Performance Evaluation of the CSS Particle Beam Interface for the Agilent Technologies, Inc. 5973 Benchtop MS System

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Abstract: The CSS Particle Beam and Agilent Technologies, Inc. 5973 Benchtop LCMS system was used to analyze samples from environmental, natural product, and clinical arenas. A polynuclear aromatic (PNA) standard was analyzed to demonstrate the utility of the particle beam for samples that are hard to detect by more popular LCMS methods. An extract of St. John's Wart (SJW) was screened for the presence of quercetin to show the utility of a library searchable spectrum in the analysis of unknown peaks or for analyses where legal defensibility is a necessity.

Experimental: The Hewlett Packard 1090 HPLC was attached to the CSS Particle Beam interface and Agilent 5973 Benchtop MS. The Hewlett Packard G1701BA Chemstation was used for instrument control and data analysis. The Wiley Registry of Mass Spectral Data was used for library searching. LCMS conditions were as follows:

Desolvation chamber temperature	80C	
MS source temperature	250C	
Nebulizing gas pressure	70psi	
Electron Energy	70eV	
HPLC flow rate	0.3ml/min.	
MS scan range	55-450amu for PNA's and 55-600amu for SJW @1scan/second	
HPLC Gradient for PNA Std.	Solvent A	Solvent B
Time = 0	30%Water	70%ACN
Time = 15	0% Water	100%ACN
Injection Vol	5ul on an Alltech 2.1mmx250mm C-18 5u	
HPLC Gradient for SJW Extract	Solvent A	Solvent B
Time = 0	100% 0.05%TFA	0%ACN
Time = 15	0% 0.05%TFA	100%ACN
Injection Vol	5ul on an Alltech 2.1mmx250mm C-18 5u	

Extract of St. John's Wart (SJW) was prepared by adding methanol to a 450mg commercial SJW tablet and grinding it into a slurry. This slurry was filtered and a 5ul aliquot was injected.

Results and Discussion: The analysis of polynuclear aromatics is an important part of many environmental monitoring systems. In this work a PNA standard was separated by liquid chromatography and detected and identified by electron impact mass spectrometry. The LCMS chromatogram for a 5ul injection of PNA standard is shown in figure 1.

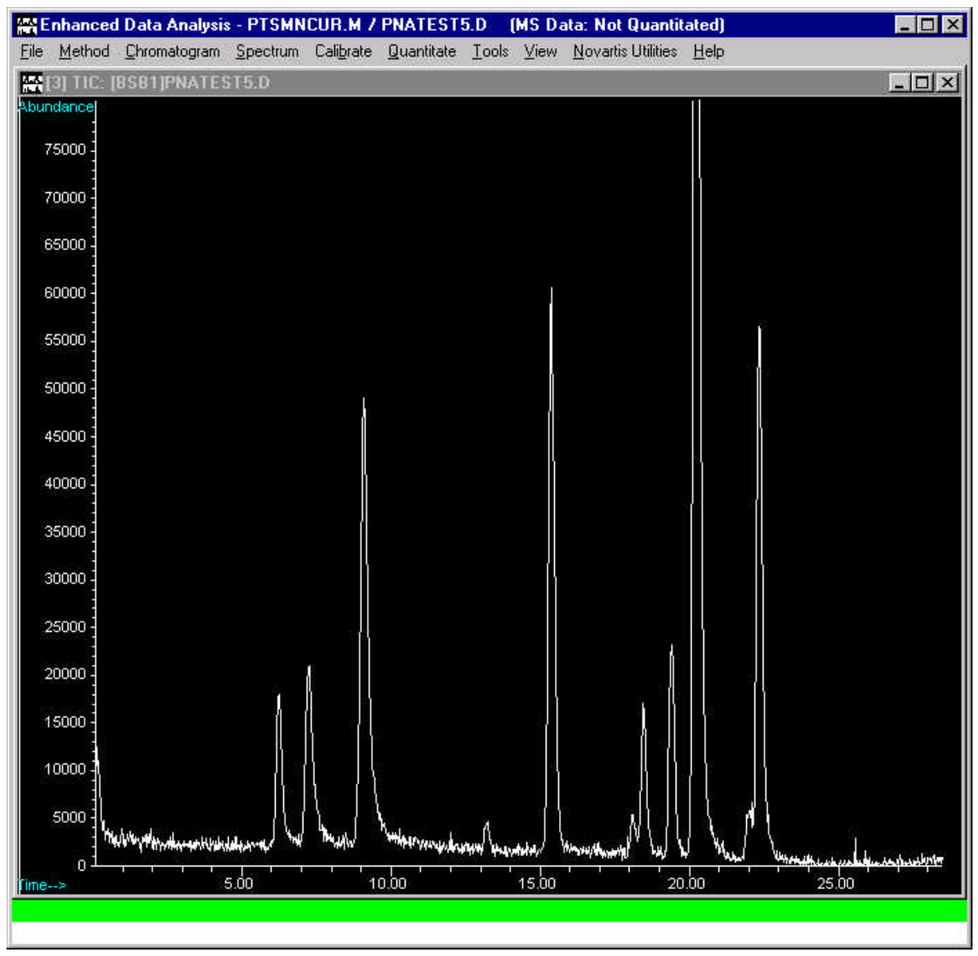

Figure 1: EI LCMS chromatogram for PNA standard.

An advantage of LCMS for the analysis of PNA's is the selectivity for the target compounds in the analysis. Figure 2 shows the mass chromatograms for the various PNA's that were extracted from the full scan electron impact data file. The LCMS chromatogram showed 11 different components of the PNA mix. By using extracted mass chromatograms, 14 different components of the mix were identified.

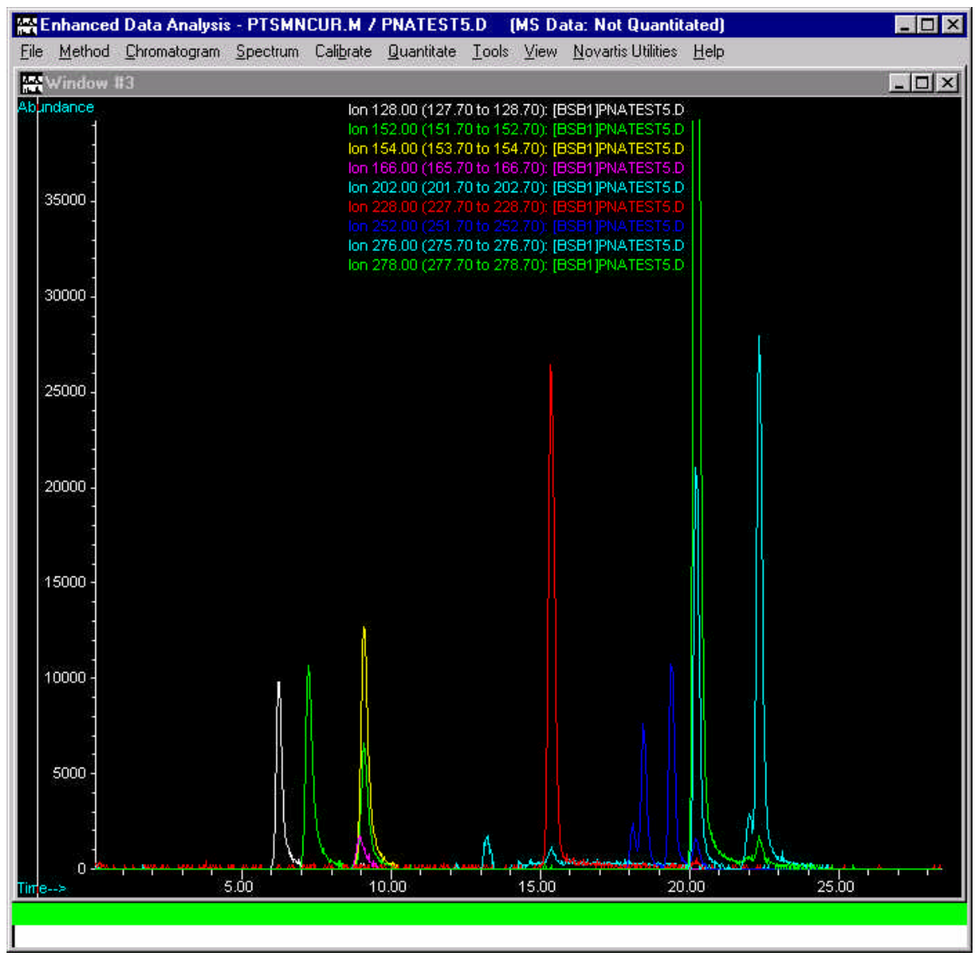

Figure 2: EI LCMS mass chromatograms extracted for the components of PNA standard.

An advantage of using electron impact for LCMS is that the unknown spectra can be searched against a database or library of known standard spectra. This will identify the unknown spectra in many cases. Figure 3 shows the unknown spectrum for several of the PNA LCMS peaks and the library match retrieved from searching the Wiley electron impact mass spectral library.

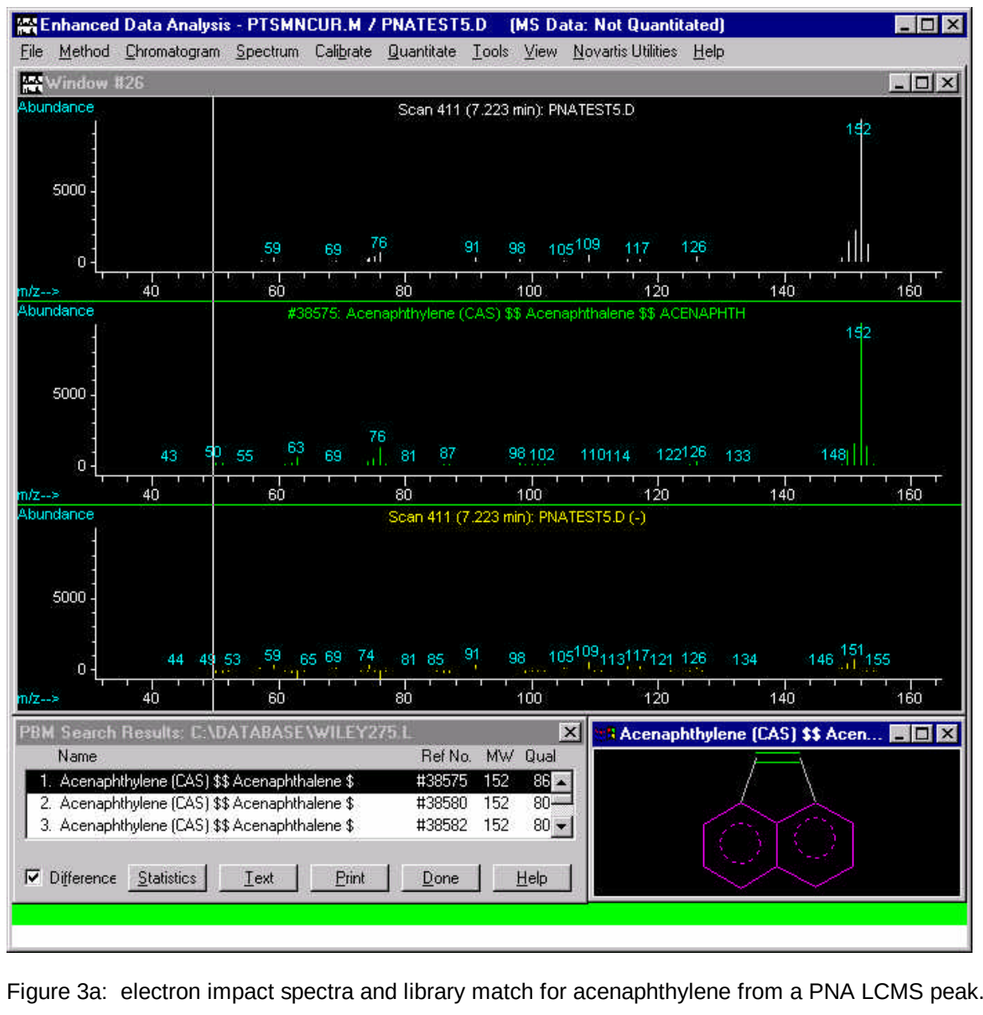

Figure 3a: electron impact spectra and library match for acenaphthylene from a PNA LCMS peak.

Acenaphthylene (CAS) $$ Acenaphthalene $$ ACENAPHTHYLEN $$ Cyclopenta[de] naphthalene $$ acenaphthylene / reference sample

Match Quality : 86
Entry Number : 38575
CAS Number : 000208-96-8
Molecular Weight : 152.06
Molecular Formula: C12H8
Retention Index : 0
Company ID : 130855
Melting Point :
Boiling Point :
Misc Information :
QI=884, Source=WS-1986-386-0, WLN=L566 1A LJ

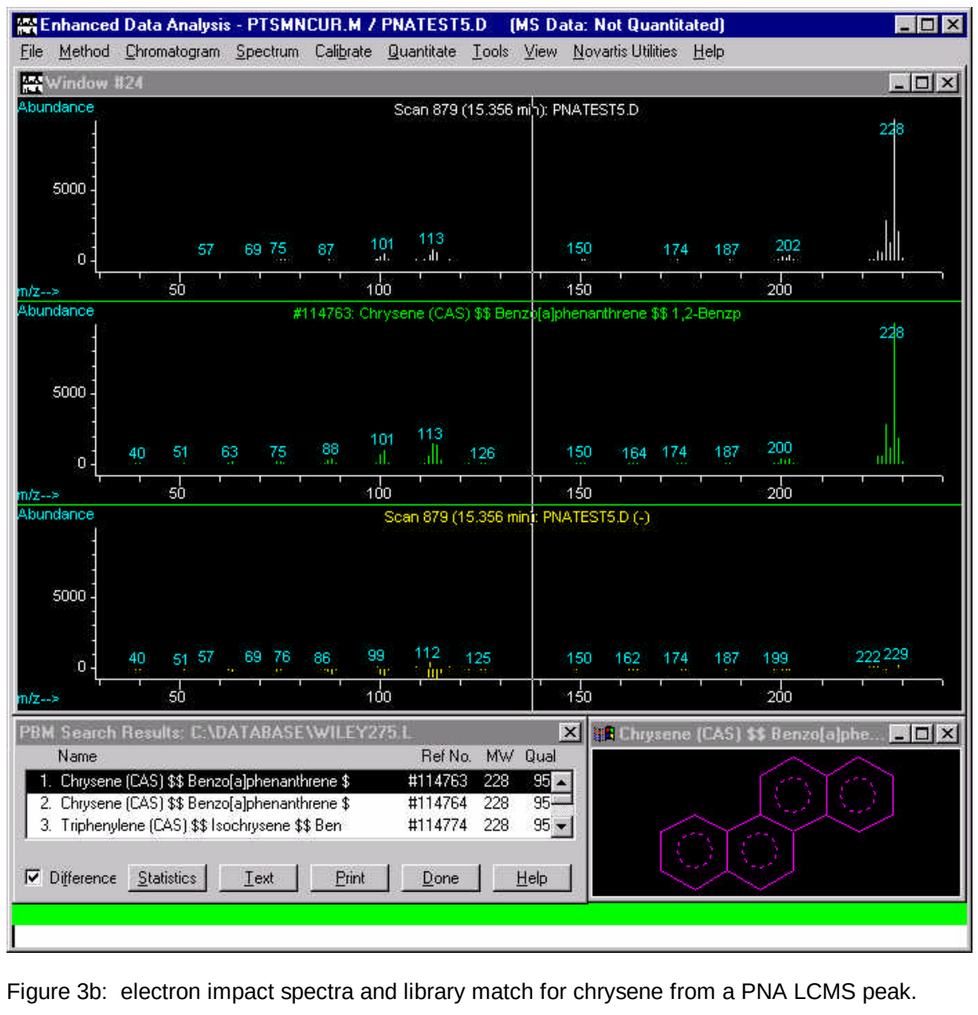

Figure 3b: electron impact spectra and library match for chrysene from a PNA LCMS peak.

Chrysene (CAS) $$ Benzo[a]phenanthrene $$ 1,2-Benzphenanthrene $$ 1,2-Benzophenanthrene $$ Benz(a)phenanthrene $$ 1,2,5,6-Dibenzonaphthalene $$ Rcra waste number U050

Match Quality : 95
Entry Number : 114763
CAS Number : 000218-01-9
Molecular Weight : 228.09
Molecular Formula: C18H12
Retention Index : 0
Company ID : 327945
Melting Point :
Boiling Point :
Misc Information :
QI=761, Source=NS-0-0-0

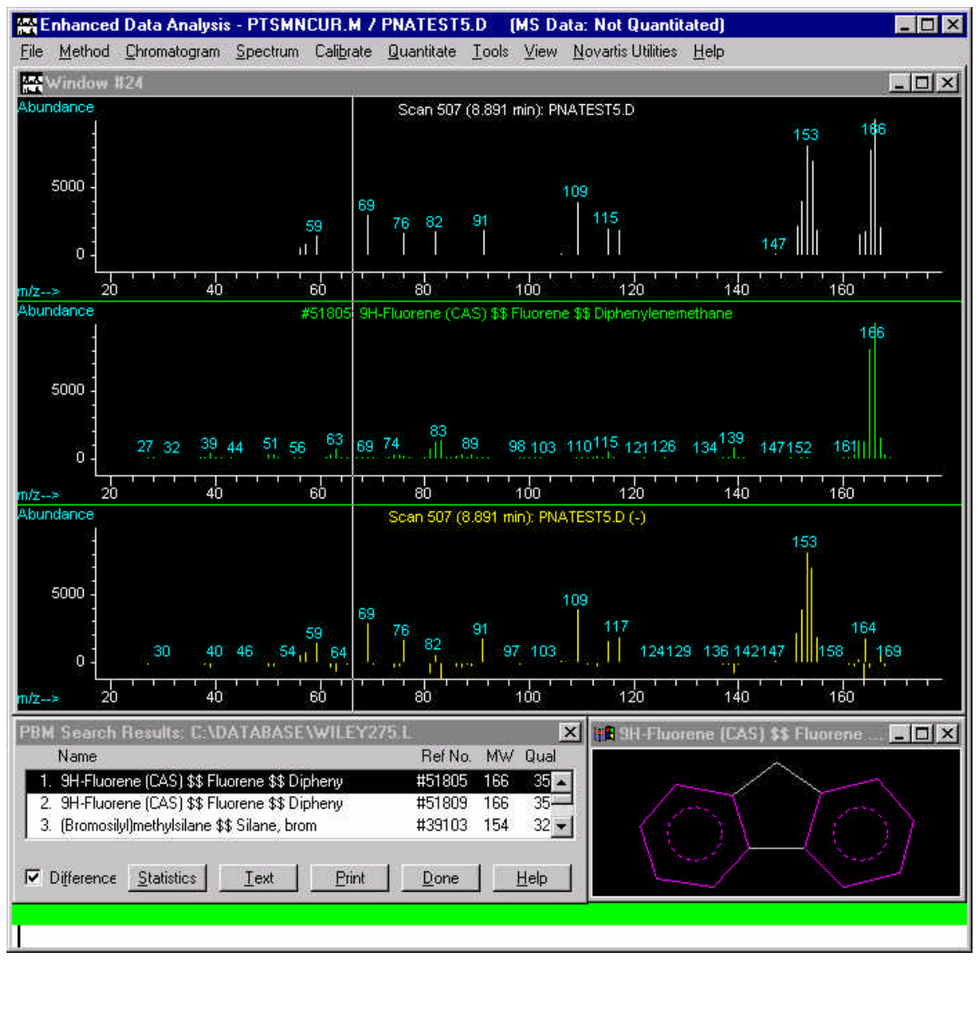

Figure 3c: electron impact spectra and library match for fluorene from a PNA LCMS peak. Matched from an impure peak.

9H-Fluorene (CAS) $$ Fluorene $$ Diphenylenemethane $$ o-Biphenylenemetha
ne $$ Methane, diphenylene- $$ 2,2'-Methylenebiphenyl $$ FLUORENE (2,3-BE
NZINDENE) $$ 2,3-Benzindene $$ o-Biphenylmethane

Match Quality : 35
Entry Number : 51805
CAS Number : 000086-73-7
Molecular Weight : 166.08
Molecular Formula: C13H10
Retention Index : 0
Company ID : 14607
Melting Point :
Boiling Point :
Misc Information :
QI=982, Source=HE-1982-0-0, WLN=L B656J

An analysis of St. John's Wart extract was performed in order to screen for the presence of quercetin, a compound of high interest in the nutriceutical field. The electon impact LCMS chromatogram for the analysis of a 5ul injection of SJW extract is shown in figure 4.

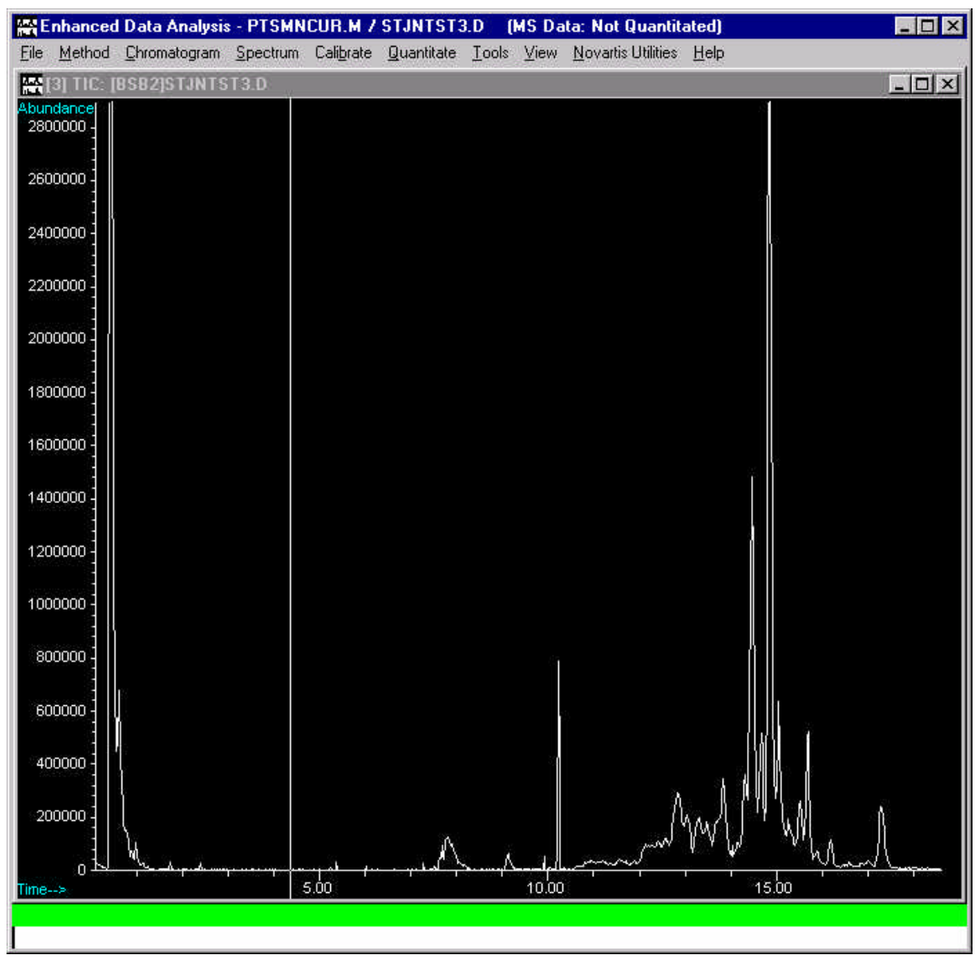

Figure 4: EI LCMS chromatogram for extract of St. John's Wart.

Analysis of the full scan electron impact spectra in the data file reveal the presence of quercetin at a retention time of 9.1 minutes. A search of the Wiley electron impact mass spectral library reveals a good match of this peak with a standard spectrum of quercetin. This is shown in figure 5.

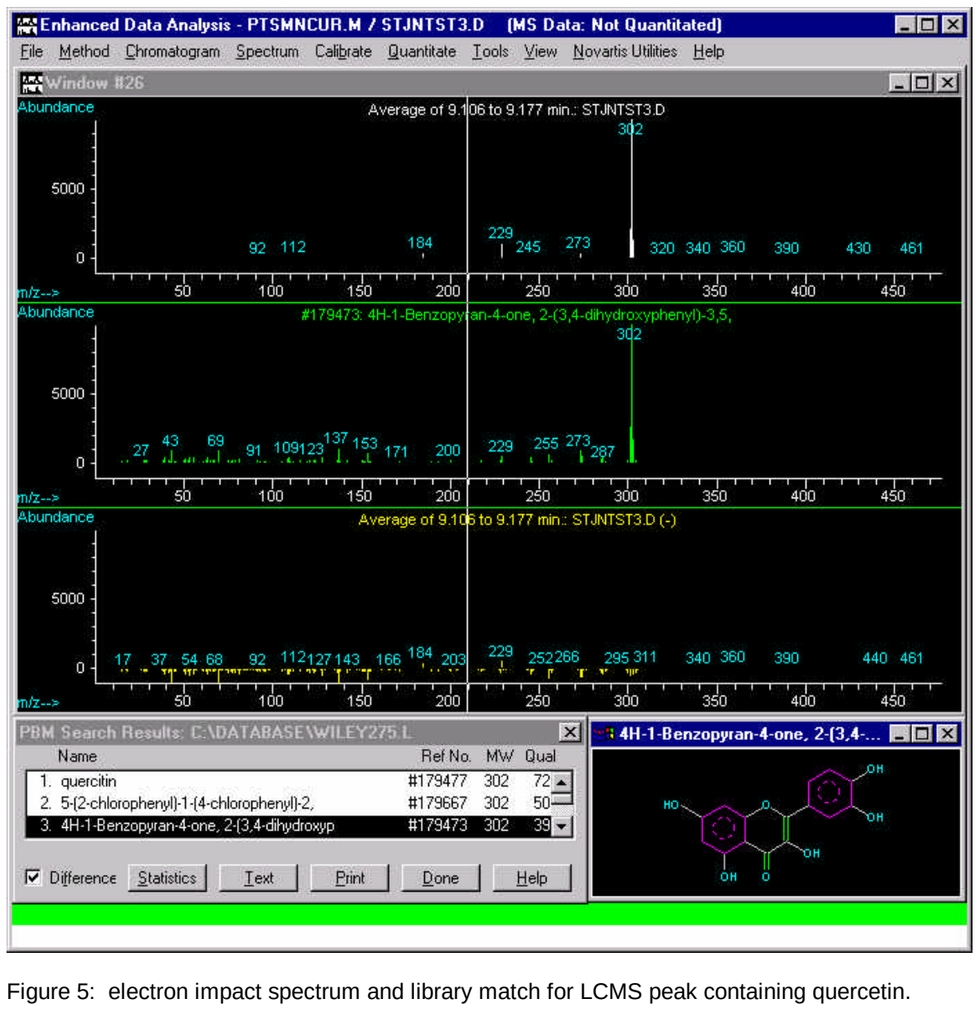

Figure 5: electron impact spectrum and library match for LCMS peak containing quercetin.

4H-1-Benzopyran-4-one, 2-(3,4-dihydroxyphenyl)-3,5,7-trihydroxy- (CAS) $$ Flavone, 3,3',4',5,7-pentahydroxy- $$ C.I. 75670 $$ C.I. Natural Yellow 10 $$ Cyanidelonon 1522 $$ Meletin $$ **Quercetin** $$ Quercetine $$ Querceto $$ Quertine $$ Sophoretin $$ Xanth

Match Quality : 72
Entry Number : 179473
CAS Number : 000117-39-5
Molecular Weight : 302.04
Molecular Formula: C15H10O7
Retention Index : 0
Company ID : 110403
Melting Point :
Boiling Point :
Misc Information :
QI=899, Source=CD-315-0-0

Conclusion: The new CSS Particle Beam and Agilent Technologies, Inc. 5973 Benchtop LCMS system is an excellent tool for the analysis of samples where traditional LCMS techniques such as electrospray or apci, have

special difficulties. In the case of PNA's ionizing the sample without difficult and time consuming hplc modifications is not possible with electrospray or apci. For sensitivity, separation, and identification purposes the CSS Particle Beam and Agilent Technologies, Inc. 5973 Benchtop LCMS system is an excellent choice. In the cases where unknown peaks may be encountered or legal defensibility is a necessity, such as the case with a sample like the St. John's Wart or perhaps a forensic application, electron impact may again be a necessity. Here even though electrospray or apci do an excellent job of detecting the peak, the information contained in the spectrum may be found lacking when a positive identification is needed. For this reason the power of library searching an electron impact spectrum becomes apparent.