LC/MS Polymer Additive Analysis by EI and APCI

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Objective

- Demonstrate how EI and API can complement each other in chemical analysis
- Develop a novel analytical method for identification and quantitation of polymer additives using acetonitrile extract from polypropylene powder as sample analyte

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Experimental Conditions

- HPLC Conditions:
  - Waters Alliance™ 2690 System
  - Waters Symmetry C_8 Column: 3.9 x 150 mm
  - Acetonitrile/Water as Mobile Phase
  - 10 µL Injection with 0.4 ml/min Flow Rate
- MS Conditions:
  - Waters Integrity System for EI
  - Micromass Platform LC System for API

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LC/MS Polymer Additive Analysis

Polymer Sample with Unknown Additive Package

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Additives Extracted from Polymer by :
Solvent Extration, Soxhlet Extraction, SFE

↓

Develop an MS Compatible LC Method

↓

EI: To Generate Lead by Library Match and Structure Elucidation

↓

API: Confirm EI Leads with MW Information

↓

Confirmation of Results by Obtaining Standards

↓

Quantitation of the Polymer Additives by API LC/MS

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Compound Identification

- Comparison of three modes of detection: PDA, EI and APCI
- Matching EI spectra with library generates lead suspects
- Obtaining molecular weight information from APCI helps to rule out wrong matches
- Generating limited fragmentation in API with Up-front Collision Induced Dissociation (CID) helps to confirm EI results

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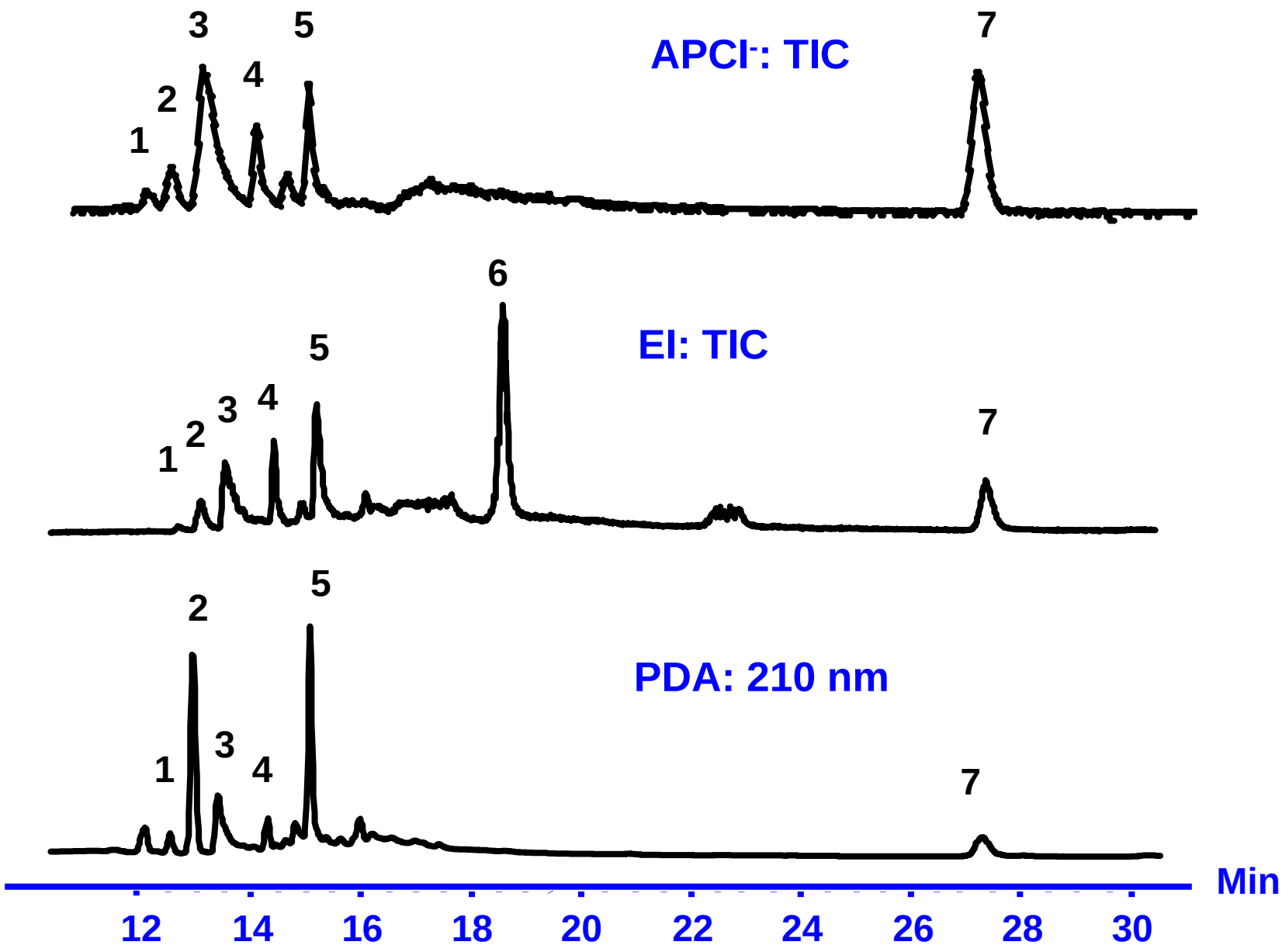

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1	Naugard Degradant	MW 436
2	NC-4	MW 414
3	3-(3,5-di-tert-butyl-4-hydroxyphenyl) propanoic acid	MW 278
4	7,9-Di-tert-butyl-1-oxaspiro(4,5) deca-6,9-deene-2,8-dione	MW276
5	Naugard	MW 696
6	1-Octodecanol	MW270
7	Irgonox 1076	MW 530

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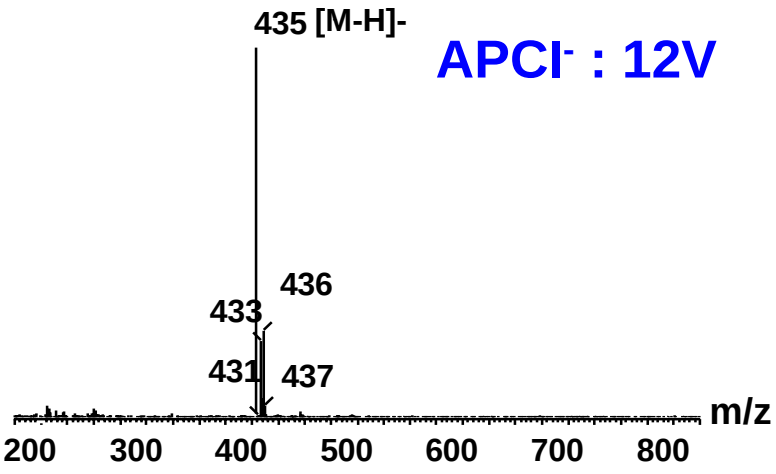
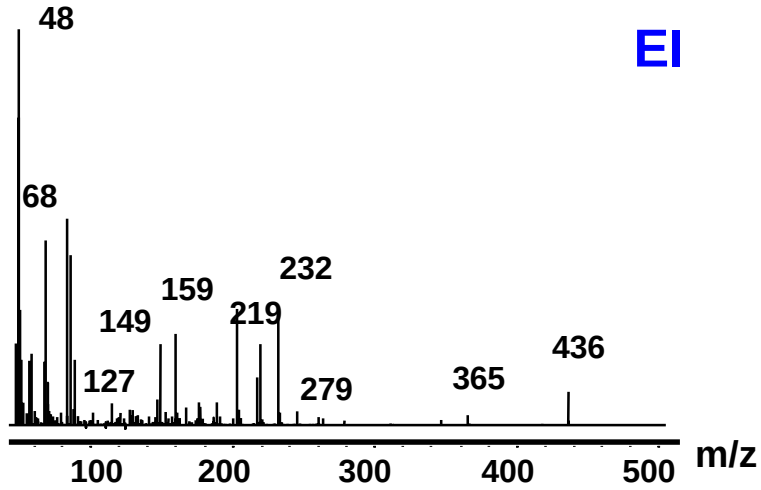
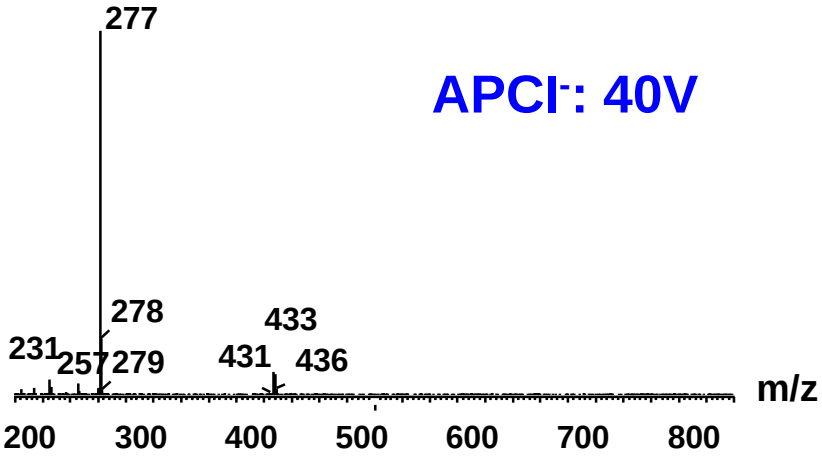
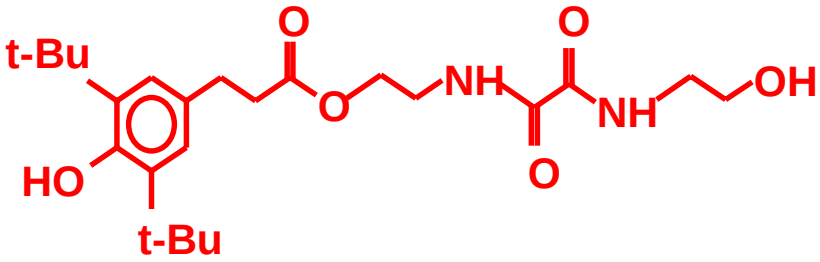
MS Spectra of Peak #1









Naugard Degradant MW 436

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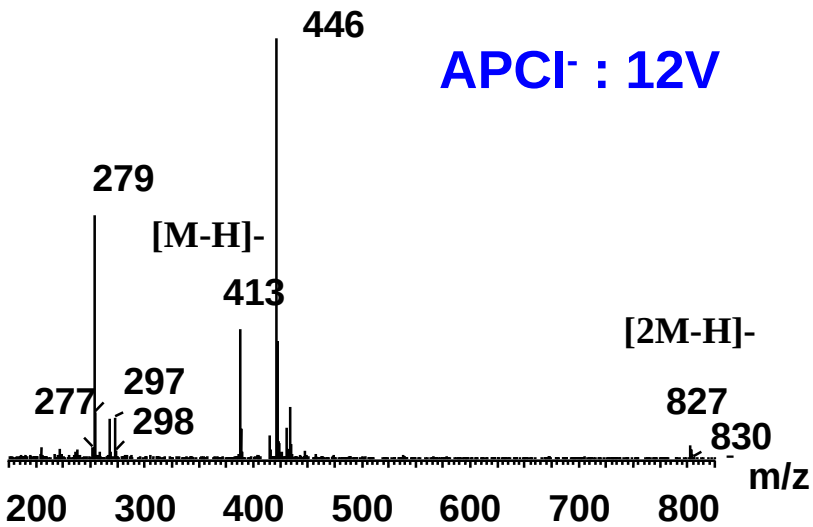
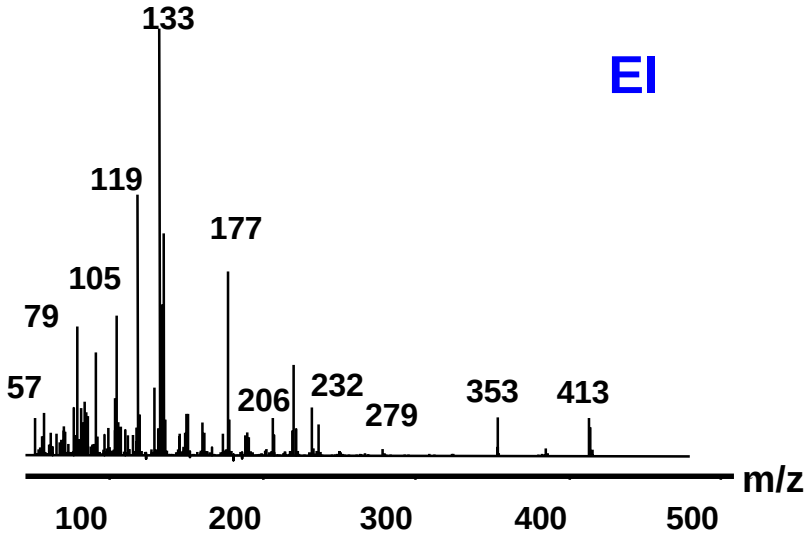
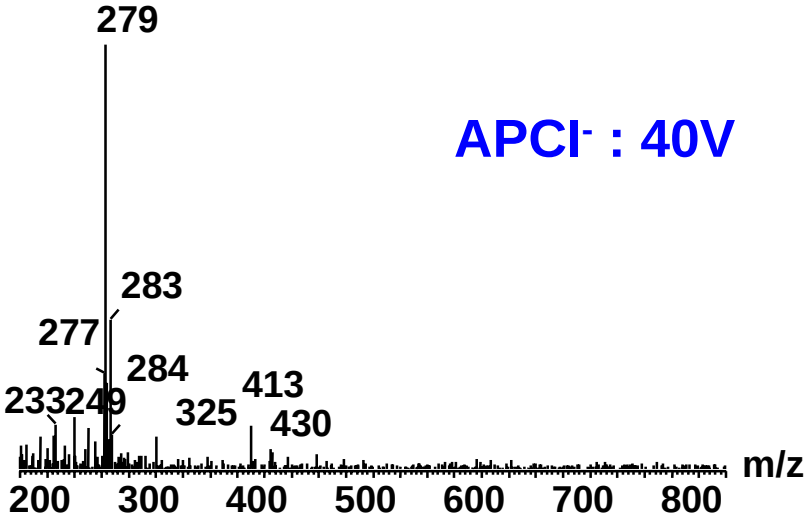
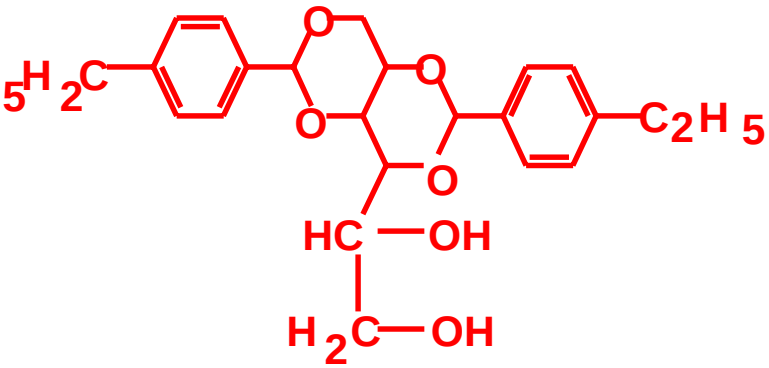
MS Spectra of the Peak #2









NC-4: MW 414

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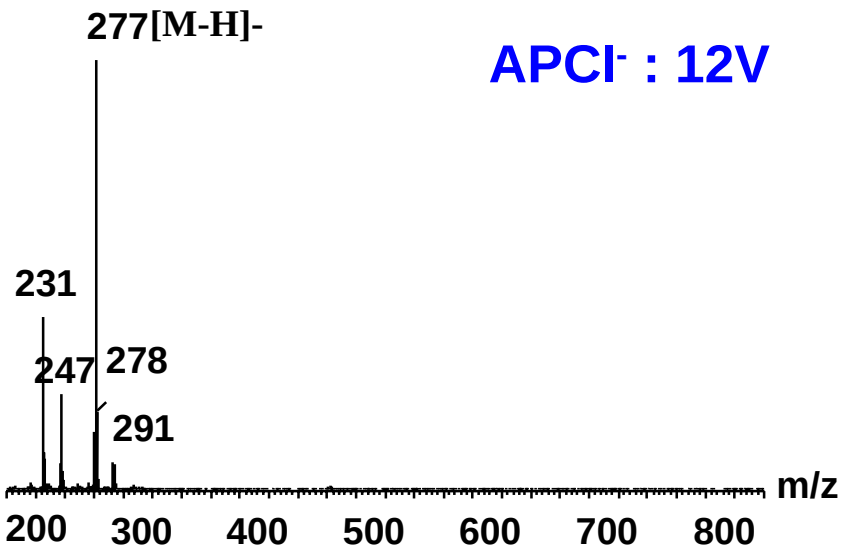
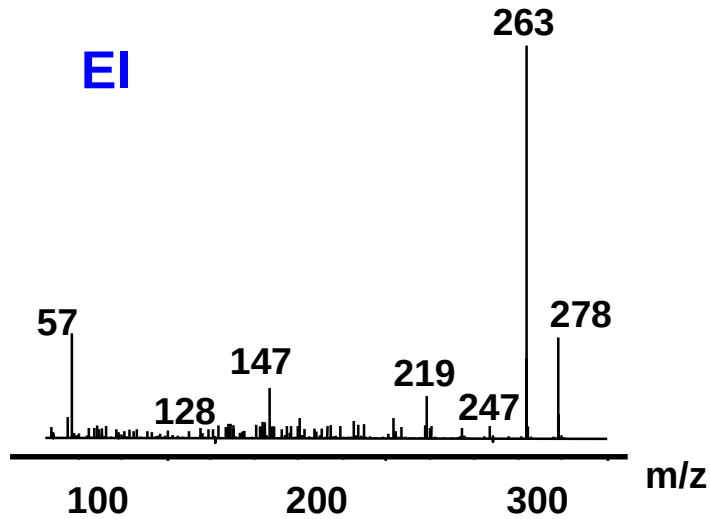
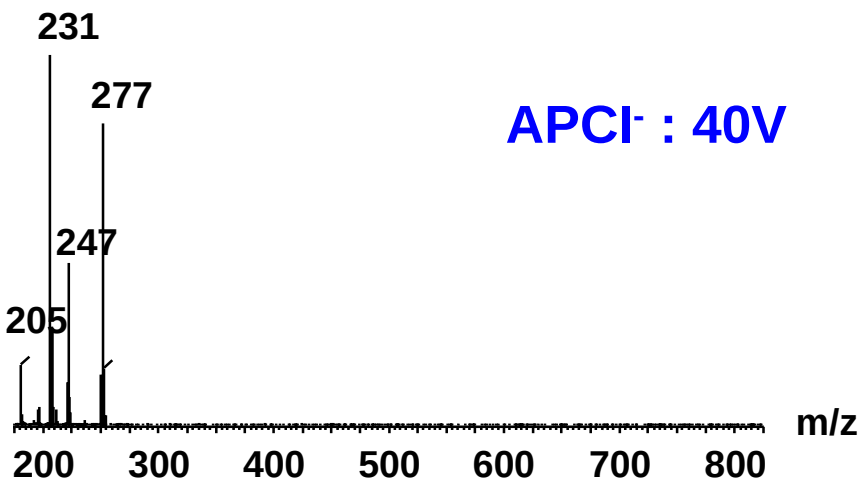
MS Spectra of Peak #3







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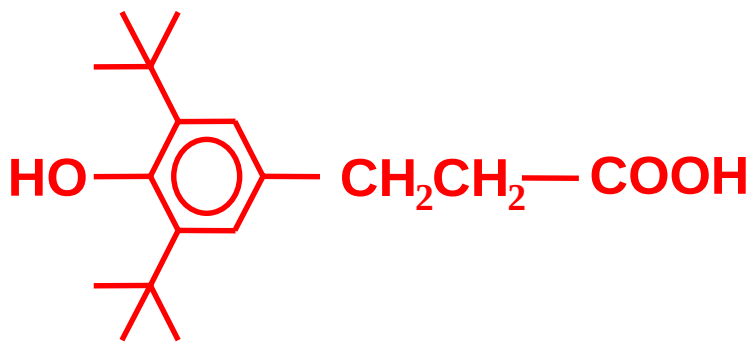
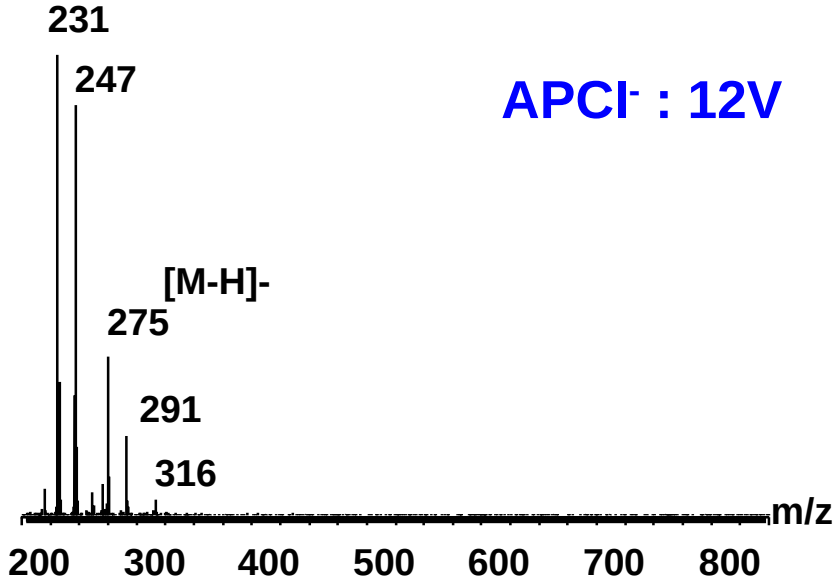
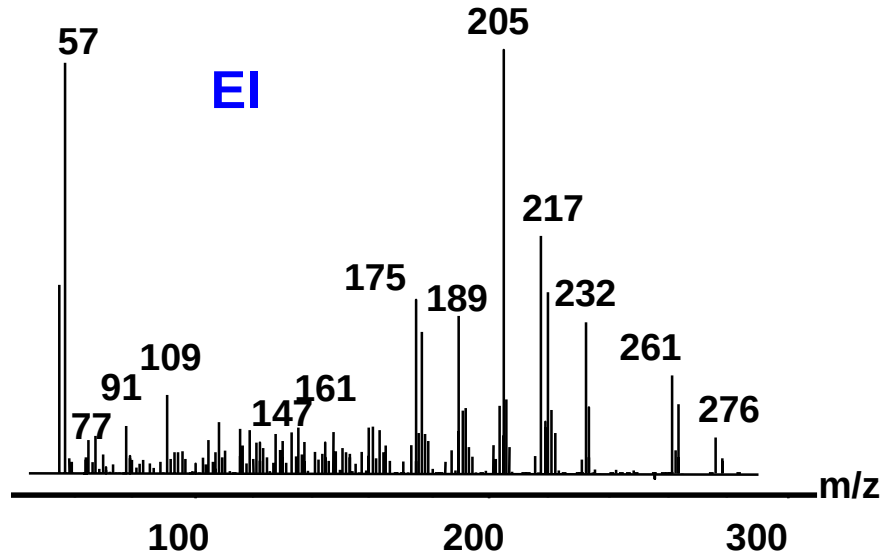


MW 278 (51% Library Match)

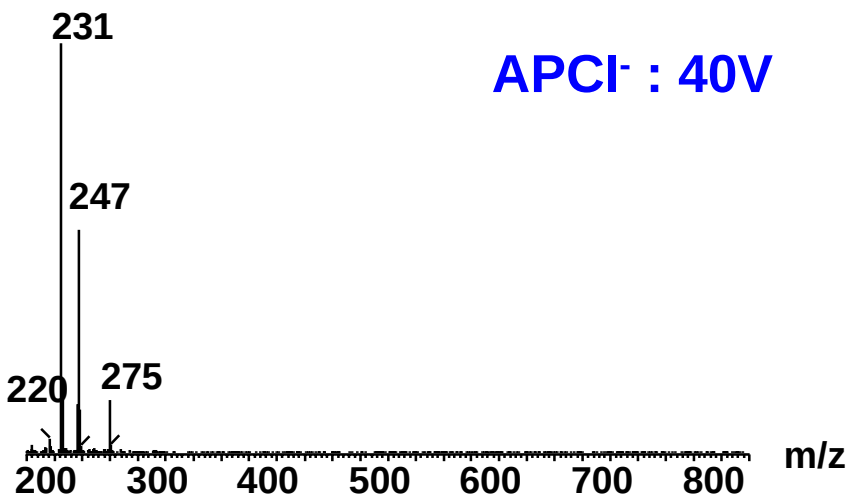
MS Spectra of Peak #4



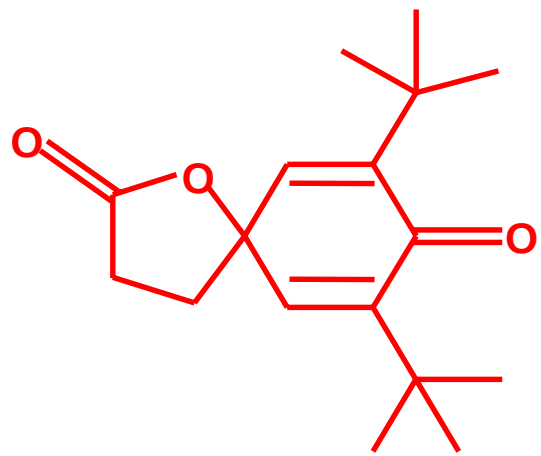
APCI$^-$: 12V



EI



APCI$^-$: 40V



MW 276 (98% Library Match)

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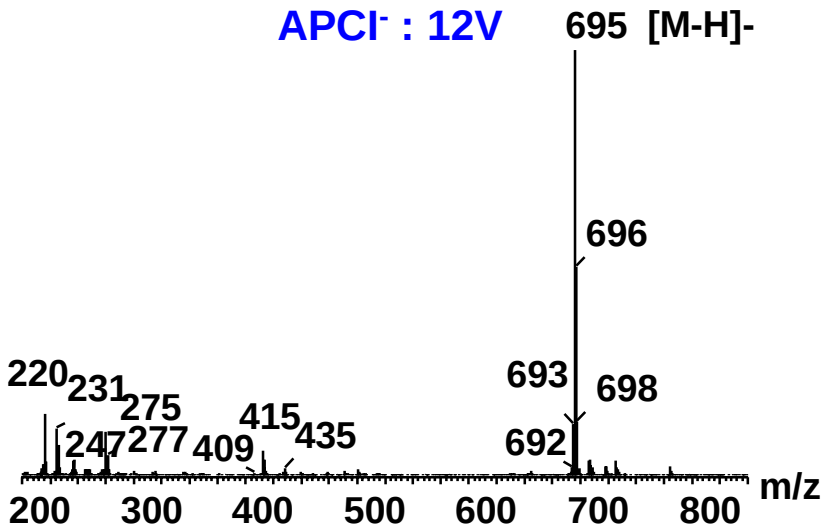
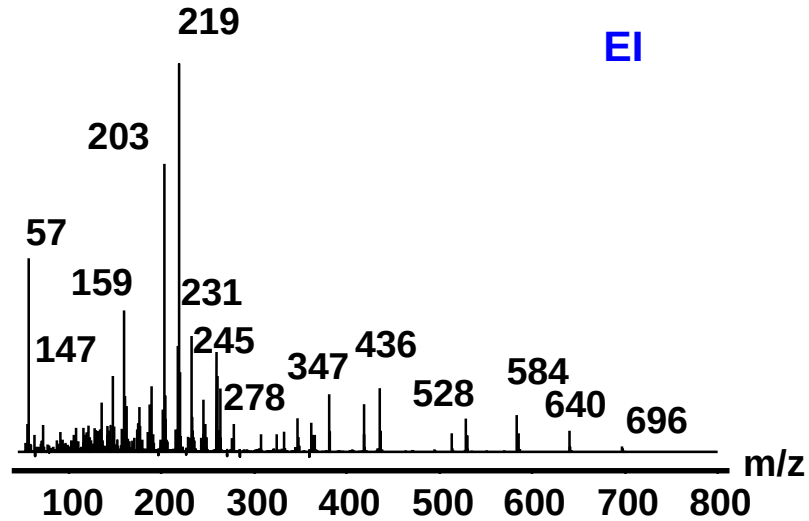
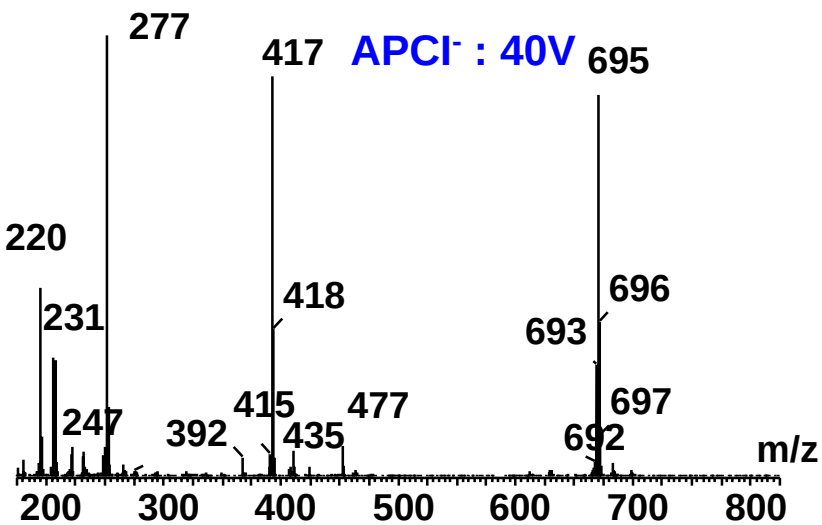
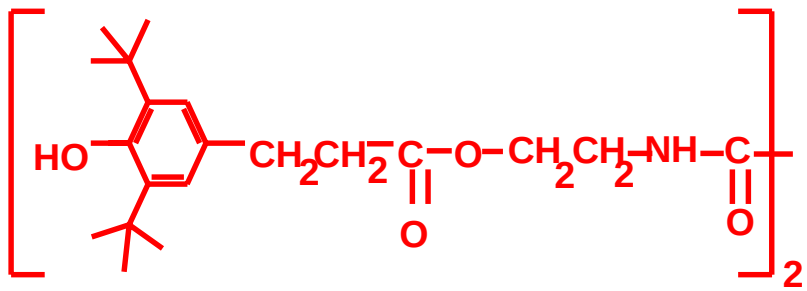
MS Spectra of Peak #5









Naugard: MW 696

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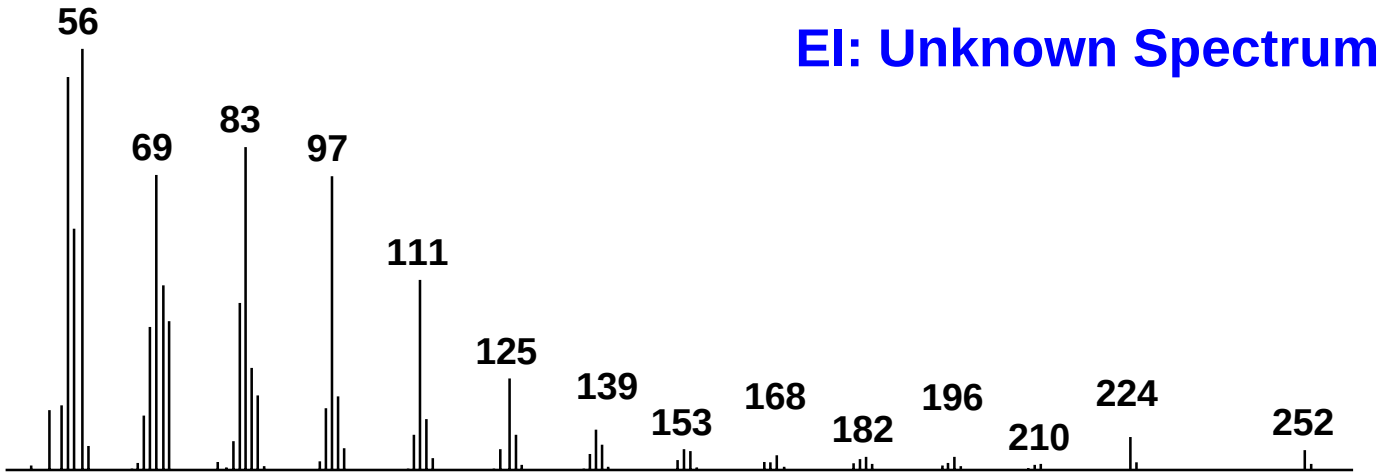
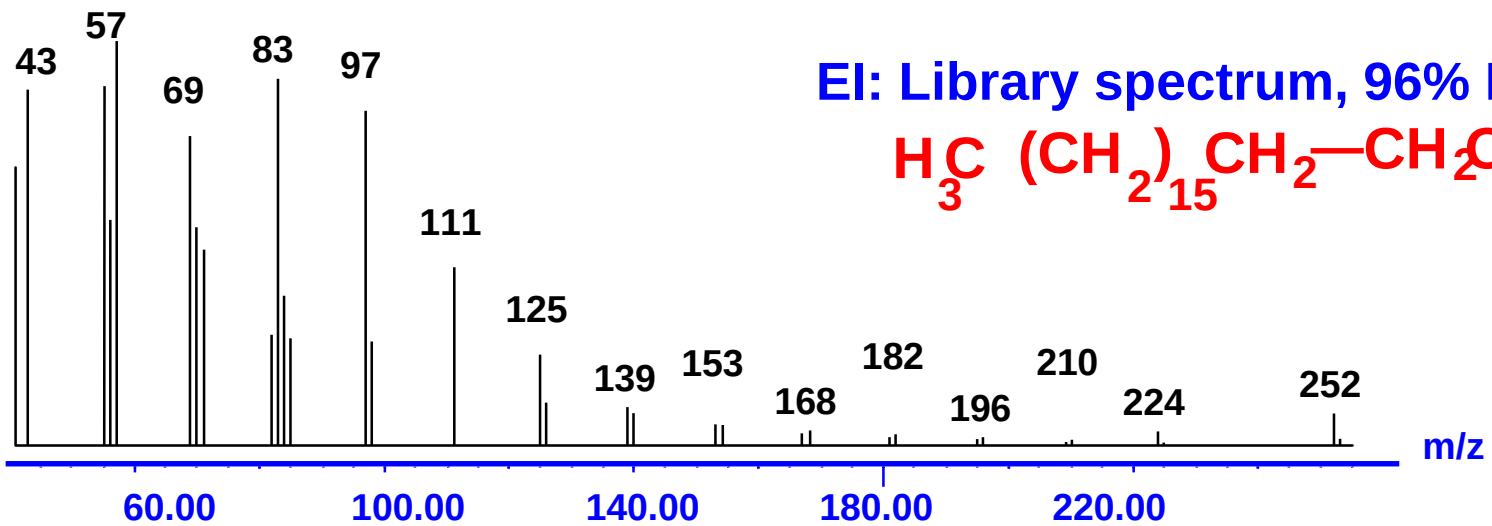
MS Spectra of Peak #6



EI: Unknown Spectrum



EI: Library spectrum, 96% Match

$H_3C\ (CH_2)_{15}CH_2—CH_2OH$

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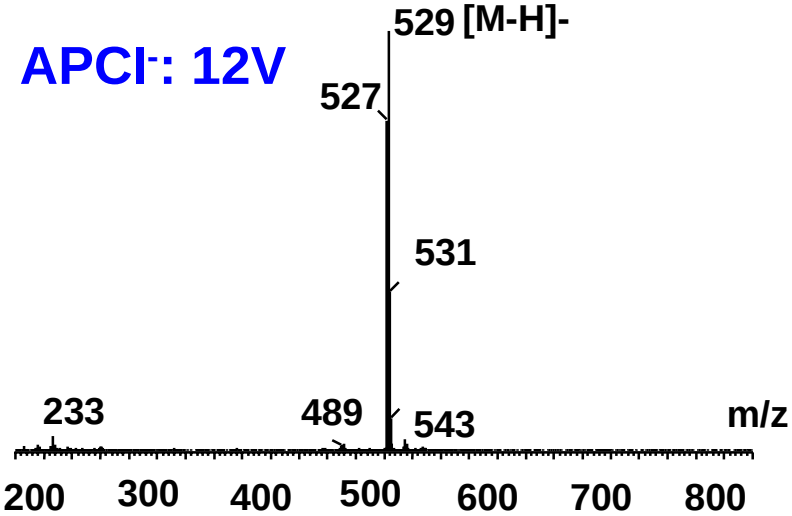
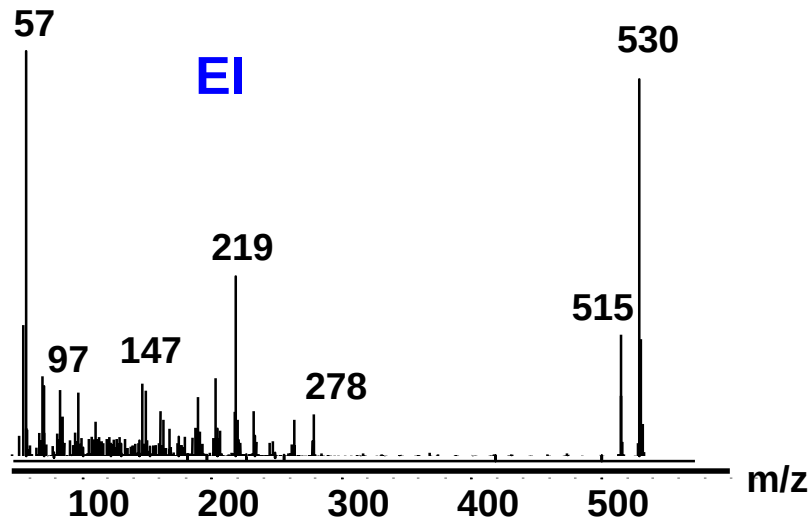
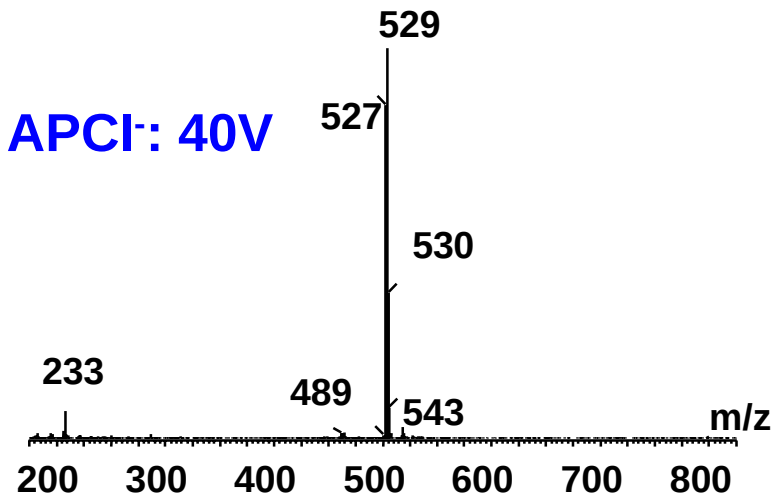
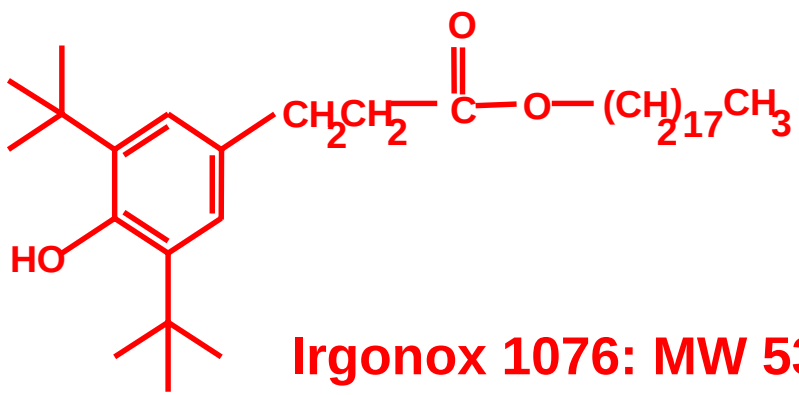
MS Spectra of Peak #7









Irgonox 1076: MW 530

(90% Library Match)

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Quantitation

- Single Ion Recording (SIR) for data collection
- Four calibration curves were made with four standards available
- $APCI^+$ was used for constructing the calibration curve
- Duplicate injection
- Linear range covers two orders of magnitude
- Amount in Original Polymer (w/w):

NC-4	Naugard-XL	1-Octadecanol	Irgonox1076
0.07%	0.04%	0.4%	0.06%

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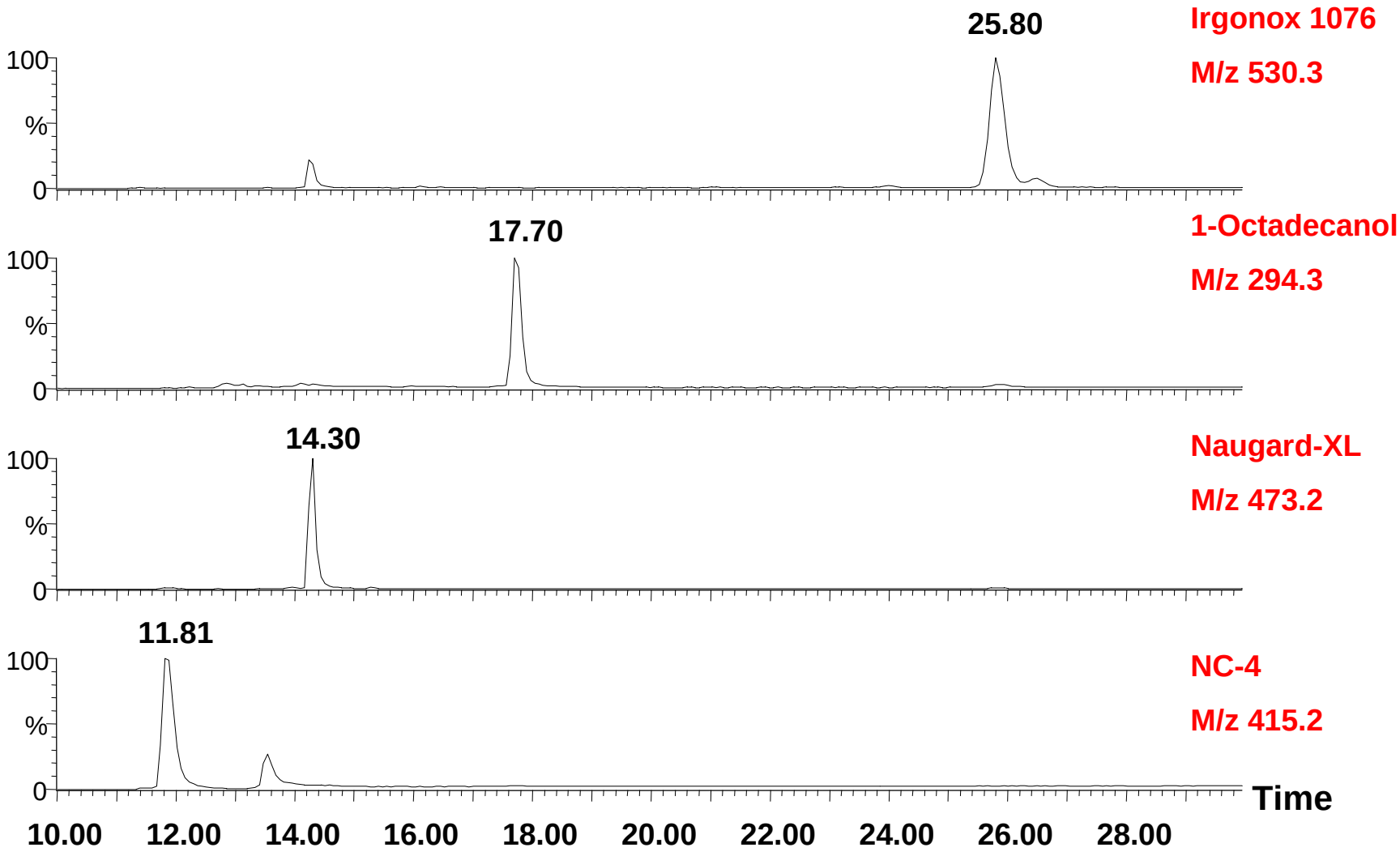
SIR Chromatograms



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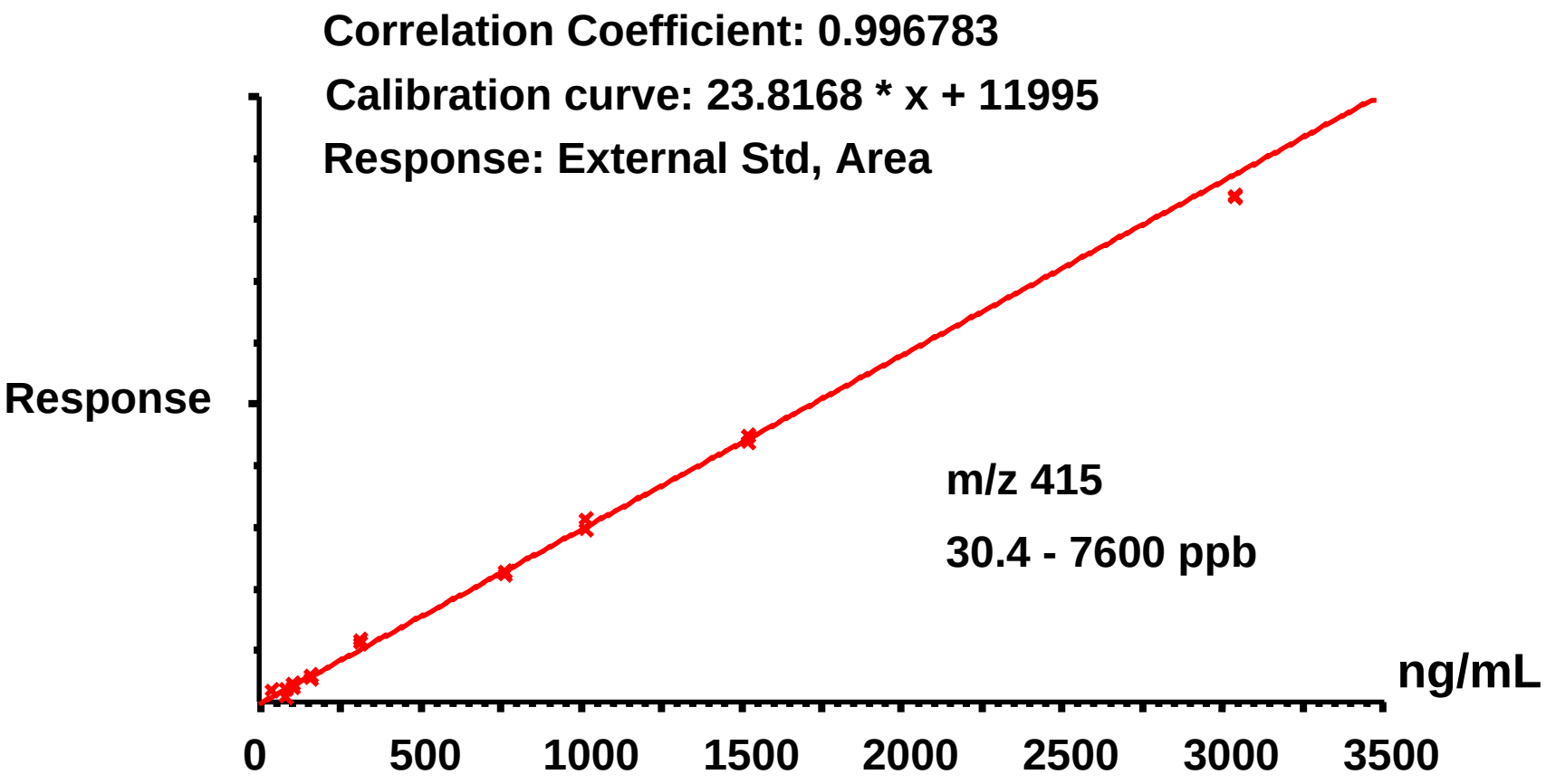
NC-4



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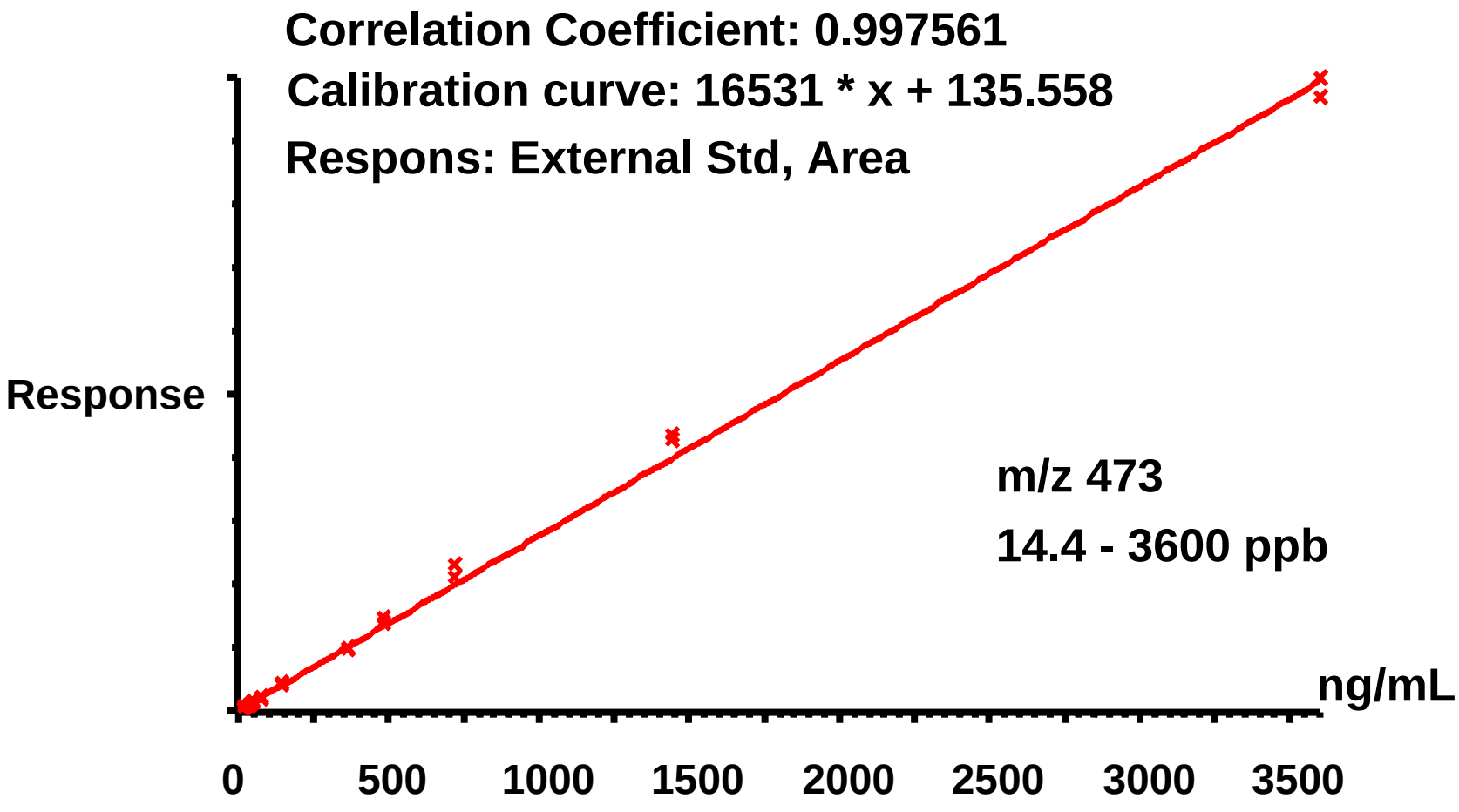
Naugard-XL



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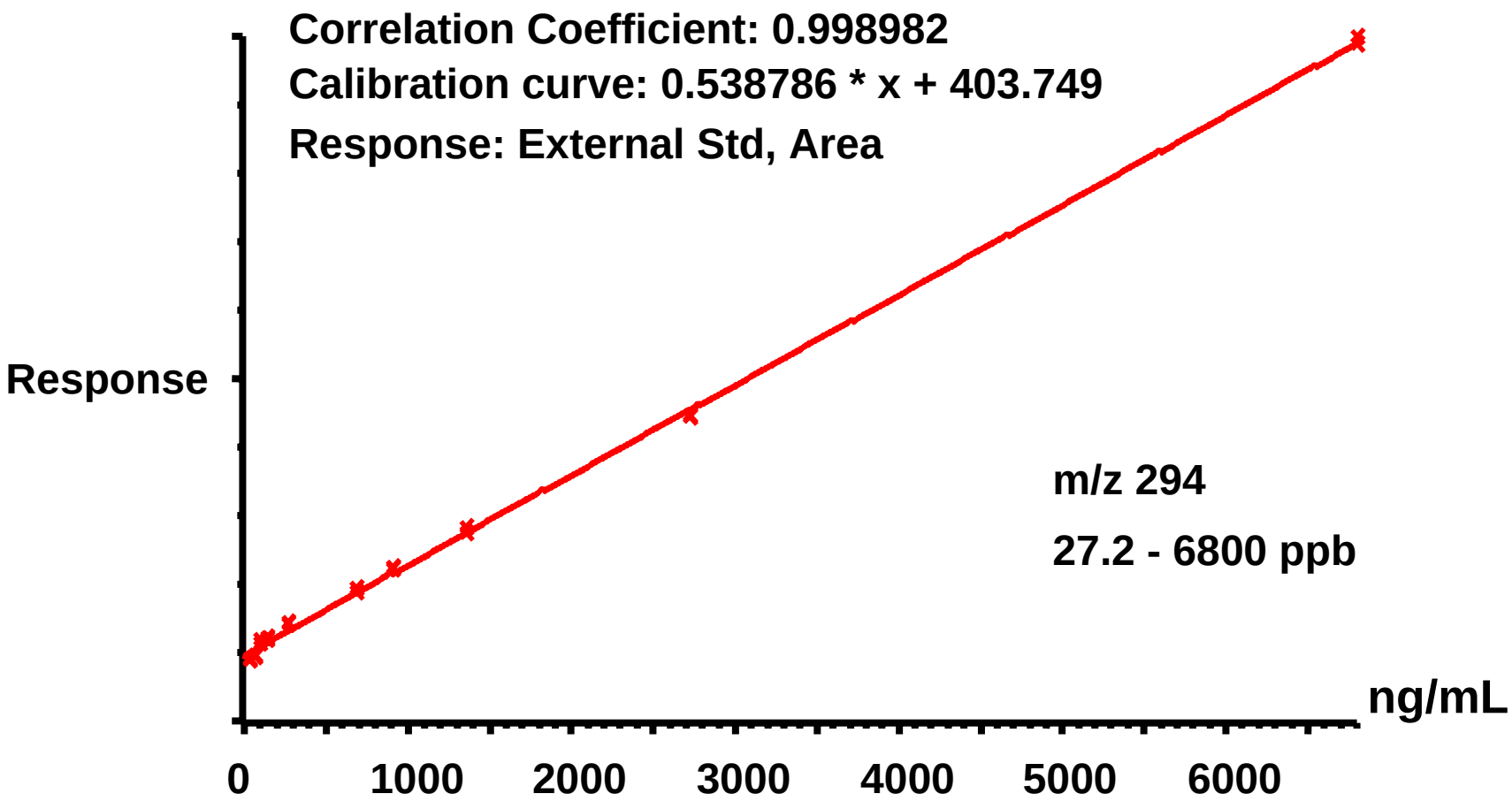
1-Octadecanol



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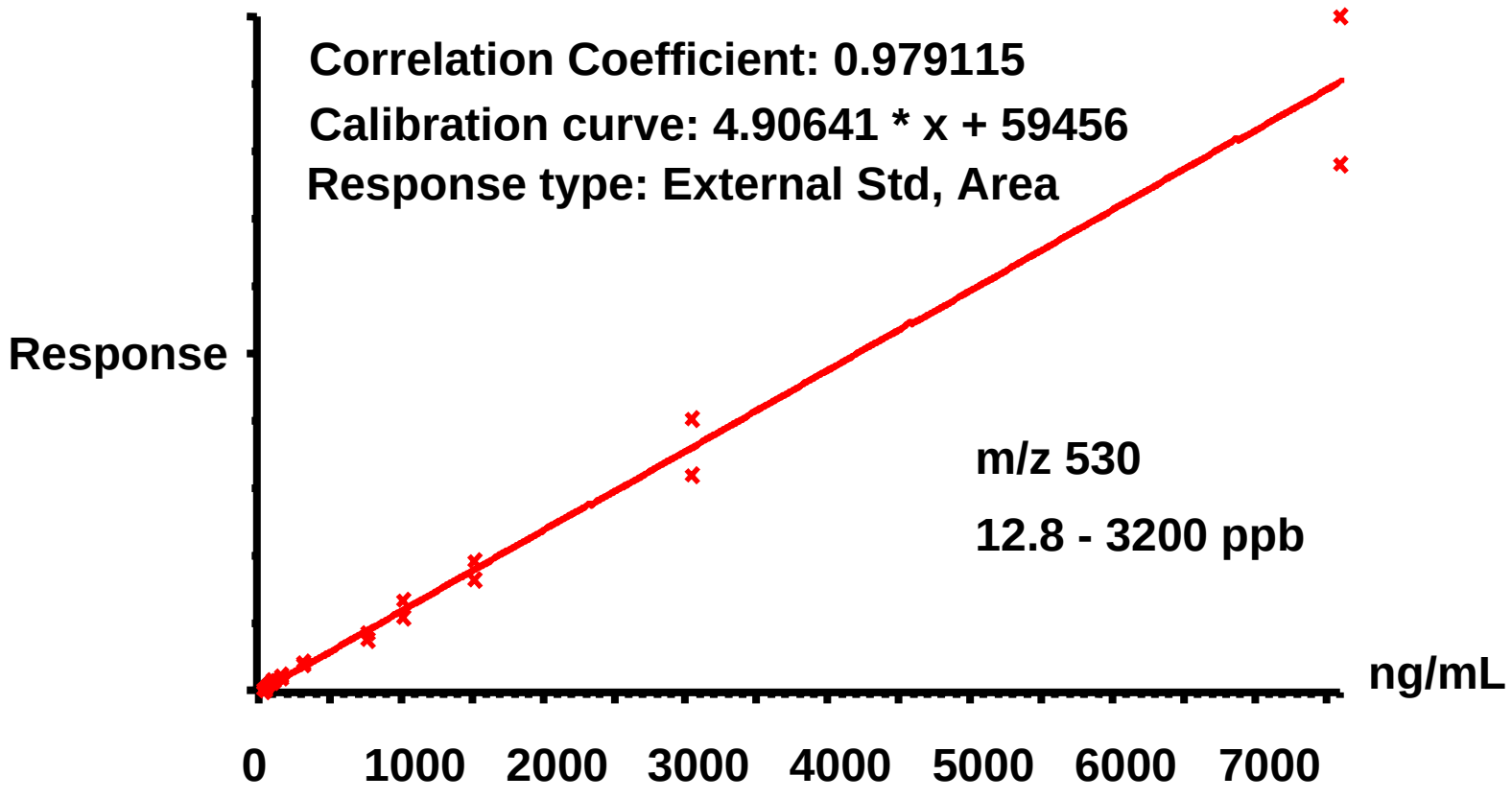
Irgonox 1076



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Conclusion

- Electron Ionization (EI) Offers Unknown Identification
  - Fragementation Pattern
  - Library Search
  - Structure Elucidation
- Atomospheric Pressure Ionization (API) Confirms EI Results
  - Molecular Weight Information
  - Limited Fragmentation by Up-front CID
- API Is Well Suited for Quantitation
  - Ion Monitoring Data Collection

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Acknowledgement

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- Dr. Craig Dorschel from Waters Corporation

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