



CASE STUDY
Quantitation of Stearamide & Antioxidants
in an Extruder Build-up

OBJECTIVE

The purpose of this work is to quantitate a series of components in an extruder buildup including stearamide and a series of antioxidants.

ANALYTICAL STRATEGY

Quantitation of the stearamide and the antioxidants (Irganox 1010, Irganox 1076) from the extruder buildup was performed by GCMS and QTOF-LCMS respectively.

Read the following report to see the full analysis.



Final Report

Jordi Labs LLC
Case Study
Quantitation of Stearamide & Antioxidants
in an Extruder Build-up

Date: xx/xx/xx

Prepared by:
Dr. Mark Jordi
President
Jordi Labs LLC

Report Number: Jxxxx

Company Name Confidential





Date

Client Name

Company Name

Address

Dear Valued Client:

Please find enclosed the test results for your samples described as:

1. Unknown Extruder Build-up

The following tests were performed:

1. Gas Chromatography Mass Spectrometry (GCMS)
2. Quadrupole Time of Flight Liquid Chromatography Mass Spectroscopy (QTOF-LCMS)

Objectives

The purpose of this work is to quantitate a series of components in an extruder buildup including stearamide and a series of antioxidants. GCMS and QTOF-LCMS were used for this purpose.

Summary of Results

GCMS analysis indicated that the quantity of stearamide in the extruder buildup was 24.33% by weight.

The sample was also examined by QTOF-LCMS in order to quantify the amount of Irganox 1010 and Irganox 1076. Irganox 1010 and 1076 were found to be present at 1.77% and 2.80% by weight, respectively.

Calibration curves and tabulated data for both techniques can be seen in the Individual Test Results section below.

Individual Test Results

A summary of the individual test results is provided below. All accompanying data, including spectra, has been mailed in the data section of this report.

GCMS

A stock solution of Stearamide was prepared at a concentration of 10.38 mg/ml in 10 ml chloroform. Dilutions of this standard were prepared at 1:10 (1.04 mg/ml) and 1:100 (0.104 mg/ml), and a calibration curve was constructed based on the peak areas observed.

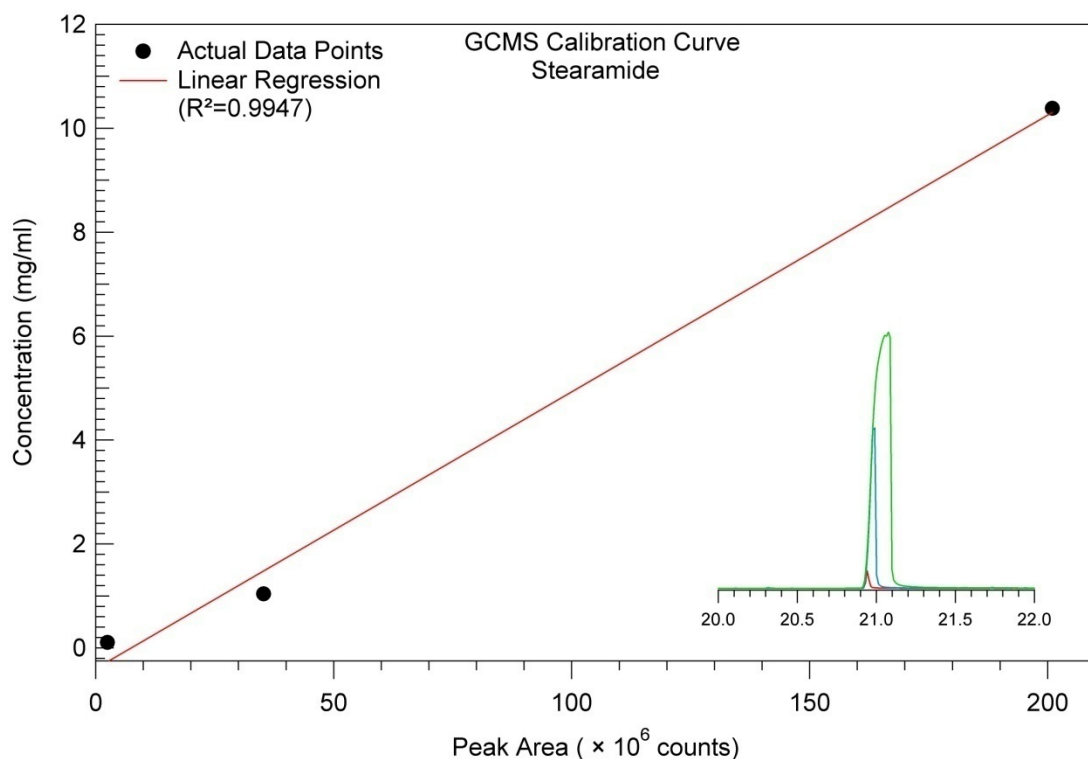


Figure 1 – Calibration curve for stearamide.

The calibration curve prepared was used to calculate the concentration of stearamide present in the sample. The equation for the linear regression line is:

$$Y = 5.320 \times 10^{-8}(X) - 0.398 \quad R^2 = 0.9947$$

Table 1 – Stearamide Content

Sample		Concentration	Peak Area	Composition (% w/w)
<i>Unknown Extruder Build-up</i>	1	1.1930	29919261	23.74
	2	1.2232	30486508	24.34
	3	1.2510	31008662	24.89
Average		1.222	30471477	24.33

QTOF-LCMS –A solution of the sample was prepared by dissolving 3.5 mg in 1 ml of a 25/25/50 Acetonitrile/Methanol/Isopropanol. A stock solution containing 1mg/ml each of Irganox 1010 and Irganox 1076 was also prepared and dilutions of this stock solution were used to create calibration curves for each component.

Background information

A number of plots are used to aid in interpreting QTOF-LCMS data. This includes Base Peak Chromatograms (BPC), Extracted Ion Chromatograms (EIC), Mass spectra (MS) and Product Ion Spectra (MSMS). A BPC is formed by plotting the most intense ion at a given retention time. This spectrum is particularly useful for identifying the retention time of unknowns. EICs are formed by plotting a single mass at all retention times. This could be considered a plot of peak intensity (~compound concentration) for a single compound (and its isomers) versus retention time.

Quantitation of a particular component relies on EIC's of the appropriate mass for the compound of interest. The areas of the EIC peak for the sample and reference standards are collected and properly scaled using the internal standard. A calibration curve is created by plotting the relative response of the LCMS versus the concentration of the standards. The relative response of the samples is then compared to this calibration curve to find the target compound concentration.

Results

Figures 2 and 3 provide the calibration curves for Irganox 1010 and Irganox 1076, respectively. **Tables 2 and 3** provide each individual injection and average concentration values for each component. The weight percent of each component in the original sample is also provided.

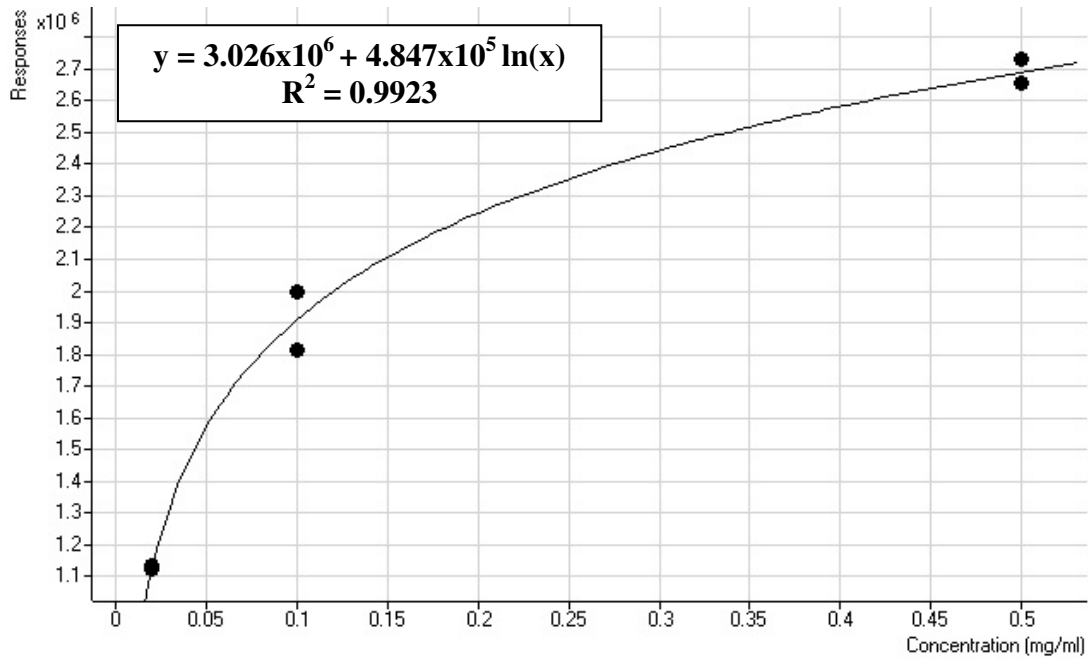


Figure 2 – Calibration Curve for Irganox 1010

Table 2 – Irganox 1010 Content

Sample		Concentration (mg/ml)	Composition (% w/w)
<i>Unknown Extruder Build-up</i>	1	0.059	1.69
	2	0.065	1.86
	3	0.063	1.80
	4	0.060	1.71
Average		0.062	1.77

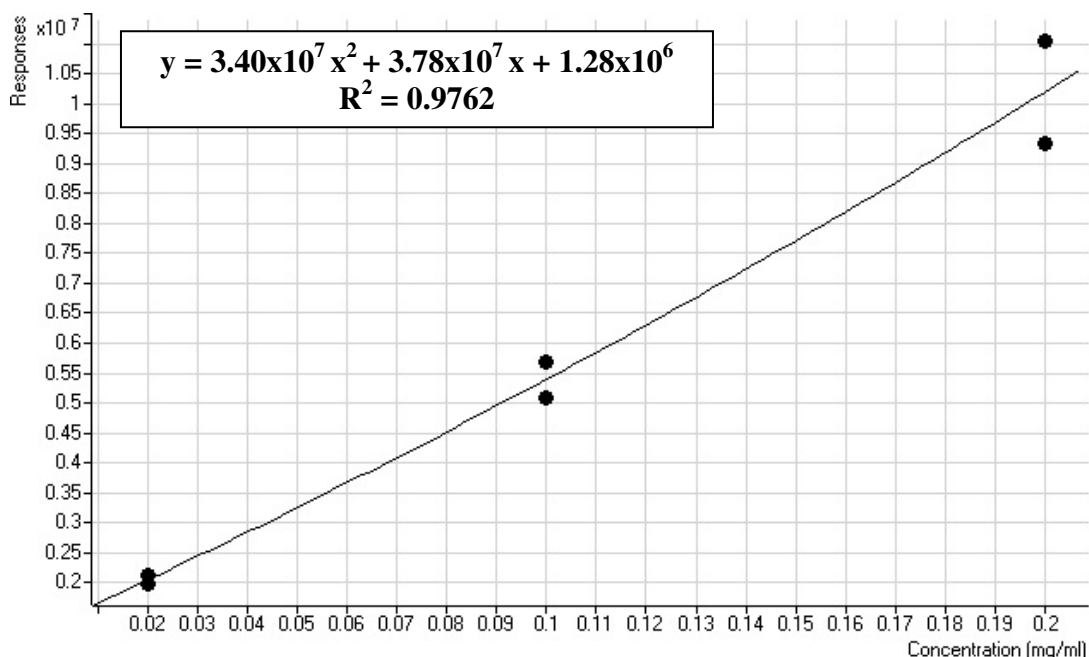


Figure 3 – Calibration Curve for Irganox 1010

Table 3 – Irganox 1076 Content

Sample		Concentration (mg/ml)	Composition (% w/w)
<i>Unknown Extruder Build-up</i>	1	0.086	2.46
	2	0.107	3.06
	3	0.103	2.94
	4	0.096	2.74
Average		0.098	2.80

Analysis Conditions

This section of a Jordi report provides information on the methods used including instrument type, temperatures, solvents, sample preparation, etc. The specific conditions have been removed for this case study.

Closing Comments

Deformulation of an unknown material is intended to provide a best estimate of the chemical nature of the sample. All chemical structures are supported by the evidence presented but are subject to revision upon receipt of additional evidence. Additional factors such as material processing conditions may also affect final material properties.

Jordi Labs specializes in polymer testing and has 30 years experience doing complete polymer deformulations. We are one of the few labs in the country specialized in this type of testing. We will work closely with you to help explain your test results and solve your problem. We appreciate your business and are looking forward to speaking with you concerning these results.

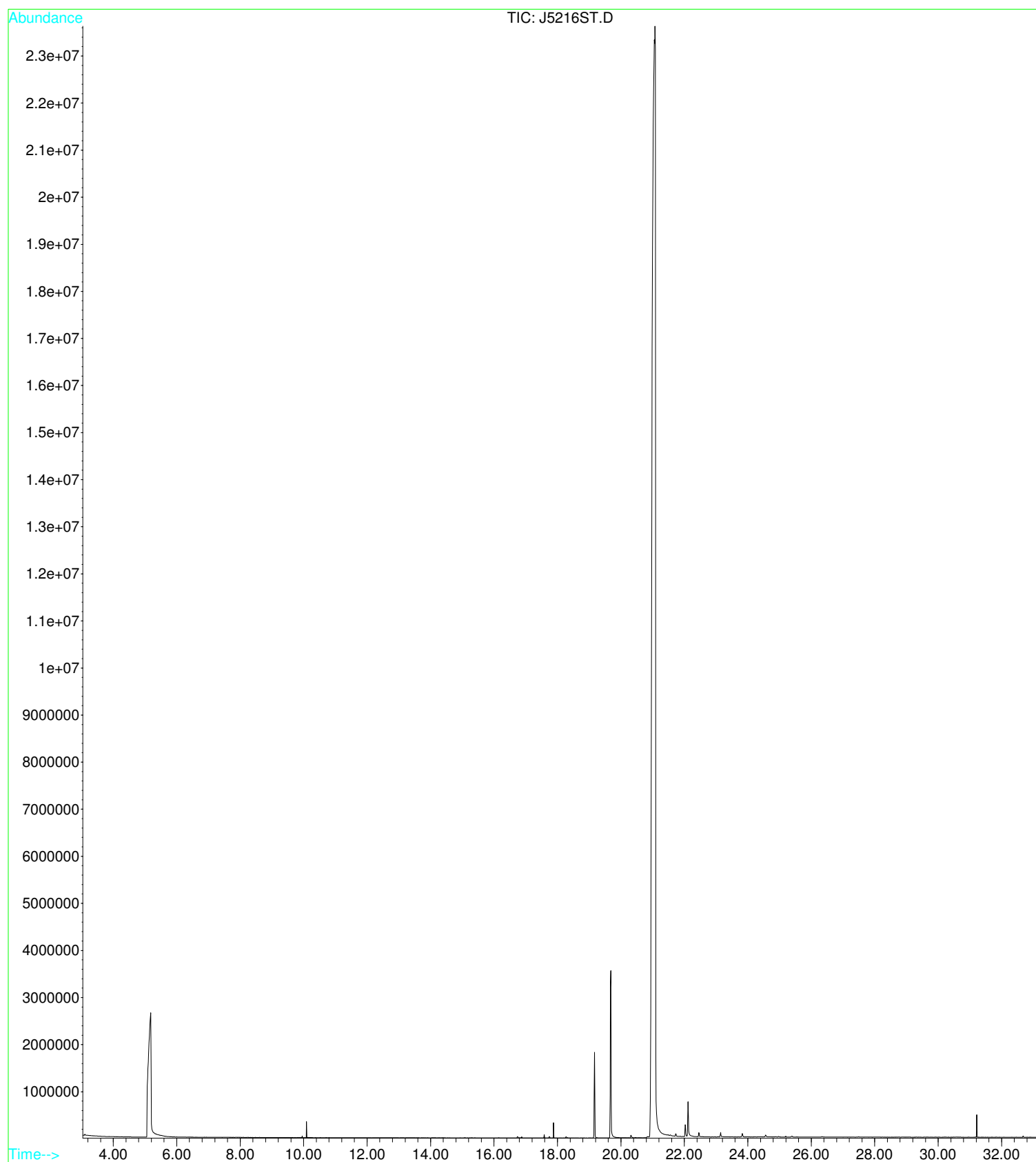
Sincerely,

Mark Jordi

Mark Jordi, Ph. D.
President
Jordi Labs LLC

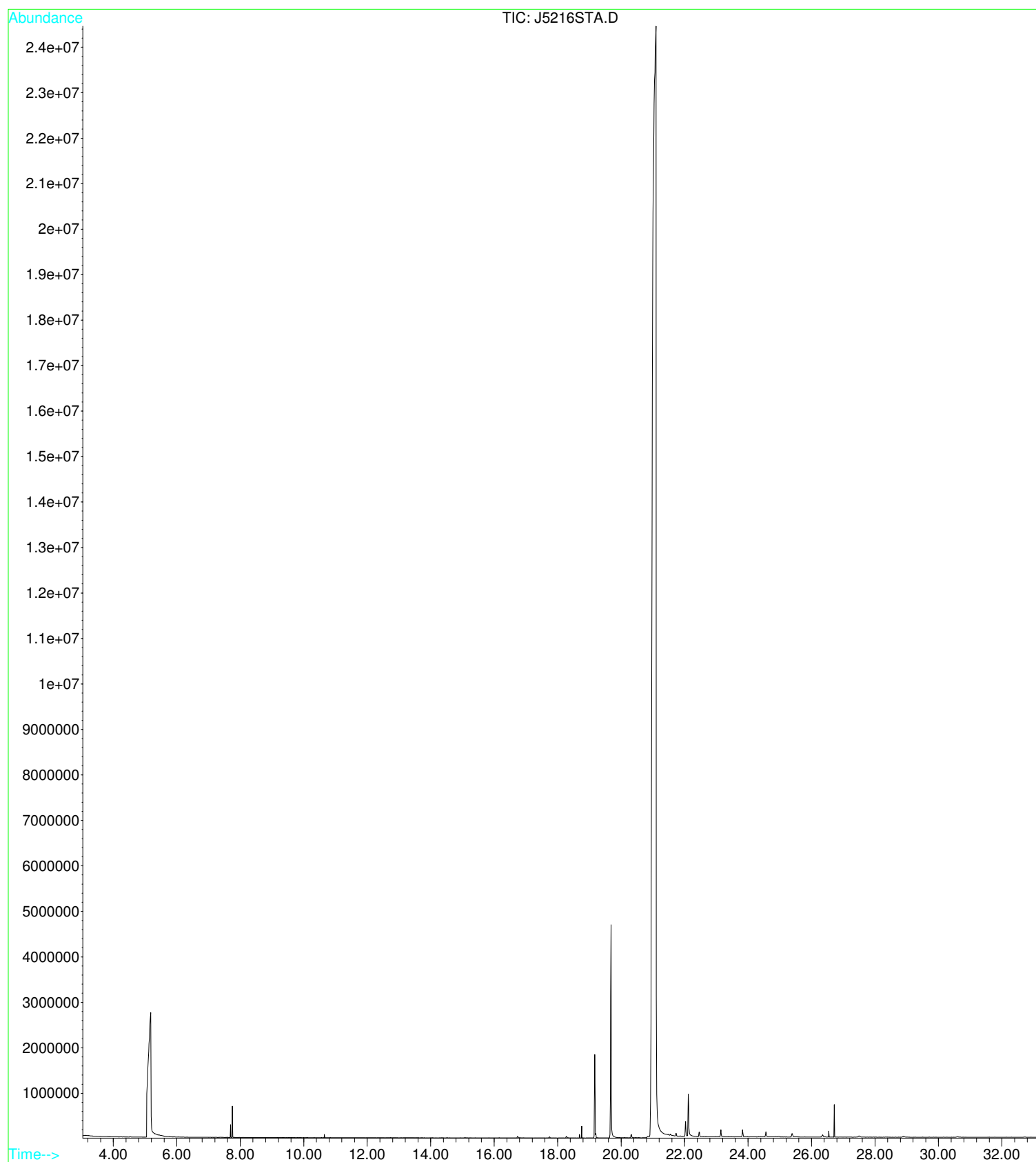
GCMS RESULTS

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Operator : Kimberly Vitiello
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Instrument : DMS Instr
Sample Name: J5216 Jordi Stearamide Stock 5mg/ml
Misc Info :
Vial Number: 10



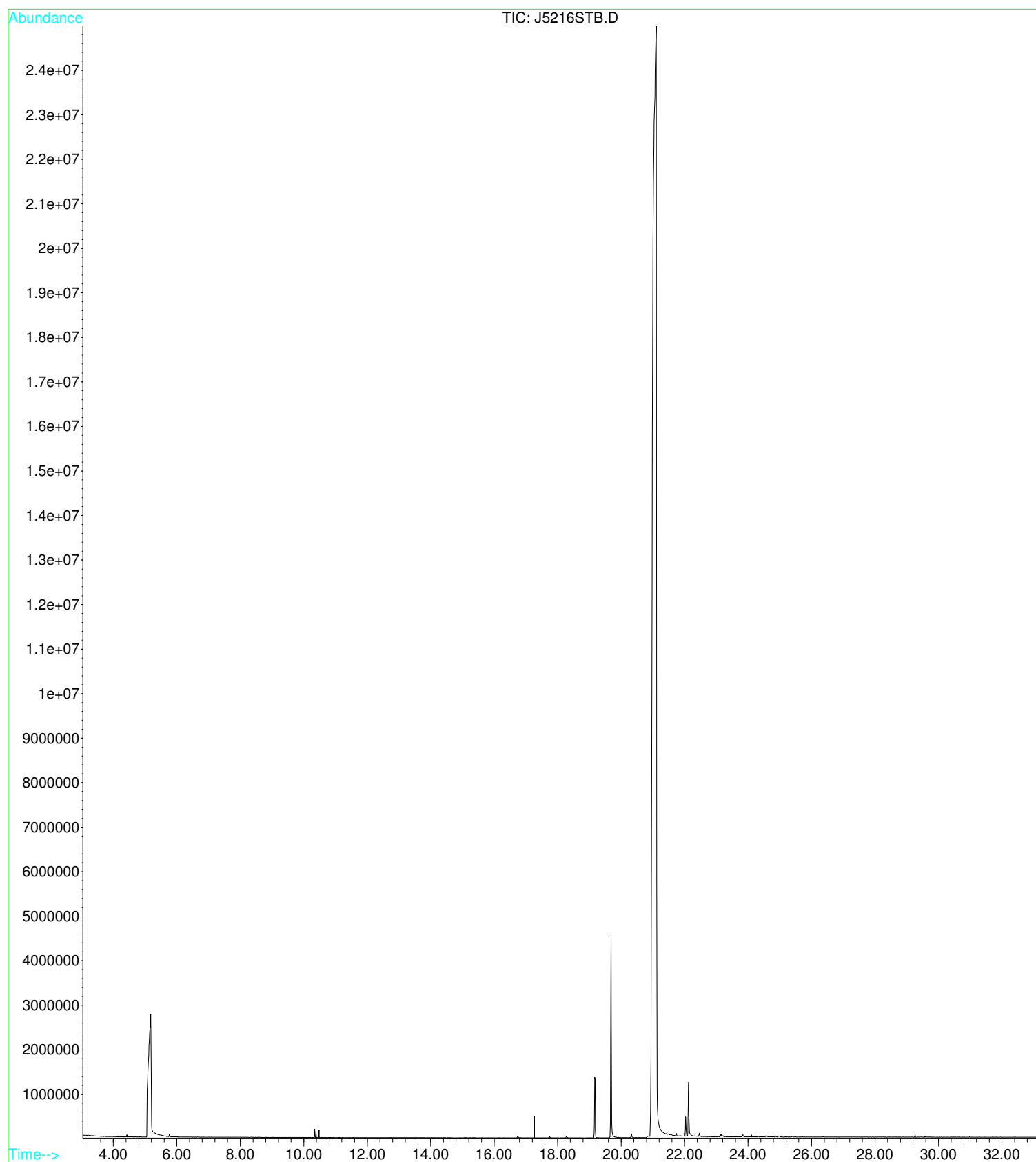
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Operator : Kimberly Vitiello
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Sample Name: J5216 Jordi Stearamide Stock 5mg/ml
Misc Info :
Vial Number: 10



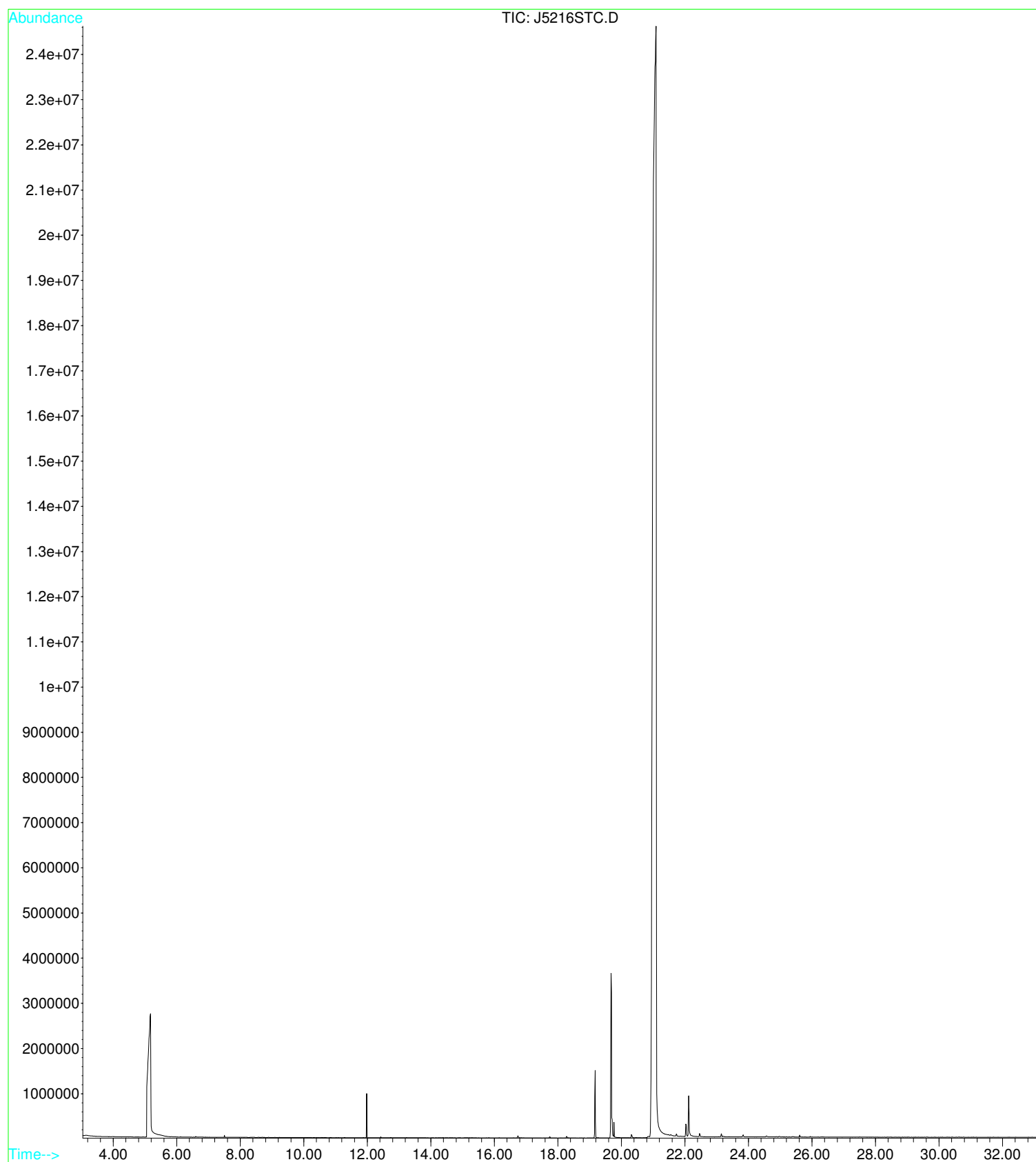
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Operator : Kimberly Vitiello
Acquired : 28 Oct 2010 15:00 using AcqMethod GCMS
Instrument : DMS Instr
Sample Name: J5216 Jordi Stearamide Stock 5mg/ml
Misc Info :
Vial Number: 10



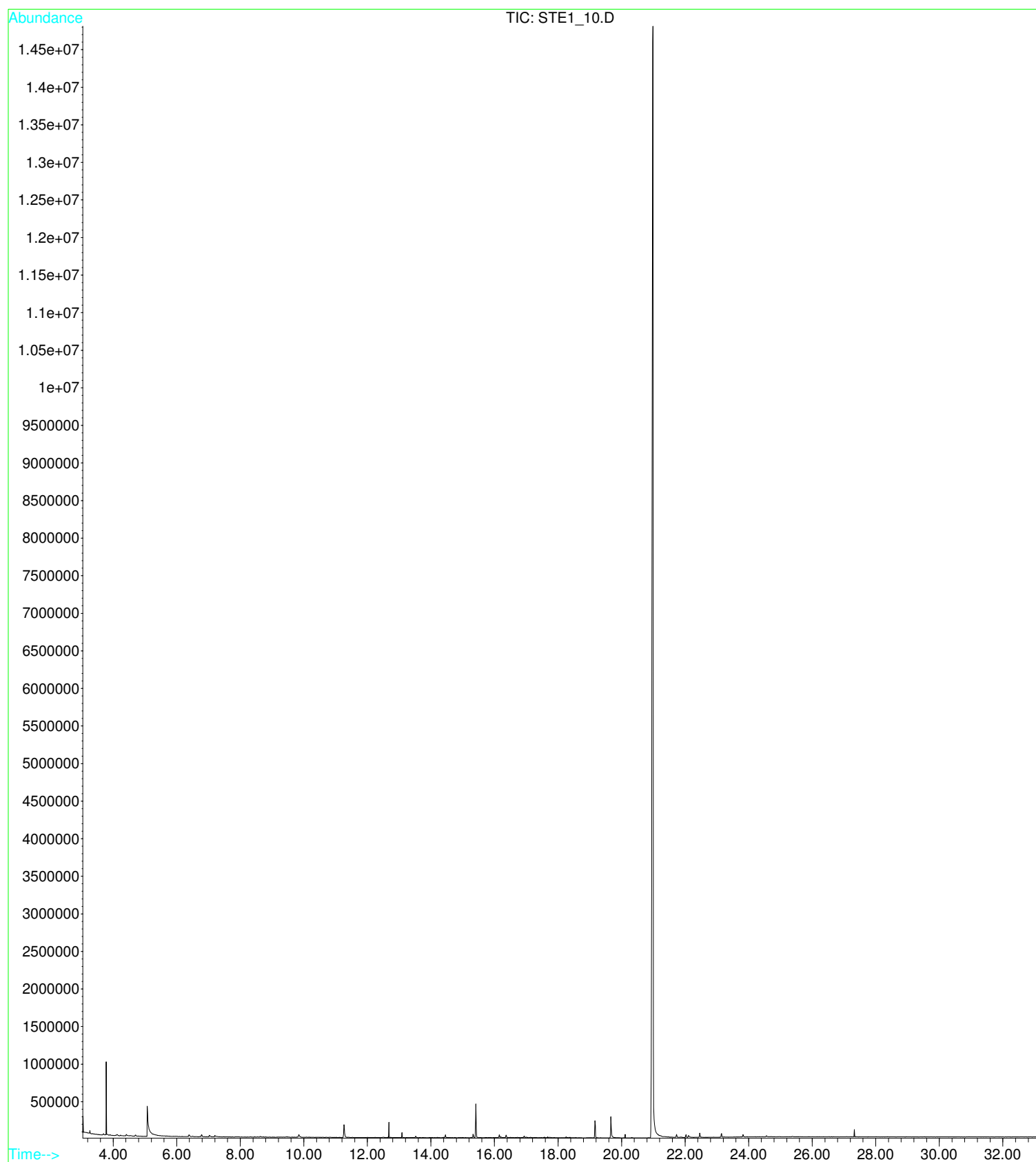
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Operator : Kimberly Vitiello
Acquired : 28 Oct 2010 15:43 using AcqMethod GCMS
Instrument : DMS Instr
Sample Name: J5216 Jordi Stearamide Stock 5mg/ml
Misc Info :
Vial Number: 10



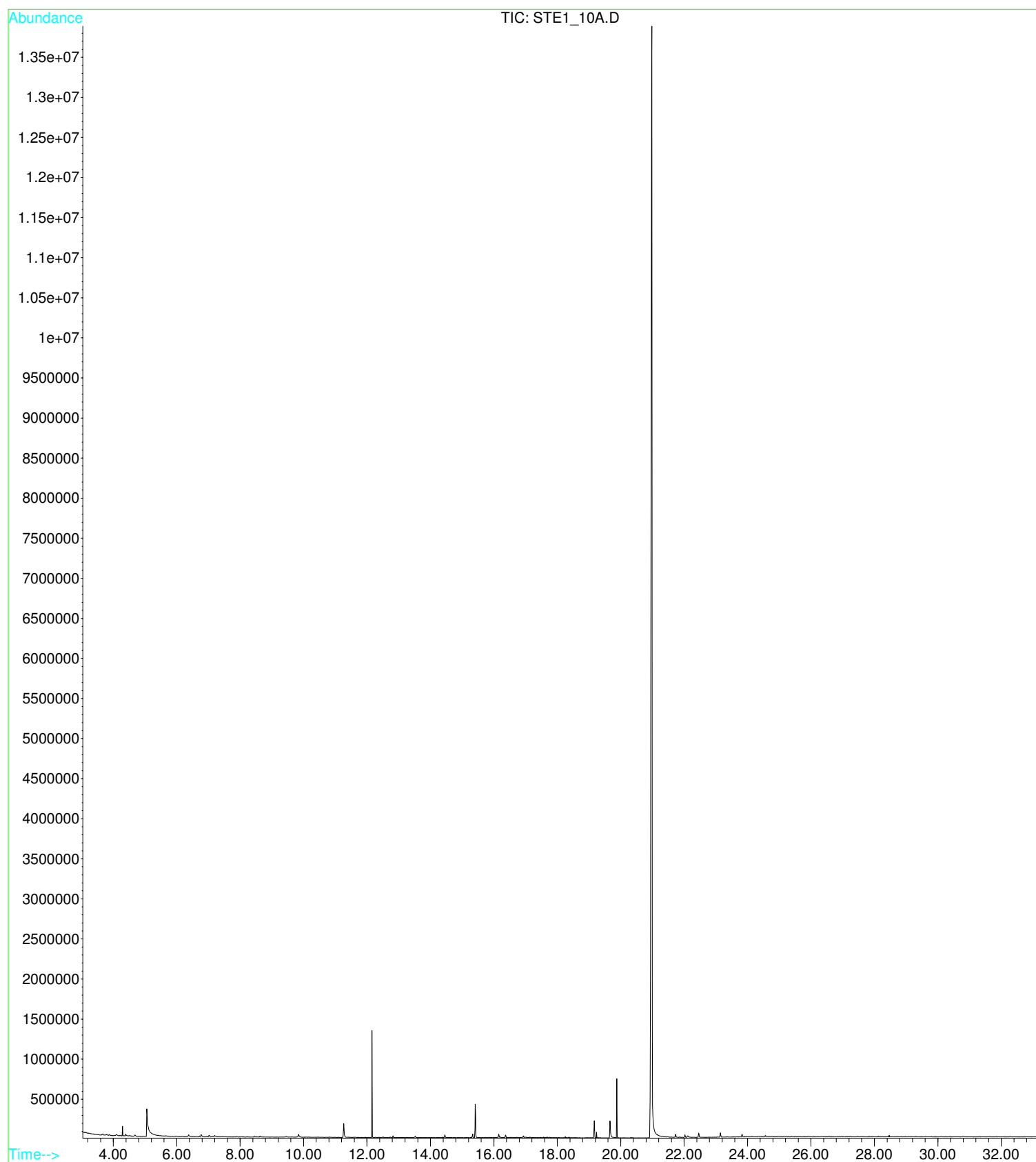
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1	21.093	rm	0.834	184735622	20.876	21.710

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Operator : Kimberly Vitiello
Acquired : 28 Oct 2010 5:07 using AcqMethod GCMS
Instrument : DMS Instr
Sample Name: J5216 Jordi Stearamide 1to10 Dilution 5mg/ml
Misc Info :
Vial Number: 11



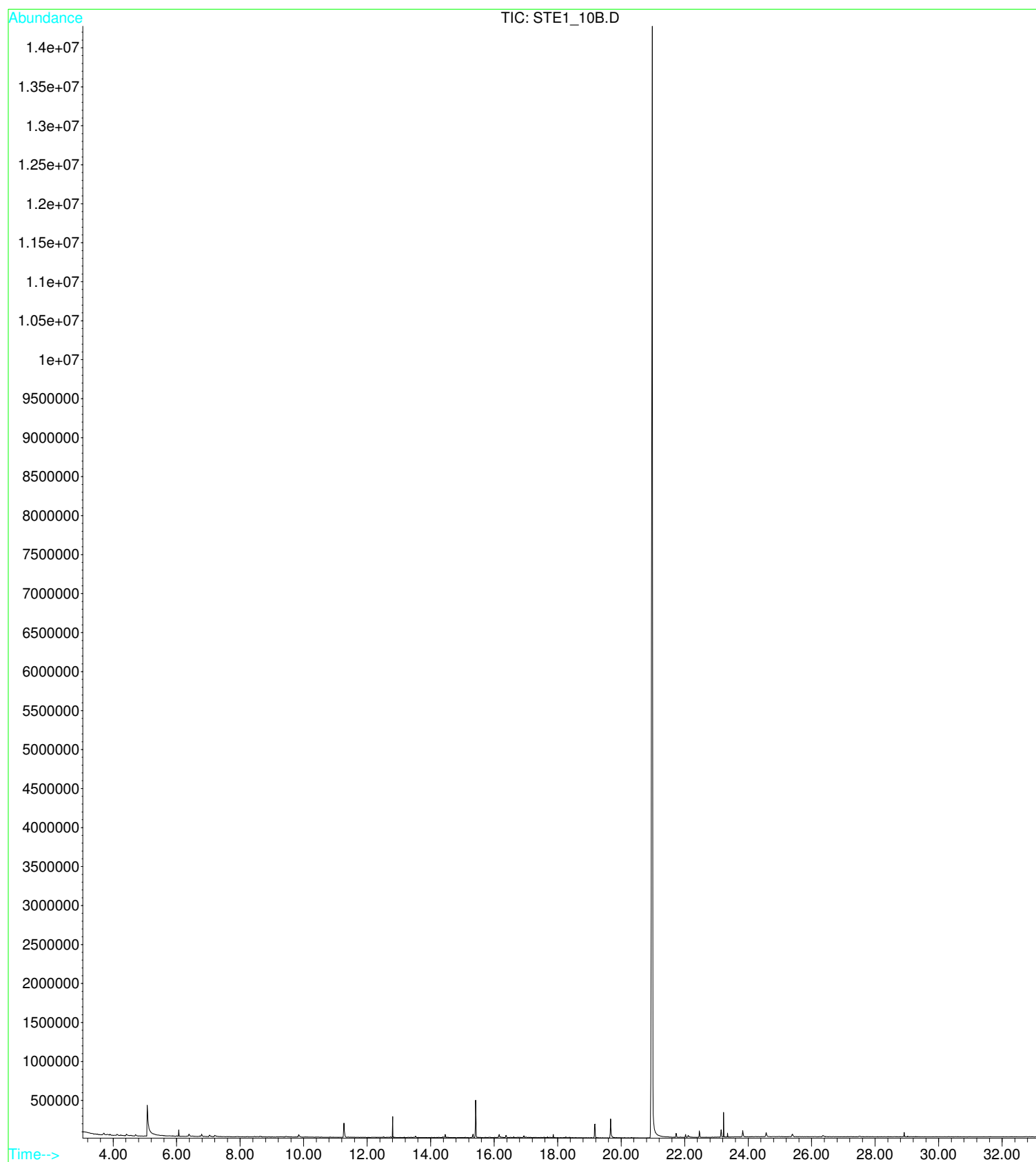
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Acquired : 28 Oct 2010 5:49 using AcqMethod GCMS
Instrument : DMS Instr
Sample Name: J5216 Jordi Stearamide 1to10 Dilution 5mg/ml
Misc Info :
Vial Number: 11



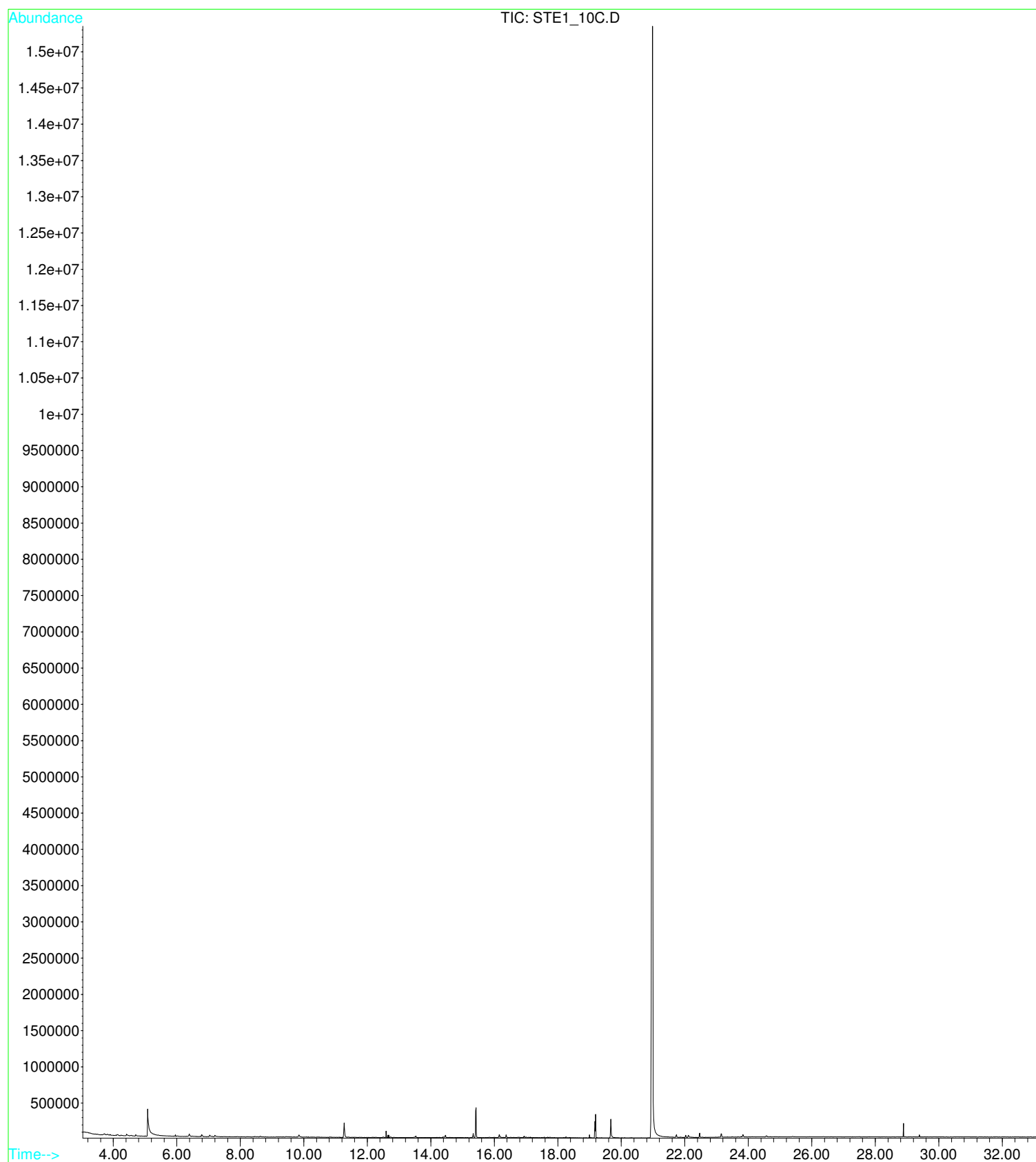
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Operator : Kimberly Vitiello
Acquired : 28 Oct 2010 13:35 using AcqMethod GCMS
Instrument : DMS Instr
Sample Name: J5216 Jordi Stearamide 1to10 Dilution 5mg/ml
Misc Info :
Vial Number: 11



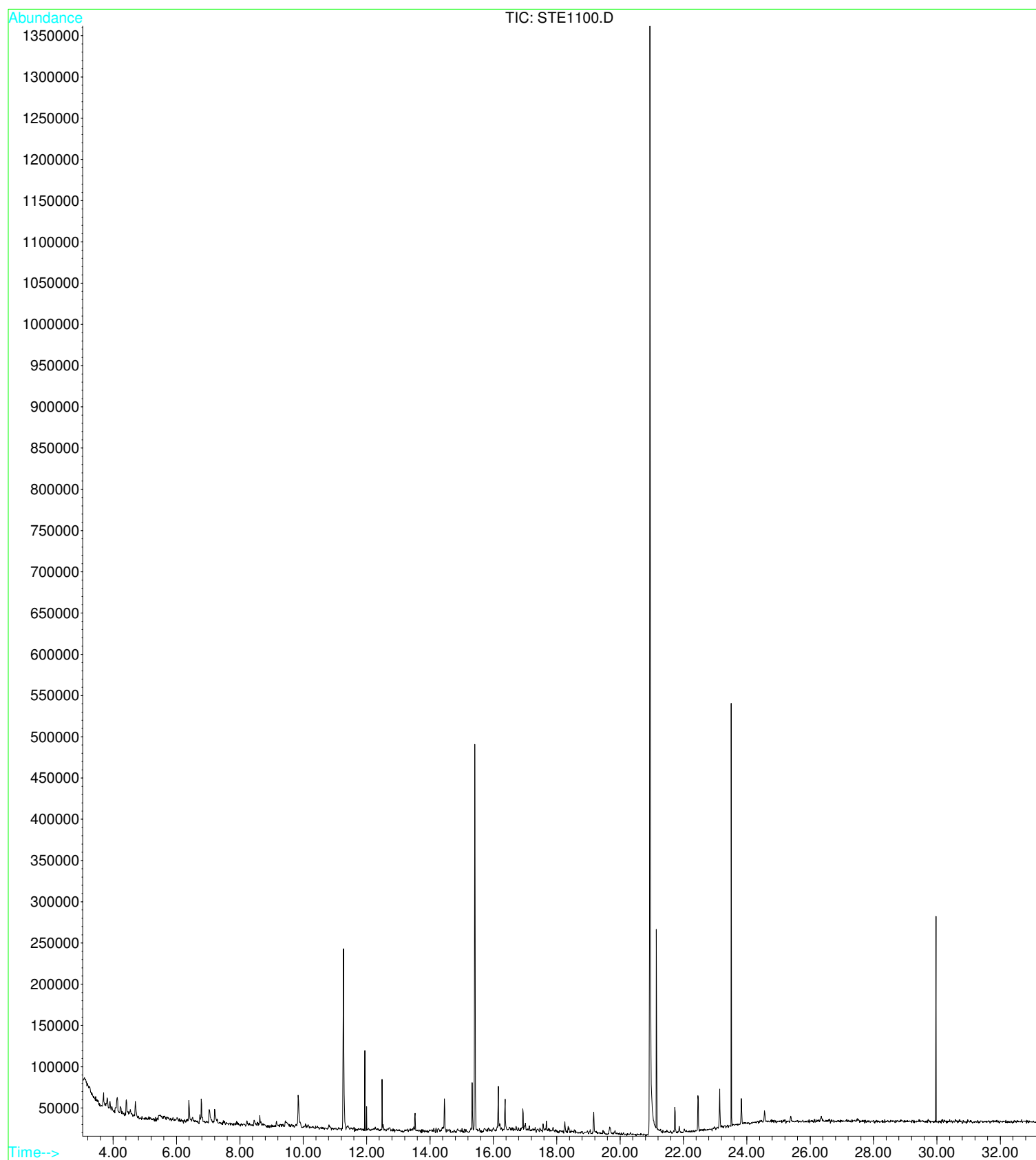
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Operator : Kimberly Vitiello
Acquired : 28 Oct 2010 14:18 using AcqMethod GCMS
Instrument : DMS Instr
Sample Name: J5216 Jordi Stearamide 1to10 Dilution 5mg/ml
Misc Info :
Vial Number: 11



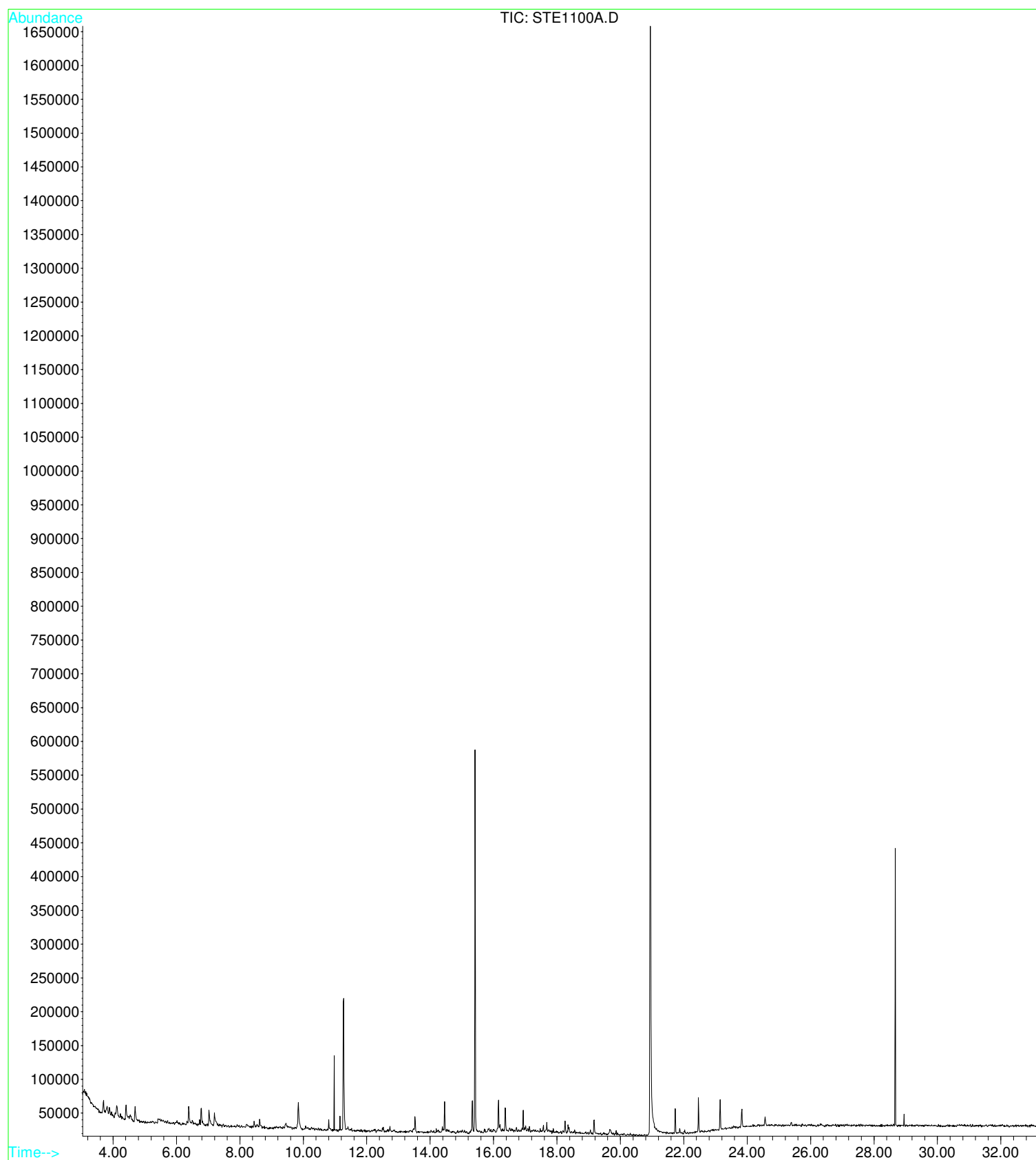
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Operator : Kimberly Vitiello
Acquired : 28 Oct 2010 3:42 using AcqMethod GCMS
Instrument : DMS Instr
Sample Name: J5216 Jordi Stearamide 1to100 Dilution 5mg/ml
Misc Info :
Vial Number: 12



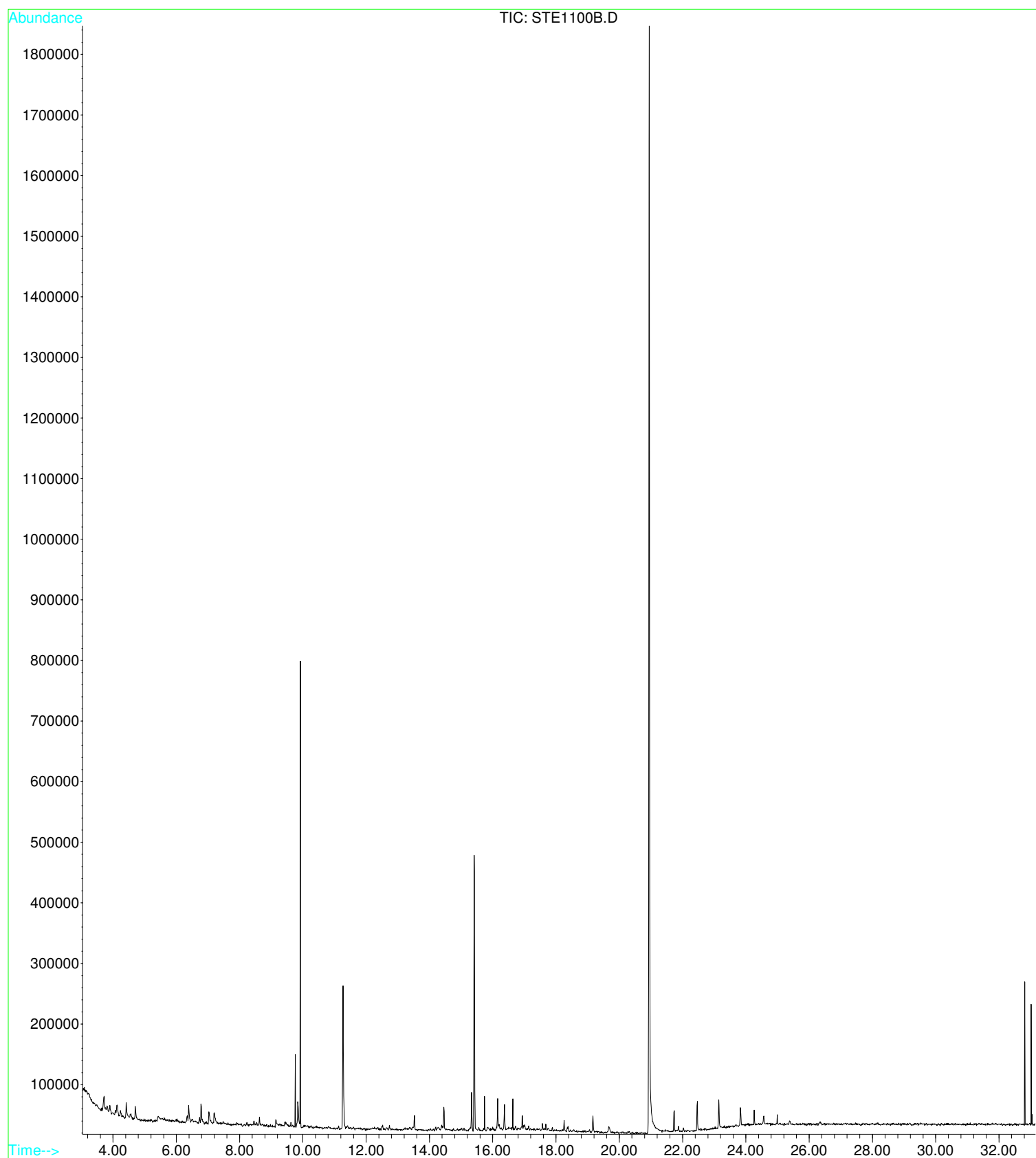
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1	20.939	rm	0.251	2302341	20.870	21.122

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Operator : Kimberly Vitiello
Acquired : 28 Oct 2010 4:24 using AcqMethod GCMS
Instrument : DMS Instr
Sample Name: J5216 Jordi Stearamide 1to100 Dilution 5mg/ml
Misc Info :
Vial Number: 12



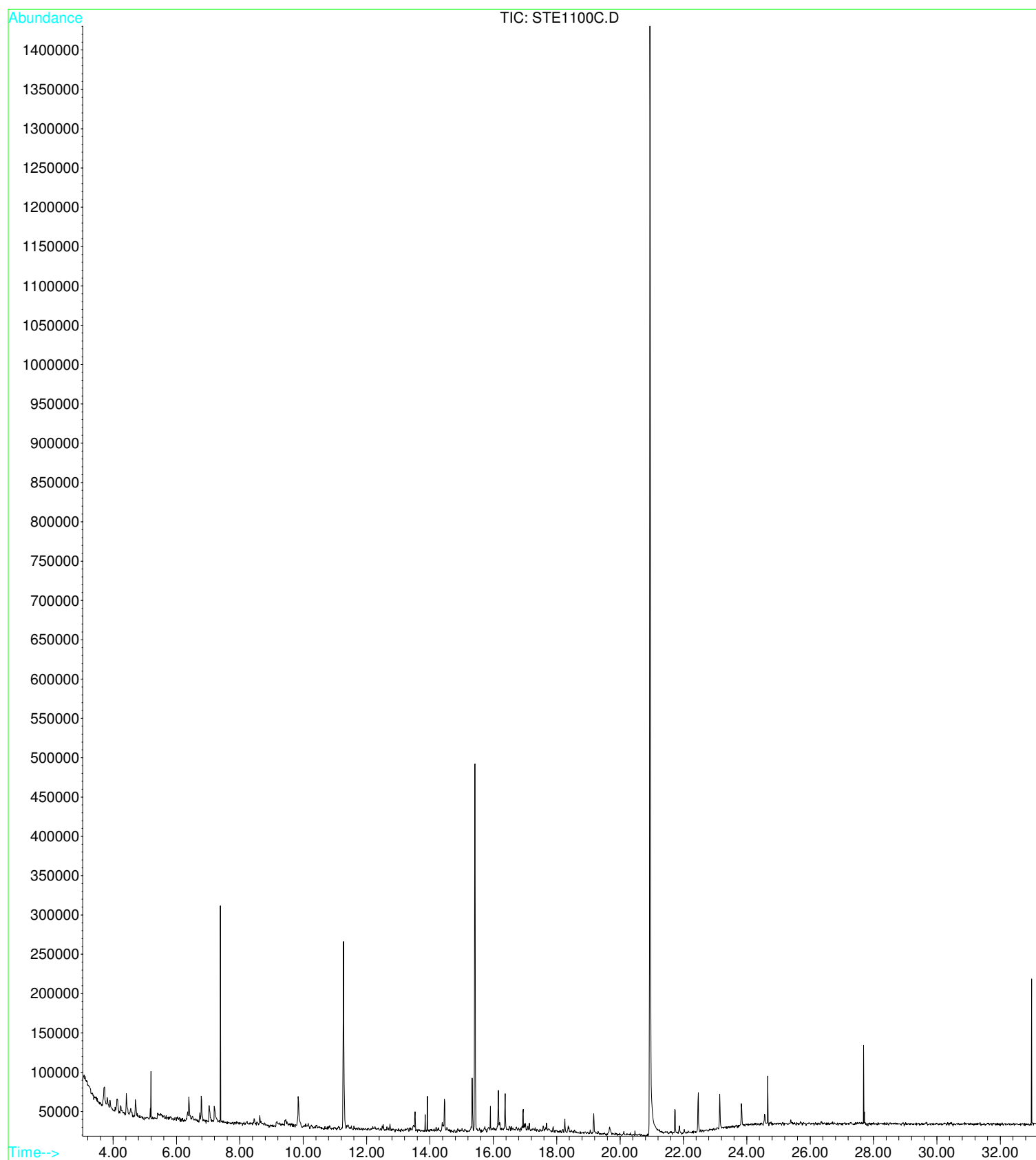
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Operator : Kimberly Vitiello
Acquired : 28 Oct 2010 12:09 using AcqMethod GCMS
Instrument : DMS Instr
Sample Name: J5216 Jordi Stearamide 1to100 Dilution 5mg/ml
Misc Info :
Vial Number: 12



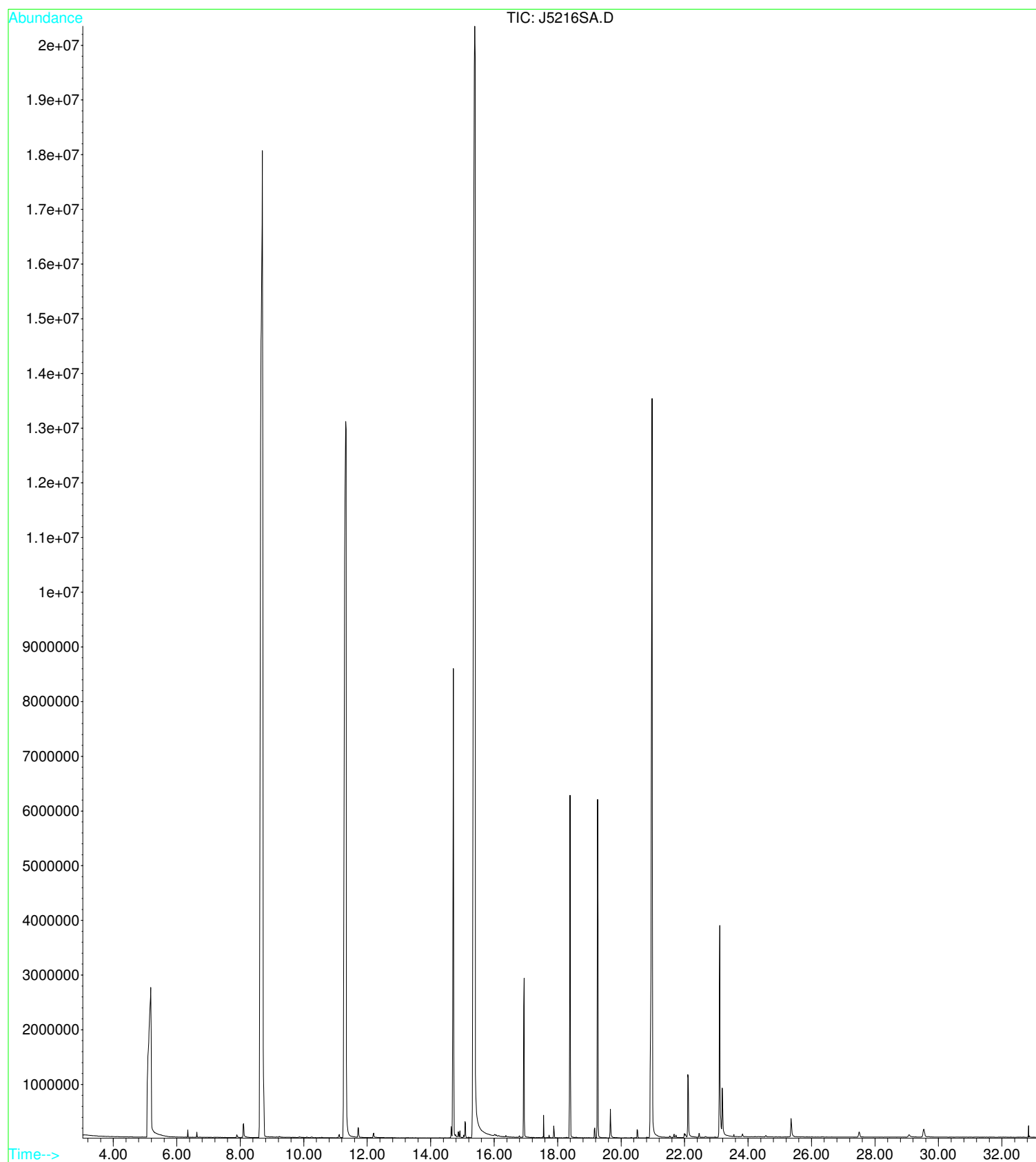
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1	20.949	rm	0.354	3219031	20.881	21.235

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Operator : Kimberly Vitiello
Acquired : 28 Oct 2010 12:52 using AcqMethod GCMS
Instrument : DMS Instr
Sample Name: J5216 Jordi Stearamide 1to100 Dilution 5mg/ml
Misc Info :
Vial Number: 12



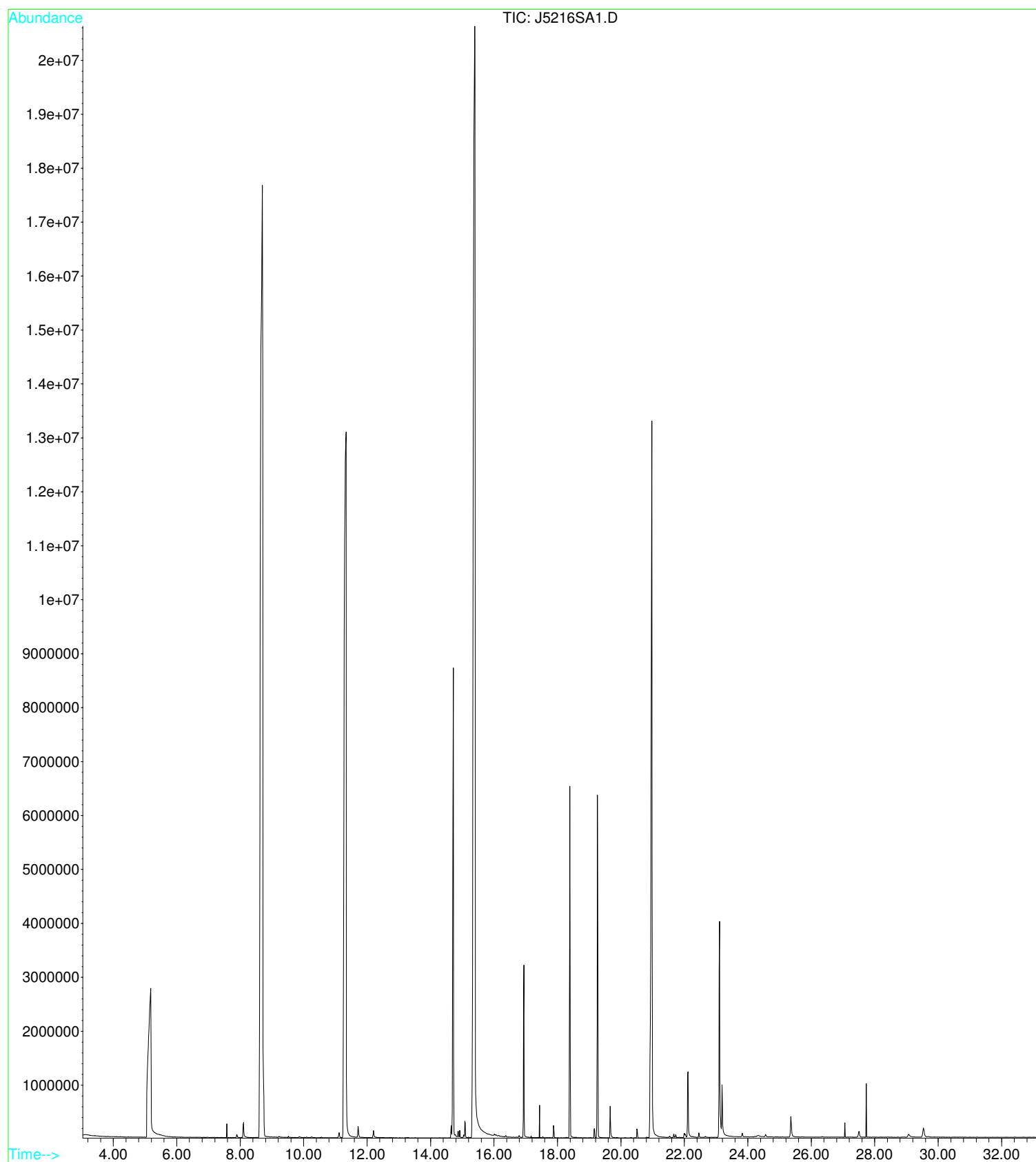
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1	20.940	rm	0.480	2589368	20.883	21.362

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Operator : Kimberly Vitiello
Acquired : 28 Oct 2010 8:37 using AcqMethod GCMS
Instrument : DMS Instr
Sample Name: J5216 Jordi Sample 5mg/ml Chloroform
Misc Info :
Vial Number: 13



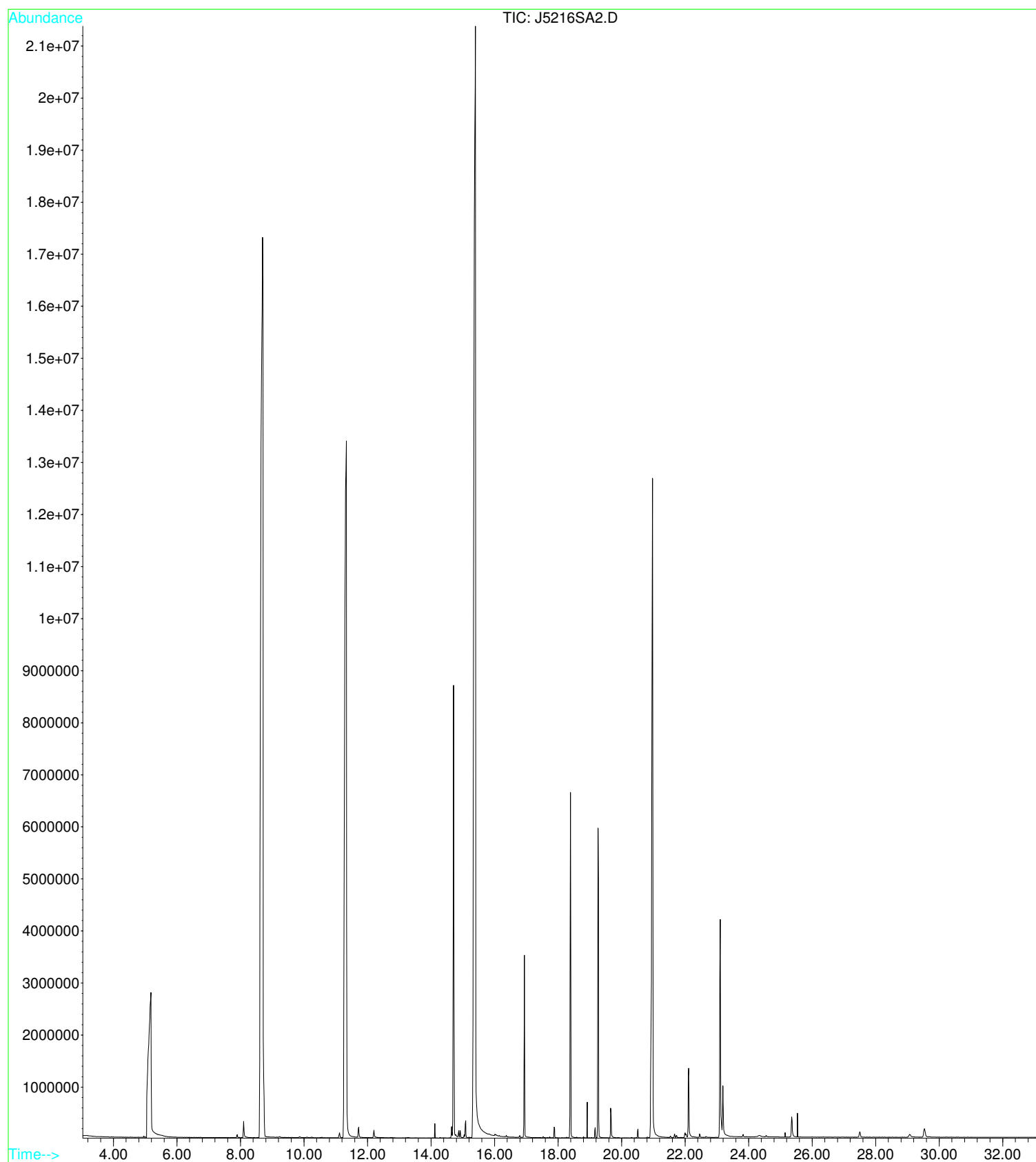
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Operator : Kimberly Vitiello
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Instrument : DMS Instr
Sample Name: J5216 Jordi Sample 5mg/ml Chloroform
Misc Info :
Vial Number: 13



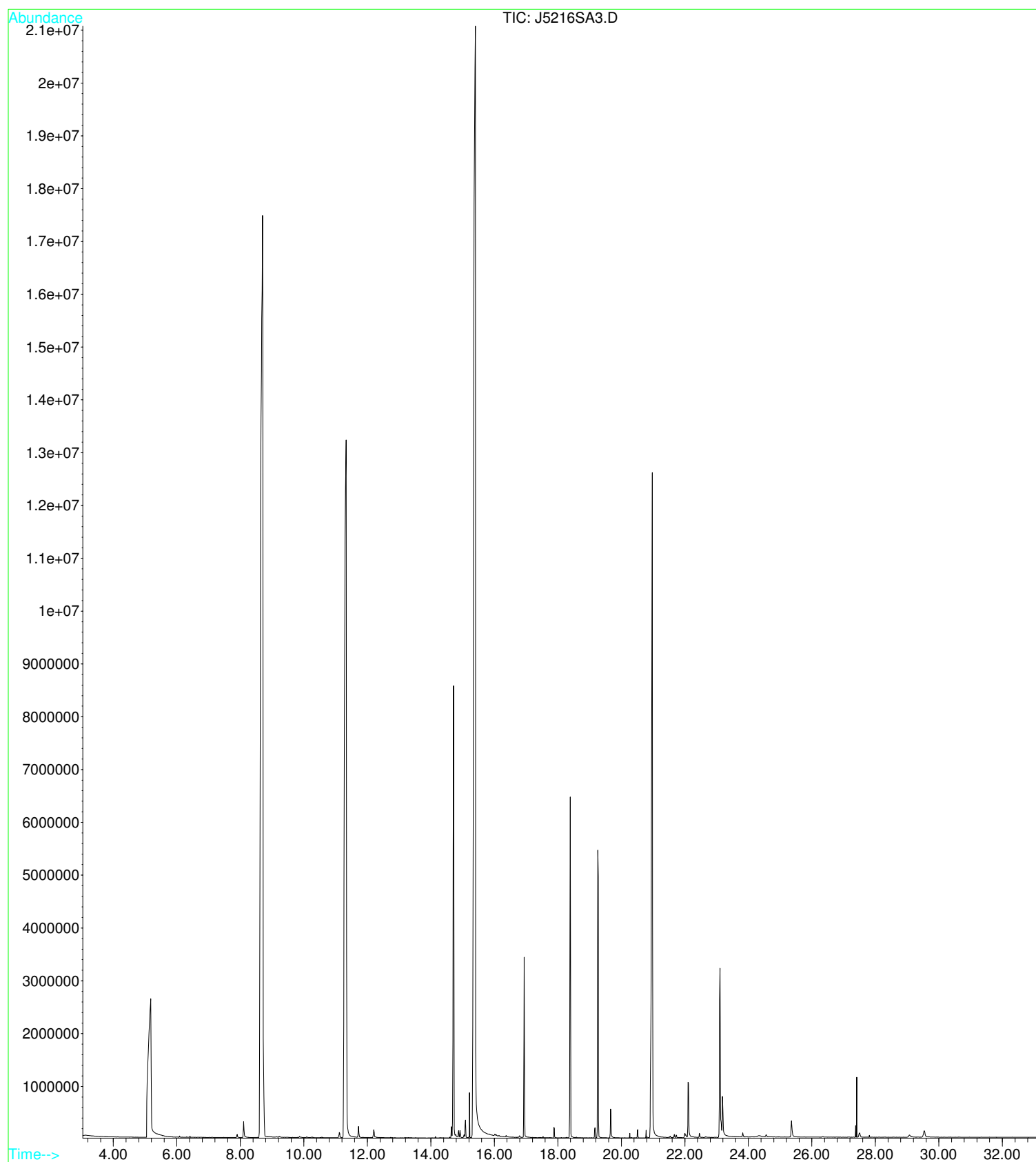
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Operator : Kimberly Vitiello
Acquired : 28 Oct 2010 10:02 using AcqMethod GCMS
Instrument : DMS Instr
Sample Name: J5216 Jordi Sample 5mg/ml Chloroform
Misc Info :
Vial Number: 13



Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	20.972	rm	0.297	31008662	20.869	21.166

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Operator : Kimberly Vitiello
Acquired : 28 Oct 2010 10:44 using AcqMethod GCMS
Instrument : DMS Instr
Sample Name: J5216 Jordi Sample 5mg/ml Chloroform
Misc Info :
Vial Number: 13



Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	20.973	rm	0.434	27570876	20.870	21.304