reaction in PETD production. Method of the experiment is decided from all the information that has been collected to achieve the objective of this research. The raw material is pentaerythritol and lauric acid and the equipment set-up design from the concept of industrial reactive distillation. The experiment runs six times for each temperature (180^{o}C – 230^{o}C). Sulphuric acid is used as catalyst in this experiment.

The parameters that have been identified is the flow regime, volume of ionic liquid, concentration of reactant, temperature, speed of agitation, catalyst particle size, catalyst loading, types of catalyst, mole ratio of the reactants was studied to optimize the reaction conditions.

The variable manipulated is temperature and decided from range 190^{o}C $^{-}$ 230^{o}C to measure the conversion of PETD. Other variable is fixed at their own criteria.

The blank test based on other experiment without catalyst.

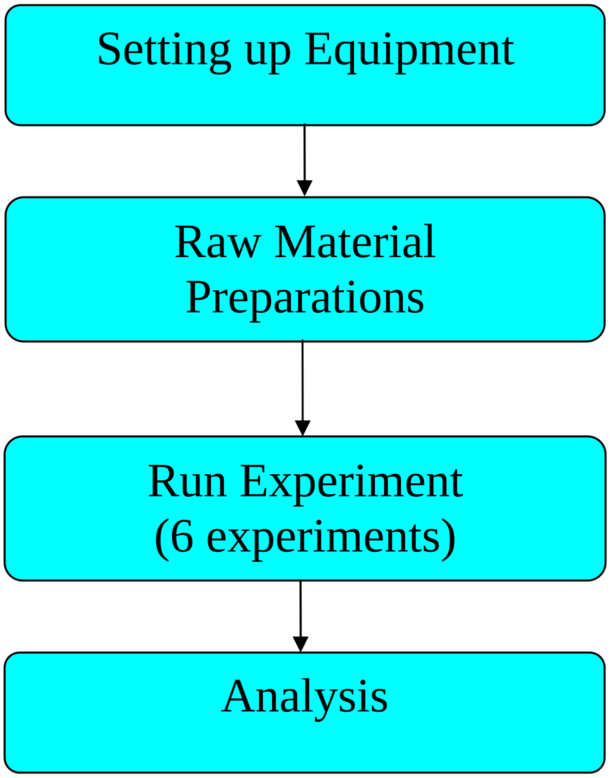


Figure 3.1: Methodology Flow Chart

3.2 Equipment Set Up

Glassware apparatus was used in this experiment. All the equipment is set-up in the lab scale as figure 3.1 has been selected based on the need to run the experiment.

1. Reactor Flask 500 ml
2. Allihn condenser
3. Fractioning column vigreux
4. Flat bottom flask short neck
5. Motor Stirrer
6. Digital thermocouple
7. Thermometer
8. Distillation chemistry kit
9. Heating Mantle

10. Retort Stand

Figure 3.2: Equipment Setup

3.3 Raw material Preparations

Based on equipment set-up, the total chemical use is only 200 ml for each experiment. The calculation is made, 29.05 gram of pentaerythritol and 170.95 gram of lauric acid is prepared to fulfill the requirement for 200 ml. Sulphuric acids (0.1 M) is prepared.

Table 3.1: Chemical Properties of raw material

Properties	Lauric Acid	Pentaerythritol
Melting Point	44^{o}C	255-259^{o}C
Boiling Point	298.9^{o}C at 760 mm Hg	276^{o}C at 30 mm Hg
Flash Point	100^{o}C	240^{o}C
MW	200.31	136.15
SG	0.86-0.87	1.396

3.4 Experiment

The reactants and a few drops of sulphuric acid that act as catalyst were inserting into the vessel. The flask was wrapped with aluminium foil to prevent heat losses. Heating mantle was switched on to start the experiment. The stirrer is set at 600 rpm. When reach at 180^oC maintain the temperature for a 16 hours. Switched off the heating mantle and the sample are put into sample bottle. The sample was ready to analyze it on GC. Repeat the experiment five times at different temperatures (190^oC, 200^oC, 210^oC, 220^oC and 230^oC).

3.5 Analysis

Six sample is run through Gac Chromatography Analysis Equipment.

3.5.1 Gas Chromatography (GC)

GC is instrumental tools for separating and analyzing organic compounds that can be vaporized without decomposition. It is use to testing the purity of a substance and separating the components of a mixture. The relative amounts of the components in a mixture may also be determined.

3.5.2 Preparation of Standard Calibration

PETD sample from Kaneka is not suitable for standard calibration because of the purity, so Lauric acid was chosen as an alternative to do the standard calibration. Three concentration 5g w/w of lauric acid has prepared with chloroform as solvent.

Table 3.2: Concentration (w/w %)

Percentage (1%)	**Lauric Acid (g)**	**Chloroform (g)**
1	0.05	4.85
2	0.10	4.90
3	0.15	4.95

3.5.3 Sample Preparation

The samples from experiment are prepared by dissolved in chloroform with concentration of lauric acid is 1 (w/w %) in 5 gram. 0.05 g of samples be dissolved in chloroform before put it in GC analysis bottle for analysis procedure.

Table 3.3: GC method

Sample	PETD
Solvent	Chloroform
Compound	Lauric Acid
Instrument	Agilent 6890N GC
Carrier	Helium 42 cm/sec, 24 psi (120°C), 1.8 ml/min constant flow
Column	HP-INNOWax (Cross-Linked PEG), 30m x 0.25mm x 0.25 µm) (Part No. 19091N-133)
Injection	Split (40:1), 1 µl, inlet 250°C
Oven	120°C for min, 100-250°C at 10°C/min, 250°C for 12 min
Detector	FID 280°C

CHAPTER 4

RESULT AND DISCUSSION

4.1 Result

The experiment was conducted and six samples had successful obtained from the experiment. The sample was set at different temperature, (180°C - 230°C). The entire sample showed the characteristic of PETD from physical observation. The sample was golden-honey like color in liquid and the shape of crystal was spherical when it solidified.

The sample was run in Gas Chromatography (GC) analysis equipment to get a proper result. The analysis was carried out by the detecting lauric acid. This purpose is to figure how many lauric acids has been consumed to create PETD. If the quantity of lauric acid is high, its mean the PETD that has been produce is small in quantity and vice versa.

Figure 4.1 to figure 4.6 exhibits the result from GC analysis. Lauric acid has retention time range between 17.009 and 17.014. Lauric acid is show by the circle at each figure.

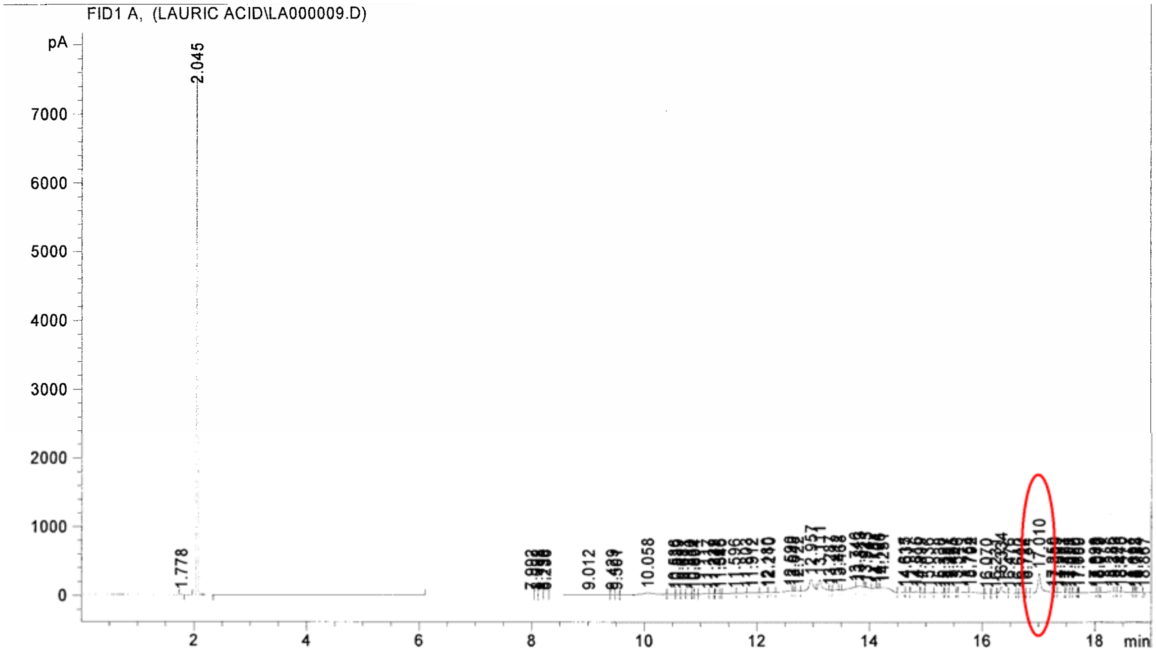


Figure 4.1: The GC analysis from sample at temperature 180°C

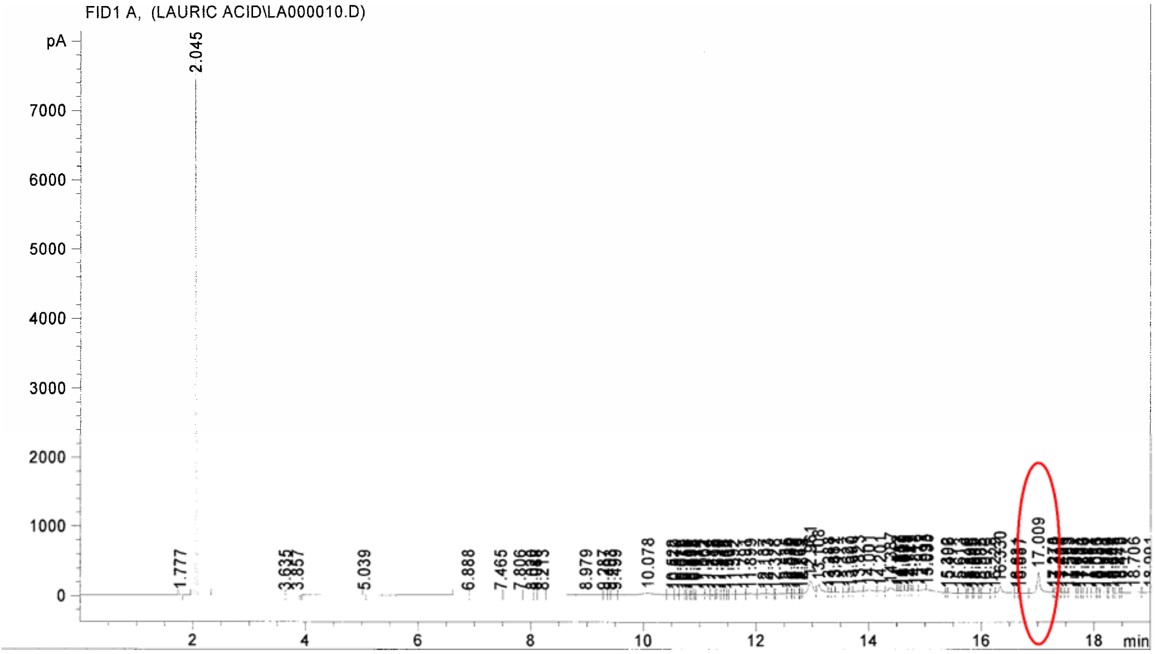


Figure 4.2: The GC analysis from sample at temperature 190°C

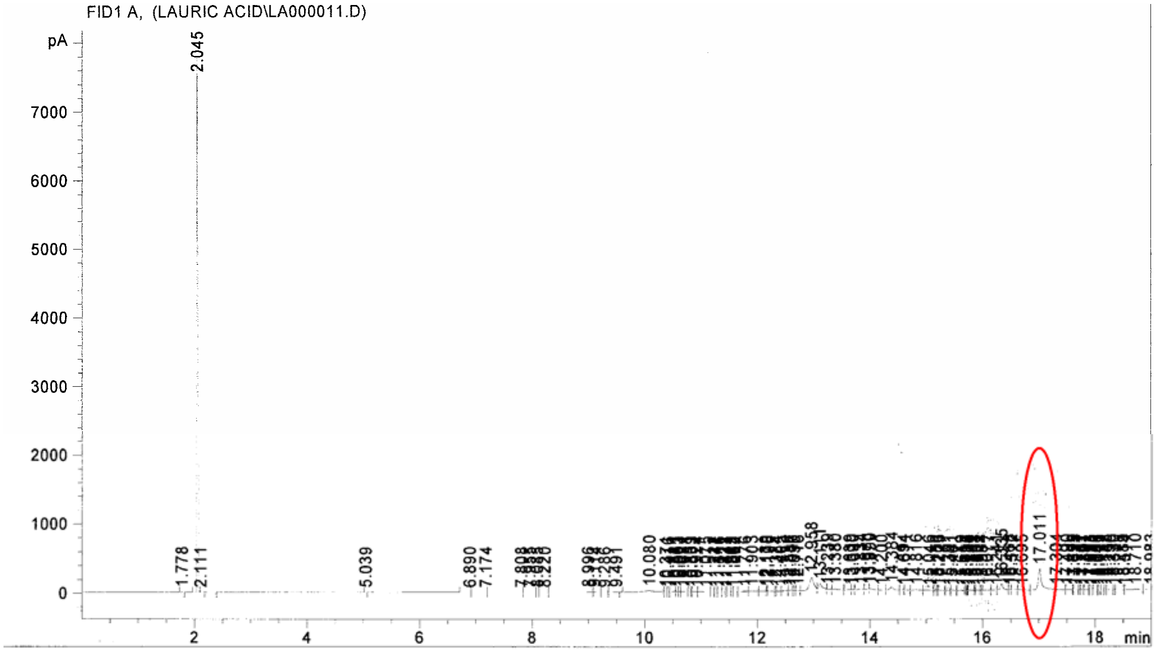


Figure 4.3: The GC analysis from sample at temperature 200°C

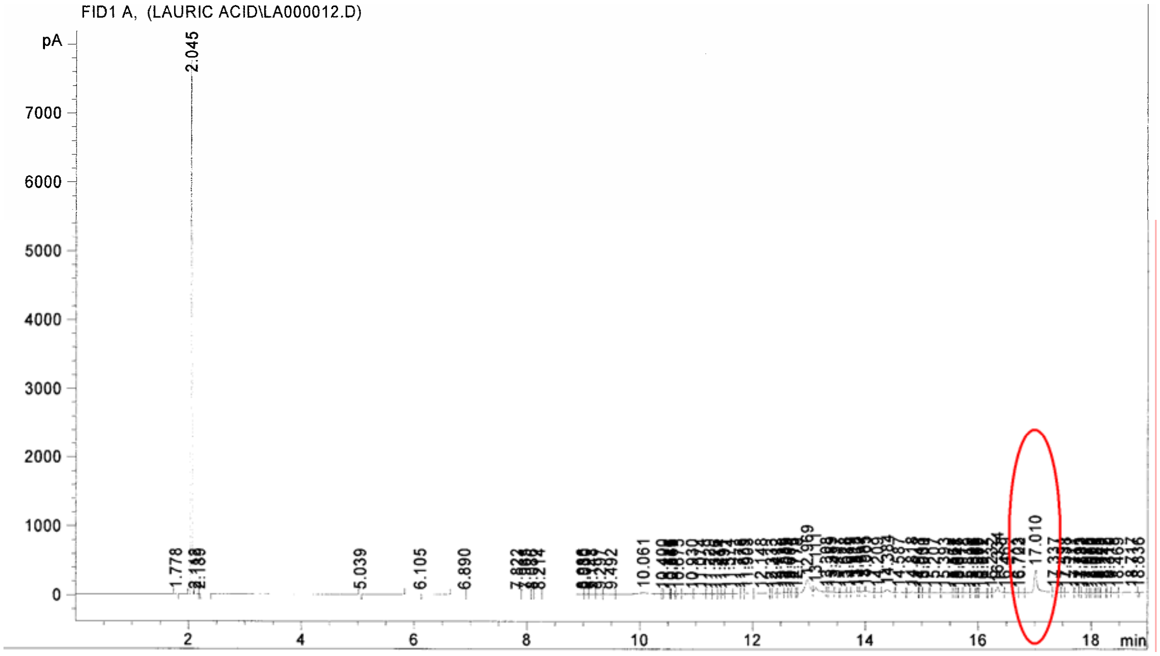


Figure 4.4: The GC analysis from sample at temperature 210°C

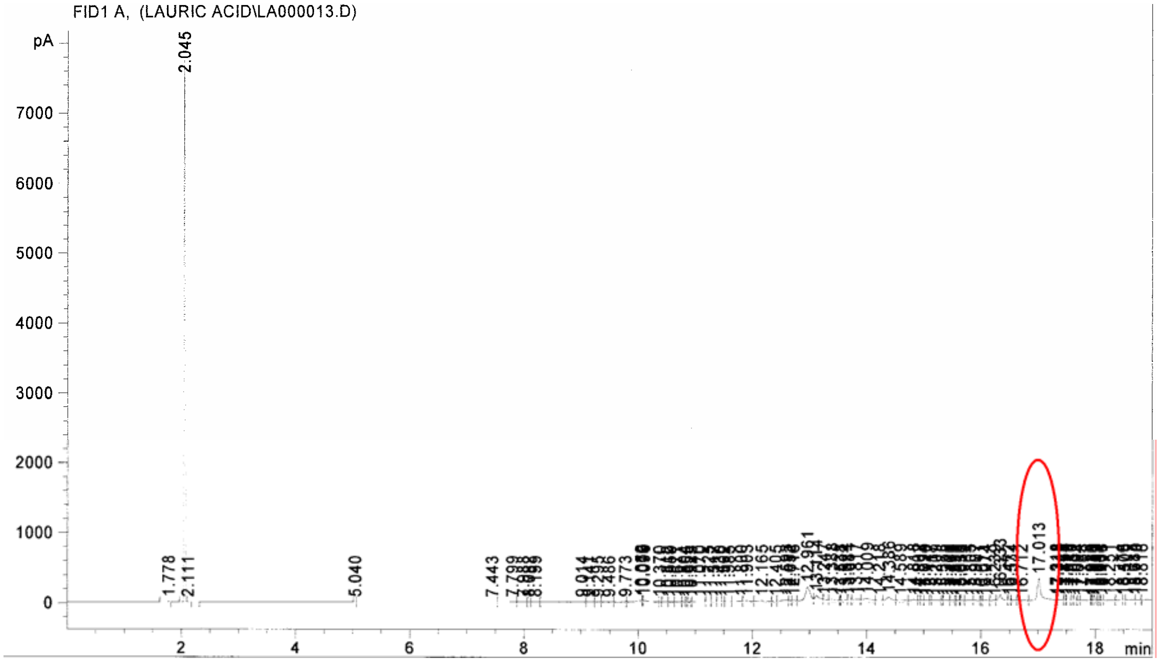


Figure 4.5: The GC analysis from sample at temperature 220°C

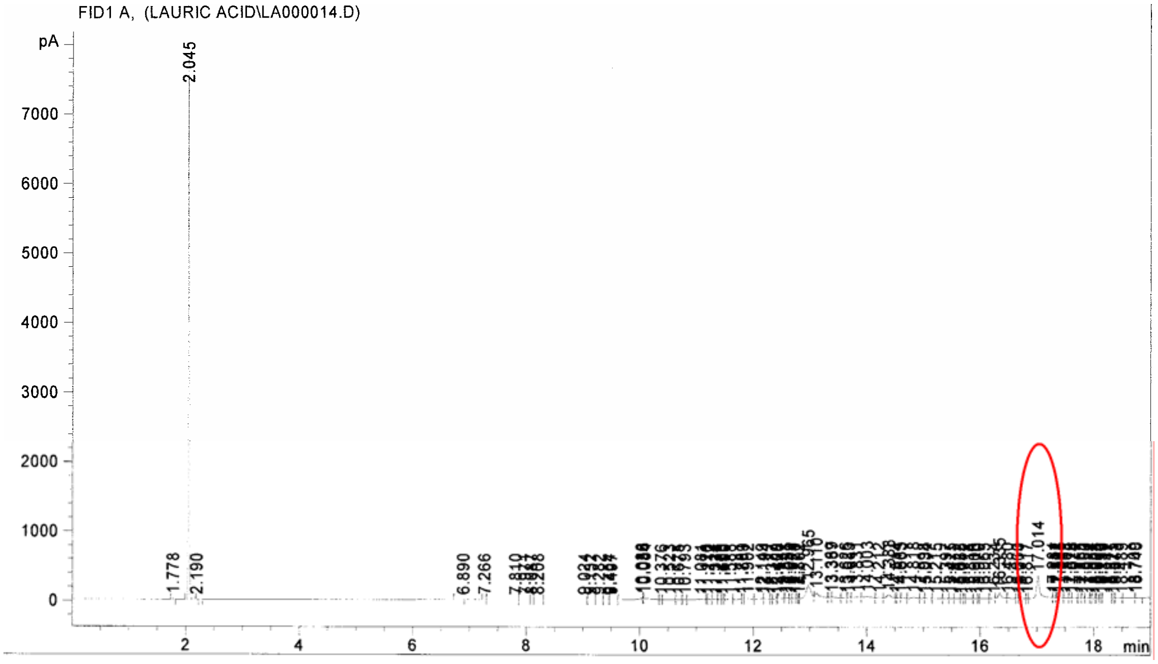


Figure 4.6: The GC analysis from sample at temperature 230°C

4.1.1 Standard calibration

All reading gathered from standard will be plotted in graph qualitative result of lauric acid (Figure 4.7) to obtain the value of concentration for each sample. Example on obtaining the concentration readings from standard calibration curve was shown in Figure 4.8.

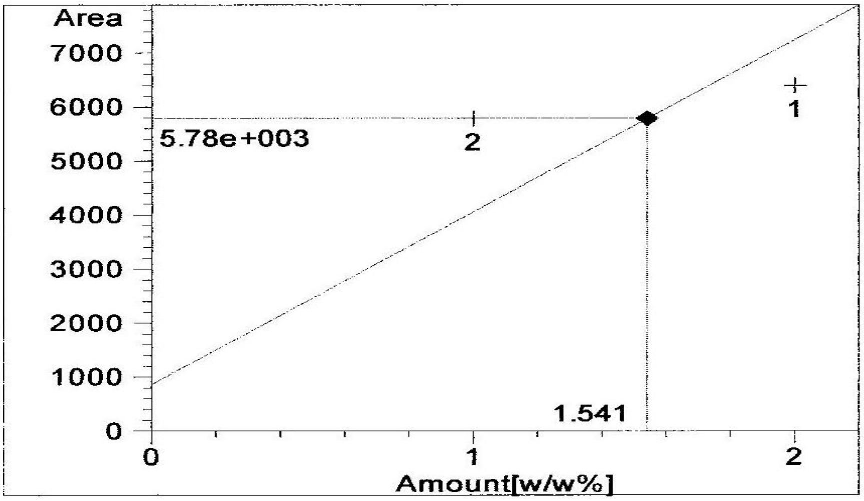


Figure 4.7: Standard calibration curve of qualitative result lauric acid based on GC analysis

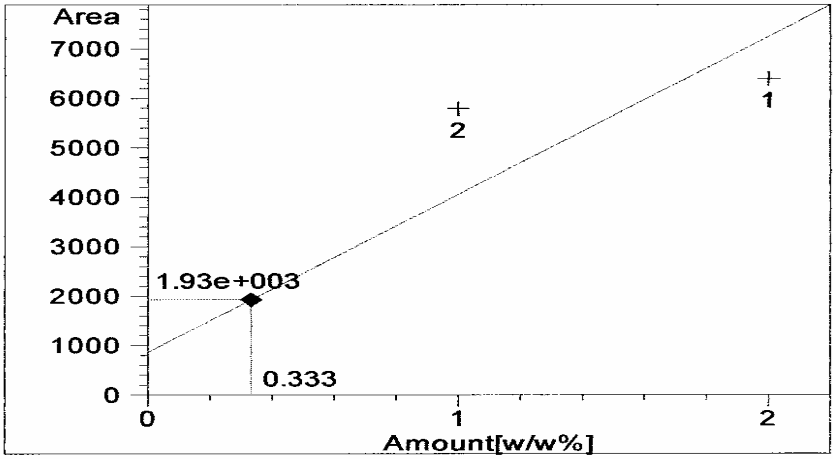


Figure 4.8: Method to obtain the concentration from calibration curve
Example: Lauric acid/chloroform 1/99 (w/w %) at temperature 180°C

4.2 Discussion

Refer to objective of this project is to study the effect of temperature on the performance of catalytic reaction in PETD production. There are three scopes to discuss in this chapter.

i) Study on the effects of difference temperature to the reaction.
ii) Determine the temperature for optimum reaction.
iii) Effect of catalyst in PETD reaction.

4.2.1 Effect Of Temperature

Refer to Le Chatelier’s Principle by increasing the temperature; it will drive the reaction to forward. The experiment has shown a significant result based on this principle. The PETD production is reversible reaction. By increased the temperature the reaction will always drive the reaction to forward and the production will always continue without any disturbance of reversible reaction. When the rection is drive forward, many lauric will be used on the reaction. Table 4.1 shows the results of samples after obtain it from Lauric acid based standard calibration curve.

Table 4.1: Percentage of lauric acid in every sample.

Temperature (°C)	Amount of lauric acid (w/w %)	Retention Time (min)
180	0.333	17.010
190	0.321	17.009
200	0.202	17.011
210	0.390	17.010
220	0.396	17.013
230	0.328	17.014

4.2.2 The Optimum Temperature

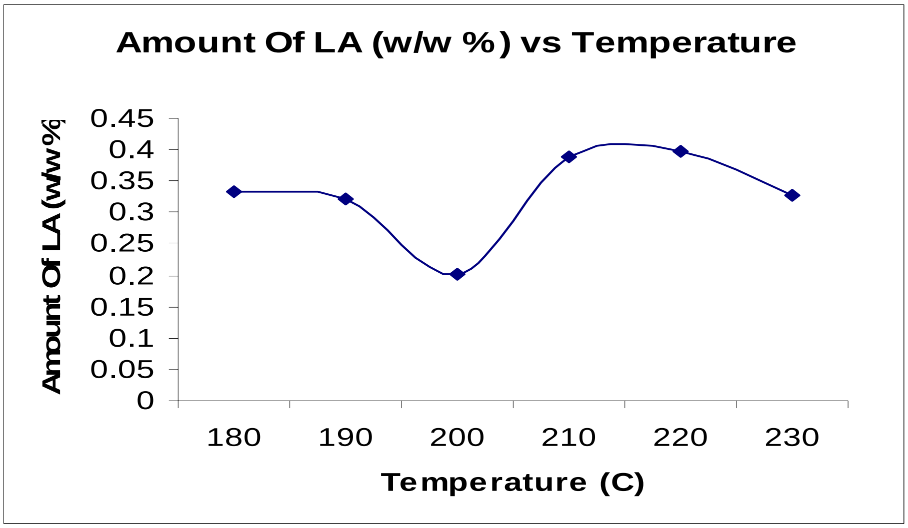


Figure 4.9: The amount of Lauric Acid vs Temperature

From the figure 4.9 we can determine the temperature for optimum reaction to produce PETD. From the first part if the figure the temperature is increased and the the amount Lauric Acid is decreased. It mean that the Lauric acid has been used to produce is high. At the second part of the graph the temperature is higher then the previous but the amount of Lauric acid used is lower than the previous temperature. It shows that after the optimum temperature the reaction still occurs but the rate is lower than before. Heat for create PETD is exceed than it supposed to be.

The optimum temperature is at 200°C. At this temperature the amount of Lauric acid is the lowest if compare that is 0.202. It signify that high amount of Lauric acid is consumed to produce PETD.

4.2.3 Effect of Catalyst

Temperature (°C)	Amount of LA (w/w %) with catalyst	Amount of LA (w/w %) without catalyst
180	0.333	0.543
190	0.321	0.459
200	0.202	0.377
210	0.390	0.761
220	0.396	0.443
230	0.328	0.969

Table 4.2: Comparison between the reaction with catalyst and without catalyst

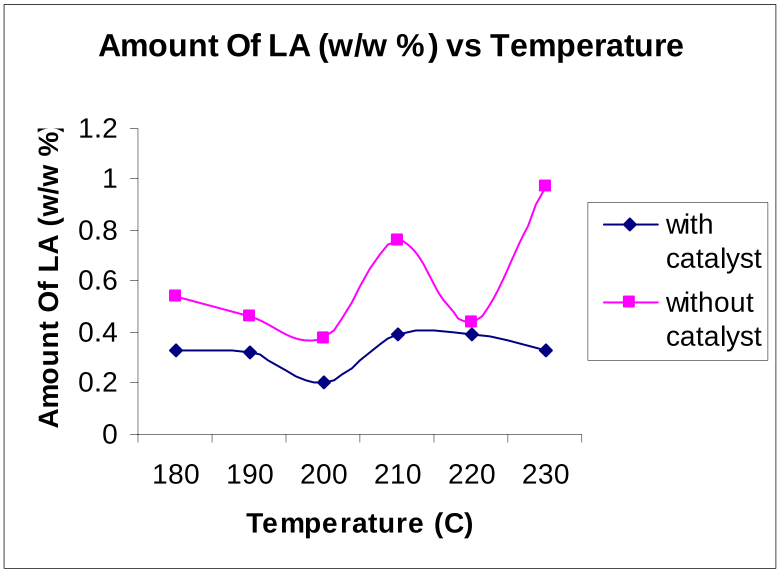


Figure 4.10: Comparison between the reaction with catalyst and without catalyst

The result in Figure 4.10 clearly shows the difference between reaction with catalyst and without catalyst. The amount of Lauric acid in reaction with catalyst is lower than reaction without catalyst. It means reaction with catalyst is more effective in producing PETD. The rate of reaction is greater because catalyst is function to lower the activation energy to produce PETD.

Another benefit using the catalyst is it will shorten the time of reaction. In the experiment, the reaction without catalyst is run 24 hours. While compare with the reaction with catalyst, it only run for 16 hours, but the amount of PETD produce is more high. This can be proving from figure 4.10.

4.3 Error and Prevention Step

The error occurred during the preparation of standard calibration. Considering the weight per weight percent, error occurred while weighting the chloroform to dilute lauric acid. The value of weight was not consistent because of the chloroform is easy to evaporate. Although, there is an error but the result in this research still available and suitable to use.

This error can be overcome from happens again by change the method. Instead of weighting it, it is suitable by convert it to volume unit. The value for chloroform that need is more accurate and prevent it from evaporate while preparing calibration standard.

CHAPTER 5

CONCLUSION

5.1 Conclusion

After the experiment was conducted and followed by analysis, all the scope of research has been fulfill to achieve the objective. From the research that has been done it can be conclude:

i. Effects of difference temperature will bring different outcome to the reaction. Temperature is manipulated variable range between 180^{o}C to 230^{o}C. From experiment, observation towards the conversion in PETD production concluded that the higher temperature will drive the reaction to forward. The relation of temperature and PETD production, is higher temperature will produce higher PETD.

ii. Normally, when the temperature increased, the product of reaction is also increased. But, when it past the optimum temperature the reaction will slowdown and rate of reaction is lower than before. The optimum temperature is the temperature that enhanced the conversion to its maximum. In this research the temperature for optimum reaction is 200°C.

iii. Catalysts reduce the activation of the energy and accelerate speed of reaction. So, the time of production is reduced to minimum as possible. This experiment clearly shows the reaction with catalyst is better than without catalyst.

PETD is highly potential specialty chemical that can enhance the magnetic characteristic of solenoid and coil (in motor and thermaionic gun). The process technology is needed to localize in Malaysia to support manufacturing needs especially in reducing the product price. This research can be considered as pioneer process in localizing the technology, scale up and commercialization of PETD production especially in Malaysia.

5.2 Recommendation

This project has a potential to develop more in the future in term of economic and industrial aspect. It is recommended to continue this project to get better view of purity, production rate, energy consumption and process minimization and others factors in PETD production.

For the better result in the future experiment, it is suggested to use equipment with accurate temperature controller for heating. The used of pure standard of PETD was also recommended to improve the result in analysis process. It is better to determine the result with actual PETD compound than used the lauric acid as an alternative.

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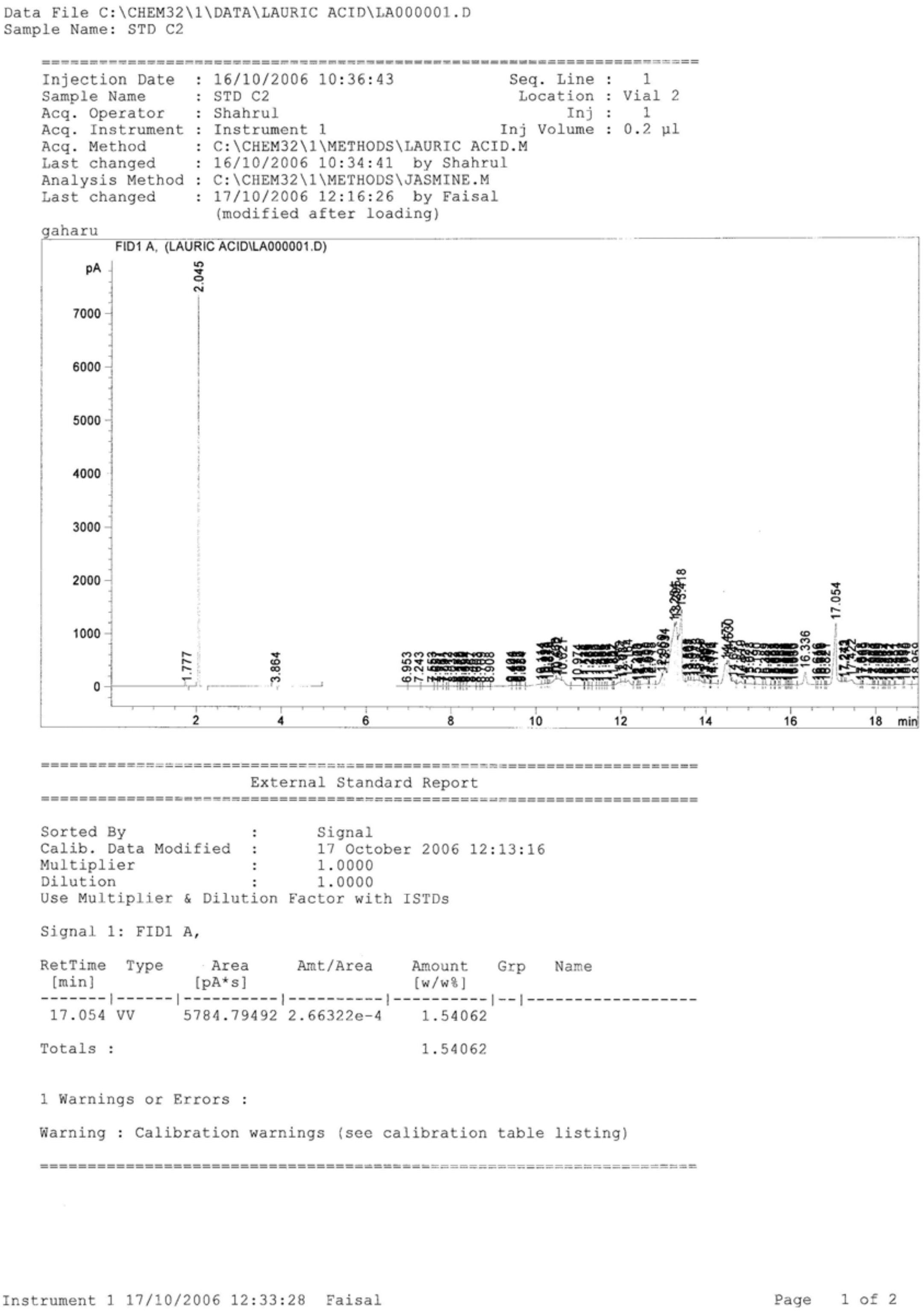
APPENDICES

Appendix A



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    Sample Name     : STD C2                         Location : Vial 2
    Acq. Operator   : Shahrul                             Inj :    1
    Acq. Instrument : Instrument 1                 Inj Volume : 0.2 µl
    Acq. Method     : C:\CHEM32\1\METHODS\LAURIC ACID.M
    Last changed    : 16/10/2006 10:34:41  by Shahrul
    Analysis Method : C:\CHEM32\1\METHODS\JASMINE.M
    Last changed    : 17/10/2006 12:16:26  by Faisal
                      (modified after loading)
    gaharu

    =====================================================================
                             External Standard Report
    =====================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      17 October 2006 12:13:16
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs

    Signal 1: FID1 A,

    RetTime  Type    Area     Amt/Area    Amount   Grp   Name
     [min]         [pA*s]                 [w/w%]
    -------|------|----------|----------|----------|--|------------------
     17.054 VV     5784.79492 2.66322e-4    1.54062

    Totals :                                1.54062

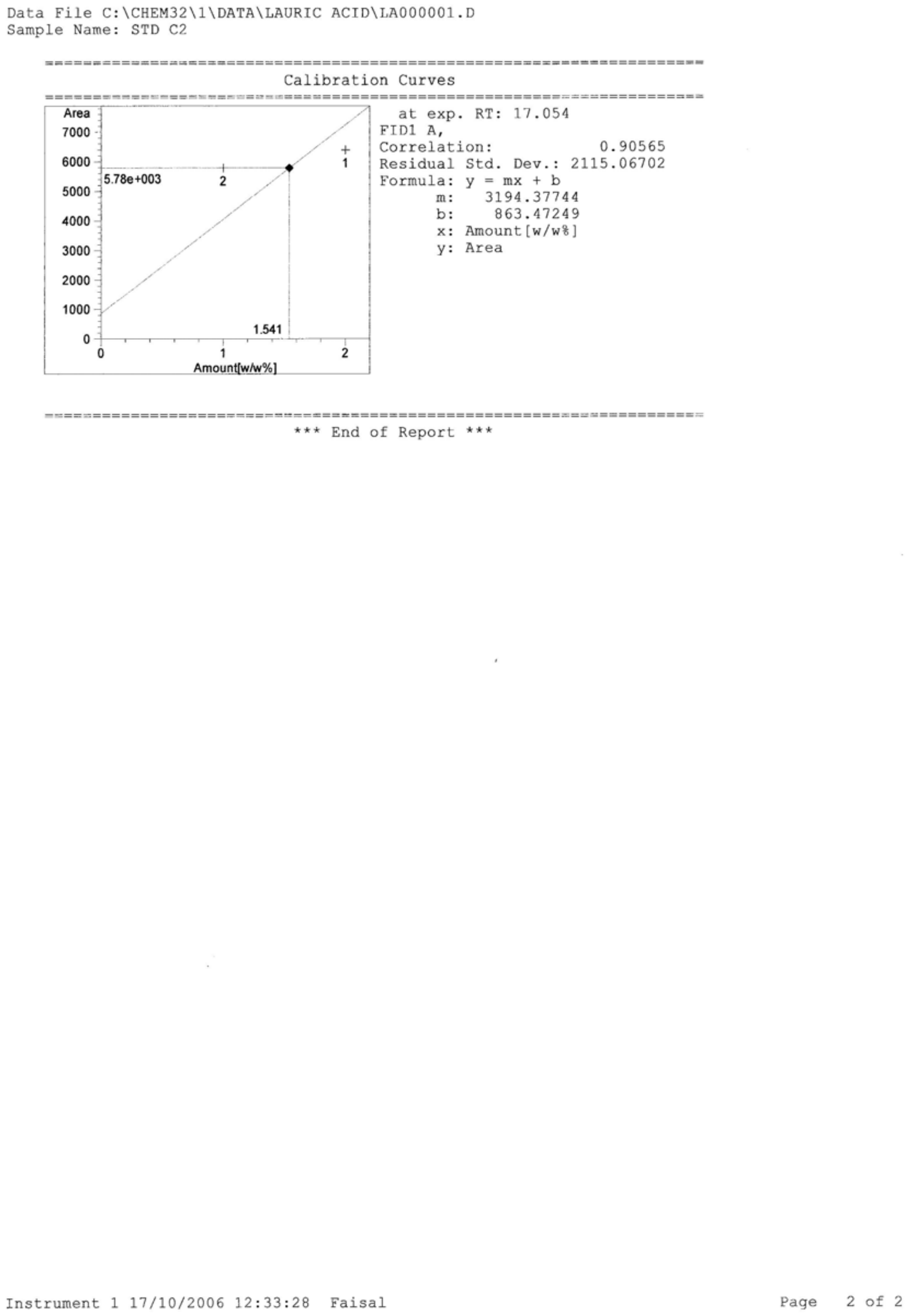
    1 Warnings or Errors :

    Warning : Calibration warnings (see calibration table listing)

    =====================================================================

Instrument 1 17/10/2006 12:33:28  Faisal                        Page   1 of 2
```

Appendix B



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Sample Name: STD C2

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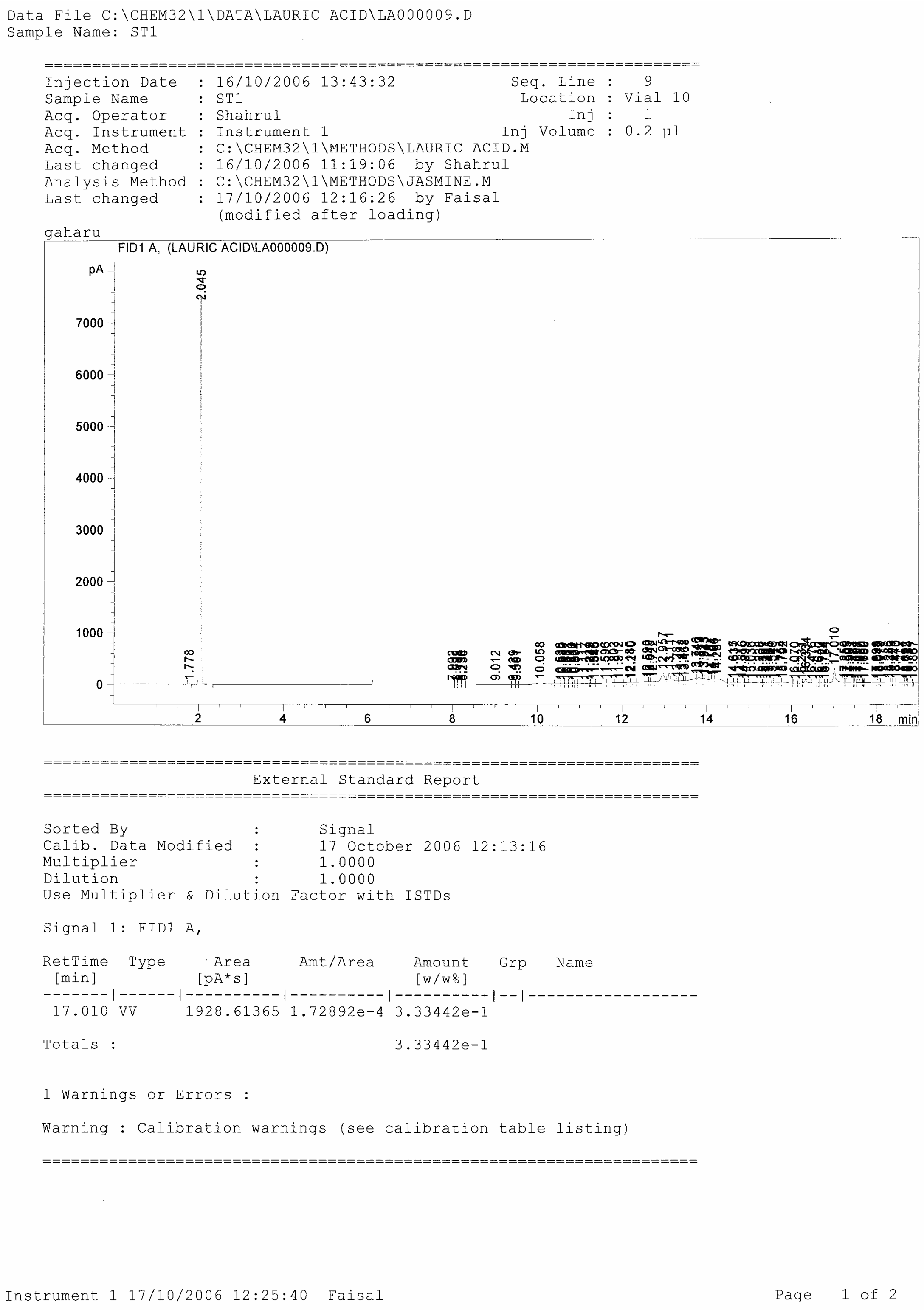
                              Calibration Curves
    =====================================================================
                                     at exp. RT: 17.054
                                   FID1 A,
                                   Correlation:           0.90565
                                   Residual Std. Dev.: 2115.06702
                                   Formula: y = mx + b
                                         m:   3194.37744
                                         b:    863.47249
                                         x: Amount[w/w%]
                                         y: Area

    =====================================================================
                             *** End of Report ***

Instrument 1 17/10/2006 12:33:28  Faisal                        Page   2 of 2
```

Appendix C



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Data File C:\CHEM32\1\DATA\LAURIC ACID\LA000009.D
Sample Name: ST1

    =================================================================
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    Sample Name     : ST1                             Location : Vial 10
    Acq. Operator   : Shahrul                              Inj :   1
    Acq. Instrument : Instrument 1                  Inj Volume : 0.2 µl
    Acq. Method     : C:\CHEM32\1\METHODS\LAURIC ACID.M
    Last changed    : 16/10/2006 11:19:06  by Shahrul
    Analysis Method : C:\CHEM32\1\METHODS\JASMINE.M
    Last changed    : 17/10/2006 12:16:26  by Faisal
                      (modified after loading)
    gaharu
```

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    =================================================================

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    Calib. Data Modified :      17 October 2006 12:13:16
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    Dilution             :      1.0000
    Use Multiplier & Dilution Factor with ISTDs

    Signal 1: FID1 A,

    RetTime  Type     Area     Amt/Area    Amount   Grp   Name
     [min]          [pA*s]                 [w/w%]
    -------|------|----------|----------|----------|--|------------------
     17.010 VV     1928.61365 1.72892e-4 3.33442e-1

    Totals :                              3.33442e-1

    1 Warnings or Errors :

    Warning : Calibration warnings (see calibration table listing)

    =================================================================


Instrument 1 17/10/2006 12:25:40  Faisal                        Page    1 of 2
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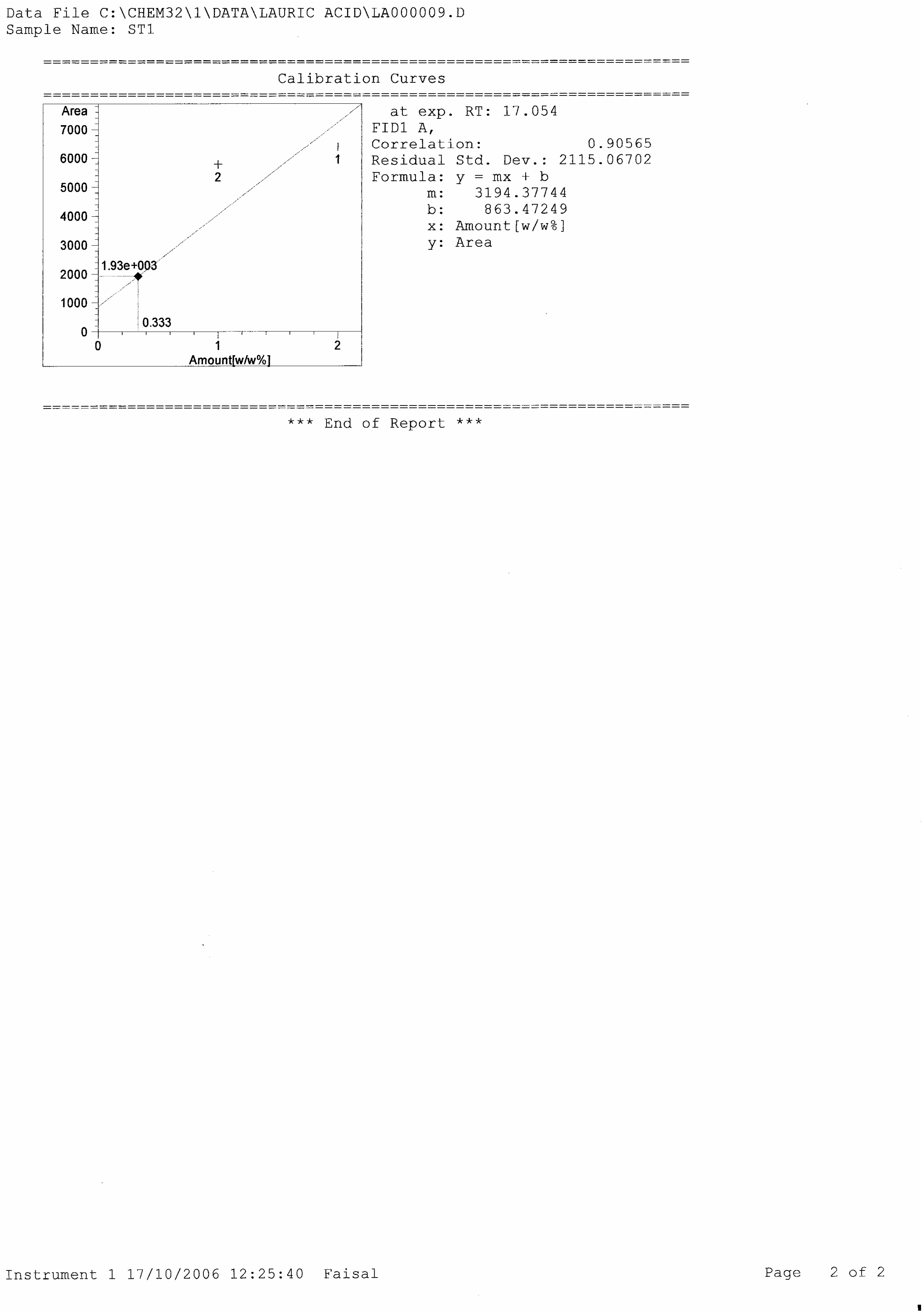
Appendix D



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Sample Name: ST1

    =====================================================================
                             Calibration Curves
    =====================================================================
                                     at exp. RT: 17.054
                                   FID1 A,
                                   Correlation:            0.90565
                                   Residual Std. Dev.: 2115.06702
                                   Formula: y = mx + b
                                         m:   3194.37744
                                         b:    863.47249
                                         x: Amount[w/w%]
                                         y: Area

    =====================================================================
                           *** End of Report ***
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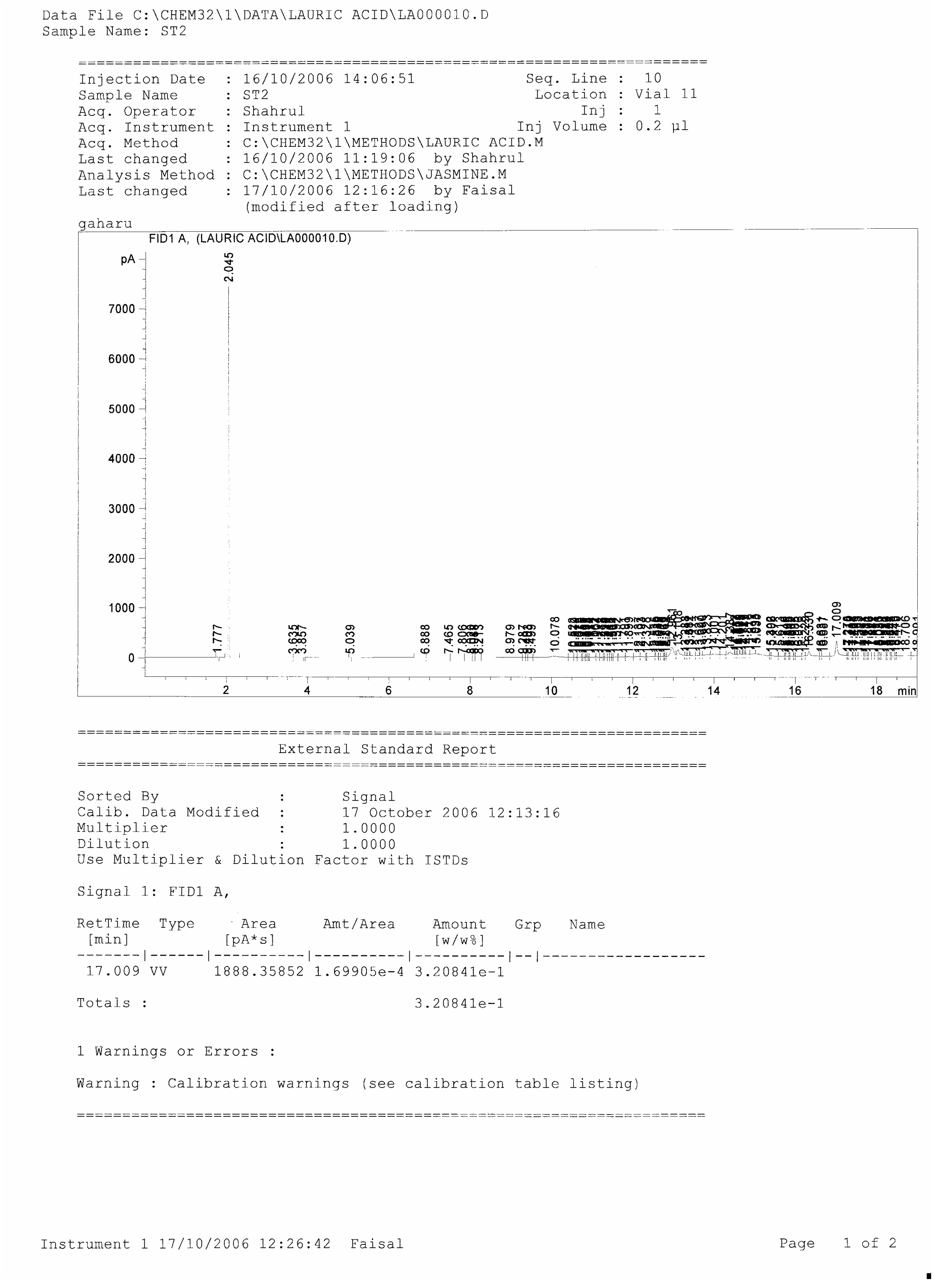
Appendix E



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Sample Name: ST2

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    Sample Name     : ST2                            Location : Vial 11
    Acq. Operator   : Shahrul                             Inj :   1
    Acq. Instrument : Instrument 1                 Inj Volume : 0.2 µl
    Acq. Method     : C:\CHEM32\1\METHODS\LAURIC ACID.M
    Last changed    : 16/10/2006 11:19:06  by Shahrul
    Analysis Method : C:\CHEM32\1\METHODS\JASMINE.M
    Last changed    : 17/10/2006 12:16:26  by Faisal
                      (modified after loading)
    gaharu
```

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    =====================================================================

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    Calib. Data Modified  :      17 October 2006 12:13:16
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs

    Signal 1: FID1 A,

    RetTime  Type     Area      Amt/Area     Amount    Grp   Name
     [min]          [pA*s]                   [w/w%]
    -------|------|----------|----------|----------|--|------------------
     17.009 VV     1888.35852 1.69905e-4 3.20841e-1

    Totals :                             3.20841e-1

    1 Warnings or Errors :

    Warning : Calibration warnings (see calibration table listing)

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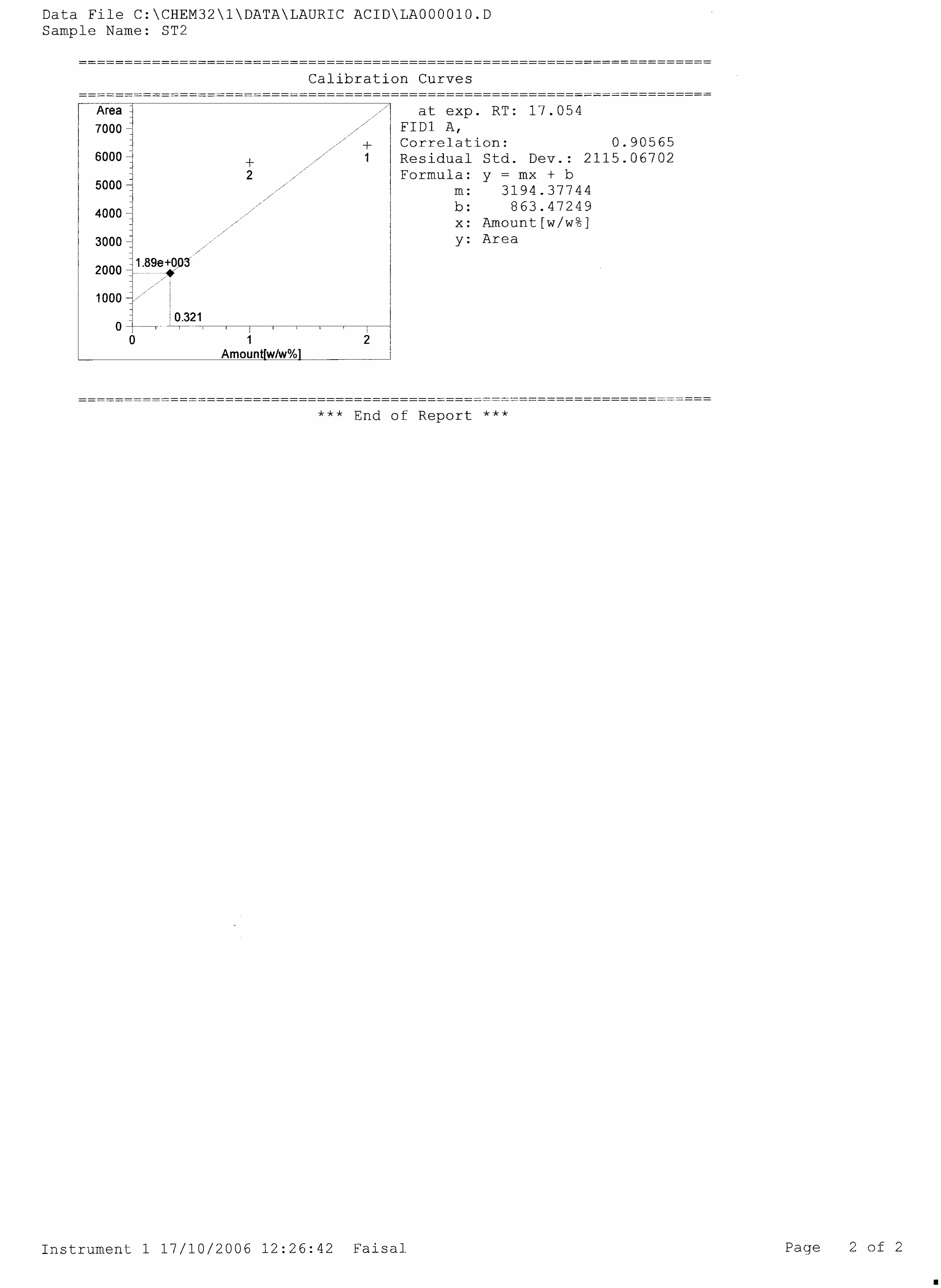
```
Instrument 1 17/10/2006 12:26:42  Faisal                          Page    1 of 2
```

Appendix F



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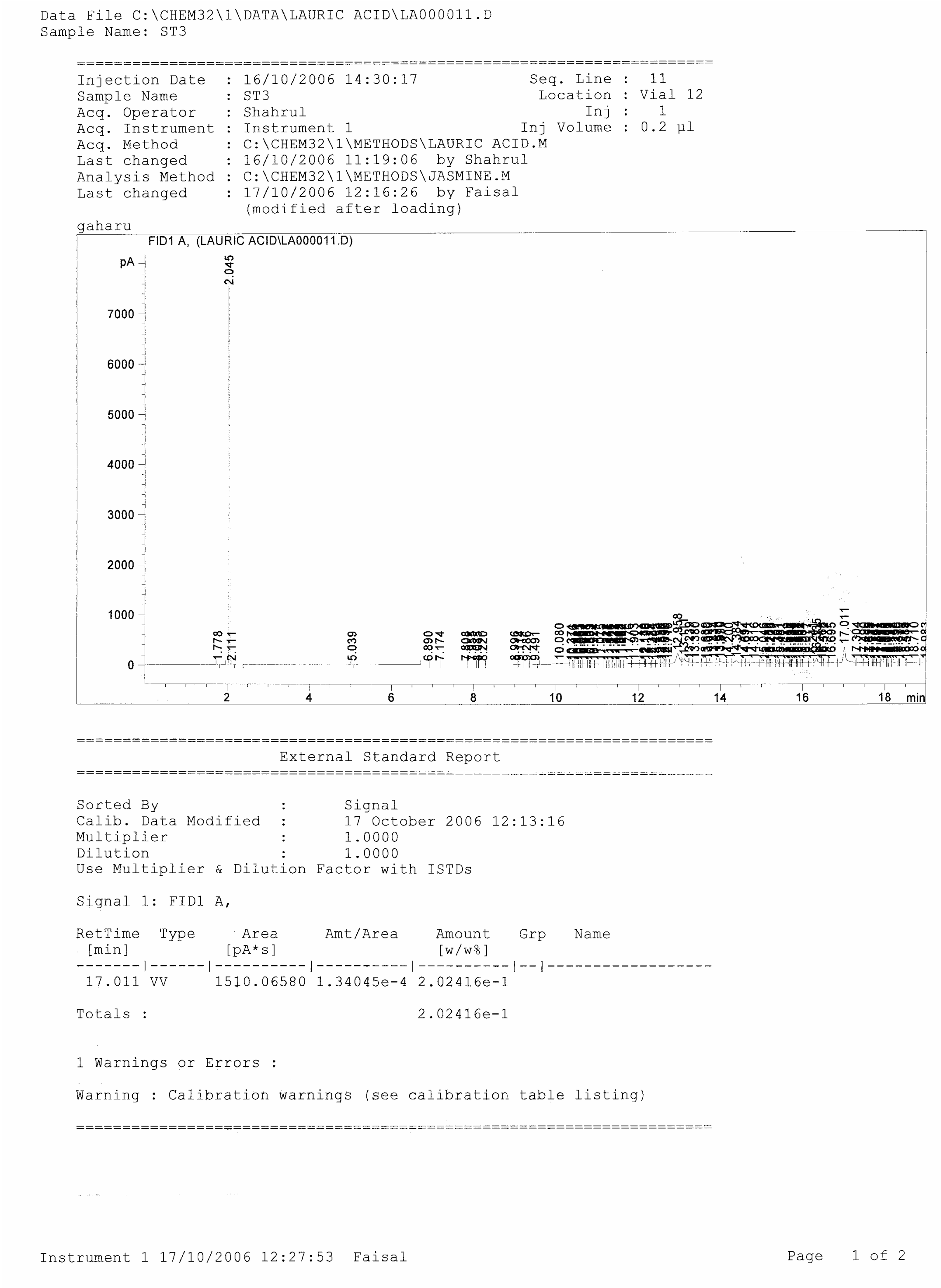
      =====================================================================
                              Calibration Curves
      =====================================================================
                                     at exp. RT: 17.054
                                   FID1 A,
                                   Correlation:           0.90565
                                   Residual Std. Dev.: 2115.06702
                                   Formula: y = mx + b
                                         m:   3194.37744
                                         b:    863.47249
                                         x: Amount[w/w%]
                                         y: Area

      =====================================================================
                          *** End of Report ***

Instrument 1 17/10/2006 12:26:42  Faisal                        Page   2 of 2
```

Appendix G



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Data File C:\CHEM32\1\DATA\LAURIC ACID\LA000011.D
Sample Name: ST3

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    Sample Name     : ST3                            Location : Vial 12
    Acq. Operator   : Shahrul                             Inj :   1
    Acq. Instrument : Instrument 1                 Inj Volume : 0.2 µl
    Acq. Method     : C:\CHEM32\1\METHODS\LAURIC ACID.M
    Last changed    : 16/10/2006 11:19:06  by Shahrul
    Analysis Method : C:\CHEM32\1\METHODS\JASMINE.M
    Last changed    : 17/10/2006 12:16:26  by Faisal
                      (modified after loading)
    gaharu
```

```
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                         External Standard Report
    =====================================================================

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    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs

    Signal 1: FID1 A,

    RetTime  Type     Area      Amt/Area     Amount   Grp   Name
     [min]          [pA*s]                   [w/w%]
    -------|------|----------|----------|----------|--|------------------
     17.011 VV     1510.06580 1.34045e-4 2.02416e-1

    Totals :                             2.02416e-1

    1 Warnings or Errors :

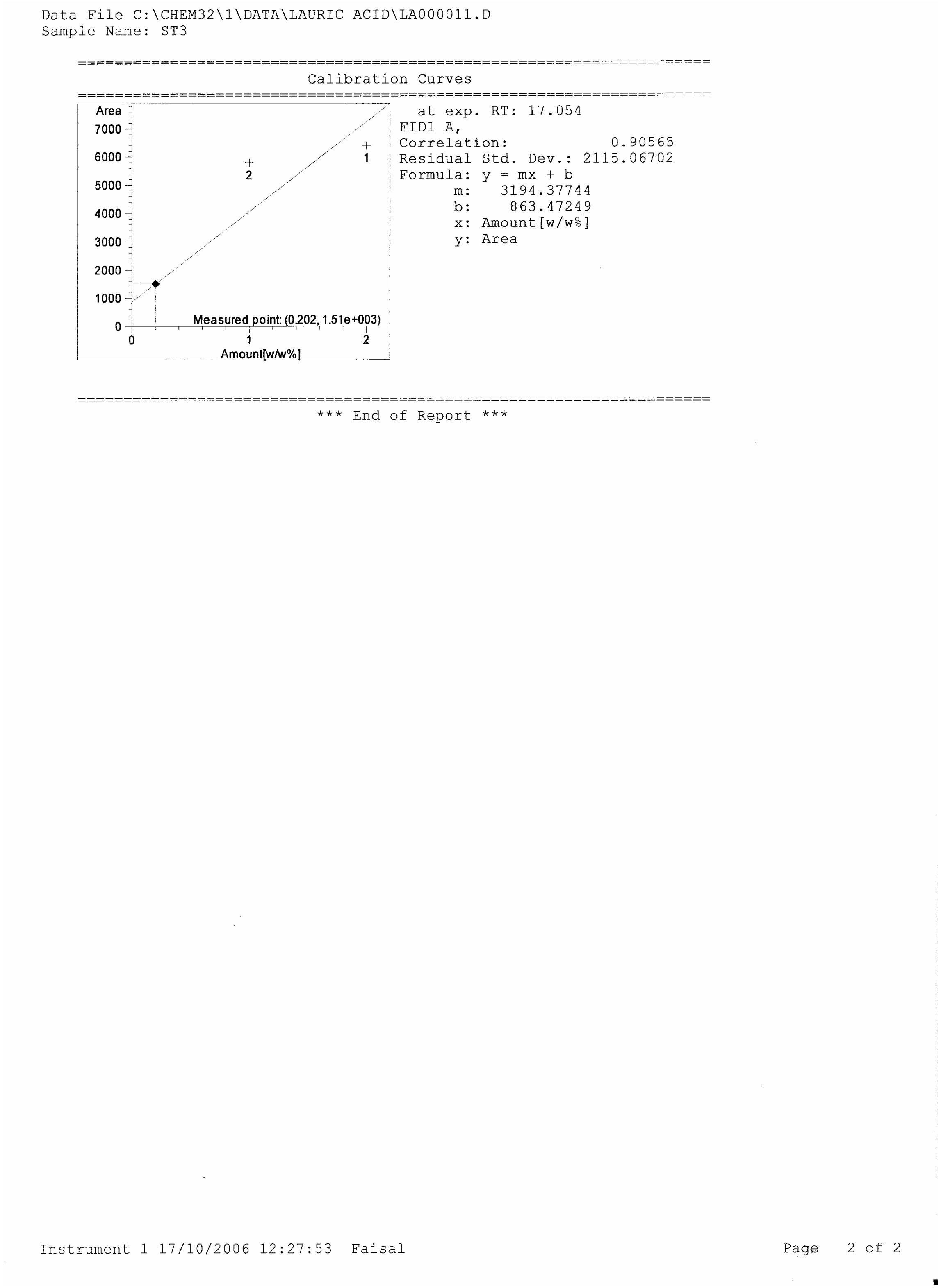
    Warning : Calibration warnings (see calibration table listing)

    =====================================================================



Instrument 1 17/10/2006 12:27:53  Faisal                        Page   1 of 2
```

Appendix H



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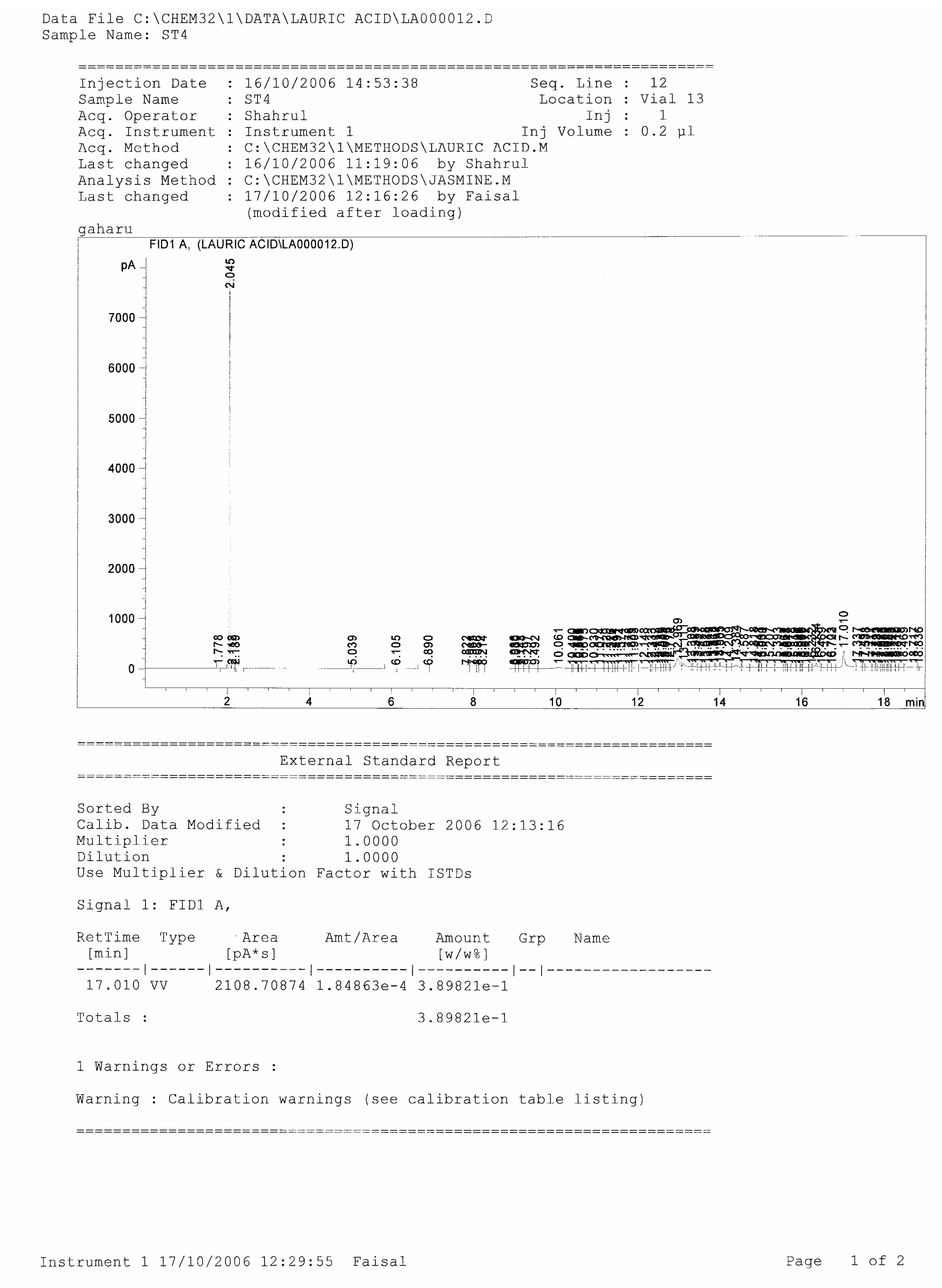
                              Calibration Curves
    ======================================================================
                                    at exp. RT: 17.054
                                  FID1 A,
                                  Correlation:              0.90565
                                  Residual Std. Dev.: 2115.06702
                                  Formula: y = mx + b
                                        m:   3194.37744
                                        b:    863.47249
                                        x: Amount[w/w%]
                                        y: Area

    ======================================================================
                              *** End of Report ***
```

```
Instrument 1 17/10/2006 12:27:53  Faisal                          Page   2 of 2
```

Appendix I



```
Data File C:\CHEM32\1\DATA\LAURIC ACID\LA000012.D
Sample Name: ST4

    =====================================================================
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    Sample Name     : ST4                             Location : Vial 13
    Acq. Operator   : Shahrul                              Inj :   1
    Acq. Instrument : Instrument 1                  Inj Volume : 0.2 µl
    Acq. Method     : C:\CHEM32\1\METHODS\LAURIC ACID.M
    Last changed    : 16/10/2006 11:19:06  by Shahrul
    Analysis Method : C:\CHEM32\1\METHODS\JASMINE.M
    Last changed    : 17/10/2006 12:16:26  by Faisal
                      (modified after loading)
    gaharu

    =====================================================================
                          External Standard Report
    =====================================================================

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    Calib. Data Modified  :      17 October 2006 12:13:16
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs

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     [min]          [pA*s]                  [w/w%]
    -------|------|----------|----------|----------|--|------------------
     17.010 VV     2108.70874 1.84863e-4 3.89821e-1

    Totals :                            3.89821e-1

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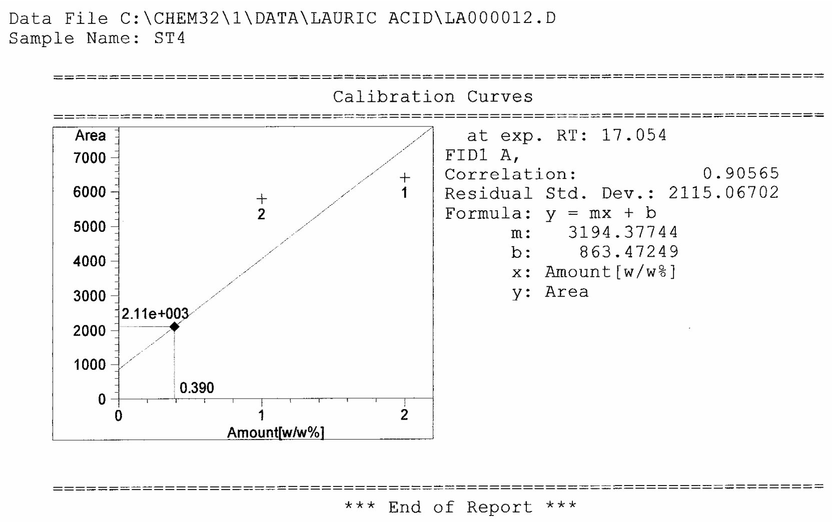
    Warning : Calibration warnings (see calibration table listing)

    =====================================================================

Instrument 1 17/10/2006 12:29:55  Faisal                          Page   1 of 2
```

Appendix J



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    =====================================================================
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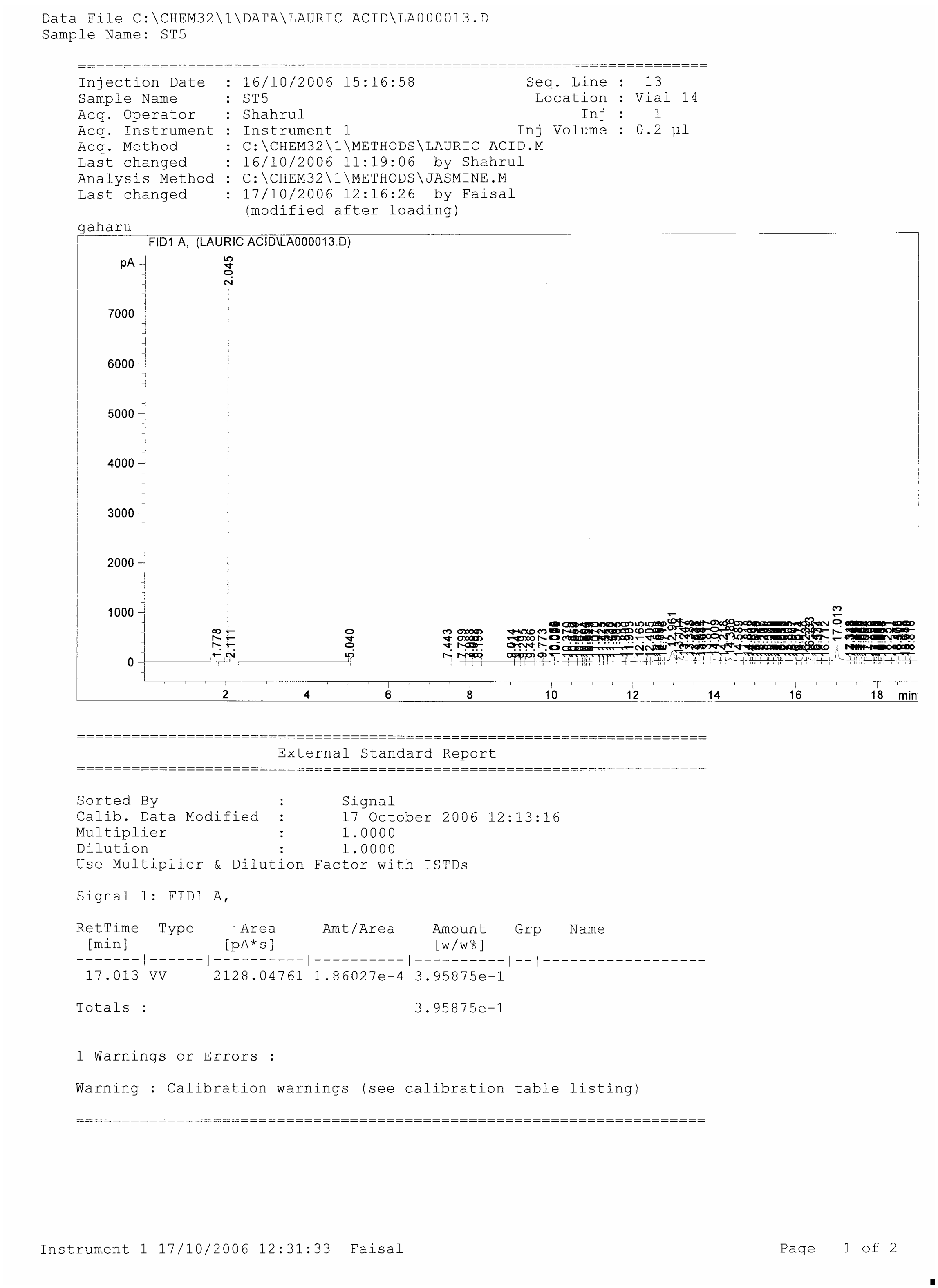
                                  FID1 A,
                                  Correlation:           0.90565
                                  Residual Std. Dev.: 2115.06702
                                  Formula: y = mx + b
                                         m:   3194.37744
                                         b:    863.47249
                                         x: Amount[w/w%]
                                         y: Area

    =====================================================================
                               *** End of Report ***
```

Appendix K



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    =====================================================================
    Injection Date  : 16/10/2006 15:16:58           Seq. Line :  13
    Sample Name     : ST5                            Location : Vial 14
    Acq. Operator   : Shahrul                             Inj :   1
    Acq. Instrument : Instrument 1                 Inj Volume : 0.2 µl
    Acq. Method     : C:\CHEM32\1\METHODS\LAURIC ACID.M
    Last changed    : 16/10/2006 11:19:06  by Shahrul
    Analysis Method : C:\CHEM32\1\METHODS\JASMINE.M
    Last changed    : 17/10/2006 12:16:26  by Faisal
                      (modified after loading)
    gaharu
```

```
    =====================================================================
                          External Standard Report
    =====================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      17 October 2006 12:13:16
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs

    Signal 1: FID1 A,

    RetTime  Type     Area      Amt/Area     Amount    Grp   Name
     [min]          [pA*s]                   [w/w%]
    -------|------|----------|----------|----------|--|------------------
     17.013 VV     2128.04761 1.86027e-4 3.95875e-1

    Totals :                             3.95875e-1

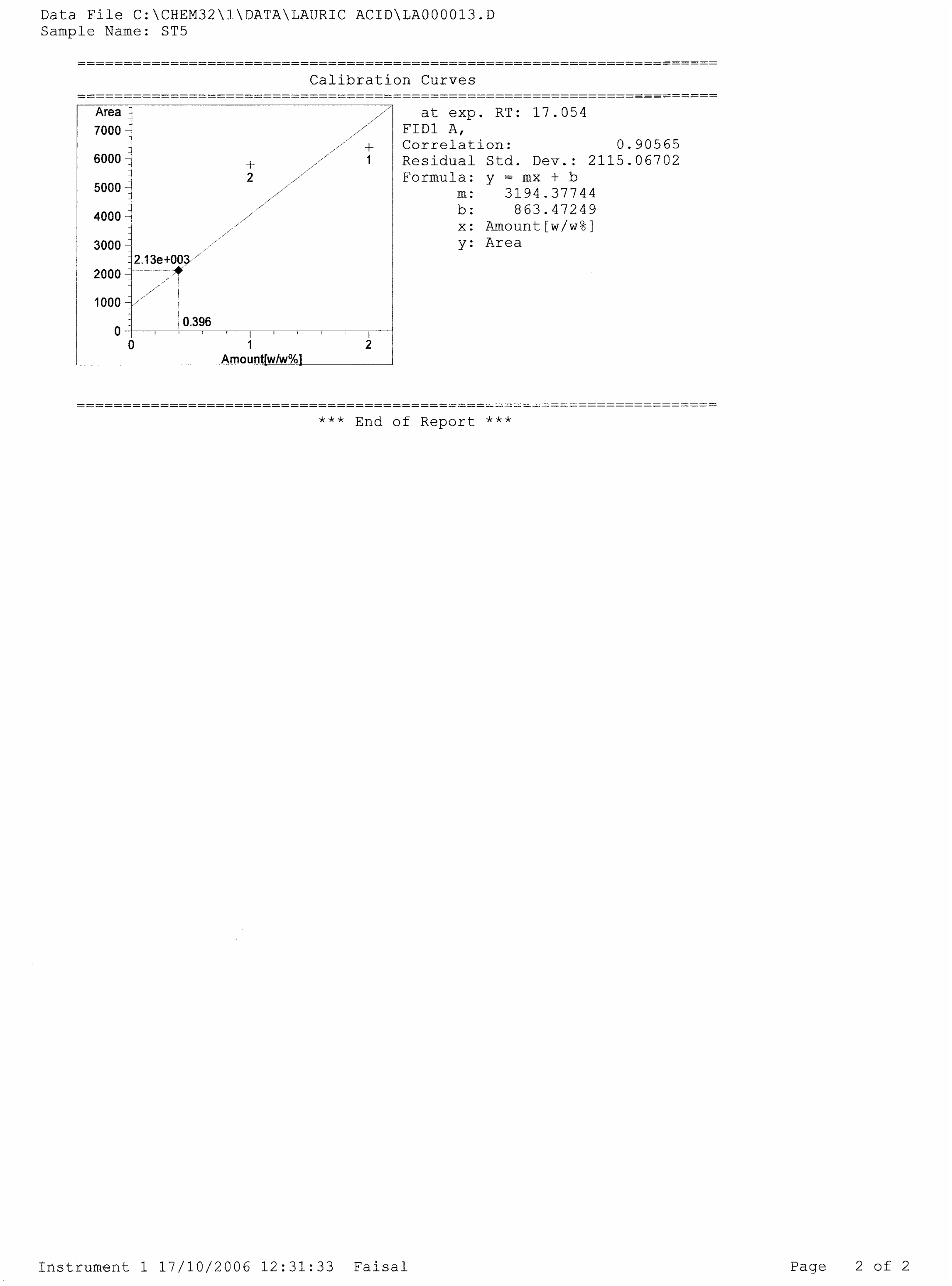
    1 Warnings or Errors :

    Warning : Calibration warnings (see calibration table listing)

    =====================================================================


Instrument 1 17/10/2006 12:31:33  Faisal                        Page    1 of 2
```

Appendix L



```
Data File C:\CHEM32\1\DATA\LAURIC ACID\LA000013.D
Sample Name: ST5

     =====================================================================
                               Calibration Curves
     =====================================================================
                                          at exp. RT: 17.054
                                         FID1 A,
                                         Correlation:           0.90565
                                         Residual Std. Dev.: 2115.06702
                                         Formula: y = mx + b
                                               m:   3194.37744
                                               b:    863.47249
                                               x: Amount[w/w%]
                                               y: Area

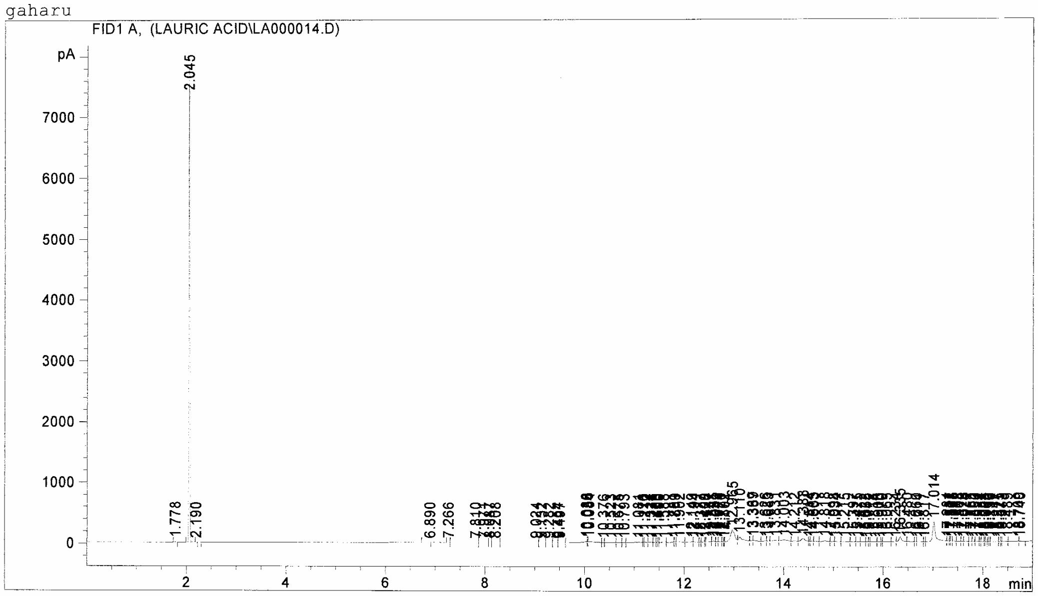
     =====================================================================
                             *** End of Report ***
```

```
Instrument 1 17/10/2006 12:31:33  Faisal                    Page   2 of 2
```

Appendix M

```
Data File C:\CHEM32\1\DATA\LAURIC ACID\LA000014.D
Sample Name: ST6

    =================================================================
    Injection Date  : 16/10/2006 15:40:25           Seq. Line :  14
    Sample Name     : ST6                            Location : Vial 15
    Acq. Operator   : Shahrul                             Inj :   1
    Acq. Instrument : Instrument 1                 Inj Volume : 0.2 µl
    Acq. Method     : C:\CHEM32\1\METHODS\LAURIC ACID.M
    Last changed    : 16/10/2006 11:19:06  by Shahrul
    Analysis Method : C:\CHEM32\1\METHODS\JASMINE.M
    Last changed    : 17/10/2006 12:16:26  by Faisal
                      (modified after loading)
    gaharu
```



```
    =================================================================
                         External Standard Report
    =================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      17 October 2006 12:13:16
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs

    Signal 1: FID1 A,

    RetTime  Type    Area      Amt/Area     Amount    Grp   Name
     [min]          [pA*s]                  [w/w%]
    -------|------|----------|----------|----------|--|------------------
     17.014 VV     1911.11011 1.71609e-4 3.27963e-1

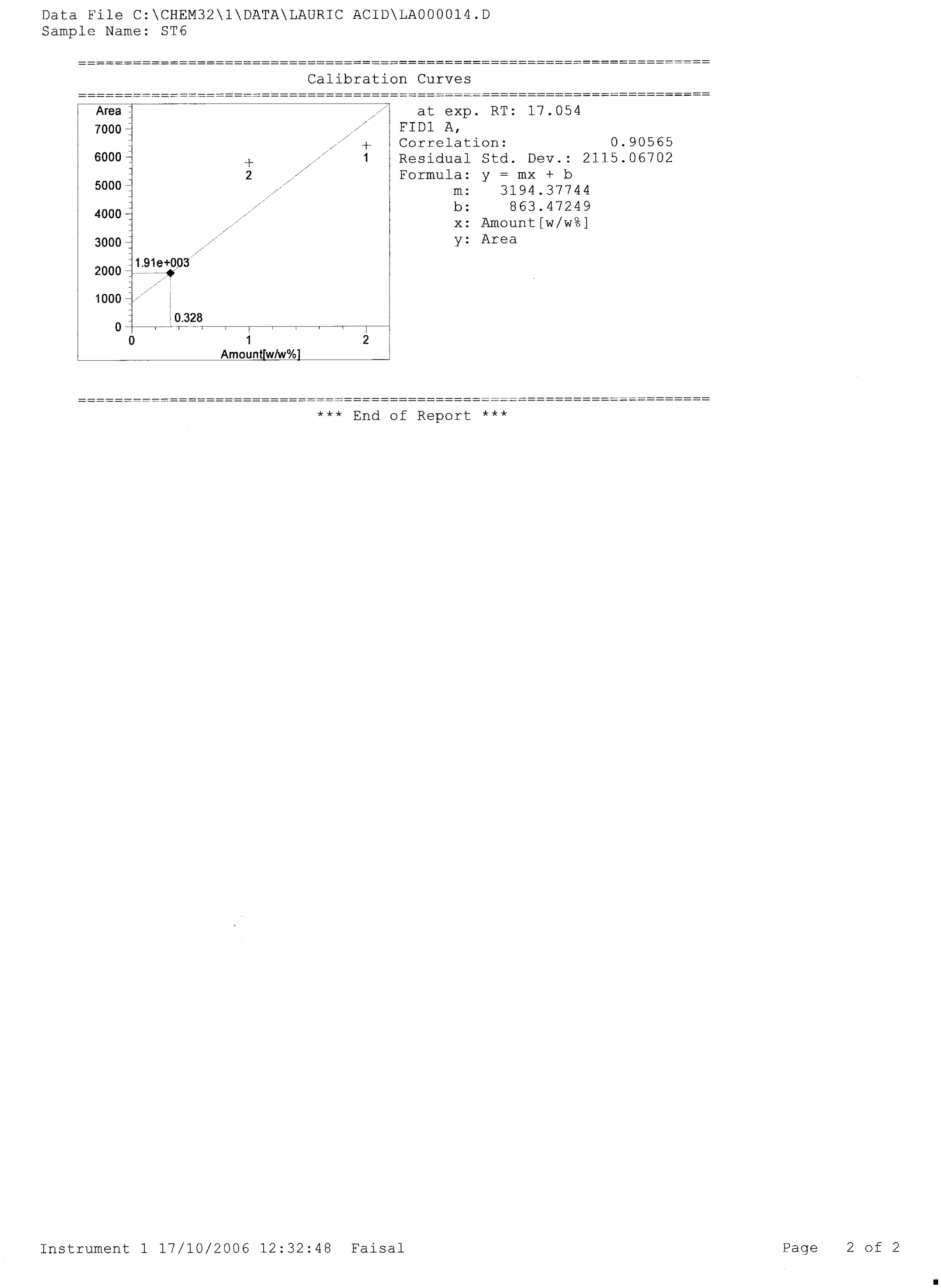
    Totals :                              3.27963e-1

    1 Warnings or Errors :

    Warning : Calibration warnings (see calibration table listing)

    =================================================================
```

Appendix N



```
Data File C:\CHEM32\1\DATA\LAURIC ACID\LA000014.D
Sample Name: ST6

    ======================================================================
                              Calibration Curves
    ======================================================================
                                      at exp. RT: 17.054
                                    FID1 A,
                                    Correlation:           0.90565
                                    Residual Std. Dev.: 2115.06702
                                    Formula: y = mx + b
                                          m:   3194.37744
                                          b:    863.47249
                                          x: Amount[w/w%]
                                          y: Area

    ======================================================================
                            *** End of Report ***
```

```
Instrument 1 17/10/2006 12:32:48  Faisal                          Page    2 of 2
```