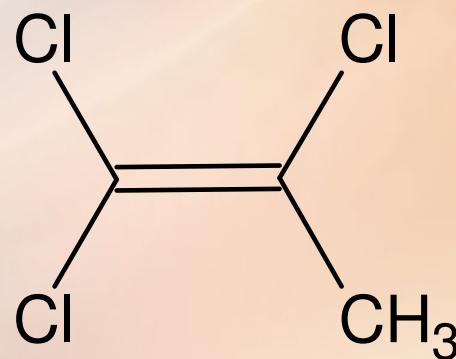


The Quantitation of Trichloroethene in Unknown Water Samples through the use of Gas-Chromatography/Mass Spectroscopy

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Introduction

- TCE is a widely used compound found in degreasers and cleaning agents. As a result of its widespread use, volatility, and limited water solubility, TCE is found extensively in the environment.
- The levels of TCE that are acceptable by the USEPA regulatory limits are 5 ng/mL in drinking water. This means that TCE is found in all forms of drinking water to a certain extent.
- The Environmental Engineering department is trying to create a way to react TCE with other chemicals in order to reduce the concentration found in drinking water.
- They tested a variety of chemicals and concentrations and then gave us the samples to be processed and analyzed.
- Gas Chromatography/ Mass Spectroscopy (GC/MS) was chosen as the process to analyze the samples.
- GC separates the chemicals by their boiling point and a significant peak should be observed.
- The intensity of the peak is determined by the concentration of the target compound.
- A ratio between the internal standard and the target compound allowed a linear calibration curve to be made to analyze the unknown compounds.



Methods: Sample Preparation

- Hexane was used as the solvent for all of the standards.
- Trichloroethene (TCE) was added to 25 mL of hexane.
- An internal standard of TCE that contained C¹³ was added to each unknown and standard sample.
- The concentration of the known samples was in parts per million (ppm). This formula is $\frac{\text{mass of sample}}{\text{total mass of solution}} \cdot 10^6$
- 25 mL of hexane contains 16.37 g hexane.
- The mass of one drop of TCE was found to be .0076 g.
- This value was used to create a 4642.6 ppm stock solution.
- This concentration was too large to work with so a dilution down to 1000 ppm was made.
- It was calculated that 3.52 g of the stock solution would be needed to make a 1000 ppm solution.
- The actual mass of the stock solution used was 3.546 g making the new solution 1005.67 ppm.
- The mass of one drop of TCE C¹³ was .0029 g. From this a stock solution of 1771.53 ppm was created.
- 9.27 g of this solution was added to 25 mL of hexane to create a 1003.4 ppm solution.
- The concentrations of 1, 5, 10, 20, 30, 40, 50, 70, 90 and 100 ppm were selected to form a calibration curve.
- 10 mL volumetric flasks contained each standard.

Methods: Sample Preparation

Estimated ppm	Amount of 1005.67 ppm added (μL)	Calculated ppm
1	10	1
5	49.8	5
10	99.5	9.99
20	199	19.99
30	299	30.04
40	398	39.99
50	498	50.04
70	697	70.04
90	896	90.04
100	995	99.99

Methods: Unknown Sample Preparation

- The samples received were already dissolved in hexane.
- 19.9 μL of the 1003.4 ppm TCE C13 solution was added to each unknown and known, to create an internal standard solution concentration of 19.95 ppm.

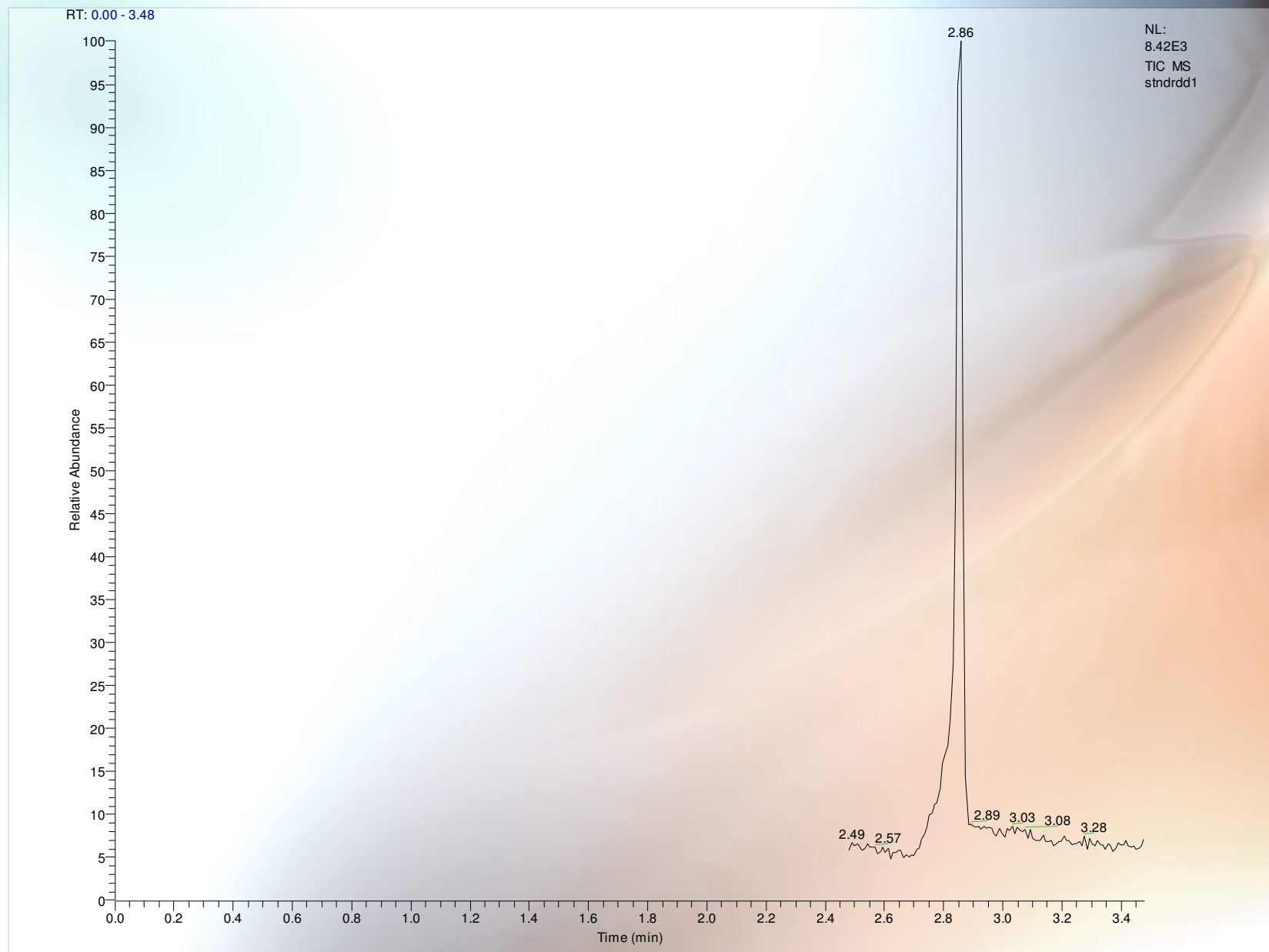
Methods: Instrument Configuration

- A Finnigan SSQ 710 mass spectrometer (MS) was used. It was coupled with a Varian 3400 gas chromatograph (GC) and a Finnigan A200S auto sampler.
- Selected Ion Monitoring (SIM) was conducted.
- A selected mass of 133 amu was selected with a range of ± 5 amu.
- The filament was set with a delay of 2.4min.
- The pressure of the helium gas flowing through was 15 psi. The tank to inlet valve pressure was 60 psi.
- The column temperature was set to 45°C. The injector and transfer line temperature were set to 250°C. The manifold pressure when the tests were ran was $6.5 \cdot 10^{-6}$ torr.
- The ion source pressure was 30.42 millitorr. A pressure of 25.81 millitorr was recorded for the forepump.
- 1 μL of each sample was injected using the A200S auto sampler. A SGE syringe was used with a total volume of 5 μL .
- The total amount of time for each test was 3.5 minutes. The GC had a stabilization time of .3 min.

Methods: Instrument Configuration

- The standard deviation of the instrument was checked with a 1ppm sample of TCE.
- This sample was tested 10 times.
- The relative standard deviation was 3.39%.

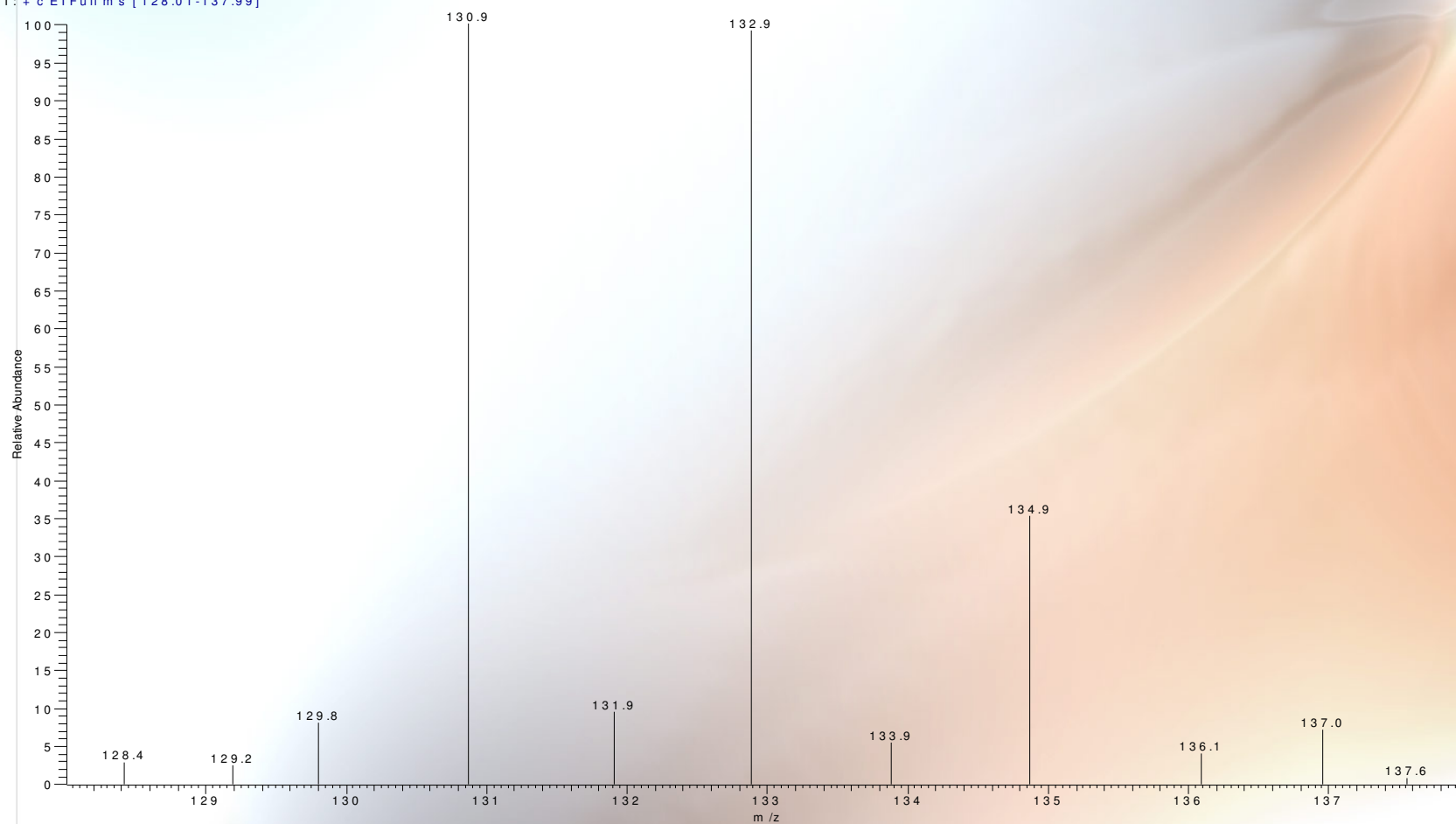
Methods: Instrument Configuration



Methods: Instrument Configuration

- The mass spectrum of the peak detected by the GC.

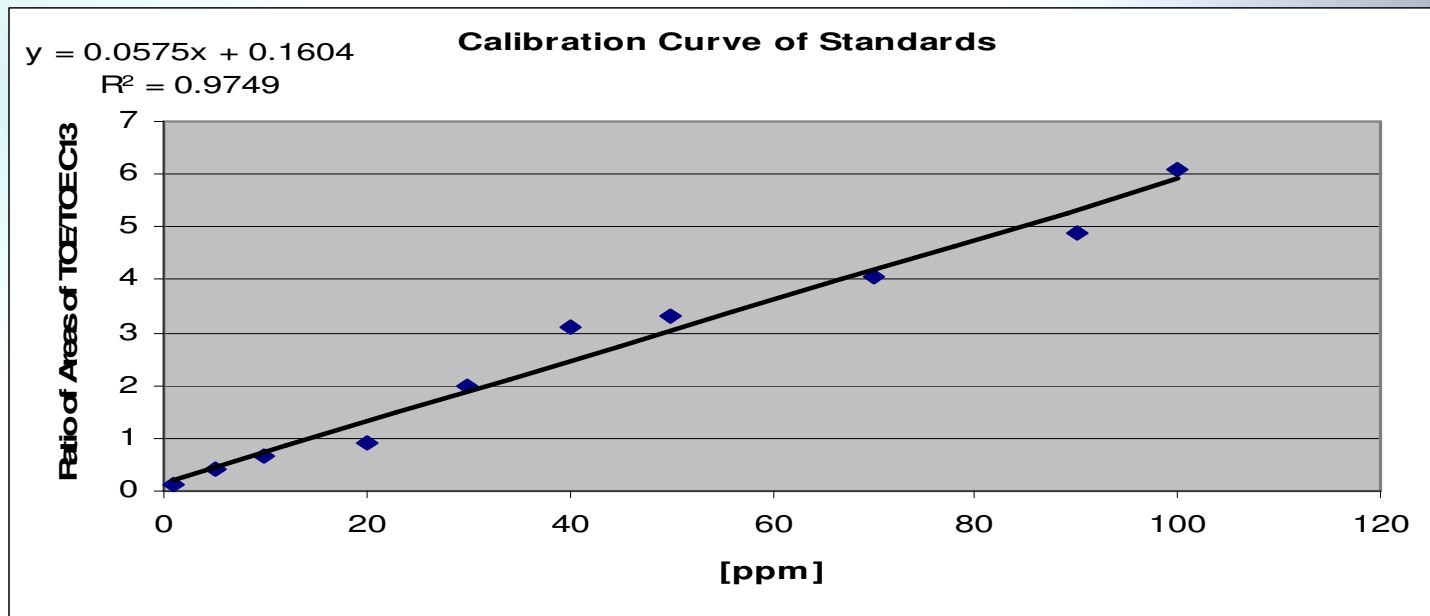
s t n d r d d 1 # 3 8 - 4 7 R T : 2 . 8 0 - 2 . 8 8 A V : 1 0 N L : 1 . 2 2 E 3
T : + c E I F u l l m s [1 2 8 . 0 1 - 1 3 7 . 9 9]



Results and Discussion

- Two programs were used for the calibration curves.
- Excel
- Xcalibur

Results Discussion: Excel



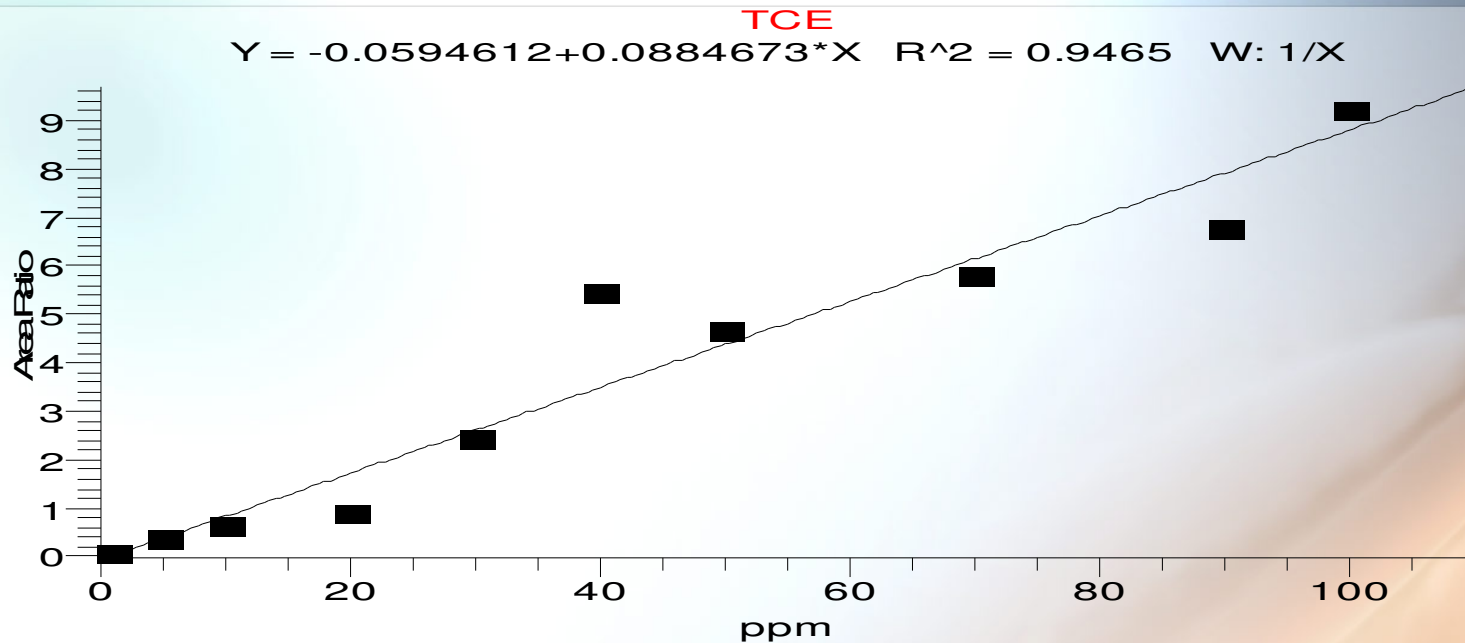
- A standard interval of the intensity of each peak was chosen.
- The sum of the TCE peaks was compared to the sums of TCE C¹³ peaks.
- This ratio was plotted against the concentration of each standard.

Results and Discussion: Excel

- Using this curve the concentrations of each unknown were calculated using the best fit equation.
- The same interval was chosen for the ratio of TCE to TCE C¹³.

Unknown	[ppm]
C1	718.9
C2	104
C3	201.3
H1	114.4
H3	113.1
H5	141.9
H7	109.8
H12	156.1
H24	105
H.01	188.8
H.03	102
L1	118.8
L3	159.5
L7	142.1
L12	73.9
L24	144
L.01	107.3
L.03	98.52
M1	135.1
M3	108.2
M5	187.1
M7	138.2
M12	108
M24	140.6
M.01	131
M.03	98.43

Results and Discussion: Xcalibur



- Xcalibur integrates the area of each peak to form the curve.
- It calculates and plots the ratio of TCE to TCE C¹³.

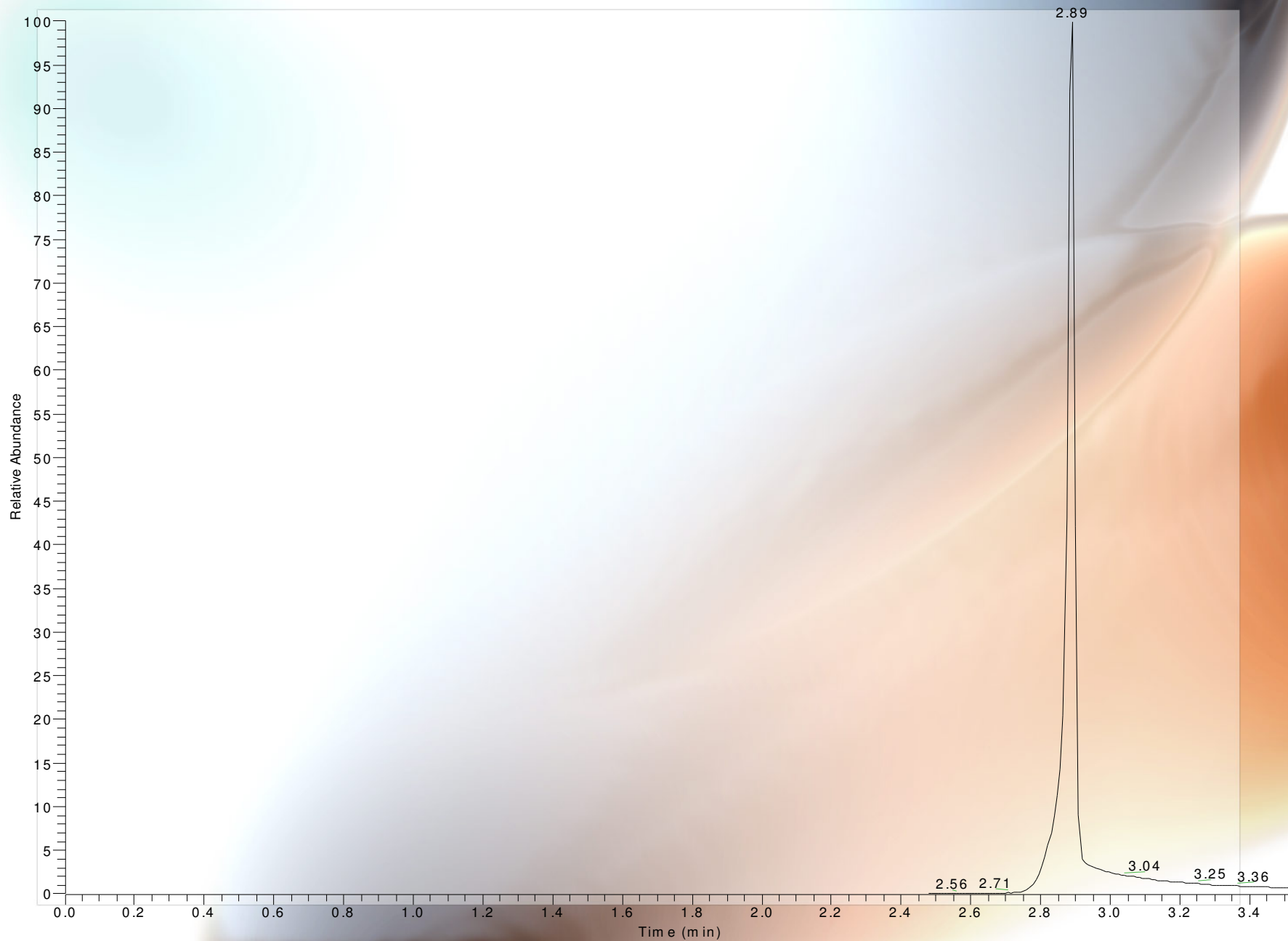
Results and Discussion: Xcalibur

- The concentrations of the unknowns were calculated by the program.
- They closely matched those calculated by Excel.
- A more accurate representation of the data.

Filename	Calc Amt (ppm)	%RSD
c1		
c2	118.338	10.9%
c3	220.679	9.1%
h01	205.367	9.2%
h03	110.508	11.3%
h1	116.631	11.0%
h12	175.773	9.5%
h24	126.987	10.5%
h3	125.959	10.6%
h5	168.601	9.5%
h7	121.144	10.8%
l01	120.387	10.8%
l03	109.560	11.3%
l1	127.527	10.5%
l12	75.909	14.6%
l24	165.579	9.6%
l3	203.762	9.2%
l7	152.232	9.8%
m01	133.578	10.3%
m03	112.253	11.2%
m1	158.175	9.7%
m12	115.452	11.0%
m24	160.055	9.7%
m3	118.566	10.9%
m5	214.295	9.1%
m7	161.076	9.7%

C1 Peak VS 100 ppm peak

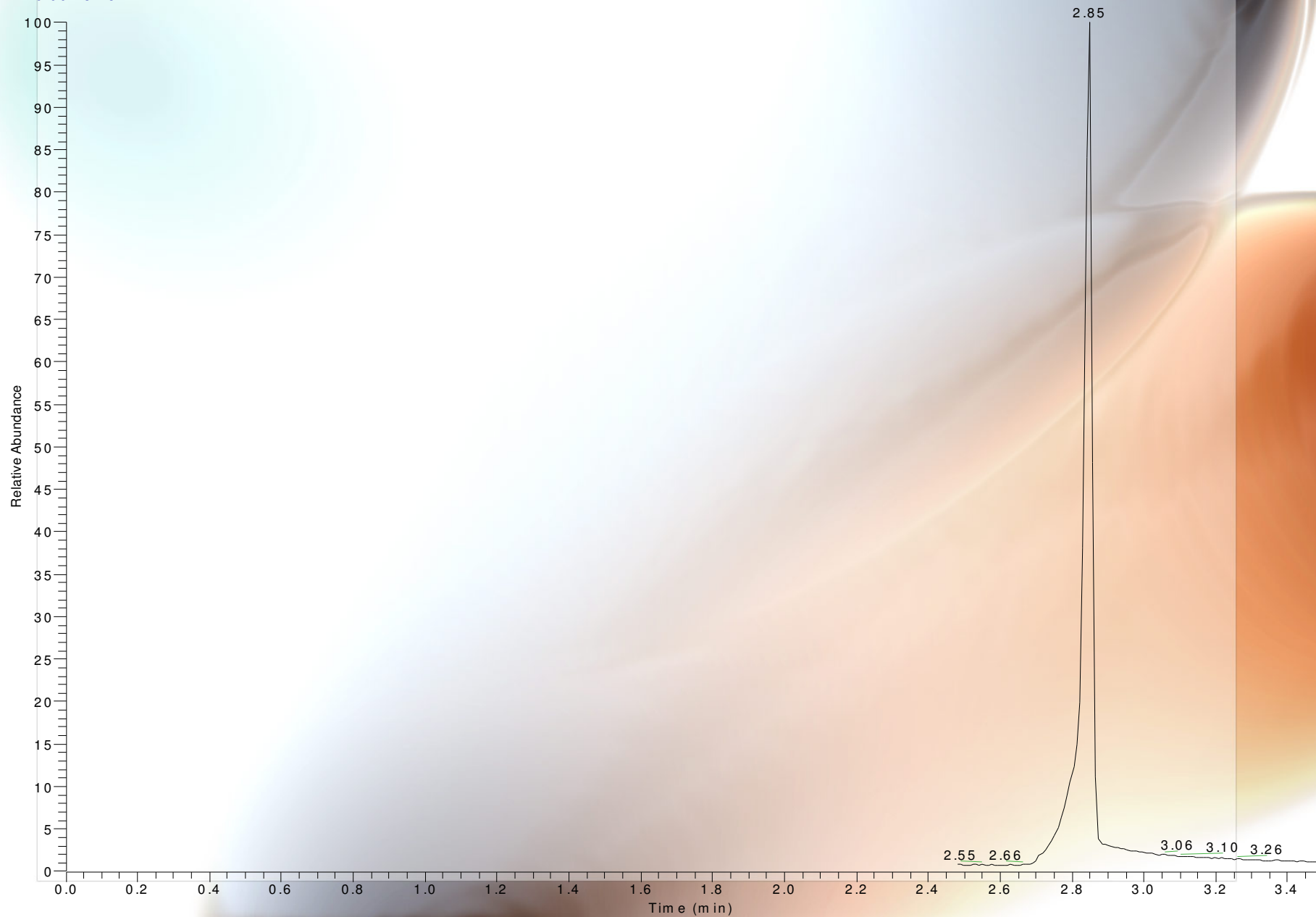
RT: 0.00 - 3.53



NL:
1.85E6
TIC MS c1

C1 Peak VS 100 ppm peak

RT: 0.00 - 3.48



NL:
3.29E5
TIC MS
quanti

Conclusion

- Although results were obtained the values were not sufficient.
- Xcalibur proved to be a more precise method of analysis.
- TCE signals were picked up by the GC/MS method.
- This method could be used to analyze other water pollutants.

Future Work

- **Using the data found in this trial, a new calibration curve could be made.**
- **Comparing this data to a Direct Liquid Injection method.**
- **Using this data as a model for conducting other tests with different types of pollutants.**

Acknowledgements

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References

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