MILLENNIUM APPLICATION BRIEF

TOPIC: Analysis of Bezoylperoxide in Acne Medications by LC-MS

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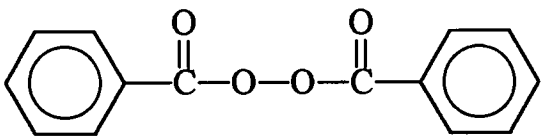
INTRODUCTION:

Benzoylperoxide (BP) is typically the main ingredient in a majority of over-the-counter acne medicines. Analysis of BP is of interest in verifying the main active ingredient in the medicines as well as identifying any other contaminants which may be present. BP is known to be thermally labile and can not be easily detected by GC-MS or SFC-MS. Analysis of BP samples was possible in under 5 minutes using the Integrity LC-MS system. Benzoylperoxide (shown below) has a molecular weight of 242 but fragments quite nicely as shown in figure. A molecular ion at mass 242 is also observed as shown in figure 2 which is a close-up of the molecular ion region of the mass spectra. This is unexpected since the peroxide bond is fairly fragile and is easily suspect to breaking, however by optimizing the MS interface for this compound a molecular ion was observed. Analysis of a BP standard and several samples was done with MS reports attached (figures 3-6). Figure 4 is a report for a BP standard at mass 242 and 122 showing the difference in intensity of the molecular ion (mass 242) and a fragment ion (mass 122) when extracted at each of those masses. The other reports are for a BP sample, Clearasil sample and Oxy-10 sample which were obtained at local stores. As you can see in the reports peak confirmation was easily obtained with the triple plot of the spectra obtained, library match spectra and their difference reported.



Benzoylperoxide (FW=242)

SAMPLE PREPARATION:

Approximately 0.12 grams of sample was weighed out into a 10.0 mL volumetric flask. To that 0.5mL of H2O was added and the sample agitated until dissolved. The sample was then brought to volume with acetonitrile. This mix was filtered and 0.3 mL of the solution diluted to 1.0 mL with acetonitrile and injected onto the HPLC.

EXPERIMENTAL:

MS Analysis

System:	Integrity LC-MS System
Mobile Phase:	Water:Methanol (40/60)
Column:	Waters Nova-Pak C18 (2.0 X 150mm)
Flow Rate:	0.25 mL/min
Detection:	MS: SCAN Mode from 50-260 m/z
Data:	Millennium 2010 Chromatography Manager, Ver. 2.2beta

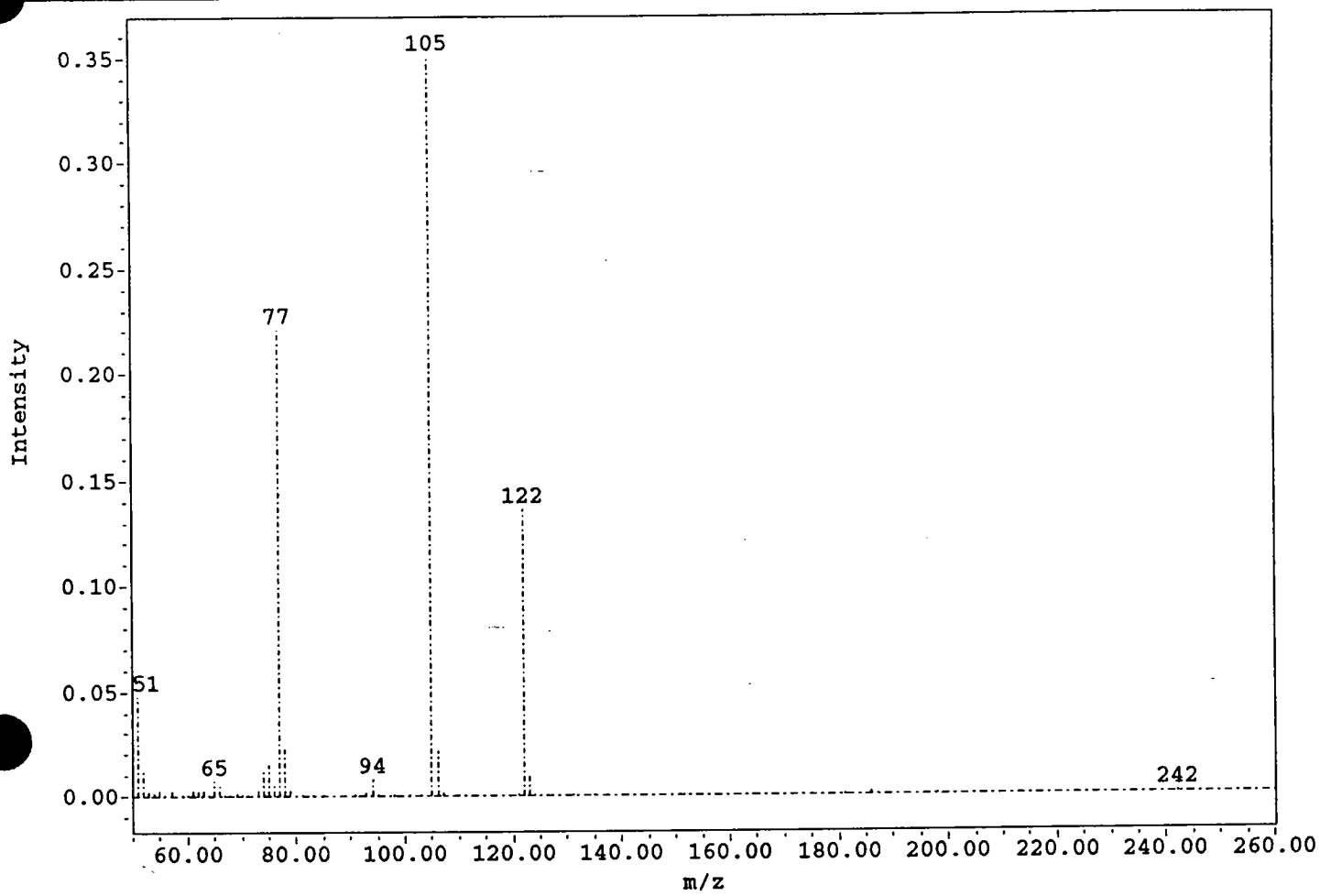
FIGURES:

Figure 1:	Mass spectra for benzoylperoxide
Figure 2:	Close-up of mass spectra region around molecular ion (mass 242)
Figure 3:	MS report for BP standard
Figure 4:	Comparison of extracted chromatograms at mass 122 and 242 for BP standard
Figure 5:	MS report for BP sample
Figure 6:	MS report for Clearasil sample
Figure 7:	MS report for Oxy 10 sample

Millennium Spectrum Review Report

Millennium v2.20GotTheTickIT

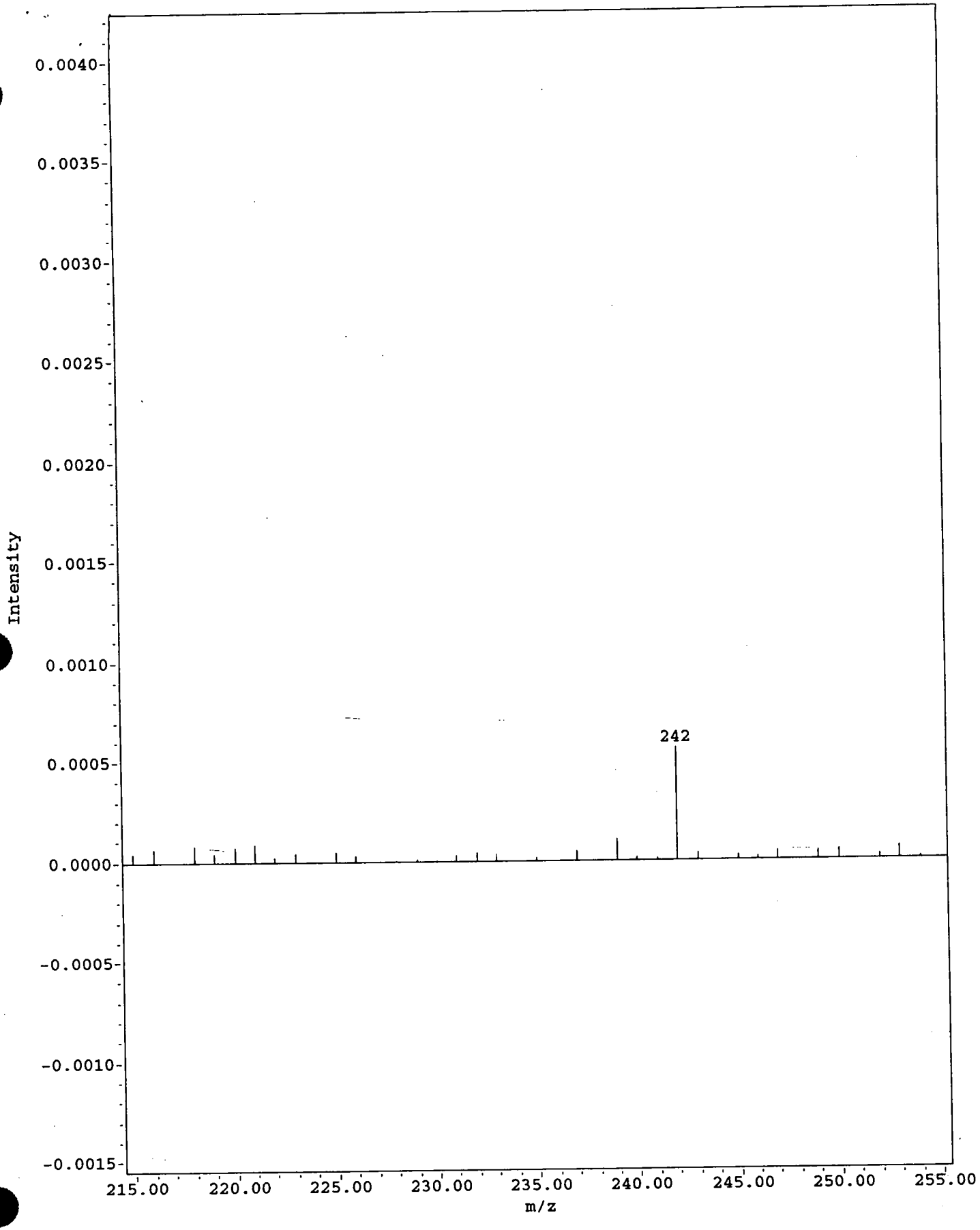
Date Printed: 11:01:34 AM, August 14, 1995



---- 4.287 minutes, 50 - 260 m/z, from BP Std-10 uL

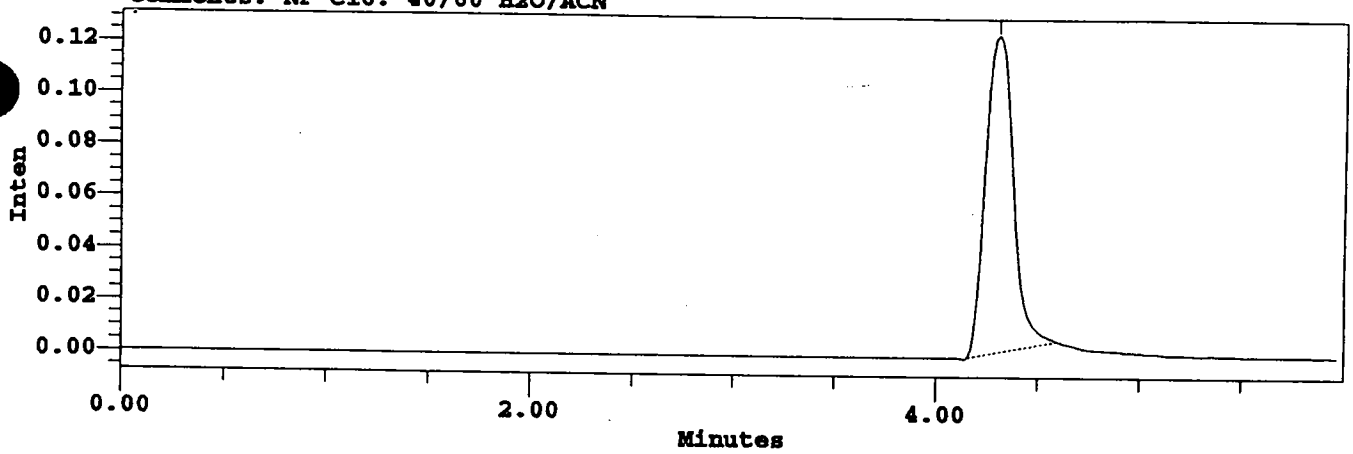
Spectral Table

#	Retention Time	Source	Baseline Correct	Searchable	Traceable	Base Peak	Maximum Intensity	Start Mass	End Mass
1	4.287	BP Std-10 uL	On	Yes	Yes	105	0.351	50	260



Millennium PDA Spectrum Plot - SampleName BP Std-10 uL, Vial 2, Inj. 1

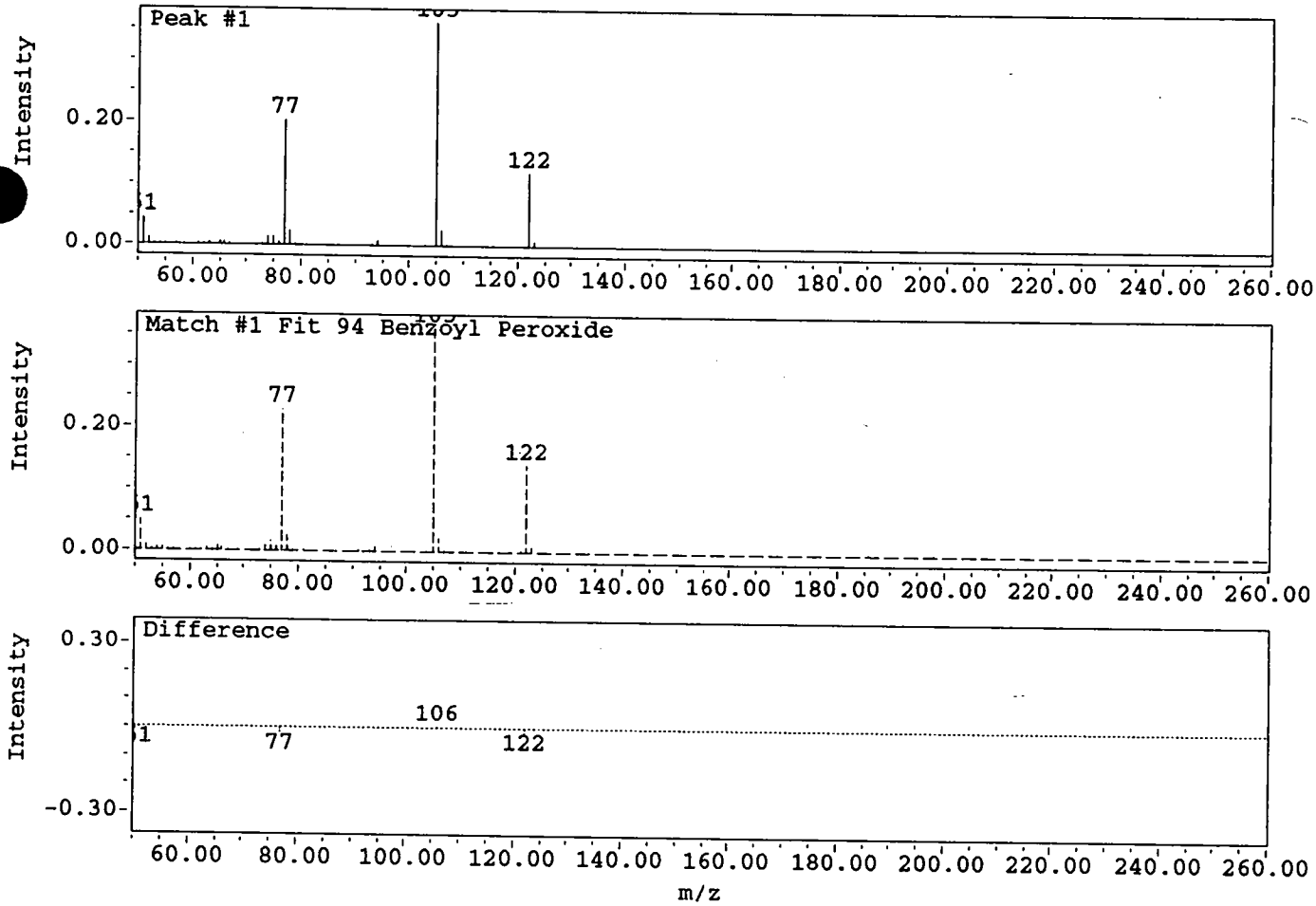
SampleName: BP Std-10 uL Channel Name: mass122
Comments: NP C18: 40/60 H2O/ACN



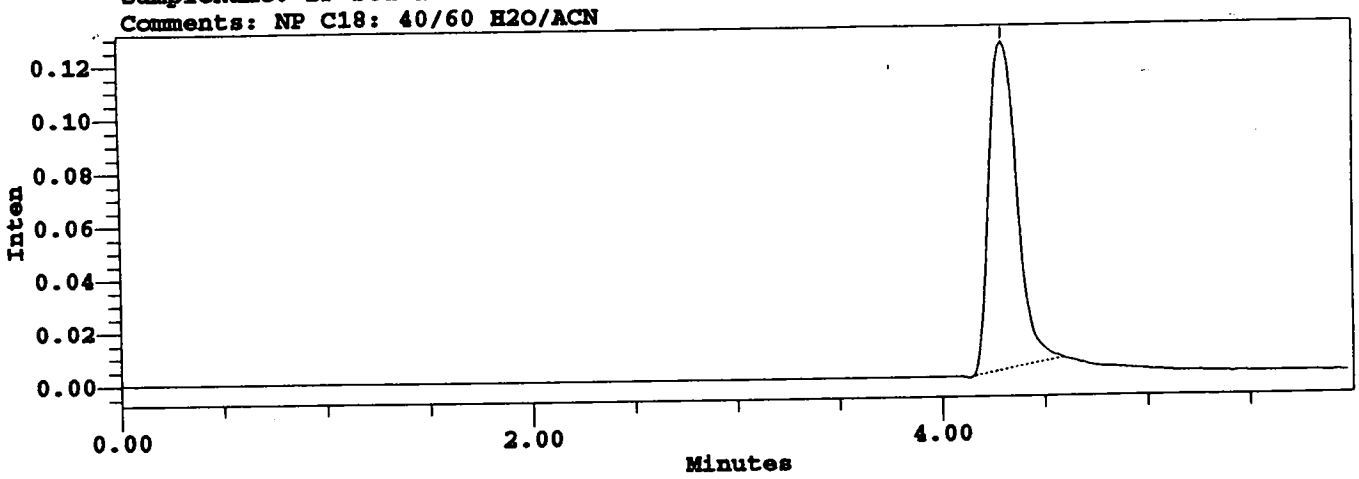
Mass Spec Info

#	Retention Time (min)	Area (uV*sec)	Height (uV)	Match1 PBM Fit	Match1 % Contam.	Match1 Spect Name
1	4.302	1156748	122734	94	0	Benzoyl Peroxide

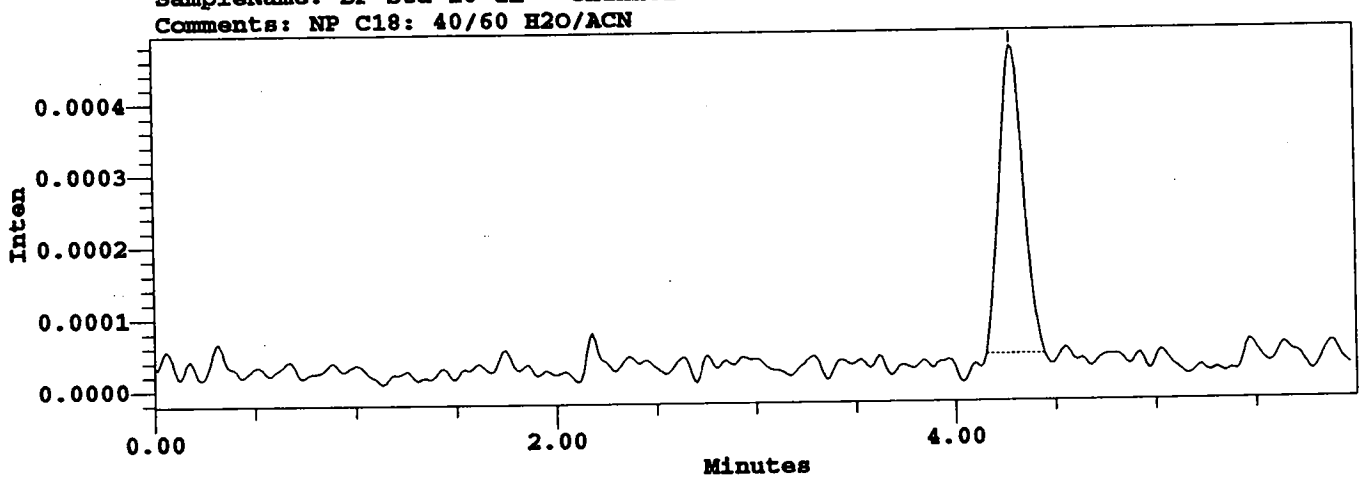
Match Plot Peak 1: Benzoylperoxide RT: 4.30

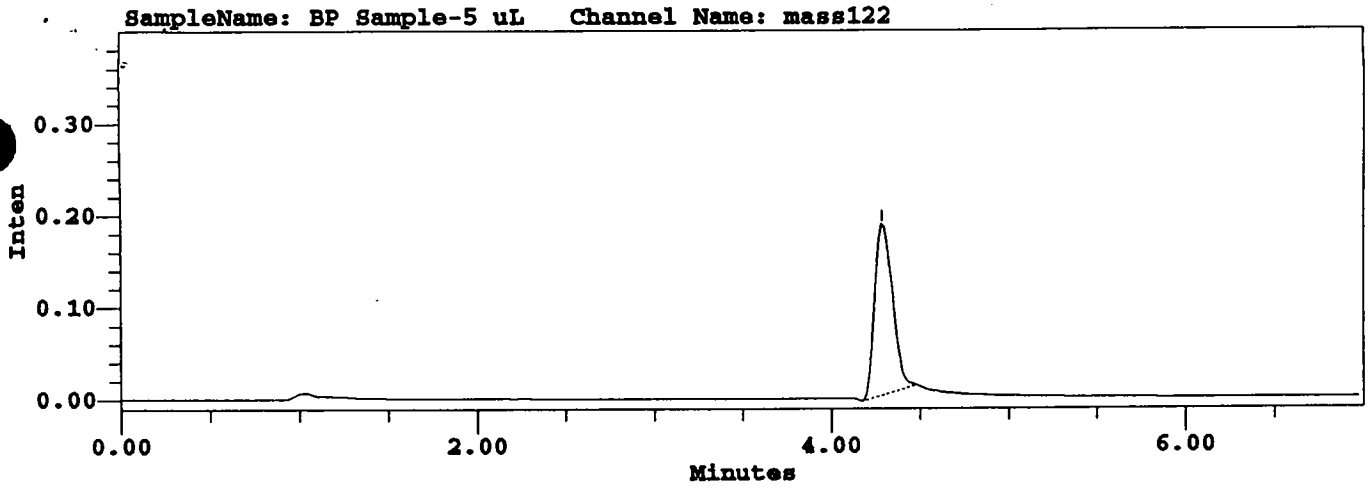


SampleName: BP Std-10 uL Channel Name: mass122
Comments: NP C18: 40/60 H2O/ACN



SampleName: BP Std-10 uL Channel Name: mass242
Comments: NP C18: 40/60 H2O/ACN

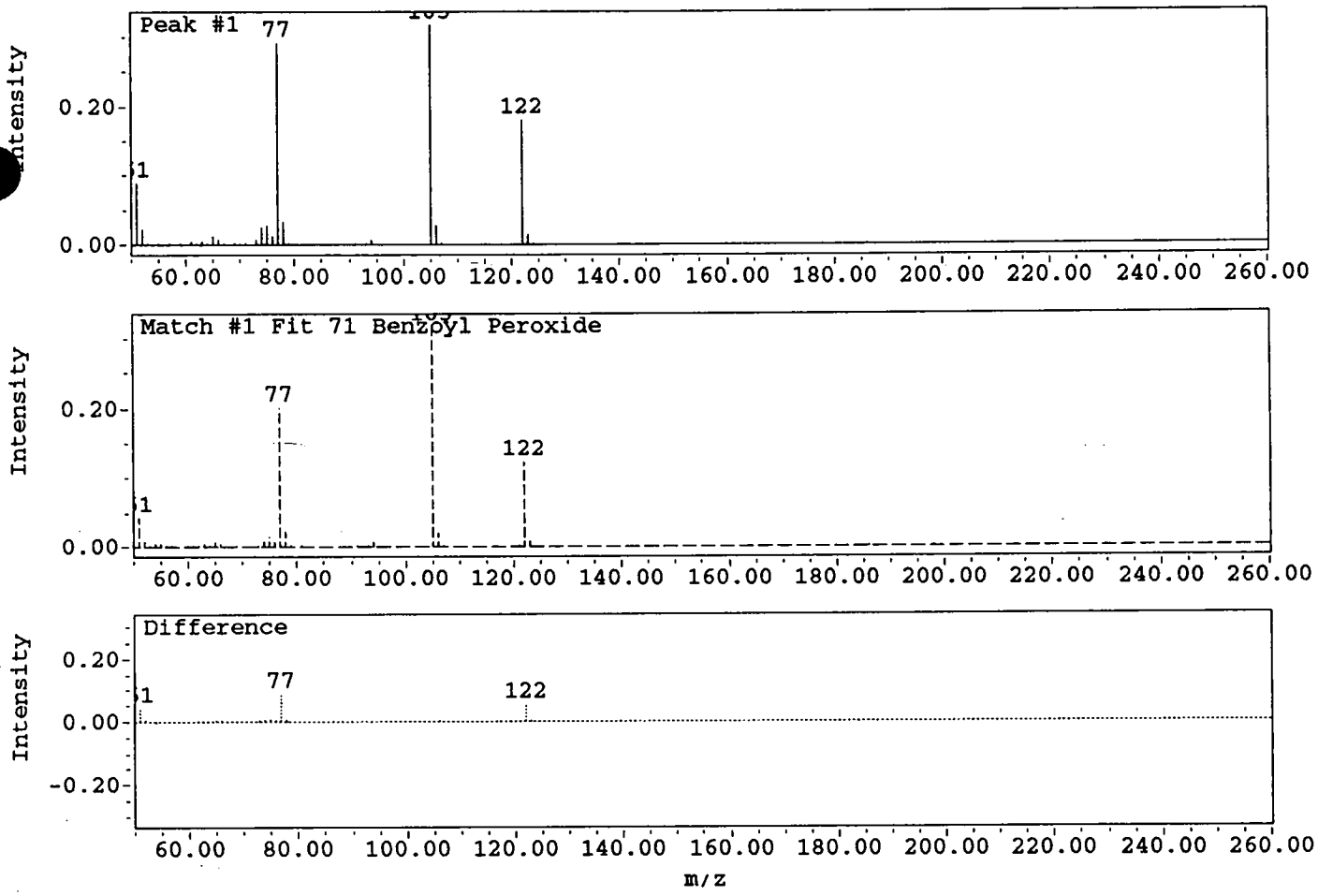


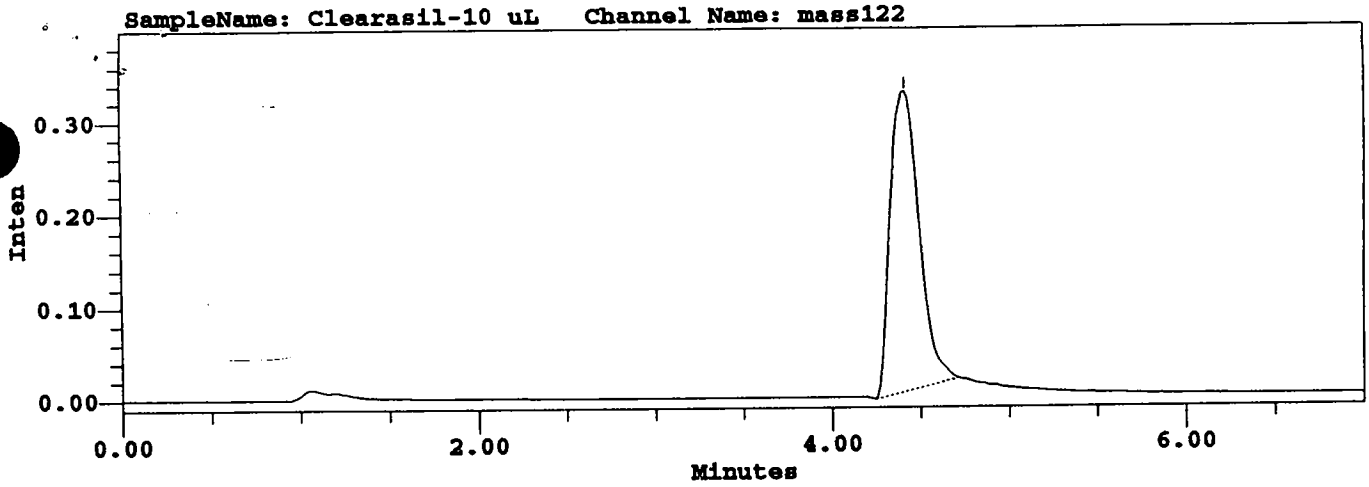


Mass Spec Info

#	Retention Time (min)	Area (uV*sec)	Height (uV)	Match1 PBM Fit	Match1 % Contam.	Match1 Spect Name
1	4.287	1295132	185941	71	0	Benzoyl Peroxide

Match Plot Peak 1: Benzoylperoxide RT: 4.29

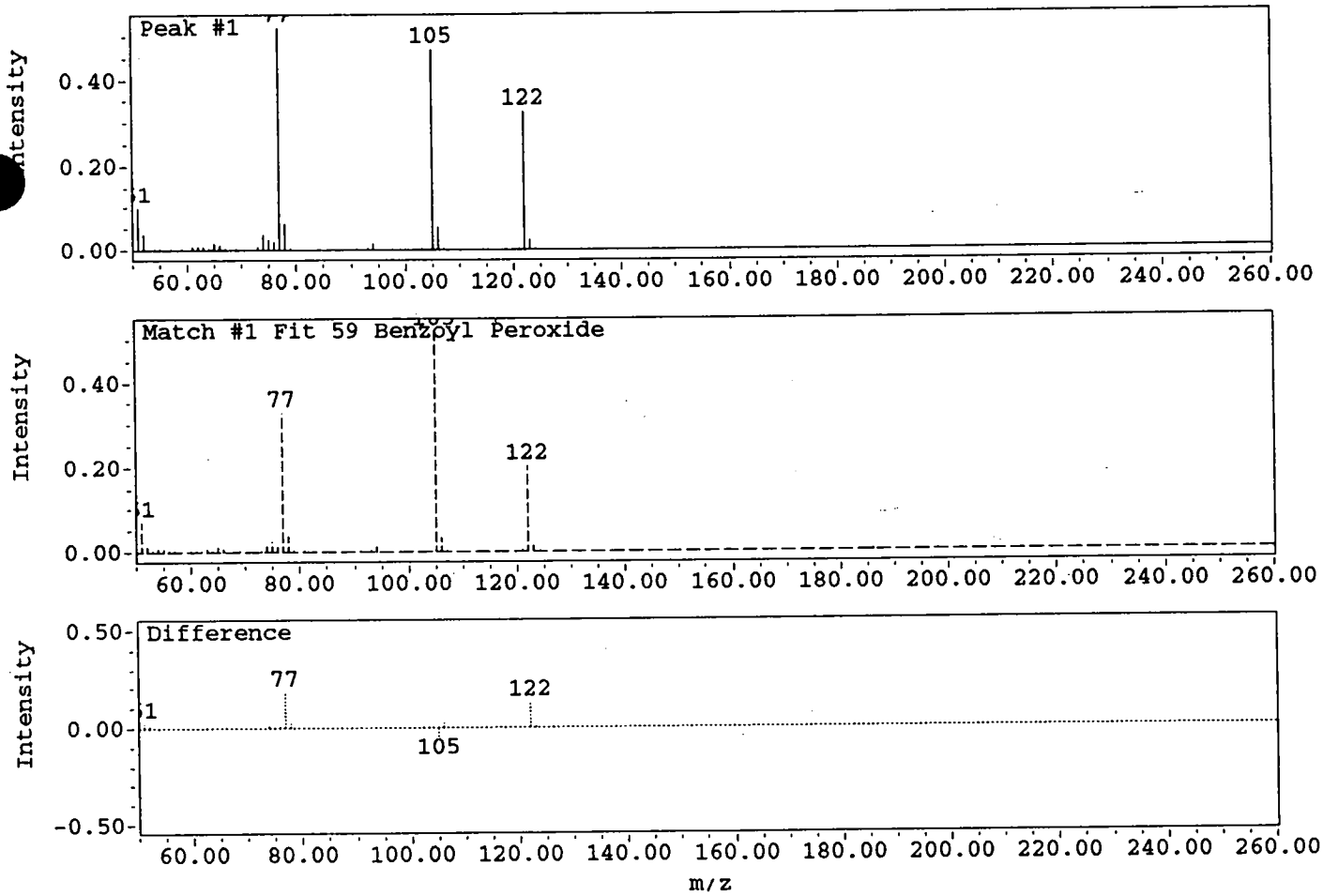


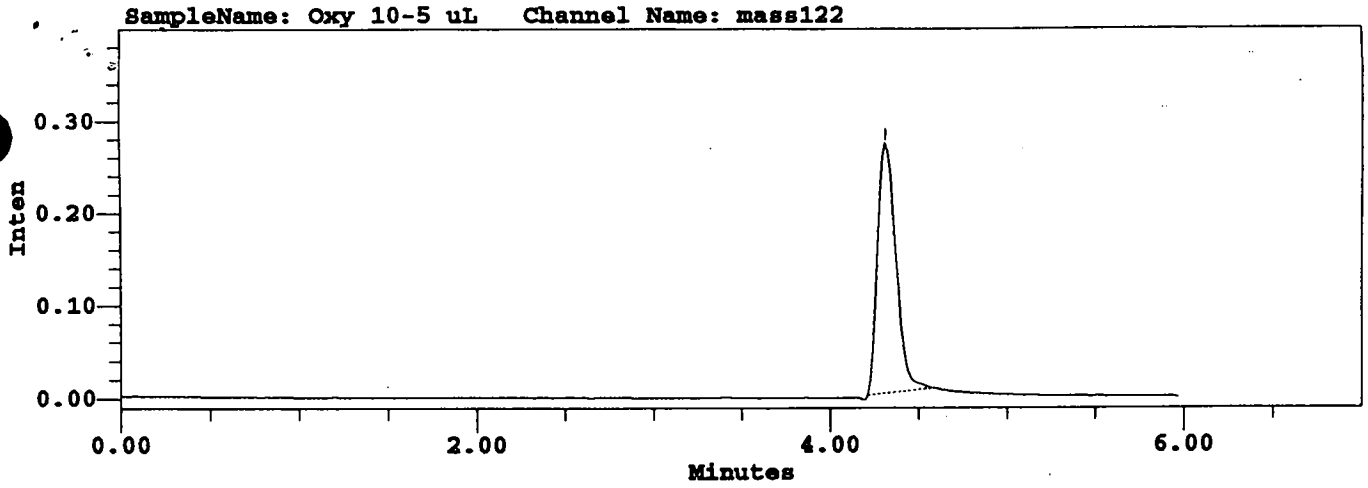


Mass Spec Info

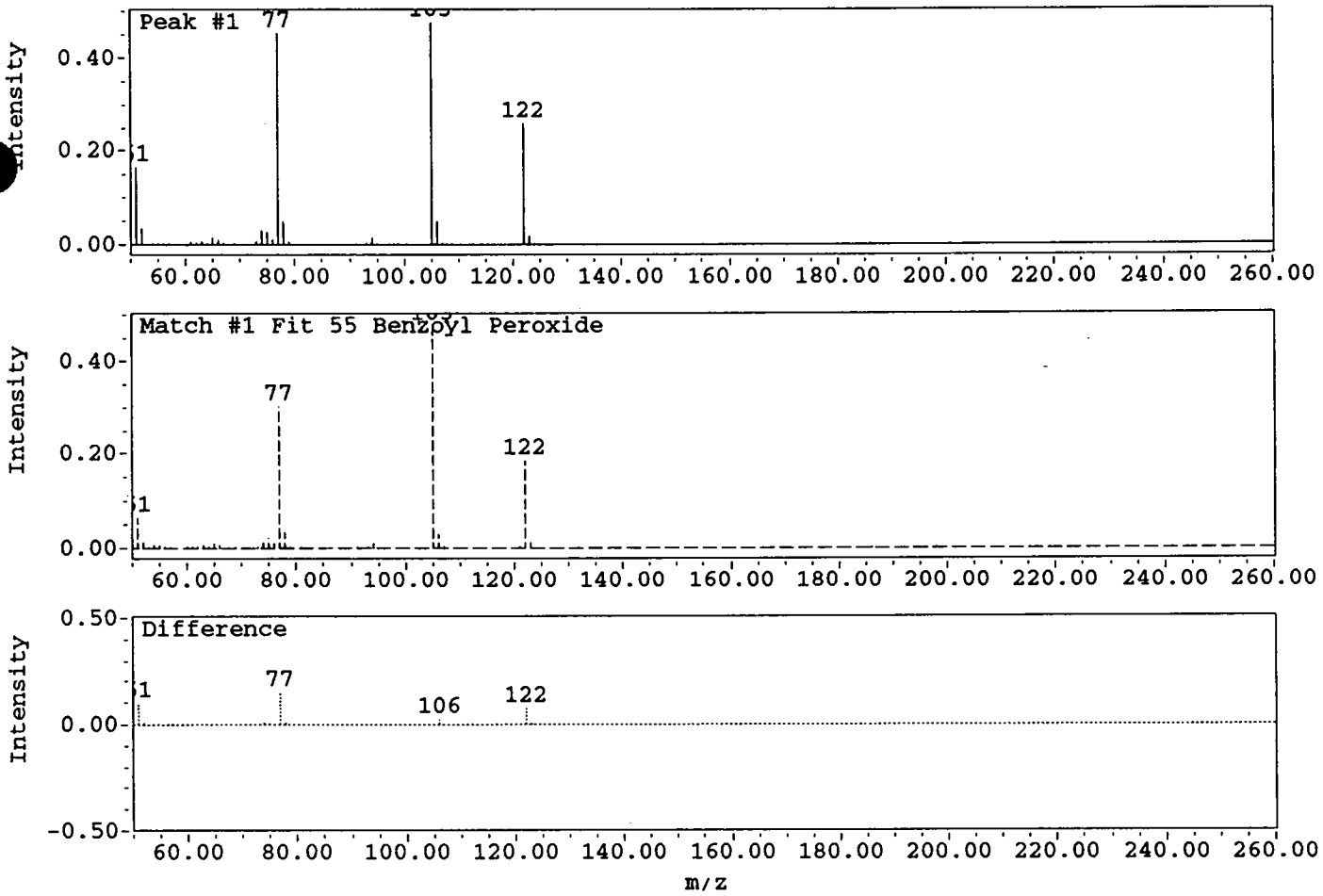
#	Retention Time (min)	Area (uV*sec)	Height (uV)	Match1 PBM Fit	Match1 % Contam.	Match1 Spect Name
1	4.418	3646952	324617	59	19	Benzoyl Peroxide

Match Plot Peak 1: Benzoylperoxide RT: 4.42





Mass Spec Info

#	Retention Time (min)	Area (uV*sec)	Height (uV)	Match1 PBM Fit	Match1 % Contam.	Match1 Spect Name
1	4.316	1940040	271676	55	20	Benzoyl Peroxide

Match Plot Peak 1: Benzoylperoxide RT: 4.32