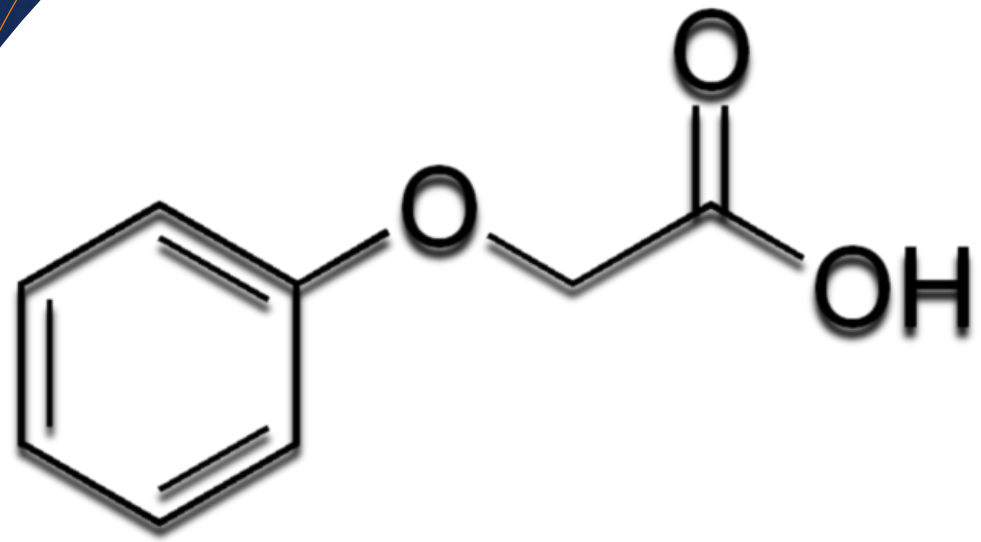
SESSION: OPTIMIZING LABORATORY OPERATIONS

Enhancing Resiliency and Sustainability in Environmental Labs – a Case Study for Herbicides Analysis



Chlorinated Phenoxy Herbicides

Julian Martinez
Technical Director, Eurofins Environment Testing

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Why This Matters?

- Case study addressing key workflow challenges.
- Modernization of chlorinated phenoxy herbicide analysis in soil and water

Environmental labs face increasing pressure from:

Global supply chain disruptions

Natural disasters

Instrumentation and facility constraints

Legacy Method – EPA 8151A (GC-ECD)

- EPA 8151A ~ Rev 1, December 1996
- Hazardous reagents to perform the method
- Relies on RT and secondary column confirmation
- GC-ECD method more subject to interference
- TAT 5-10 days on average
- Complex method preparation; poor recoveries

~~Diazomethane/Diazald~~

Why method 8321 (LC-MS/MS)

- Retains 8151A target list
- Greater specificity with MRM detection
- Reduced sample volume and simpler prep
- Safer reagents: no derivatization, no chlorinated solvents
- Shorter TAT without compromising sensitivity

Greener, Streamlined Sample Prep

Elimination of potential harmful solvents and reagents

Only MeOH & Water are used

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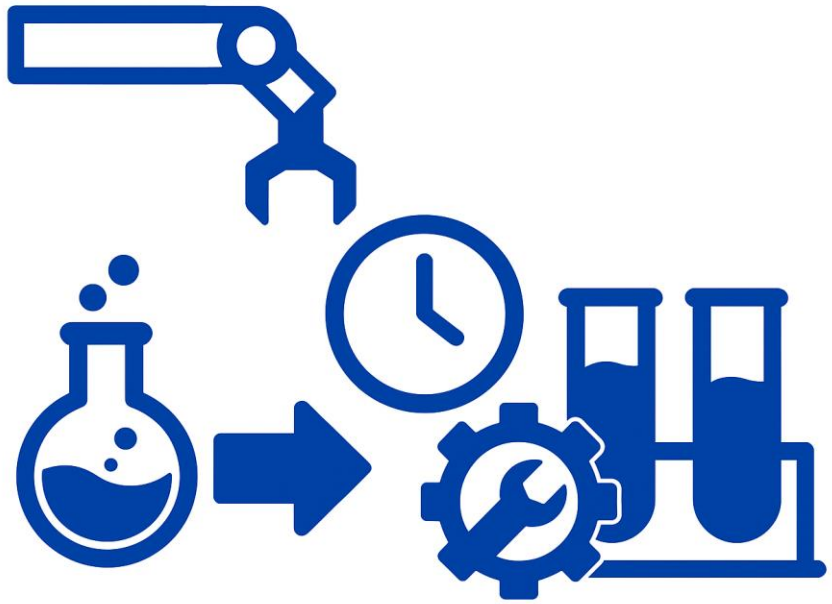
Greener, Streamlined Sample Prep

Key Advantages:

- 6x reduction in workflow time
- No hazardous solvents: Only methanol, acetonitrile, and water
- Increased analyst safety and simplified compliance
- 25 samples processed in 4 hours



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FAST analysis by LC-MS/MS

- Short run time (7 minutes cycle time)
  - 205 injections in 24 hours
  - MRM (Multiple Reaction Monitoring)
- LC column
  - Good for separating polar compounds
  - Stable for use in acid mobile phase (pH 2-3)
- Mobile phase (0.04% acid)
  - Acid selection (volatile)
    - Acetic acid (CH3COOH)

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Validation Summary

- Low level detections
- Good recoveries
- Accurate & Precise
- Method designed for water & soil
- Direct Comparison between GC Method and LC - MSMS method
- Parallel testing study between 8321 & 8151
- Superior PT performance

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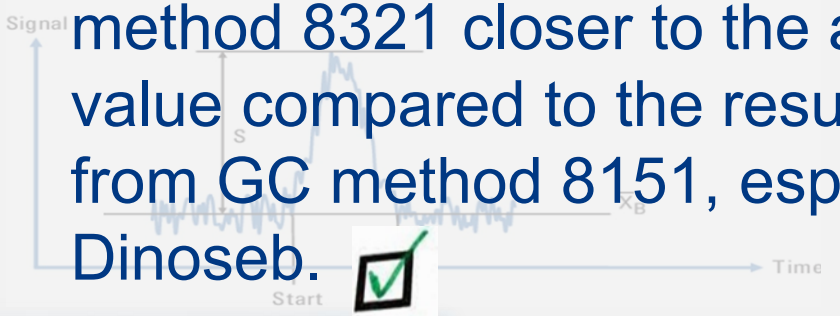
➢ *Superior PT performance*

Herbicides in Soil		Phenova Lot#7088-18					
NELAC Analyte Code	Analyte	Units	8321 Reported Value	8151 Reported value	Assigned Value	Acceptance Limits	8321 Analysis Date
8545	2,4-D	ug/Kg	647	156	594	59.4-998	5/9/2024
8560	2,4-DB	ug/Kg	397	232	363	36.3-504	5/9/2024
8555	Dalapon	ug/Kg	<10	<100	<100	0.00-100	5/9/2024
8595	Dicamba	ug/Kg	198	85.2	195	19.5-315	5/9/2024
8605	Dichlorprop	ug/Kg	678	163	534	53.4-775	5/9/2024
8620	Dinoseb	ug/Kg	197	107	199	19.9-285	5/9/2024
7775	MCPA	ug/Kg	<10	<1000	<1000	0.00-1000	5/9/2024
7780	MCPP	ug/Kg	<10	<1000	<1000	0.00-1000	5/9/2024
8655	2,4,5-T	ug/Kg	775	154	701	70.1-1000	5/9/2024
8650	2,4,5-TP	ug/Kg	988	286	835	83.5-1500	5/9/2024
6605	Pentachlorophenol	ug/Kg	832	328	844	84.4-928	5/9/2024

Herbicides in Water		ERA	Lot#042324P			
NELAC Analyte Code	Analyte	Units	8321 Reported Value	Assigned Value	Acceptance Limits	8321 Analysis Date
8545	2,4-D	ug/L	2.15	3.48	0.348-5.55	4/25/2024
8560	2,4-DB	ug/L	7.13	9.48	1.15-15.6	4/25/2024
8555	Dalapon	ug/L	3.24	4.85	0.00-7.64	4/25/2024
8595	Dicamba	ug/L	7.36	8.55	2.05-11.9	4/25/2024
8605	Dichlorprop	ug/L	3.66	4.89	0.941-7.26	4/25/2024
8620	Dinoseb	ug/L	5.82	5.02	0.00-7.83	4/25/2024
7775	MCPA	ug/L	<1.0	<10.0	0.00-10.0	4/25/2024
7780	MCPP	ug/L	<1.0	<10.0	0.00-10.0	4/25/2024
8655	2,4,5-T	ug/L	4.36	7.94	1.78-11.4	4/25/2024
8650	2,4,5-TP	ug/L	2.28	3.26	0.814-4.93	4/25/2024
6605	Pentachlorophenol	ug/L	8.93	8.00	1.06-11.9	4/25/2024

➢ Aqueous and soil PT results for method 8321 closer to the assigned value compared to the result obtained from GC method 8151, especially for Dinoseb. ☑



Reported value: 197 ug/kg
Assigned: 199 ug/kg

Reported value: 5.82 ug/L
Assigned value: 5.02 ug/L

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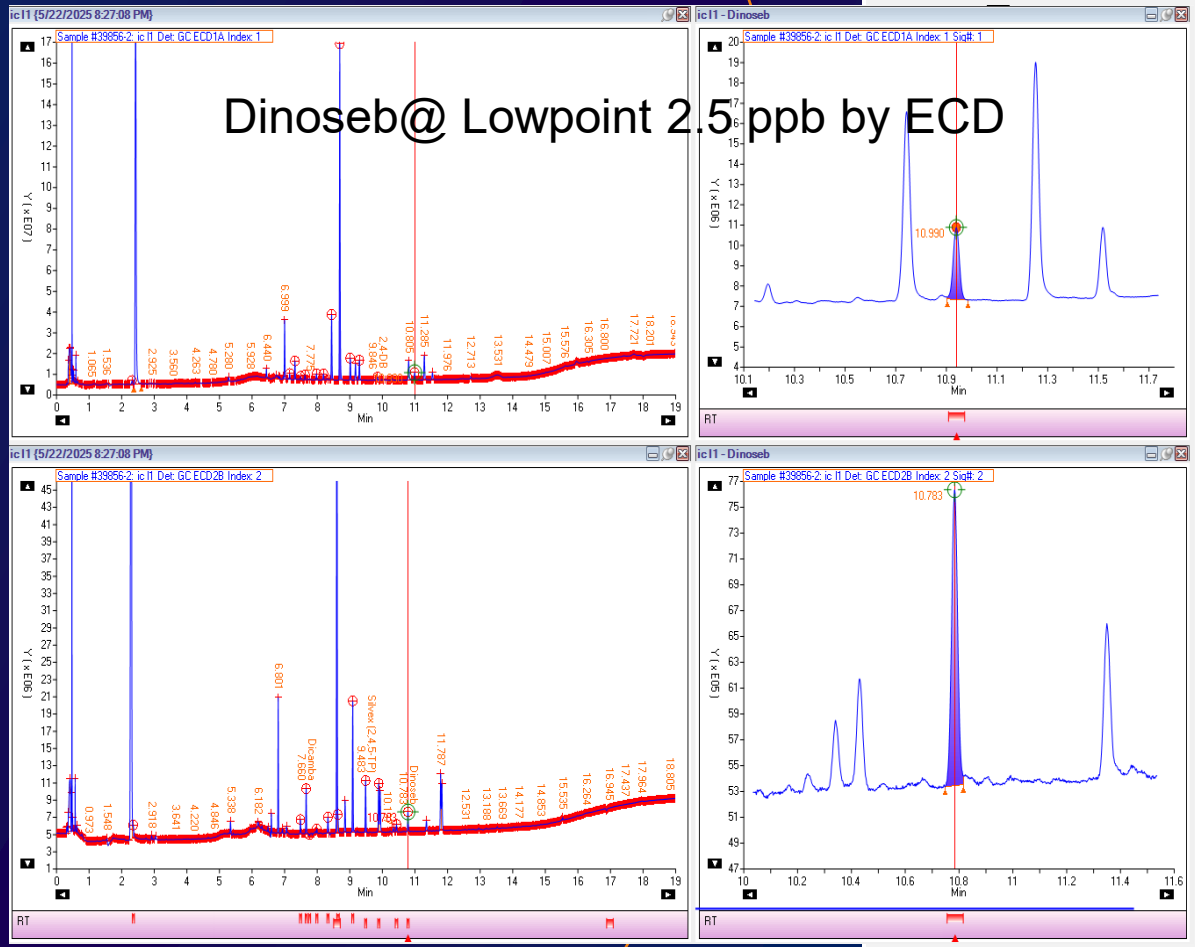
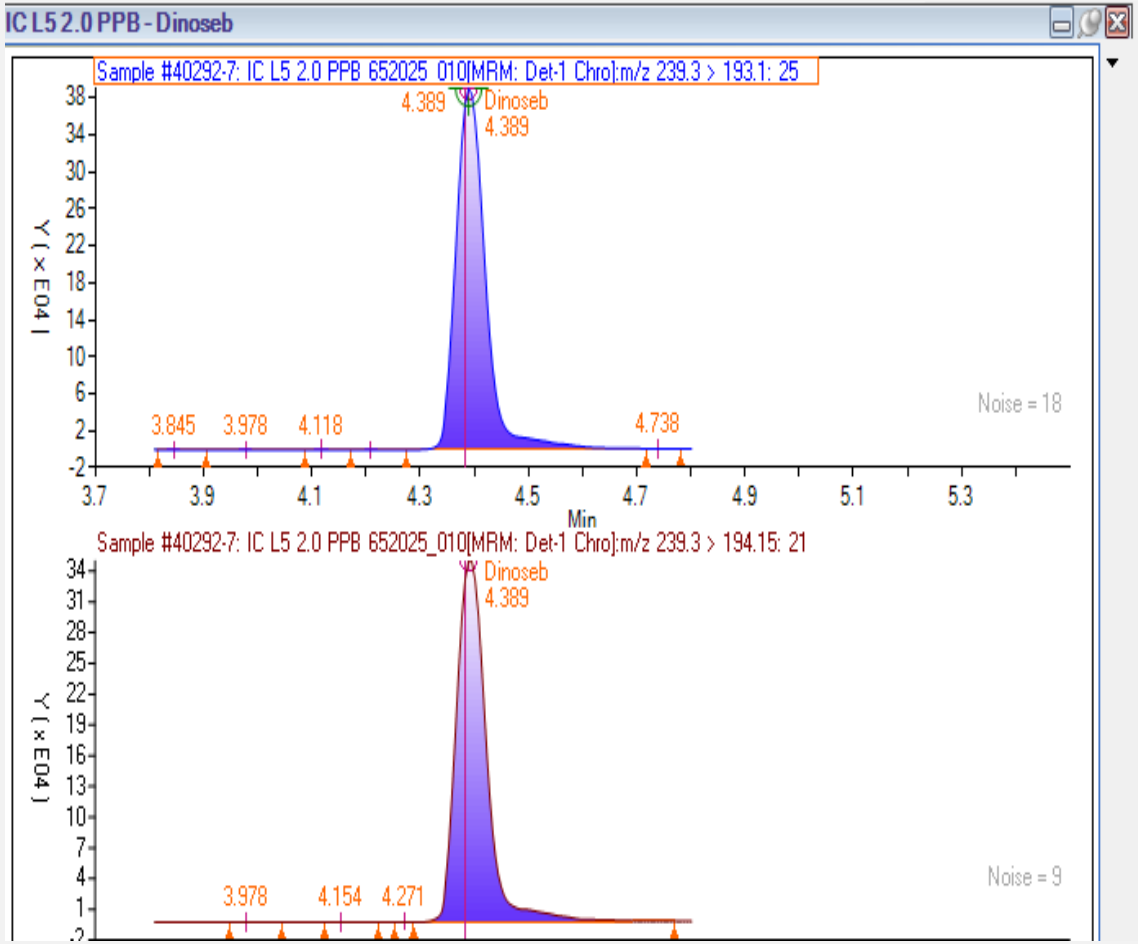
- **Peak Identification**
- %R of LCS/D, MS/D

Problematic Compounds: DINOSEB & DALAPON

Dinoseb@ Lowpoint 2.5 ppb by ECD



Dinoseb@ Midpoint 2.0 ppb by LC/MSMS



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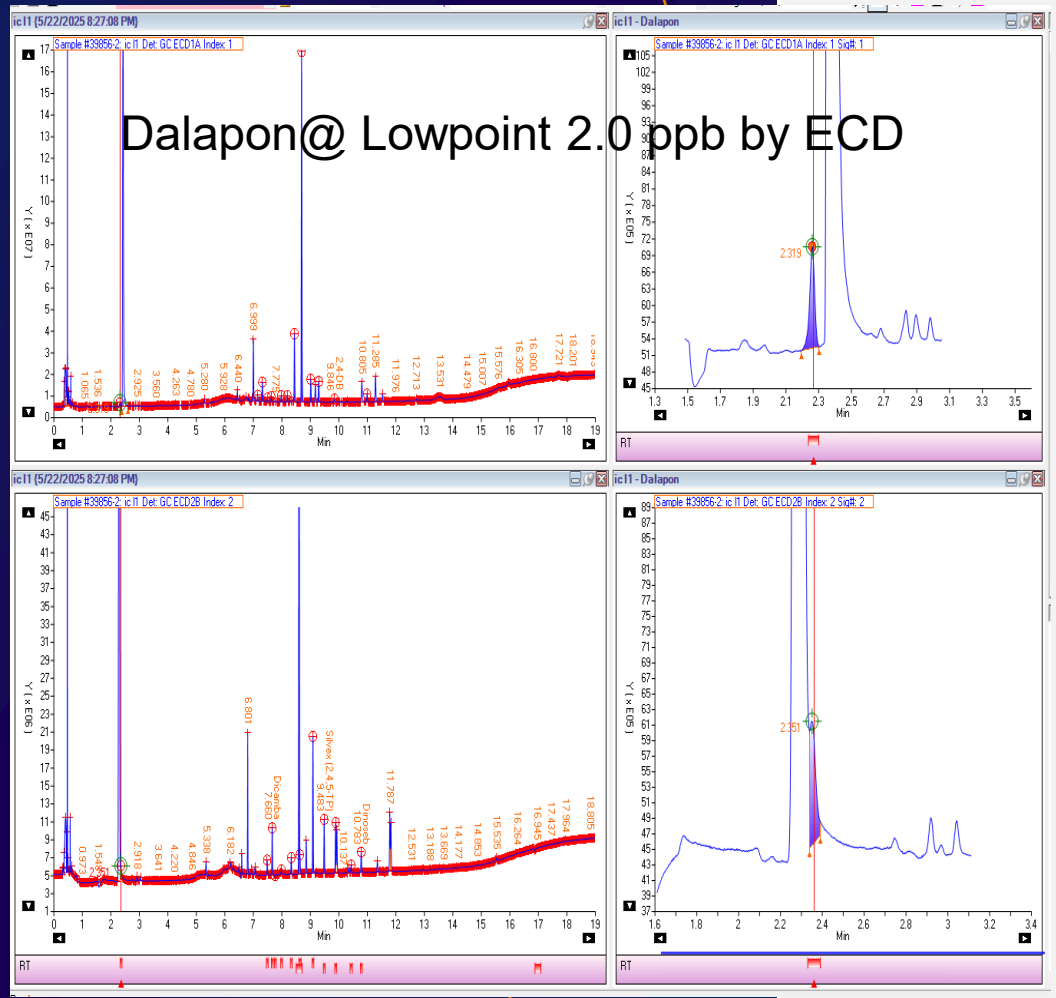
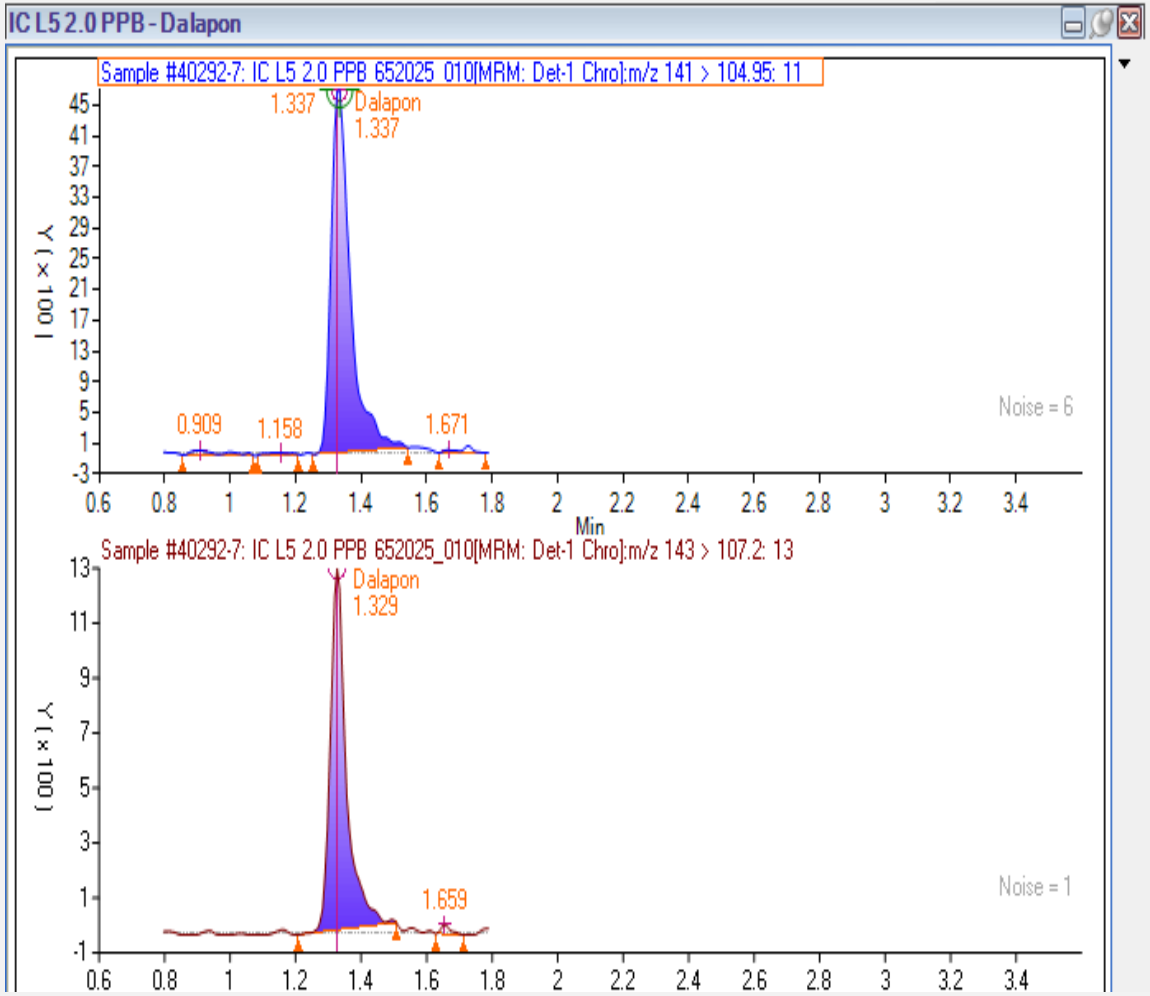
- **Peak Identification**
- %R of LCS/D, MS/D

Problematic Compounds: DINOSEB & DALAPON

Dalapon@ Lowpoint 2.0 ppb by ECD



Dalapon@ Midpoint 2.0 ppb by LC/MSMS



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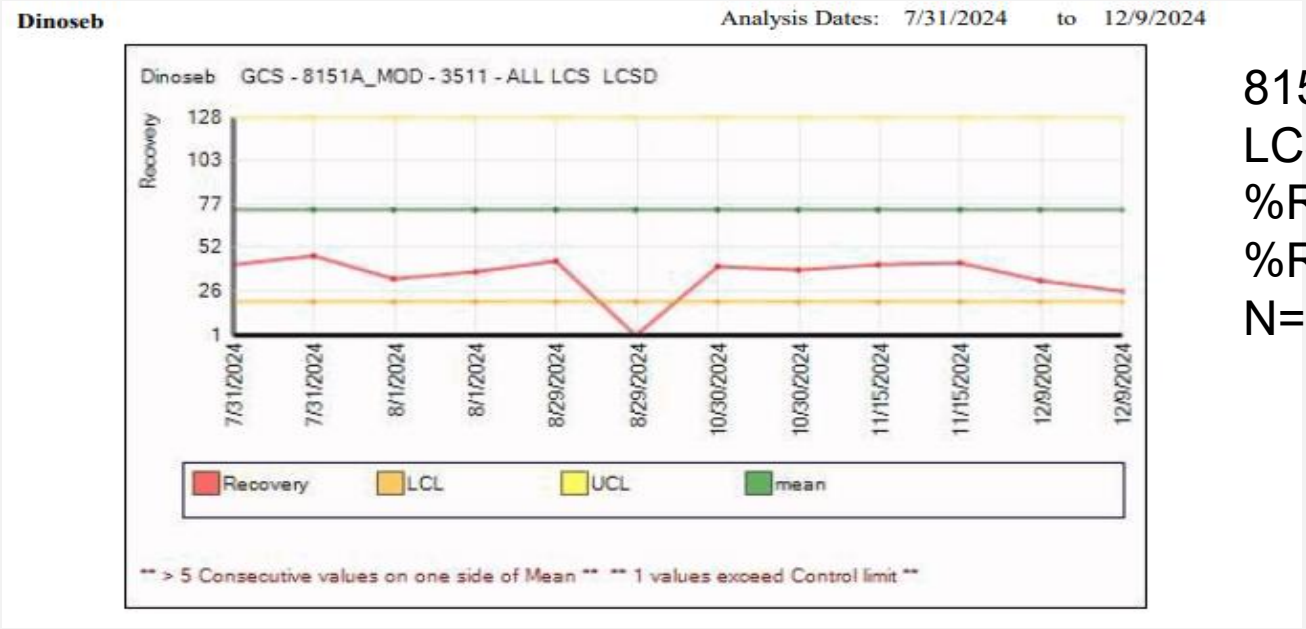
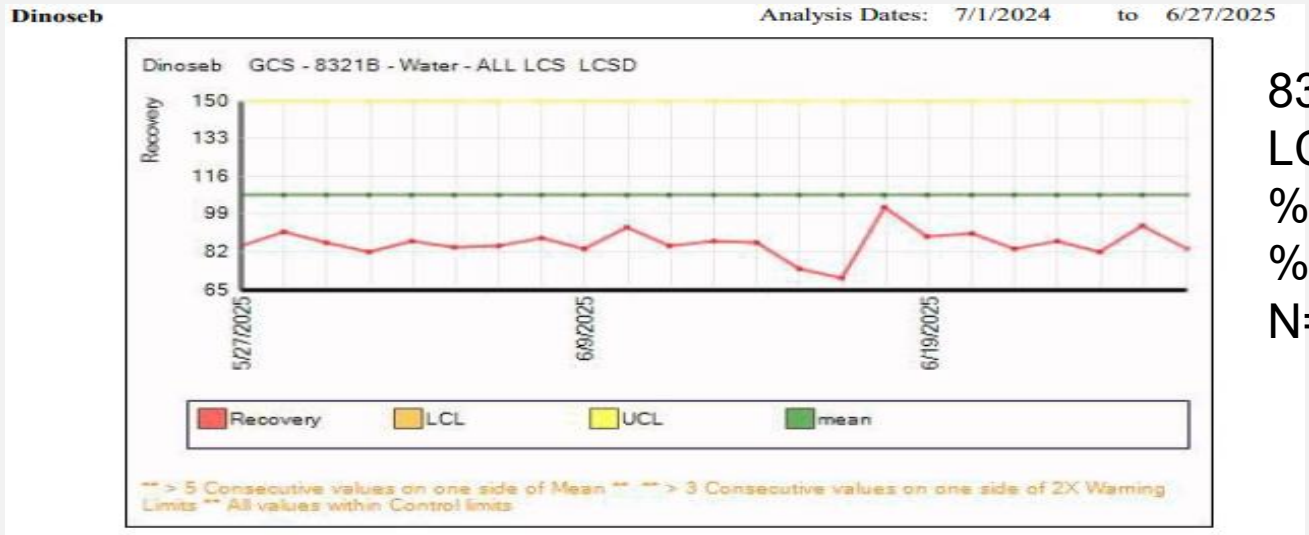
Problematic Compounds: DINOSEB
8151 vs 8321 Water Matrix

- Peak Identification
- **%R of LCS/D, MS/D**



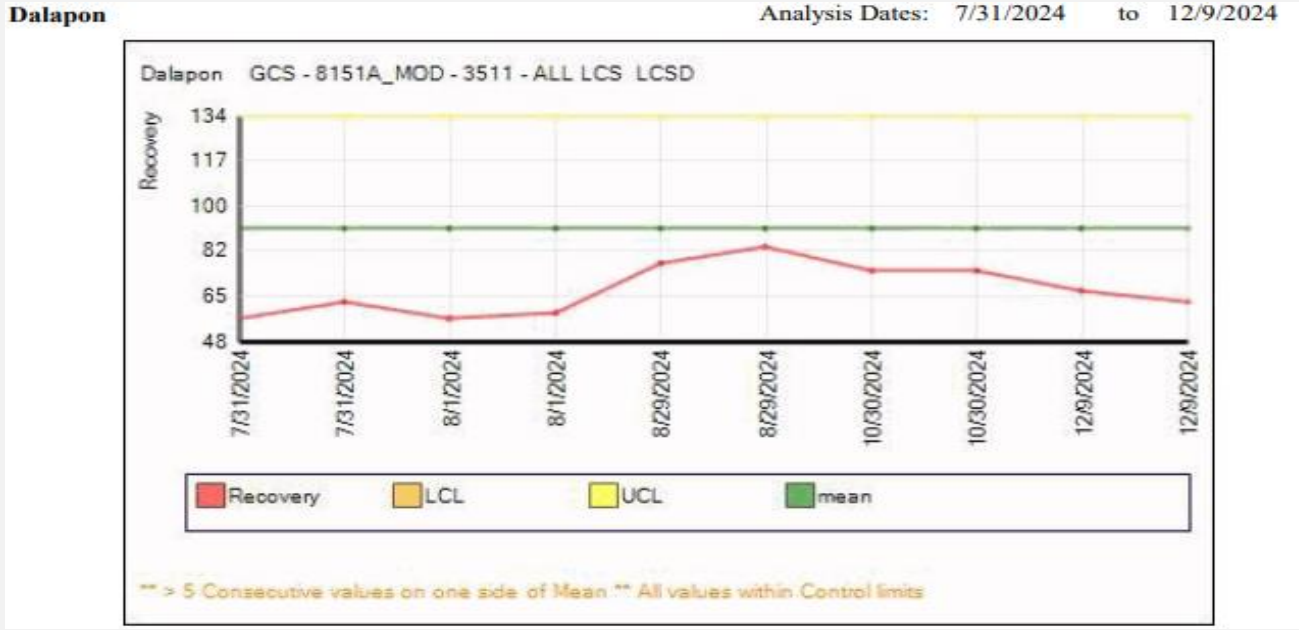
8151
LCS/LCSD
%R: 35.29%
%RSD: 35.19%
N=200



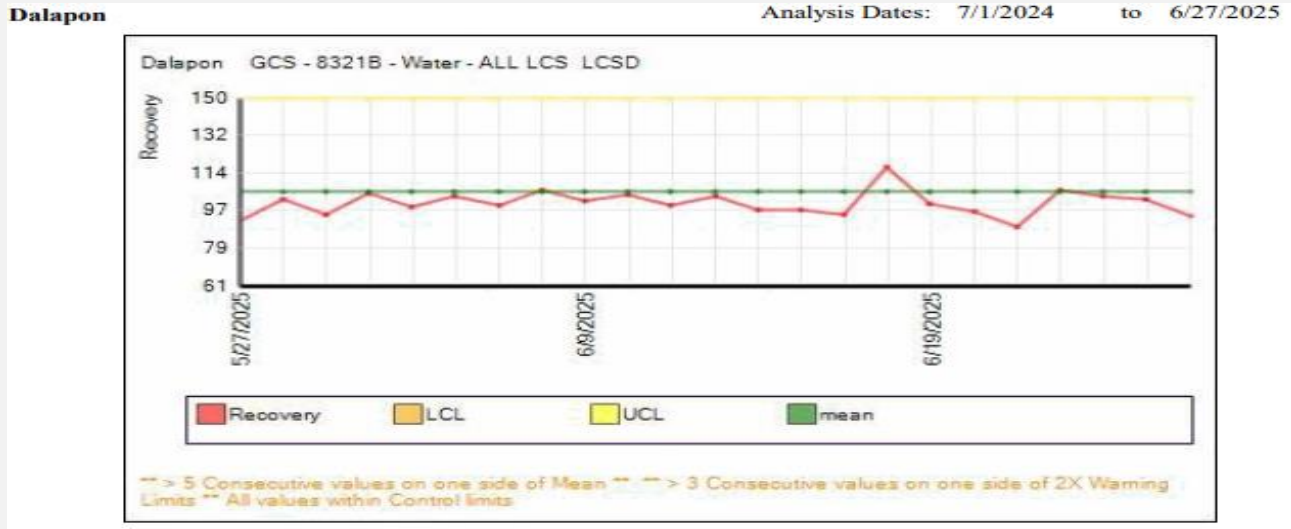
8321
LCS/LCSD
%R: 106%
%RSD: 15.25%
N=200

Problematic Compounds: DALAPON
8151 vs 8321 Water Matrix

- Peak Identification
- **%R of LCS/D, MS/D**



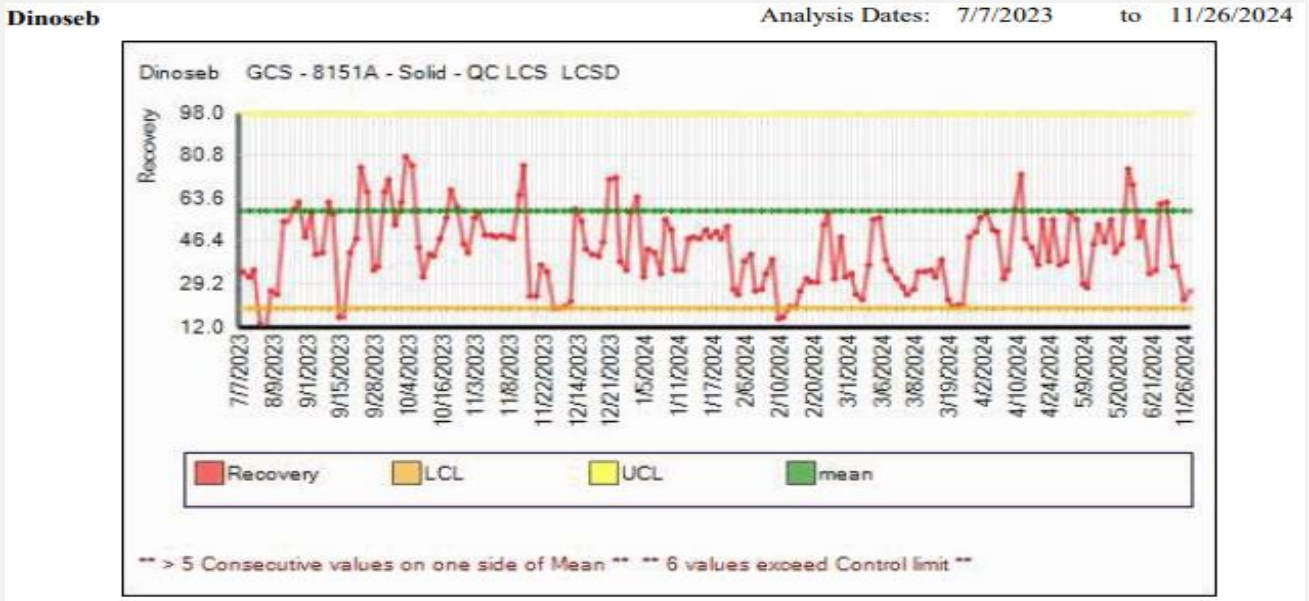
8151
LCS/LCSD
%R: 67%
RSD: 14%
N=200



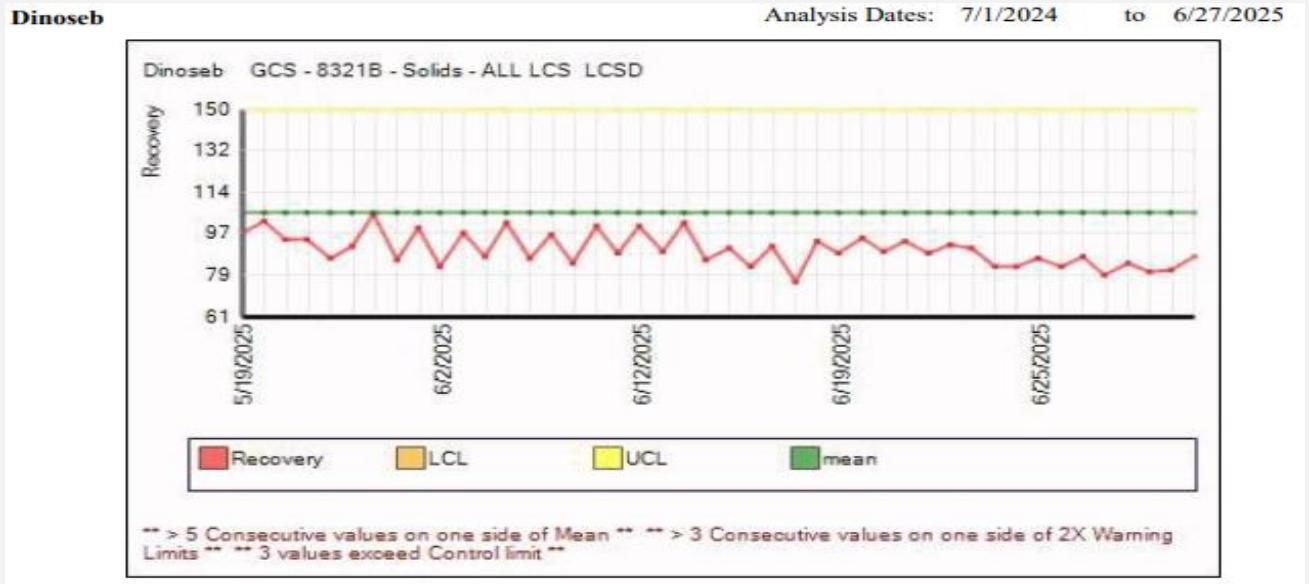
8321
LCS/LCSD
%R: 110%
RSD: 12.66%
N=200

Problematic Compounds: DINOSEB
8151 vs 8321 Solid Matrix

- Peak Identification
- **%R of LCS/D, MS/D**



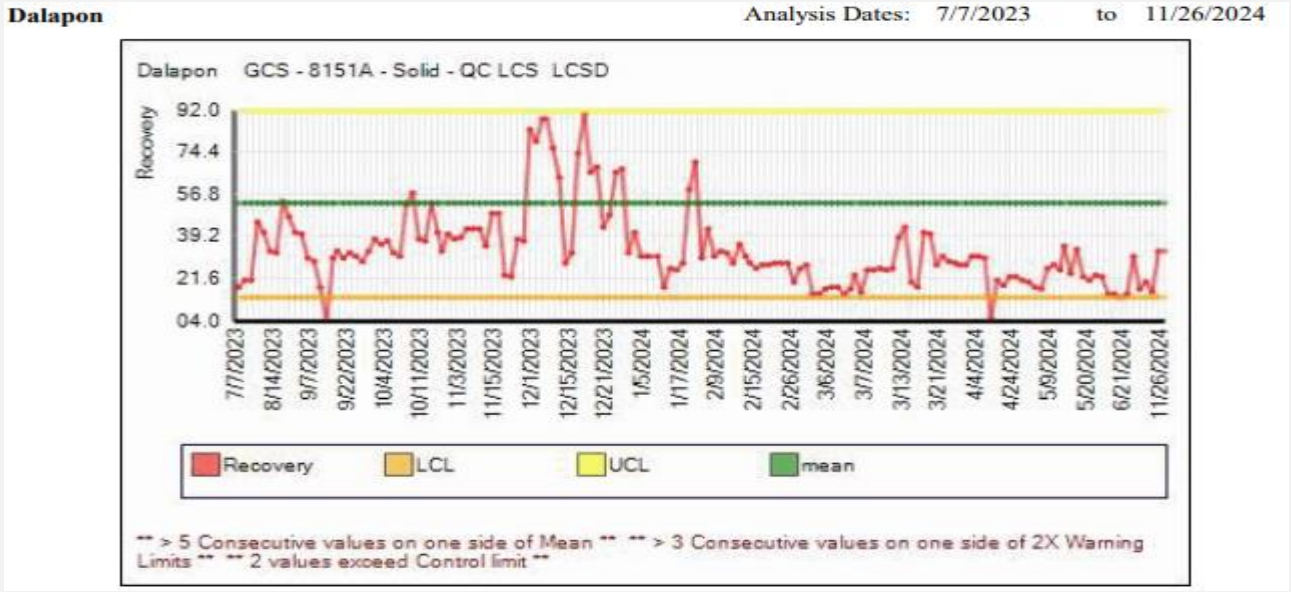
8151
LCS/LCSD
%R: 42.7%
%RSD: 35.4%
N=200



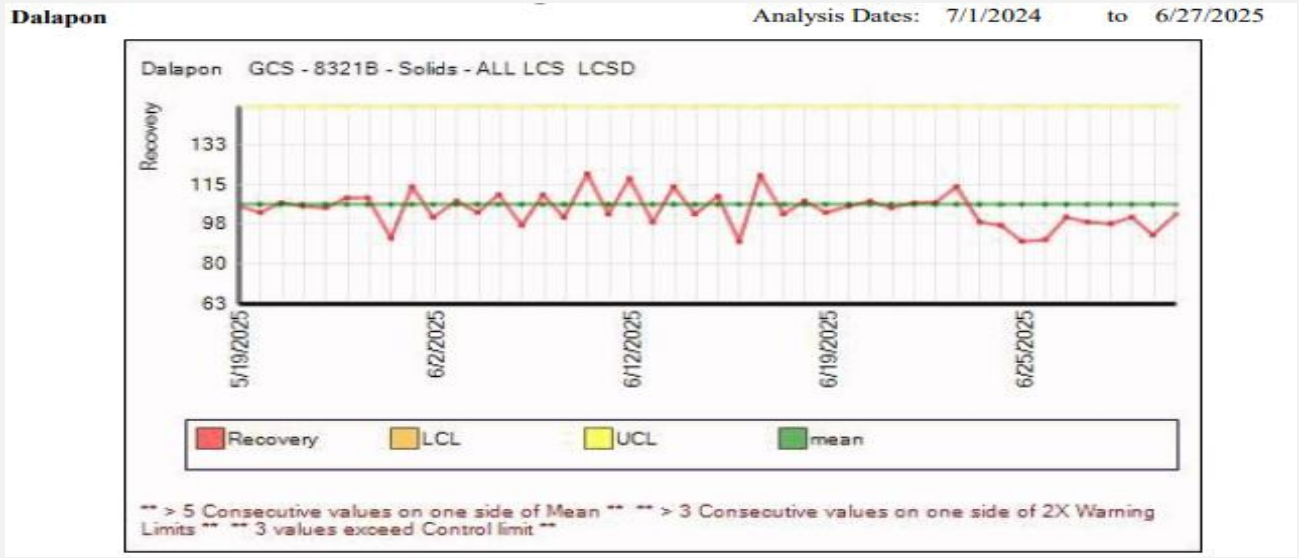
8321
LCS/LCSD
%R: 110%
%RSD: 17.7%
N=200

Problematic Compounds: DALAPON
8151 vs 8321 Solid Matrix

- Peak Identification
- **%R of LCS/D, MS/D**



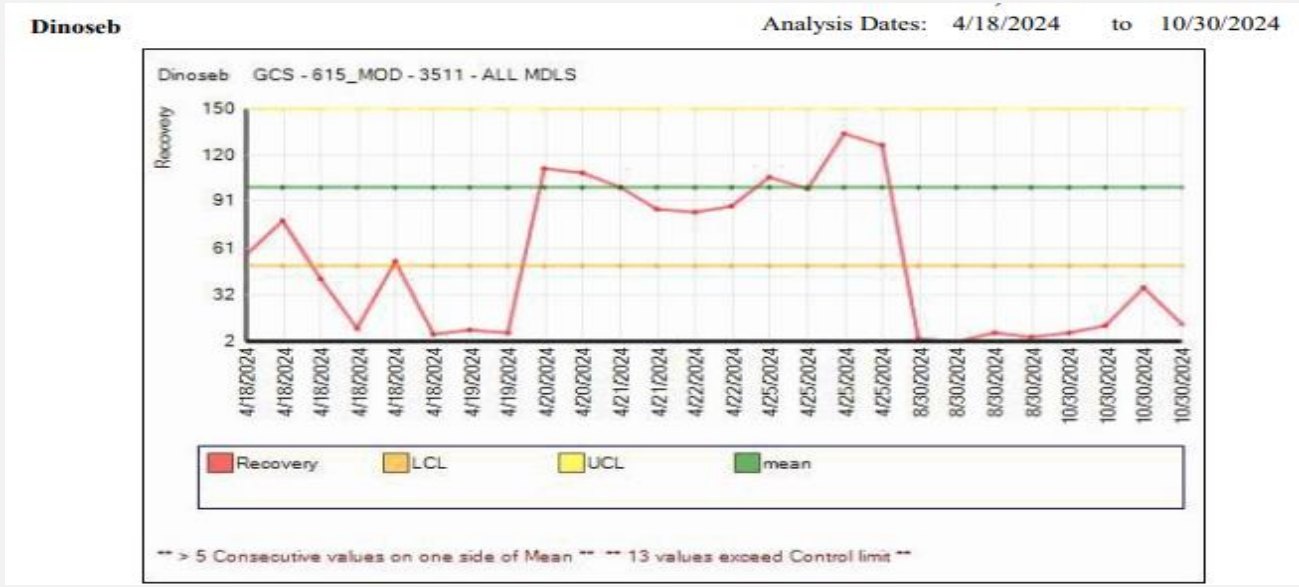
8151
LCS/LCSD
%R: 33.4%
%RSD: 48.8%
N=200



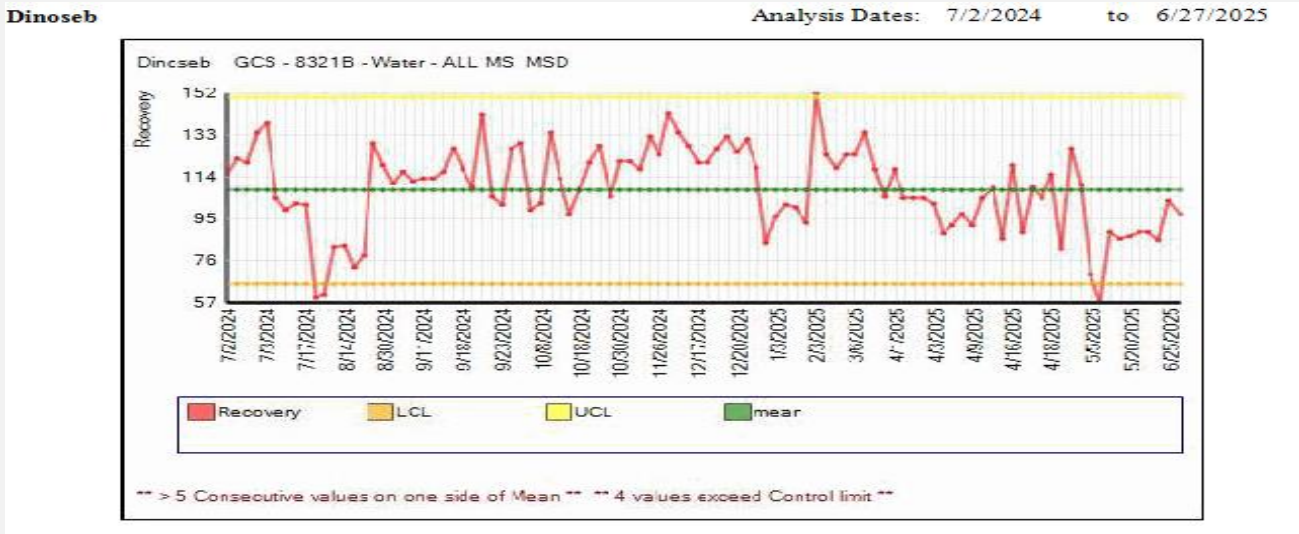
8321
LCS/LCSD
%R: 116%
%RSD: 14.11%
N=200

Problematic Compounds: DINOSEB
8151 vs 8321 Water Matrix

- Peak Identification
- **%R of LCS/D, MS/D**



8151
MS/MSD
%R: 53.3%
%RSD: 86.45%
N=200

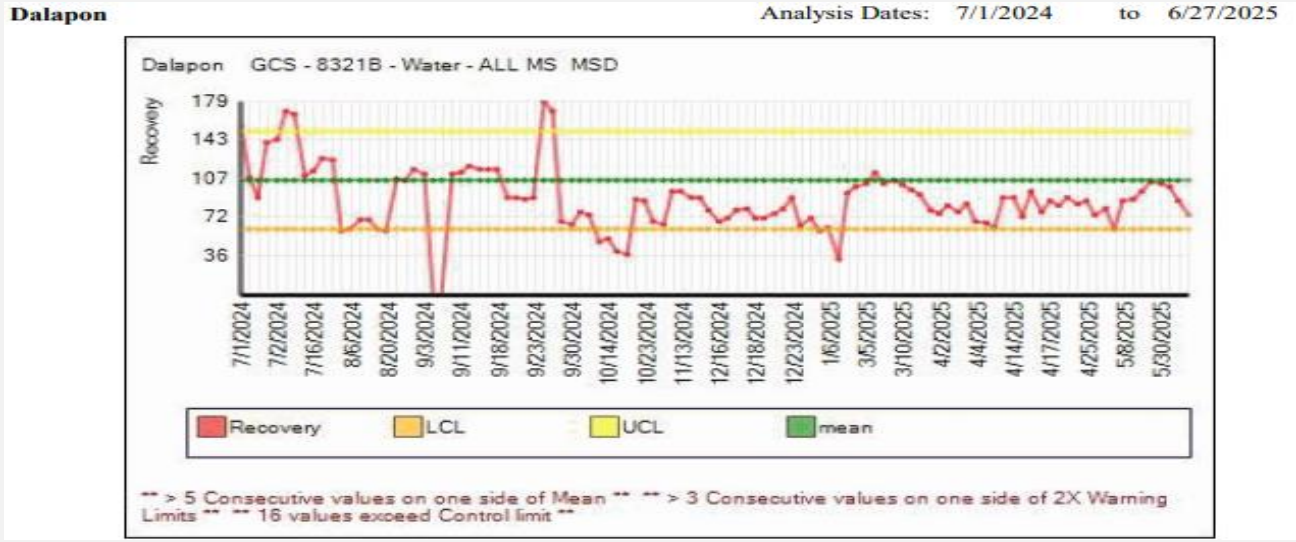


8321
MS/MSD
%R: 109.1%
%RSD: 17.51%
N=200

Problematic Compounds: DALAPON
8151 vs 8321 Water Matrix

- Peak Identification
- **%R of LCS/D, MS/D**

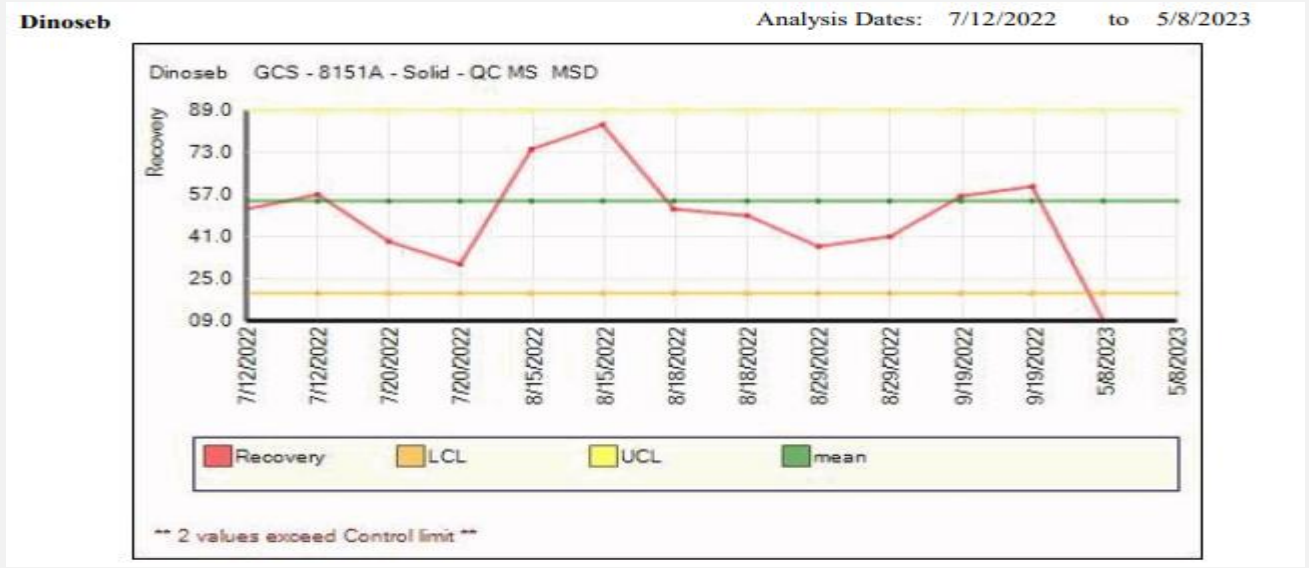
8151
MS/MSD
%R: 82.9%
%RSD: 30.13%
N=200



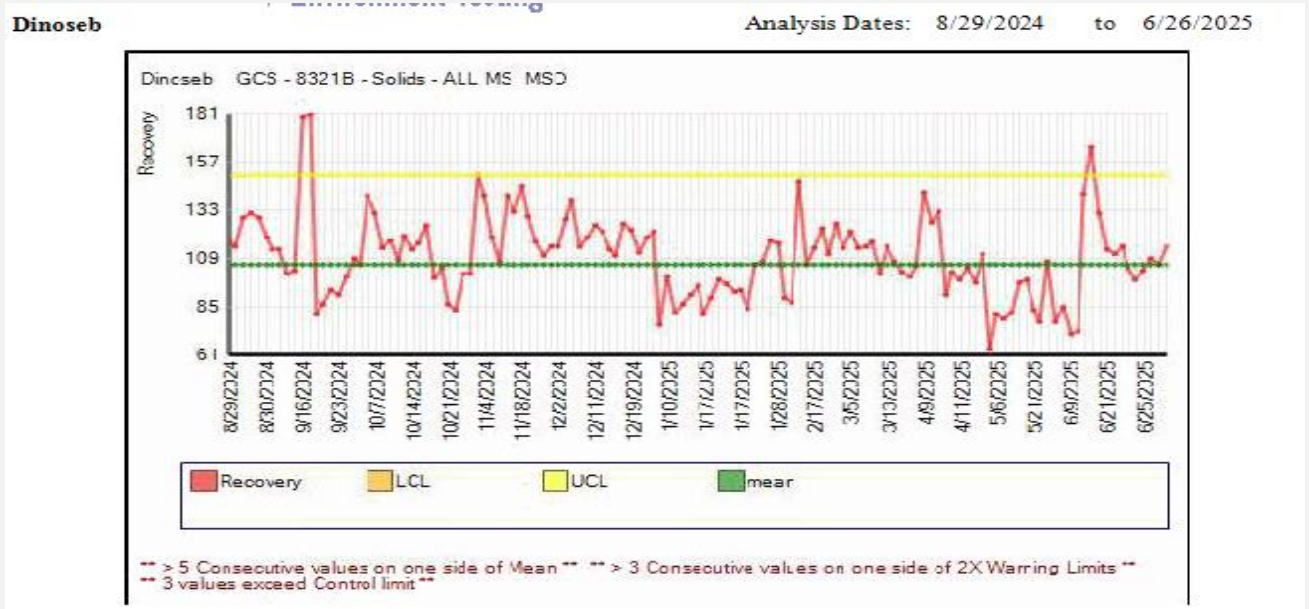
8321
MS/MSD
%R: 87.92%
%RSD: 27.85%
N=200

Problematic Compounds: DINOSEB
8151 vs 8321 Solid Matrix

- Peak Identification
- **%R of LCS/D, MS/D**



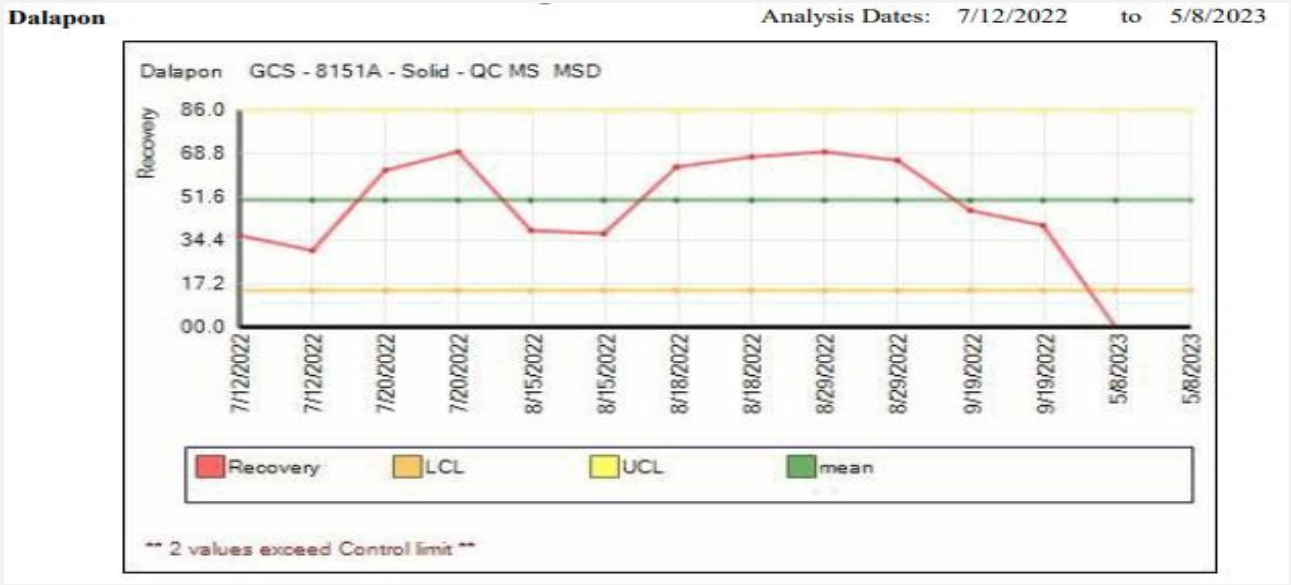
8151
MS/MSD
%R: 46.12%
%RSD: 45.68%
N=200



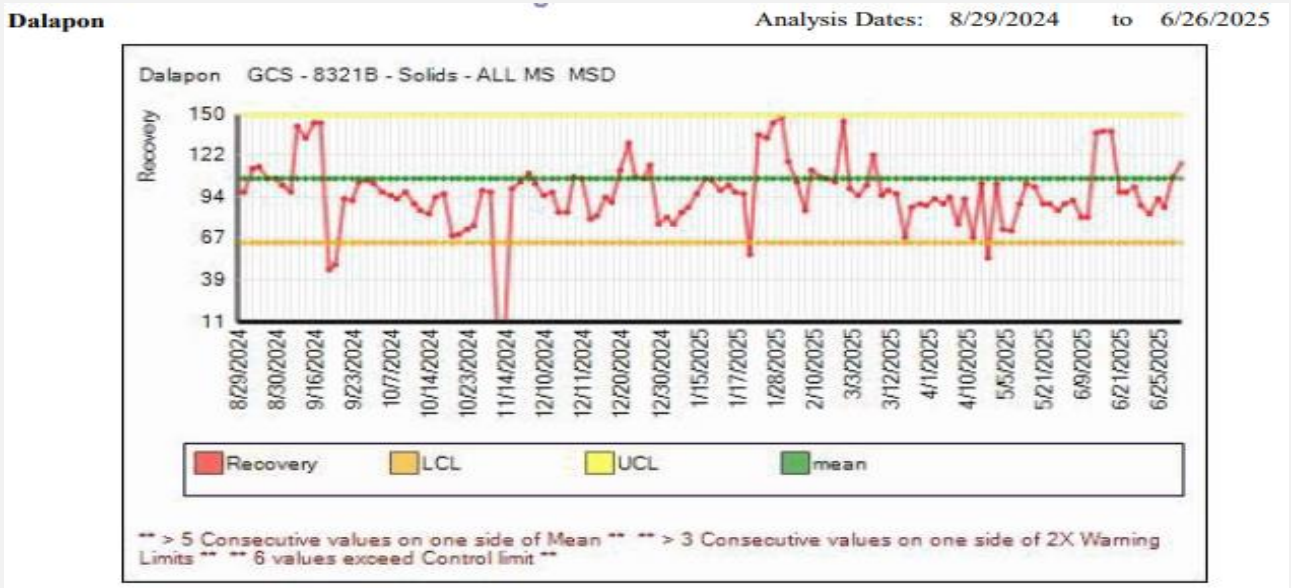
8321
MS/MSD
%R: 109%
%RSD: 18.65%
N=200

Problematic Compounds: DALAPON
8151 vs 8321 Solid Matrix

- Peak Identification
- **%R of LCS/D, MS/D**



8151
MS/MSD
%R: 46.53%
%RSD: 42.14%
N=200



8321
MS/MSD
%R: 95.6%
%RSD: 24.09%
N=200

What Changes?

- Superior specificity and sensitivity
- Fast sample preparation
- Elimination of excessive organic solvent use
- 1 day TAT possible

- Acceptable performance for accuracy and precision
- Extraction recoveries for most common herbicides are excellent and routinely fall within 30% of the true value
- High throughput

Julian Martinez
Eurofins Environment Testing

Julian.Martinez@ET.EurofinsUS.co

THANK YOU

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