

CPSC Symposium on Phthalates Screening and Testing Methods



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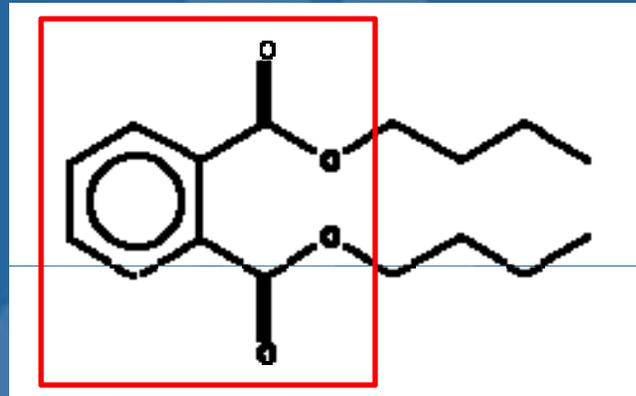
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Symposium Overview

- Presentations
 - Phthalate Testing Methods
 - Screening and/or Analysis Tools
- Break for Lunch
- Discussion Panel

Phthalates: Brief Background

- Class of chemical compounds often used as a plasticizer



- Plasticizers impart flexibility to otherwise rigid plastic



← PVC Pipe is rigid

PVC toy with plasticizer is pliable →



Section 108 of the CPSIA

- Permanent ban on children's toys or child-care articles containing more than 0.1% of DBP, BBP, or DEHP
- Interim ban on mouthable children's toys or child care articles containing more than 0.1% of DnOP, DINP, or DIDP
 - Effective until CHAP completes study and Commission issues final rule
- Phthalate limits apply to plasticized, accessible component parts

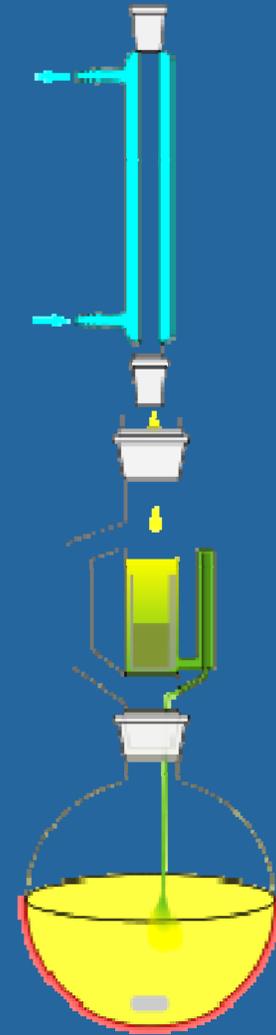
Phthalates Test Methods

- Methods consist of extraction and analysis steps
- CPSC-CH-C1001-09.3:
 - Dissolution in THF, precipitation with hexanes
 - 15-30 minutes with sonication
 - Analysis by GC-MS
 - Allows for several alternative methods



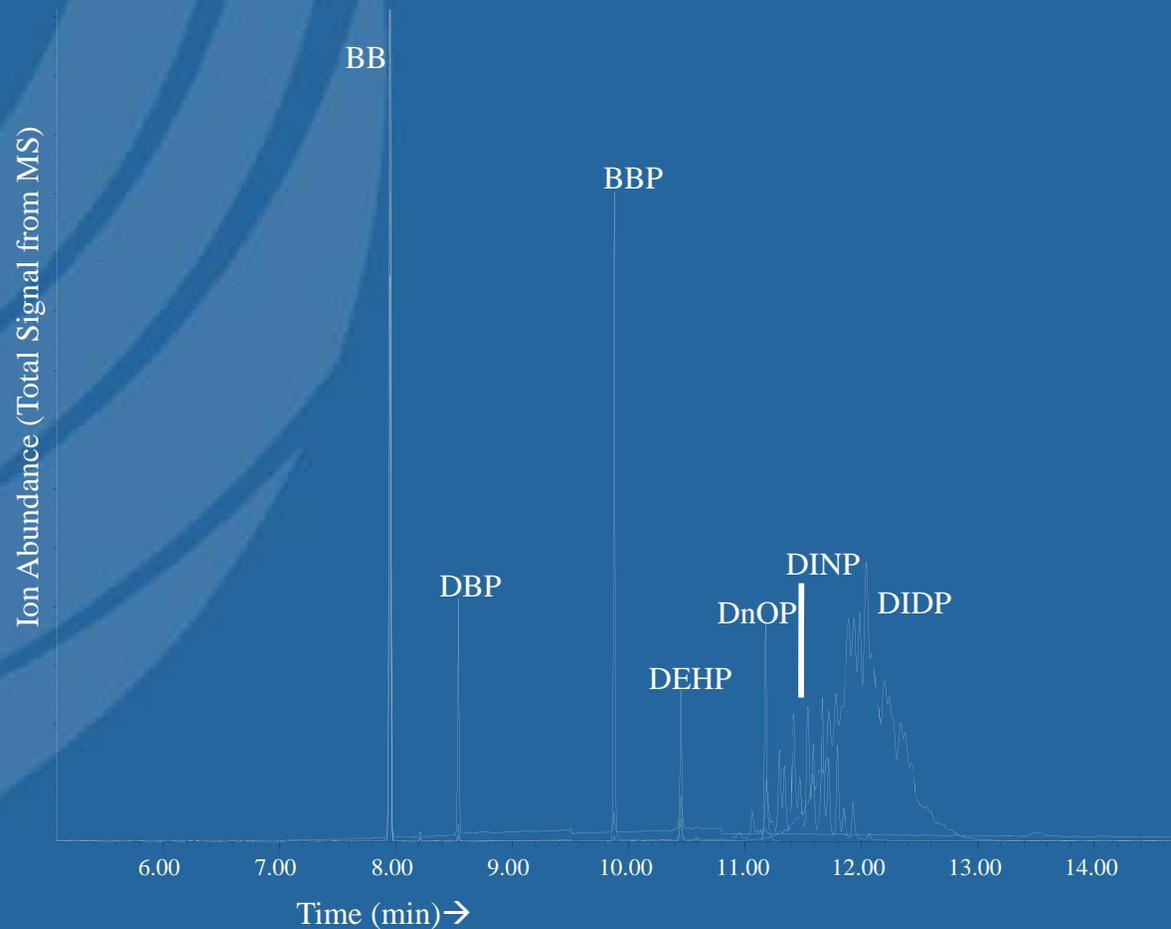
Alternative Methods

- Extraction Techniques:
 - Fluid (15 min to 12 hrs)
 - California Dept. of Toxic Substances Control, Health Canada C-34
 - Soxhlet (~6 hrs)
 - GB/ T 22048(Chinese method), EN14372(European method), EPA 3540C, etc.
 - Other
 - EPA microwave, PFE, ultrasonic
- Analysis by GC-MS



Analysis

- GC-MS is preferred analysis technique
 - Qualitative and quantitative
 - Can separate, differentiate phthalates and alternatives



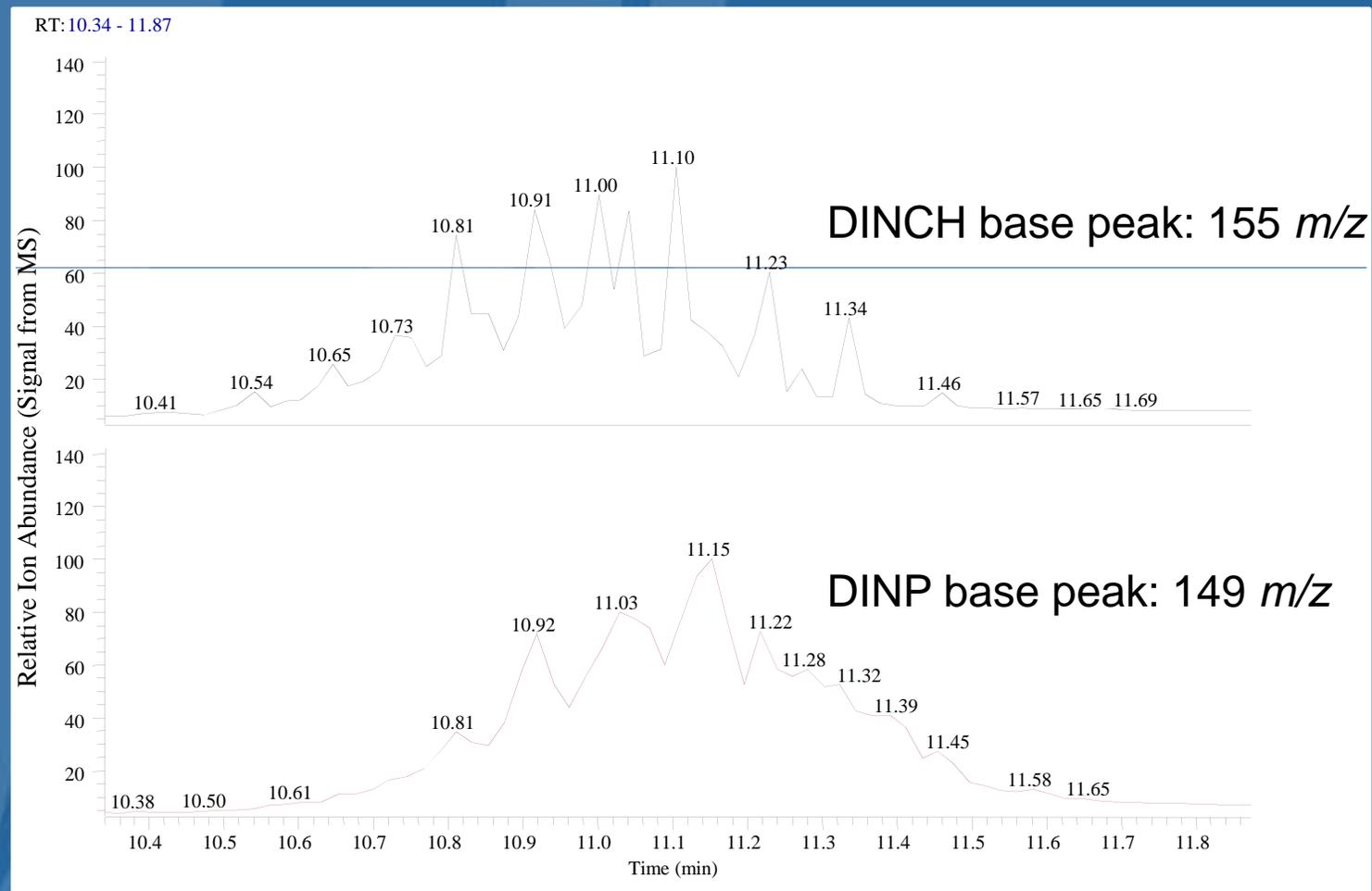
Common Problems

- Availability of Standard Reference Materials (SRM) of Phthalates in PVC
- False Positive Result
- Contamination
- Identity of DINP, DIDP
 - Different CAS #s

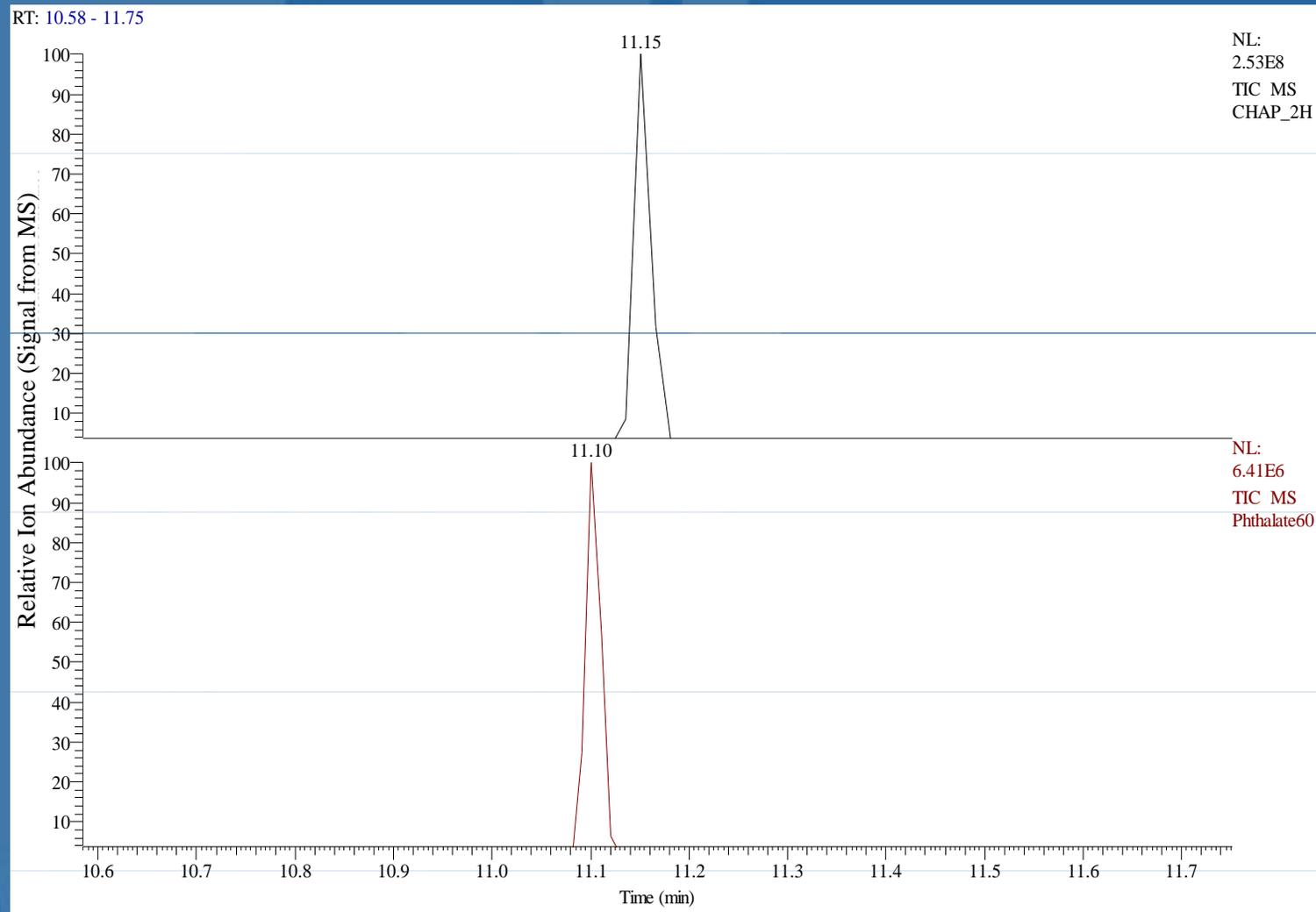
Addressing Common Problems

- Follow most current method (CPSC-CH-C1001-09.3)
- NIST developing SRM
- Contamination
- Qualitative Analysis by Technical Staff
 - Retention time match with standard
 - Mass spectrum match
 - Blank analysis
 - Ensure chromatogram peak shape match for DINP, DIDP

DINP vs. DINCH

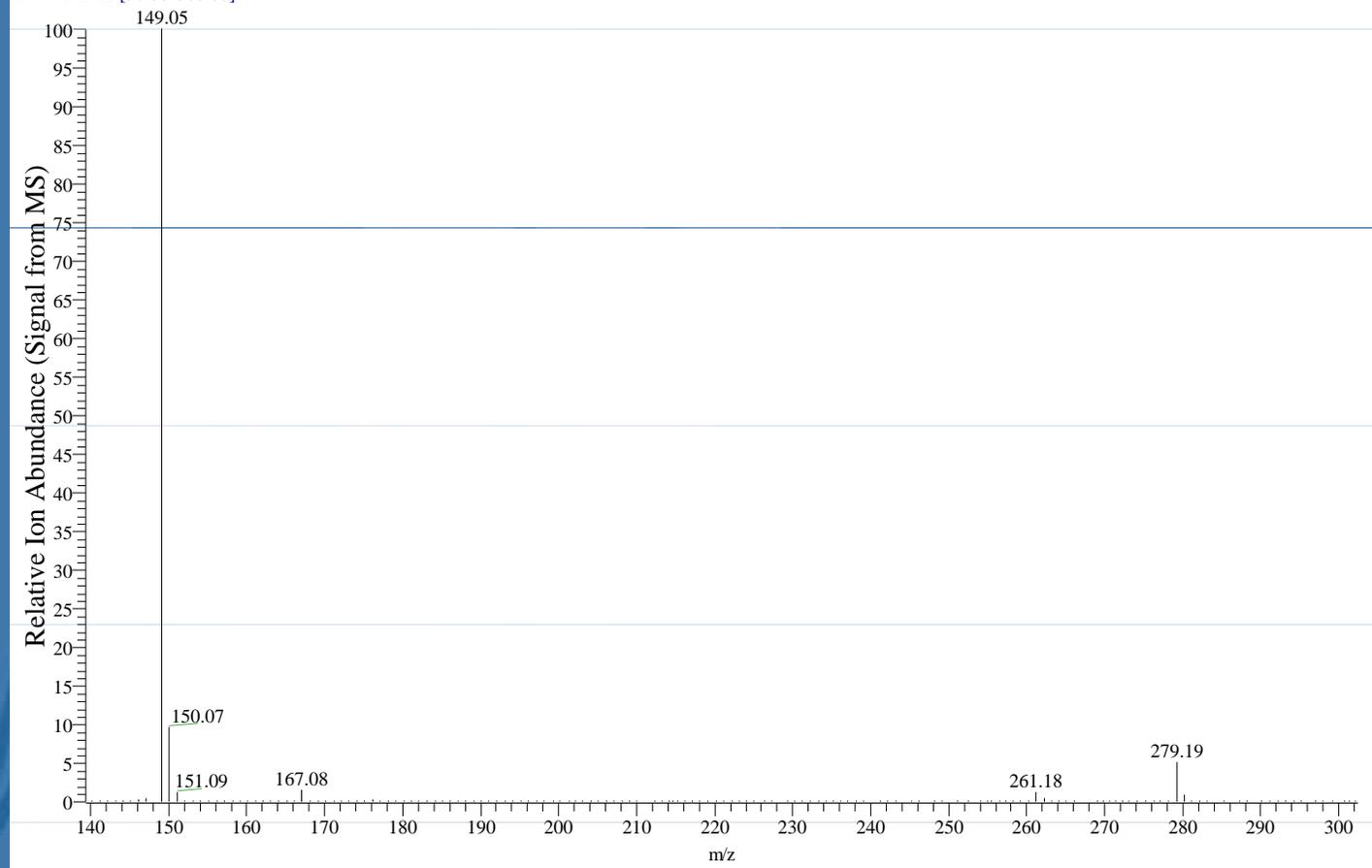


DnOP vs. DOTP



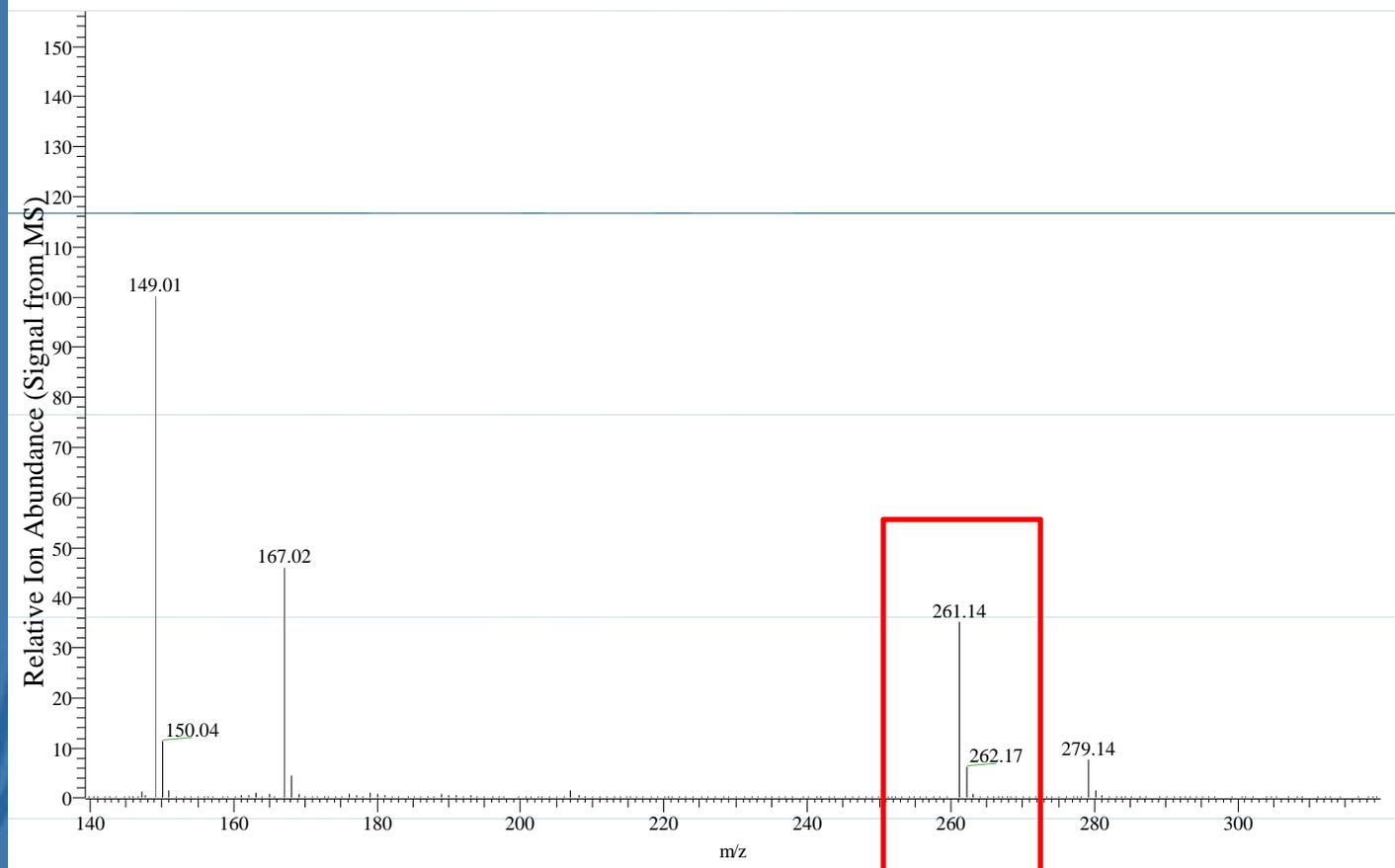
DnOP Mass Spectrum

300ppm_111207152118 #336 RT: 11.24 AV: 1 SB: 20 11.15-11.22, 11.27-11.47 NL: 1.76E8
T: + c Full ms [50.00-500.00]



DOTP Mass Spectrum

CHAP_2H #336 RT: 11.15 AV: 1 NL: 2.05E7
T: + c Full ms [50.00-500.00]



Emerging Technologies

- Portable Spectroscopy
- Colorimetric¹
 - Modified gold nanoparticles change color in water solution when phthalates present
- DART-MS²
 - No sample prep; real time data
 - No quantification method (...yet)
 - Cannot distinguish isomers (...yet)

1) Zhang et al., Chem. Commun., 2011; 47: 11849-11851

2) Rothenbacher et al., Rapid Commun. Mass Spectrom. 2010; 24: 21-29; *multiple others*

Other Sources of Information

- www.cpsc.gov/phthalates
- Chronic Hazard Advisory Panel (CHAP)
 - www.cpsc.gov/about/cpsia/chapmain.html
- ASTM D20: Workgroup WK25759
- EPA Design for the Environment: Alternatives to Phthalates
 - www.epa.gov/dfe