

Utilization of Hydrogen Carrier Gas on a High Resolution GC-TOFMS System: An Application Compendium

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Background

Both the costs associated with a dwindling helium supply and the need for high sample throughput have fueled the desire to develop fast gas chromatography methods using hydrogen as a carrier gas. This poster demonstrates the ability to utilize hydrogen carrier gas on a high resolution GC-TOFMS system, the Pegasus® GC-HRT. Methods and data from multiple application markets, including specialty chemicals, forensics, and metabolomics, will be displayed. Various polymer additives were used to represent the specialty chemical market, drugs of abuse were used to demonstrate capabilities in the forensic market, and a derivatized metabolite mixture designed for metabolomics research was used for the metabolomics market.

GC-HRT methods utilizing hydrogen carrier gas were developed for analysis of representative specialty chemical, forensic, and metabolomic markets.

- Polymer additives were dissolved in chloroform prior to analysis.
- Drugs of abuse were dissolved in organic solvents and analyzed underivatized.
- Metabolites were derivatized prior to GC-HRT analysis using an optimized two-step procedure: 1) Treatment with methoxylamine hydrochloride and 2) MSTFA.



Helium

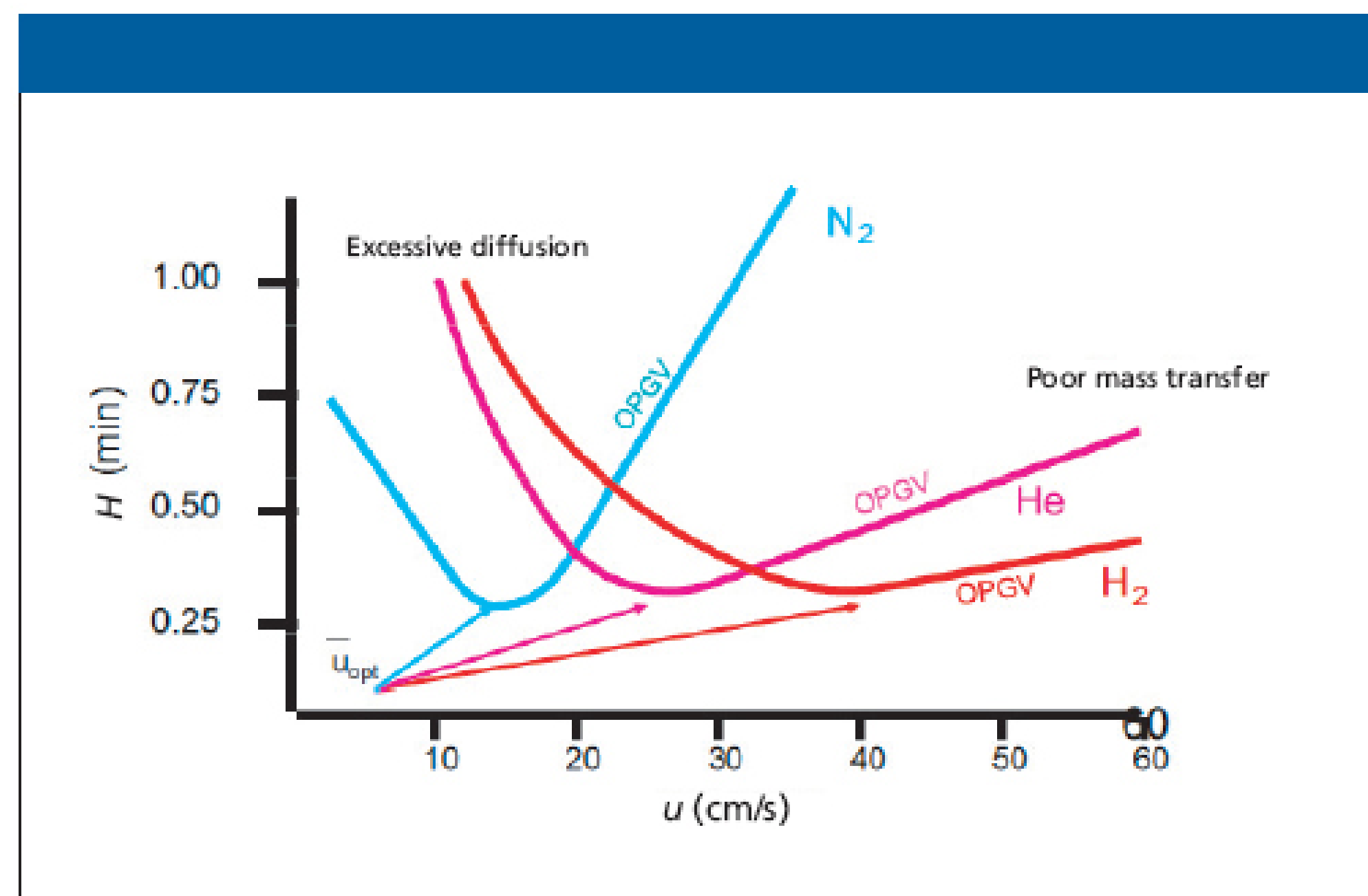


Figure 1. Van Deemter curves for He, N₂, and H₂. OPGV = Optimum practical gas velocity.

Instrumentation



Figure 2. Pegasus GC-HRT High Resolution TOFMS.

Pegasus GC-HRT Capabilities

- High Resolution (Up to R=50,000)
- Mass Accuracy (<1 ppm)
- Fast Acquisition Rates (Up to 200 spectra/second)
- High Resolution Deconvolution™ (HRD™)
- H₂ Carrier Gas Compatible

Experimental

GC-HRT Methods (Polymer Additives)

GC Conditions
 Column: Rxi-1ms 20 m x 0.18 mm x 0.18 micron film (Restek)
 Carrier: Hydrogen @ 0.8 mL/min
 Injection: 1 µL, split 20:1 @ 300°C
 Oven: 50°C (0.5 min) to 300°C @ 50°C/min
 Tr. Line: 300°C

HRT Conditions

Resolution: High (25k)
 Mass Range: 40-700 m/z
 Ionization: EI

GC-HRT Methods (Drugs of Abuse)

GC Conditions
 Column: Rxi-1ms 20 m x 0.18 mm x 0.18 micron film (Restek)
 Carrier: Hydrogen @ 0.8 mL/min
 Injection: 1 µL, split 50:1 @ 280°C
 Oven: 40°C (2 min) to 300°C @ 30°C/min
 Tr. Line: 300°C

HRT Conditions

Resolution: High (25k)
 Mass Range: 40-400 m/z
 Ionization: EI

GC-HRT Methods (Metabolomics)

GC Conditions
 Column: Rxi-1ms 20 m x 0.18 mm x 0.18 micron film (Restek)
 Carrier: Hydrogen @ 0.8 mL/min
 Injection: 1 µL, split 10:1 @ 250°C
 Oven: 80°C to 300°C @ 40°C/min
 Tr. Line: 300°C

HRT Conditions

Resolution: High (25k)
 Mass Range: 50-600 m/z
 Ionization: EI

Results

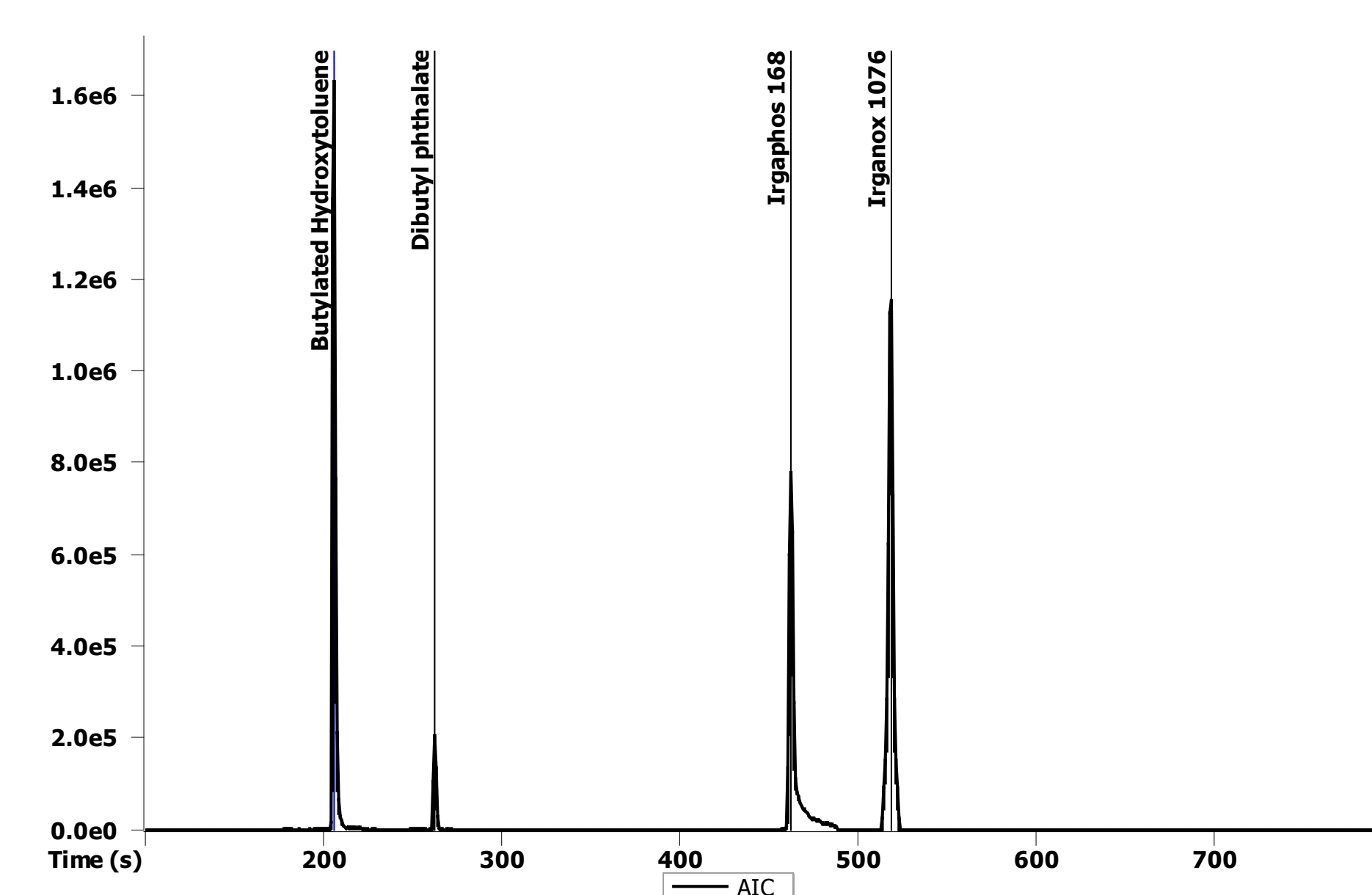


Figure 3. AIC Showing Representative Polymer Additives.

Table I. Tabular Data for Polymer Additives

Compound	Retention Time (min)	Experimental m/z	Observed m/z	Mass Accuracy (ppm)	Formula
Butylated Hydroxytoluene	813	230.1022	230.1021	1.2	C ₁₄ H ₁₈ O ₂
Dibutyl phthalate	875	278.1513	NA	NA	C ₁₈ H ₂₆ O ₄
Irganox 168	688	646.0709	646.0711	0.32	C ₁₈ H ₂₀ O ₂
Irganox 1076	566	570.0921	570.0920	0.49	C ₁₈ H ₂₀ O ₂

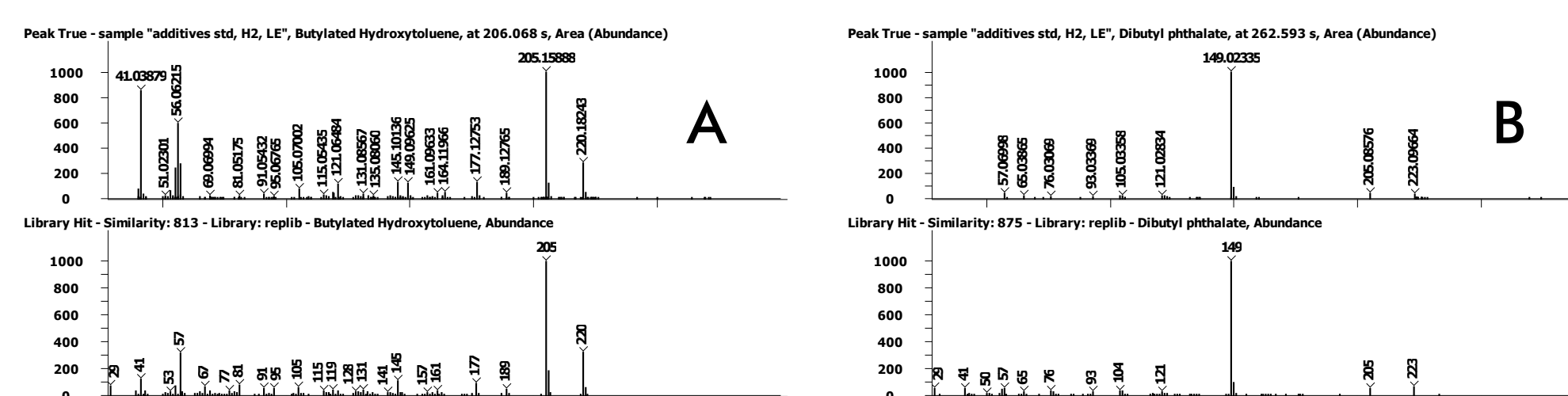


Figure 4. Deconvoluted (Peak True) and Library Hit Spectra for Butylated Hydroxytoluene (A) and Dibutyl Phthalate (B).

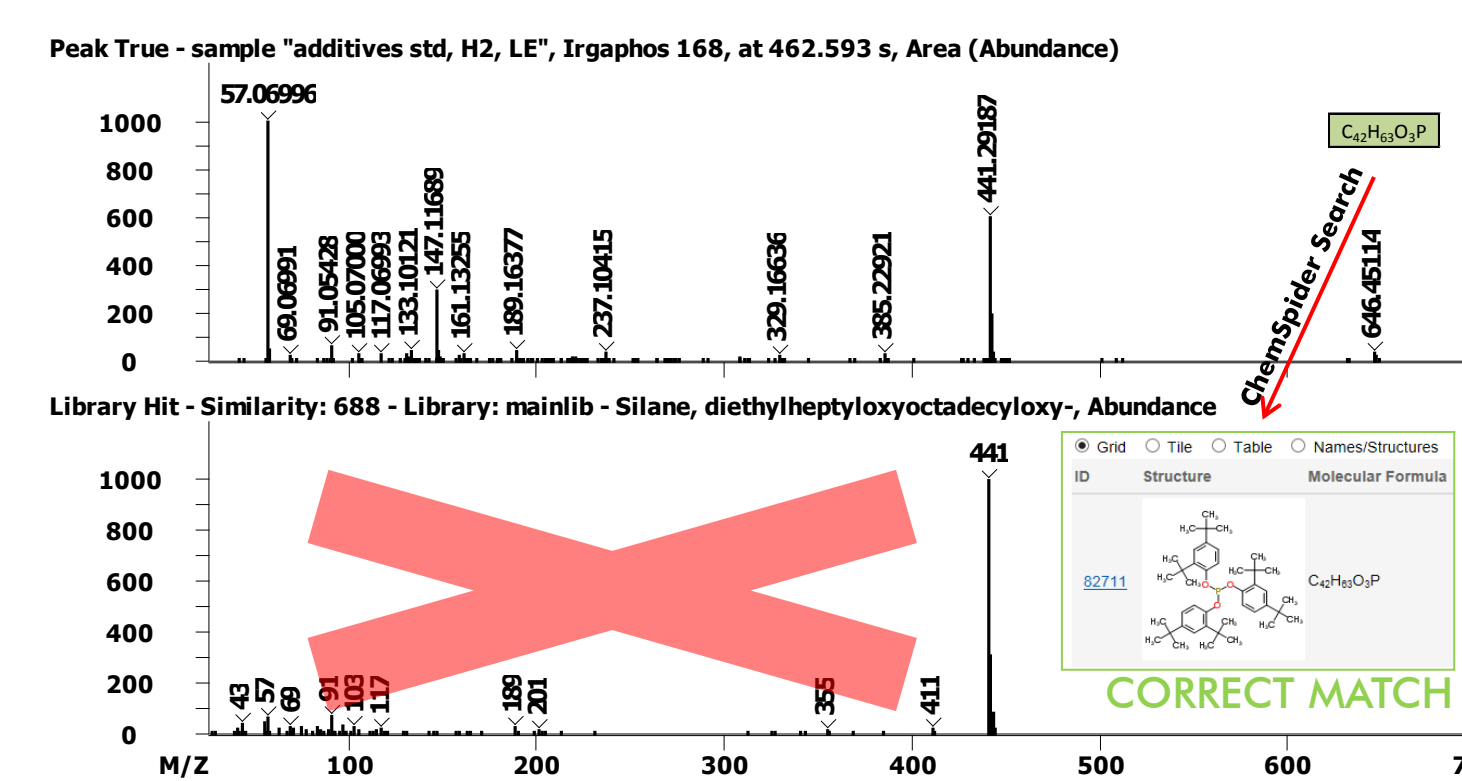


Figure 5. Example of Accurate Mass Data Used to Correctly Identify an Incorrect NIST Library Match.

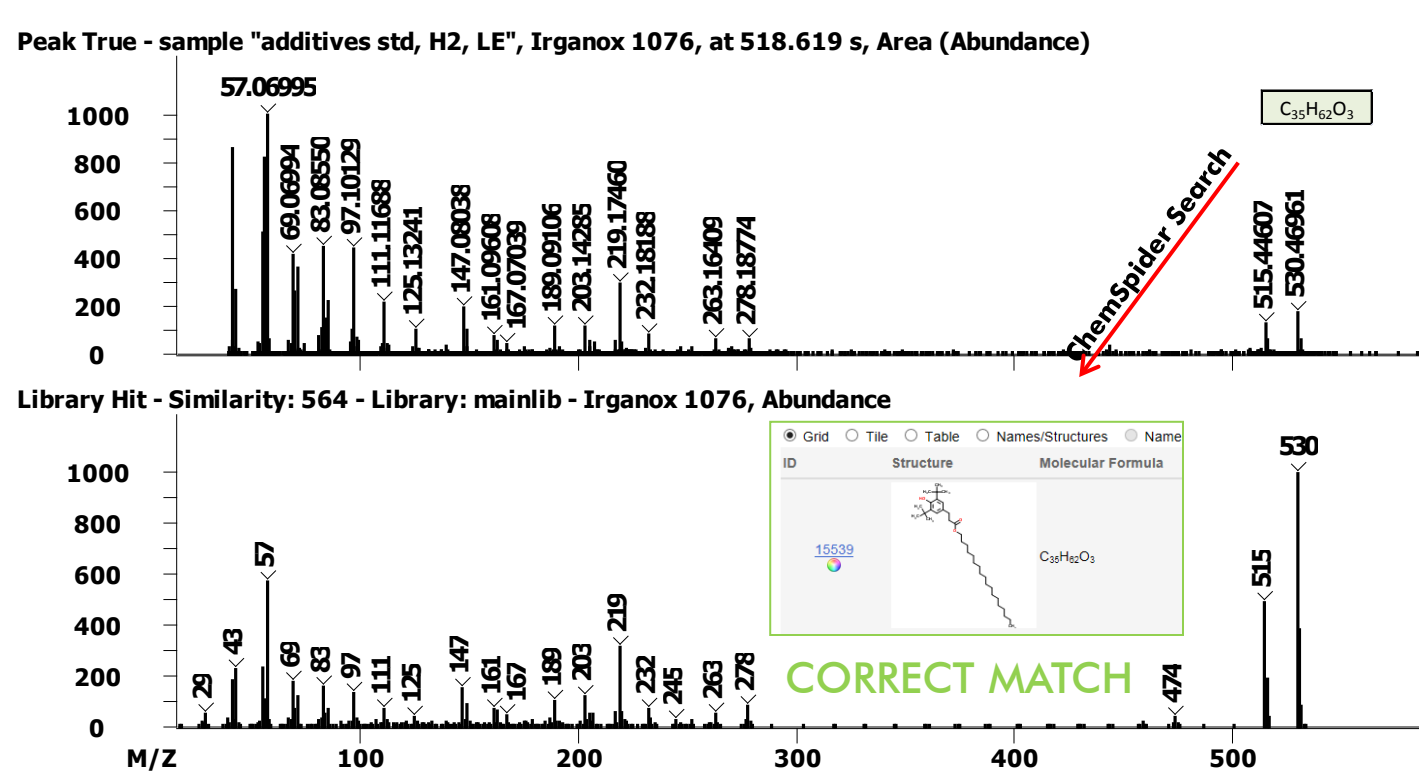


Figure 6. Example of Accurate Mass Data Used to Confirm a NIST Library Match.

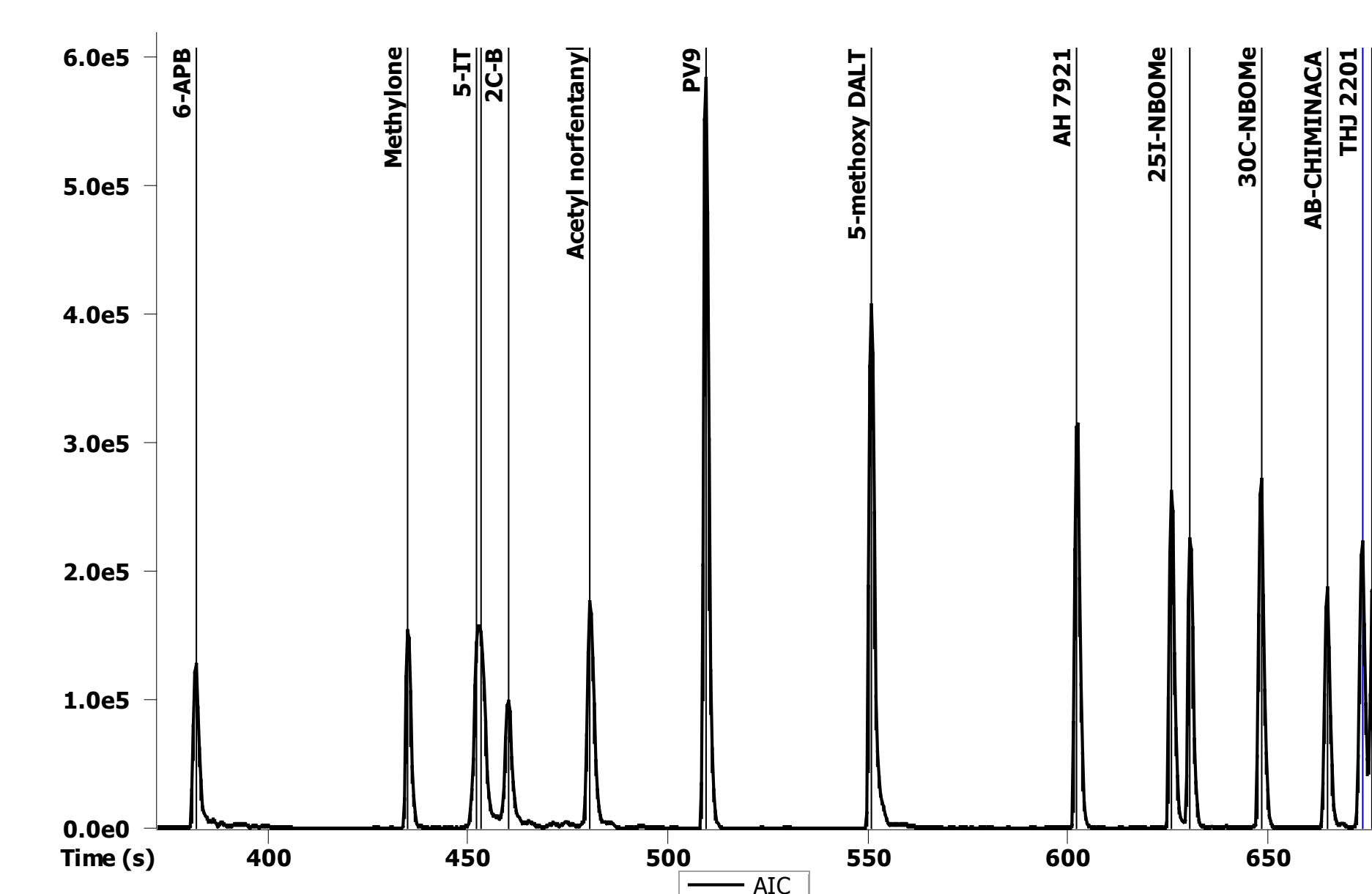


Figure 7. AIC Showing Representative Drugs of Abuse.

Table II. Tabular Data for Drugs of Abuse

Drug	Retention Time (min)	Experimental m/z	Observed m/z	Mass Accuracy (ppm)	Formula
6-APB	682	375.0920	375.0921	-0.59	C ₁₂ H ₁₅ NO
Methylene	780	207.0889	NA	NA	C ₁₁ H ₁₇ N ₃ O ₂
5-IT	719	174.1151	174.1155	2.26	C ₁₁ H ₁₅ N ₃
N,N-DMT	566	188.1308	188.1307	-0.27	C ₁₁ H ₁₇ N ₃
2C-B	874	259.0222	259.0205	0.98	C ₁₁ H ₁₅ N ₃ O
Acetyl norfentanyl	779	218.1414	218.1416	1.27	C ₁₁ H ₁₅ N ₃ O
PV9	809	273.2087	NA	NA	C ₁₁ H ₁₅ NO
5-methoxy DALT	763	270.1727	270.172	-2.3	C ₁₁ H ₁₇ N ₃ O
AH 7921	761	328.1104	NA	NA	C ₁₁ H ₁₇ N ₃ O
25I-NBOMe	799	427.0639	NA	NA	C ₁₁ H ₁₇ N ₃ O
MT-45	762	348.256	NA	NA	C ₁₁ H ₁₇ N ₃ O
3OC-NBOMe	462	NA	NA	NA	C ₁₁ H ₁₇ ClN ₃ O
AB-CHMINACA	384	NA	NA	NA	C ₁₁ H ₁₇ N ₃ O ₂
THU 2201	737	360.1627	360.1627	-1.5	C ₁₁ H ₁₇ N ₃ O
AM201 benzimidazole analog	857	360.1627	360.162	-3.34	C ₁₁ H ₁₇ N ₃ O

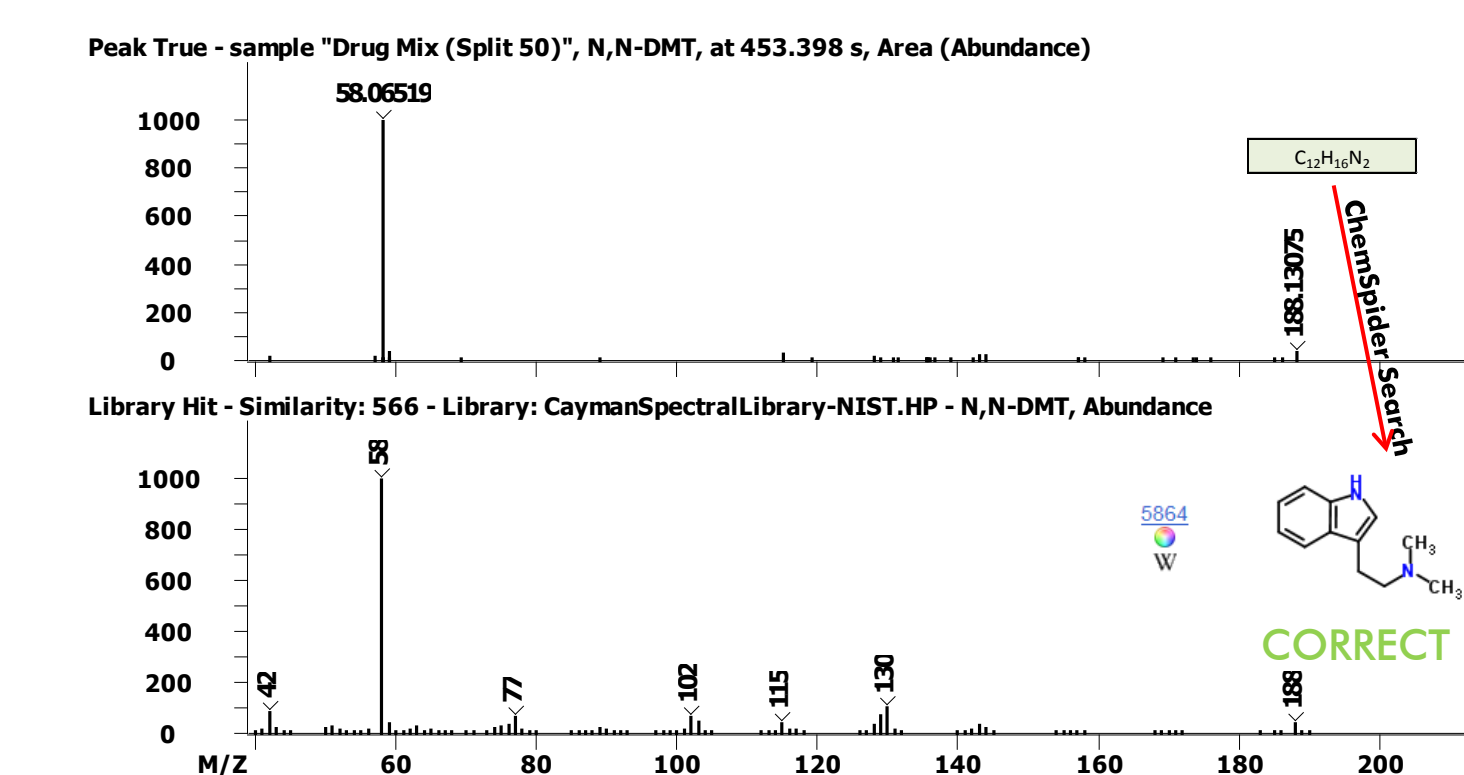


Figure 8. Deconvoluted (Peak True) and Library Spectra with Accurate Mass Utilized for Confirmation.

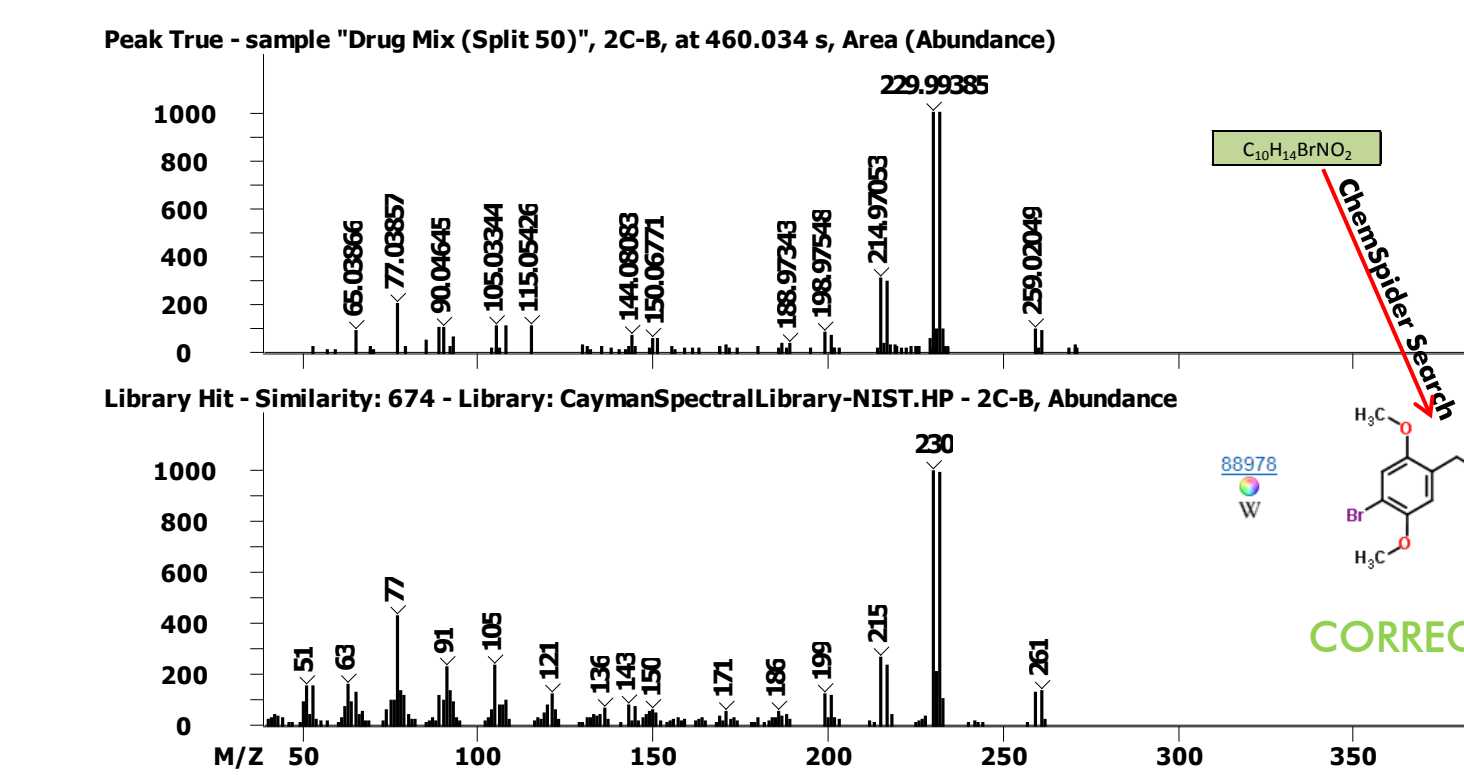


Figure 9. Deconvoluted (Peak True) and Library Spectra with Accurate Mass Utilized for Confirmation.

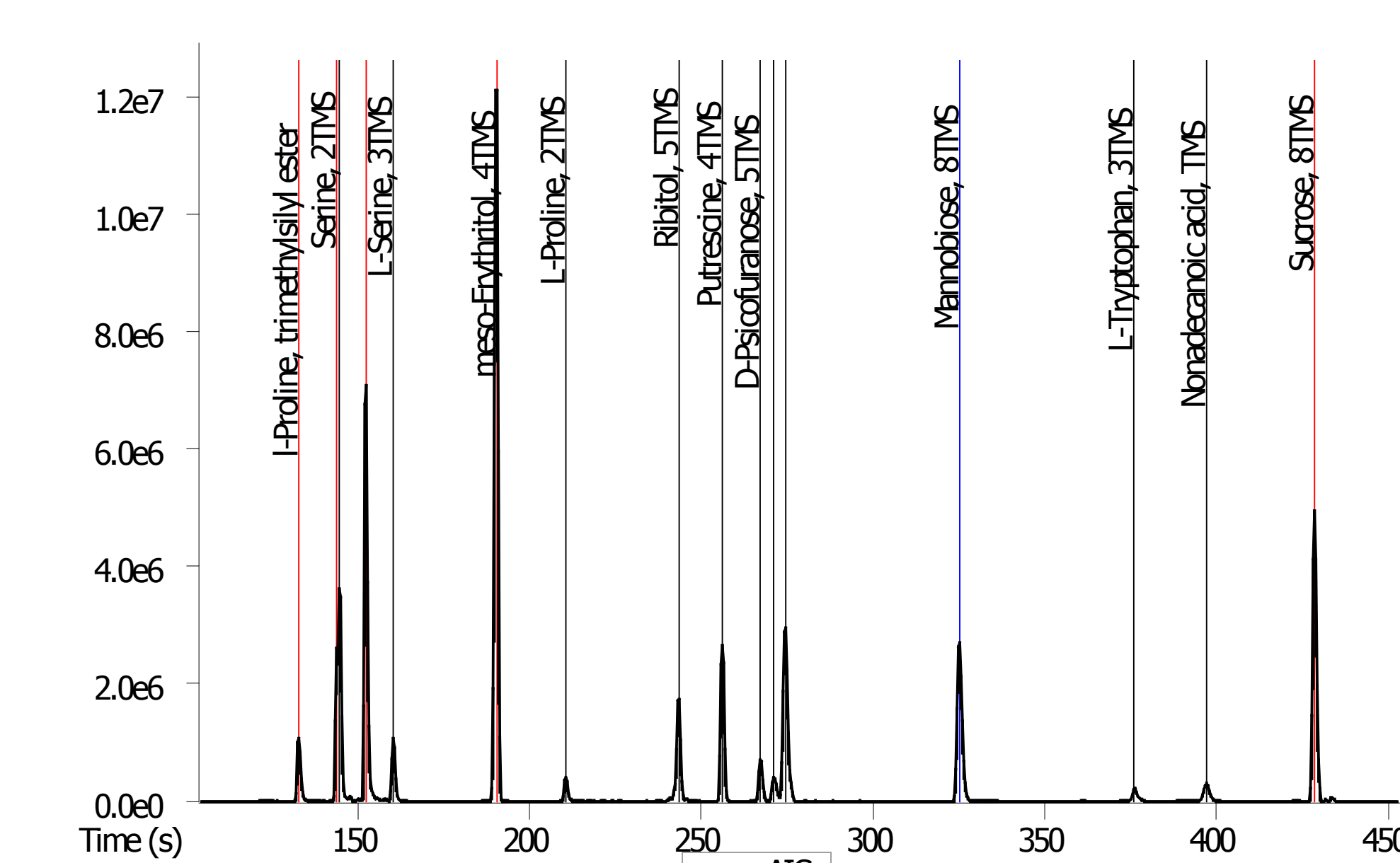


Figure 10. AIC Showing Representative Metabolites.

Table III. Tabular Data for TMS Derivatized Metabolites

Metabolite	Retention Time (min)	Experimental m/z	Observed m/z	Mass Accuracy (ppm)	Formula
L-Proline, trimethylsilyl ester	771	187.1023	187.1023	0.04	C ₁₀ H ₁₉ NO ₃
Serine, 2TMS	761	249.1211	NA	NA	C ₁₀ H ₂₁ NO ₃
Silanol, trimethyl-, phosphate (E:1)	898	314.0983	314.0953	1.172	C ₁₀ H ₂₁ O ₅ PSi ₃
Glycine, 3TMS	826	291.1006	291.1008	-0.588	C ₁₀ H ₂₁ NO ₃
L-Serine, 3TMS	833	321.1006	NA	NA	C ₁₀ H ₂₁ NO ₃
meso-Erythritol, 4TMS	864	430.2147	NA	NA	C ₁₀ H ₂₁ O ₅
L-Proline, 2TMS	747	273.1211	NA	NA	C ₁₀ H ₂₁ NO ₃
Ribitol, 5TMS	896	512.2006	NA	NA	C ₁₀ H ₂₁ O ₅
Putrescine, 4TMS	899	378.2506	378.2613	-3.971	C ₁₀ H ₂₁ O ₅
D-Fructofuranose, 5TMS	836	543.2047	NA	NA	C ₁₀ H ₂₁ O ₅
D(-)-Fructofuranose, 5TMS	781	543.2047	NA	NA	C ₁₀ H ₂₁ O ₅
OTIC ACID, 4TMS	848	480.1846	NA	NA	C ₁₀ H ₂₁ O ₅
3-β-Norbornene, 6TMS	765	318.1188	NA	NA	C ₁₀ H ₂₁ O ₅
L-Tryptophan, 3TMS	726	420.2075	NA	NA	C ₁₀ H ₂₁ O ₅
Nonadecanoic acid, TMS	740	370.1206	NA	NA	C ₁₀ H ₂₁ O ₅
Sucrose, 6TMS	806	518.4188	NA	NA	C ₁₀ H ₂₁ O ₅

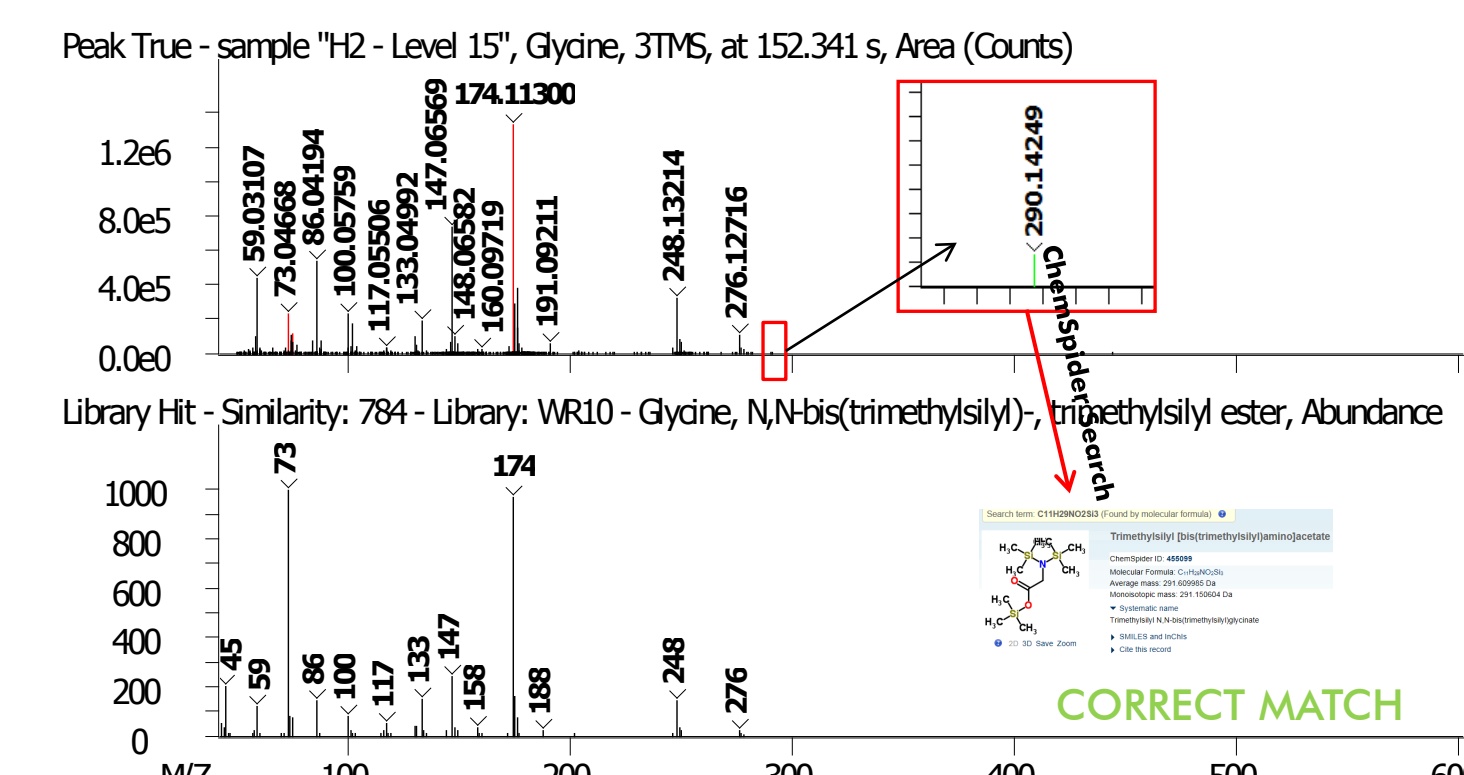


Figure 11. Deconvoluted (Peak True) and Library Spectra with Accurate Mass Utilized for Confirmation.

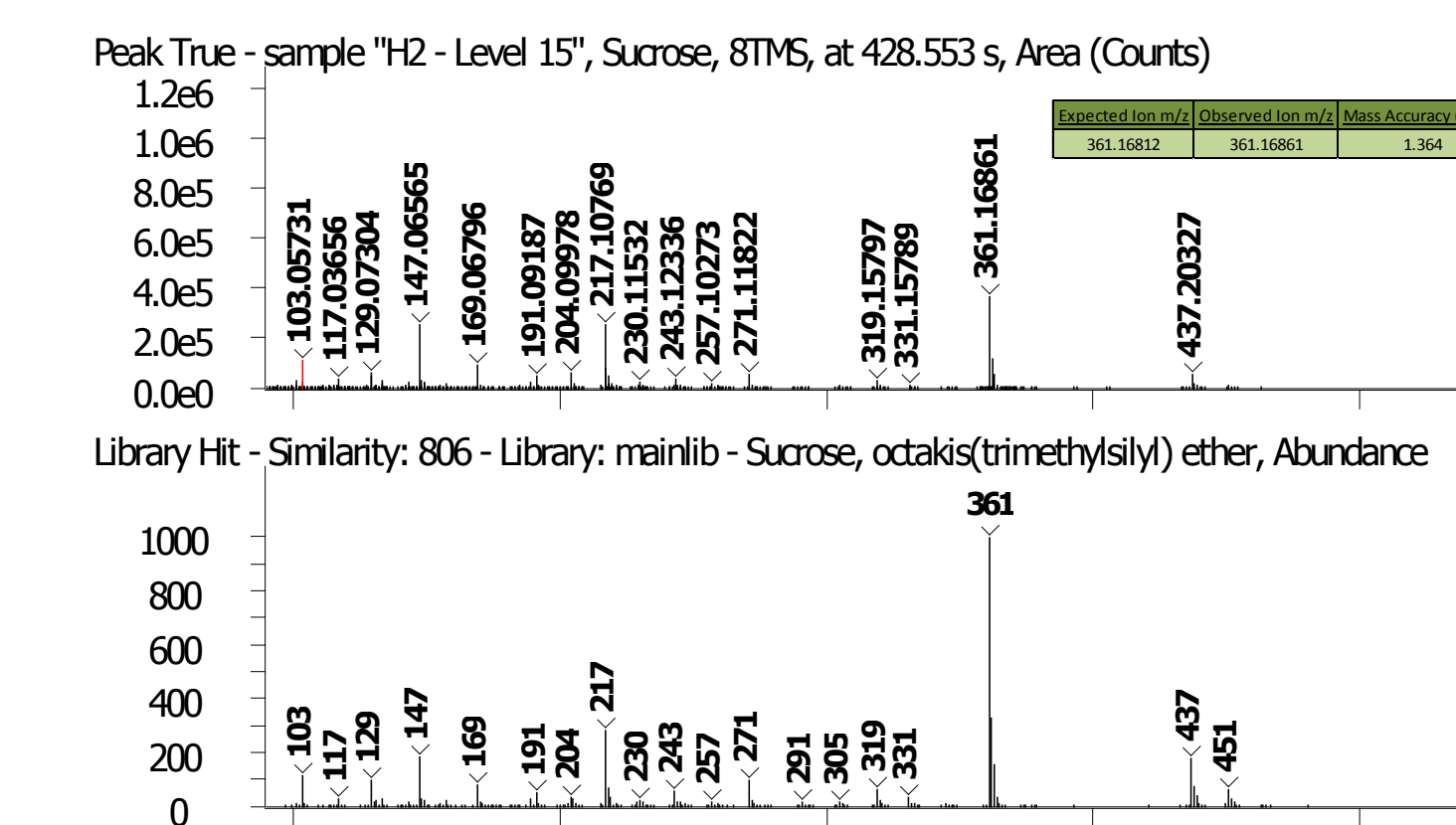


Figure 12. Deconvoluted (Peak True) and Library Spectra for Sucrose. Accurate Mass Measurement of m/z 361.16861 Confirms Chemical Formula of C₁₂H₂₂O₁₁, Which is Part of the Sucrose Structure.