Improved Antimony Recoveries in Soil Matrices by 3050B/6010B

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Agenda

- ✓ Past Method Performance for Antimony
- ✓ Current Method Performance for Antimony
- ✓ Method 3050B and procedure change
- ✓ Method 3050B effect on other elements
- ✓ Conclusions

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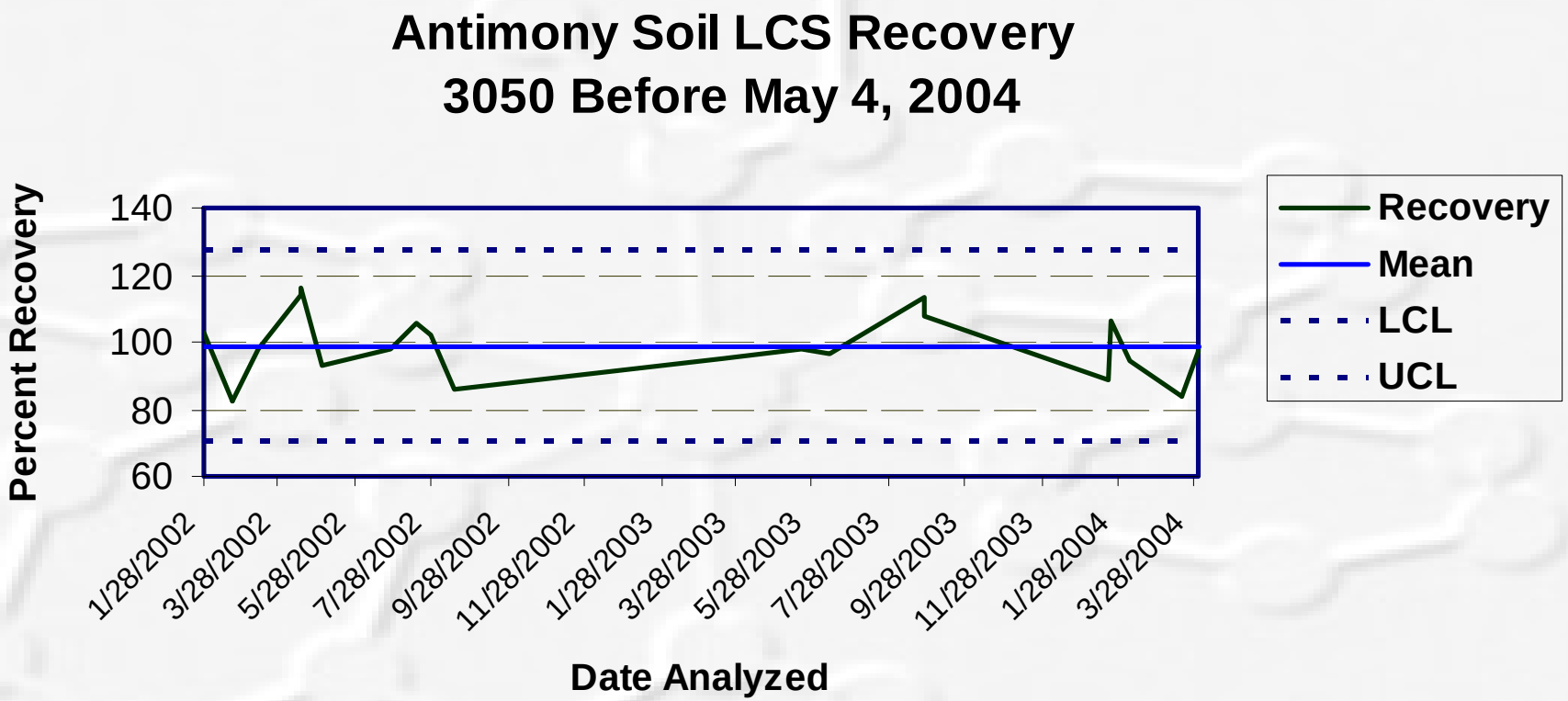
Past LCS Performance



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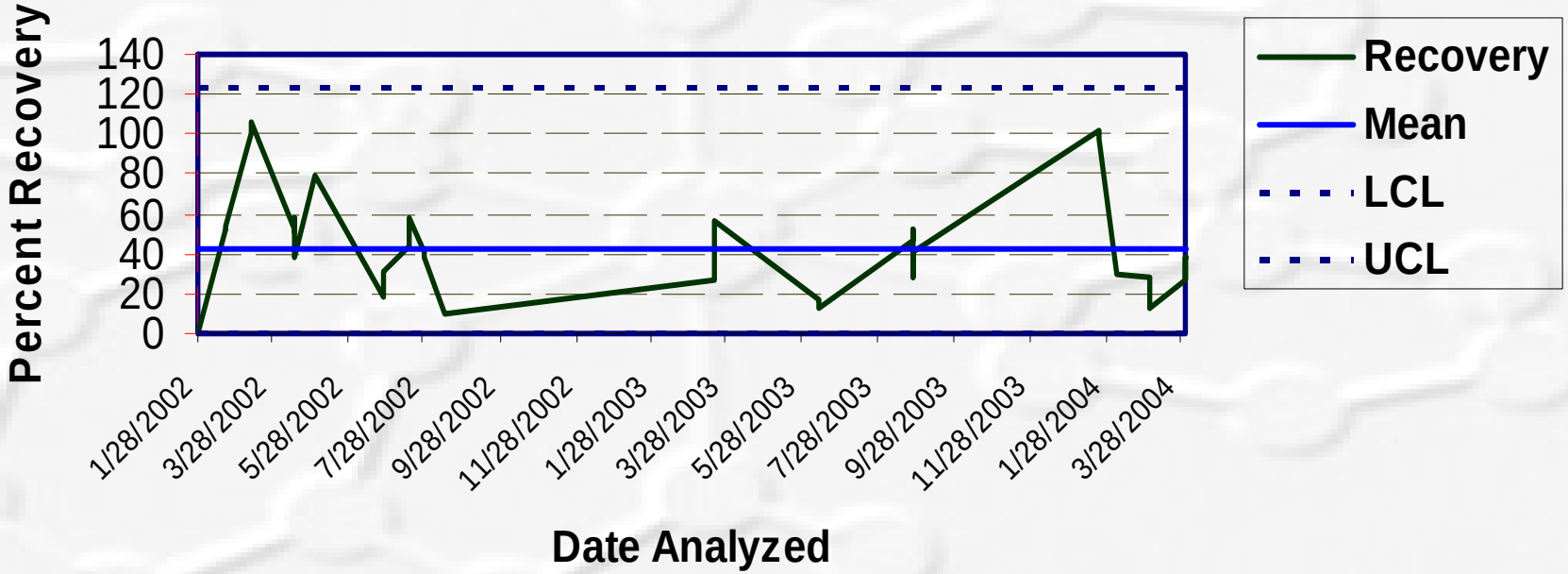
Past MS/MSD Performance

Antimony Soil Matrix Spike Recovery
3050 Before May 4, 2004



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LCS Soil

INORGANIC REFERENCE MATERIAL
SOLID LABORATORY CONTROL SAMPLE
LCS (0996)

The solid laboratory control sample, LCS (0996) was prepared by the UNLV Quality Assurance Laboratory, and is being distributed by IT Corporation, under contract to the EPA. The "True Value" concentrations were derived from the results of an EPA multi-laboratory analysis of the solid material by Contract Laboratory Program procedures. The "True Value" concentrations are listed in Table 1.

TABLE 1. "TRUE VALUE" CONCENTRATIONS AND CONTROL LIMITS FOR THE ELEMENTS IN THE SOLID LABORATORY CONTROL SAMPLE, LCS (0996)

Element	"True Value" (mg/kg)	Control Limits (mg/kg)		
Al	309	193.1	to	424.2
Sb	213	129.4	to	297.2
As	930	613.6	to	1247
Ba *	[5.3]	2.5	to	8.1
Be	18.8	15.3	to	22.2
Cd	41.6	32.1	to	51.1
Ca	184000.5	142933.0	to	225376.0
Cr	96.5	77.8	to	115.2
Co	140	115.4	to	165.6
Cu	6680	5727.3	to	7633.1
Fe	21000	16831.3	to	25193.0
Pb	224	167.6	to	280.5
Mg	113000	97493.0	to	128886.0
Mn	201	167.9	to	234.4
Hg	12.3	7.8	to	16.9
Ni	56.8	43.5	to	70.1
K *	[102.4]	0	to	379.3
Ag	20.9	13.2	to	28.5
Se	37.0	17.6	to	56.4
Na *	[92.8]	0	to	277.4
Tl	38.1	24.6	to	51.6
V	65.8	53.0	to	78.6
Zn	175	127.7	to	222.1

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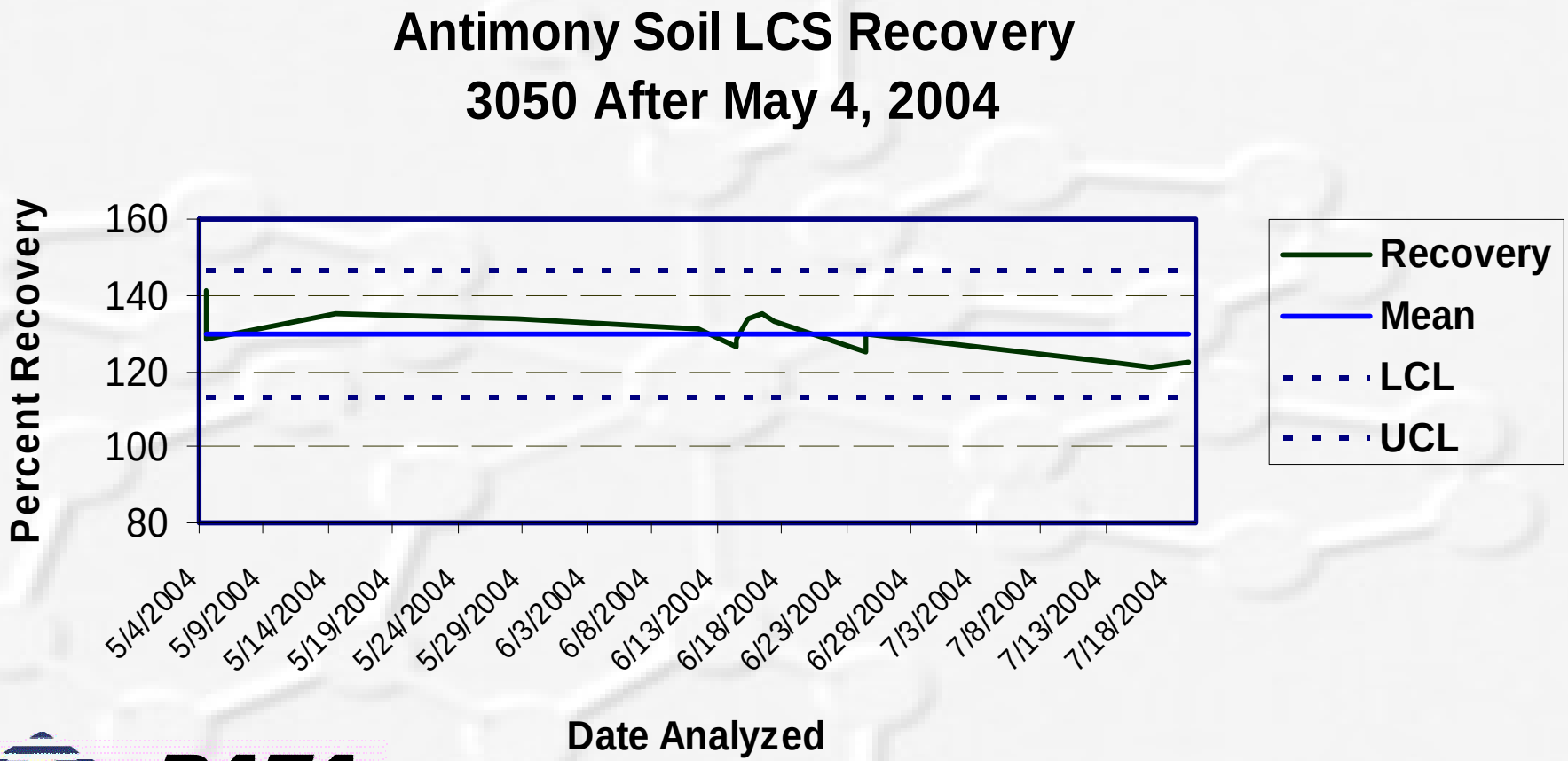
Current LCS Performance



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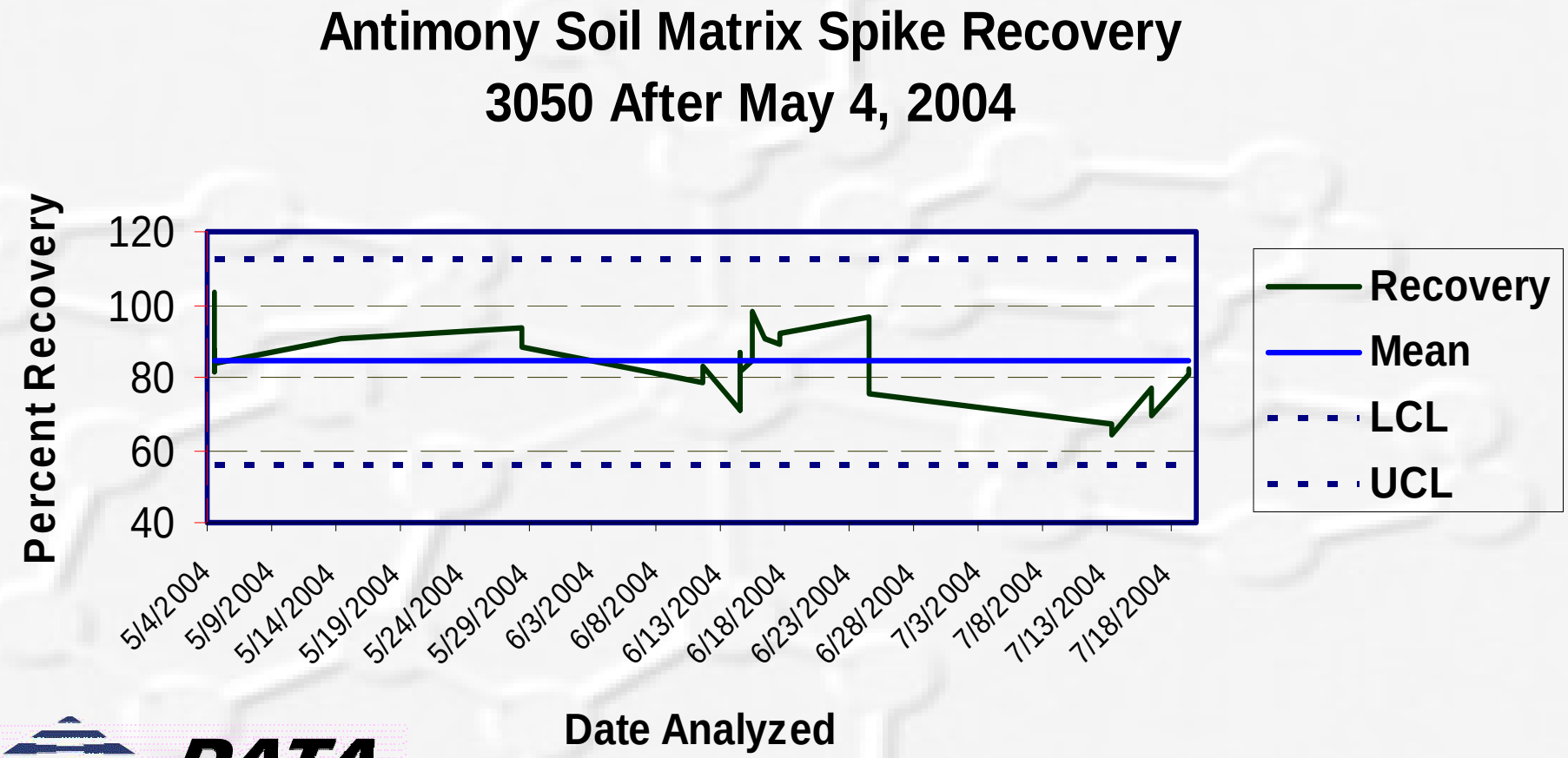
Current MS/MSD Performance



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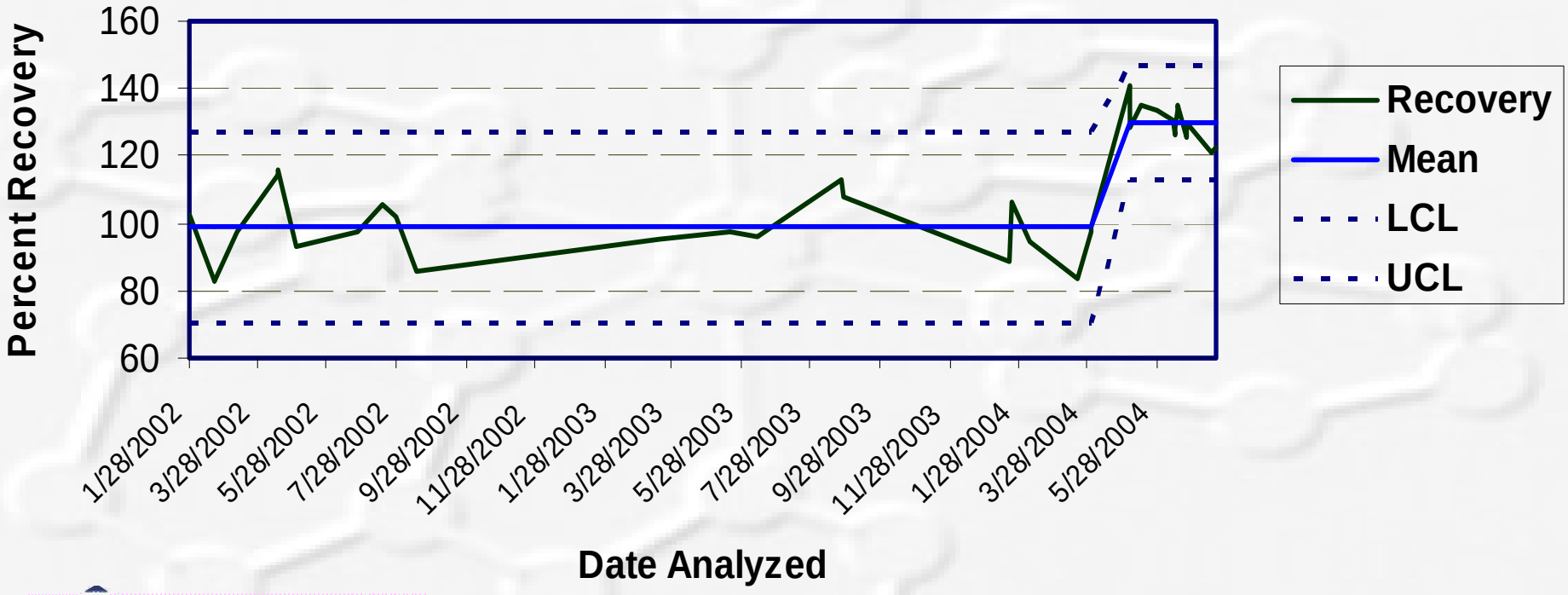
Overall LCS Performance

Antimony Soil LCS Recovery



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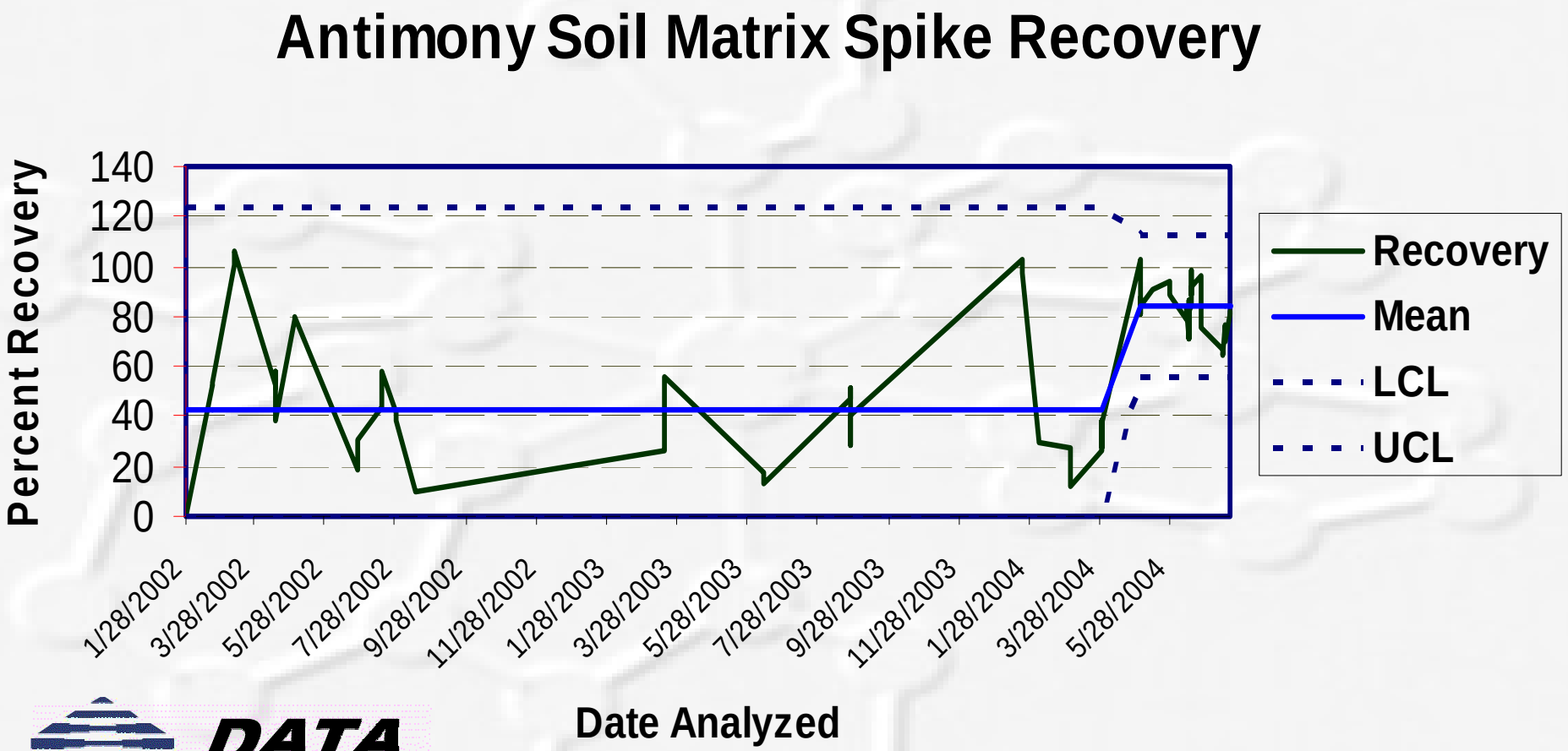
Overall MS/MSD Performance



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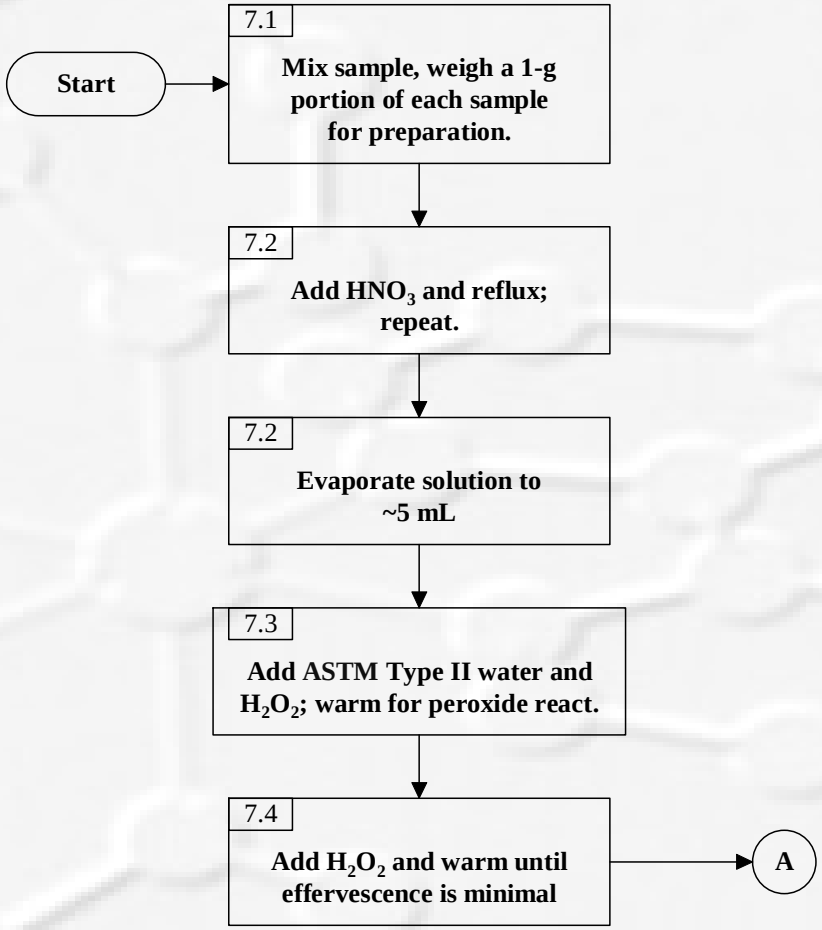
Method 3050B Procedure



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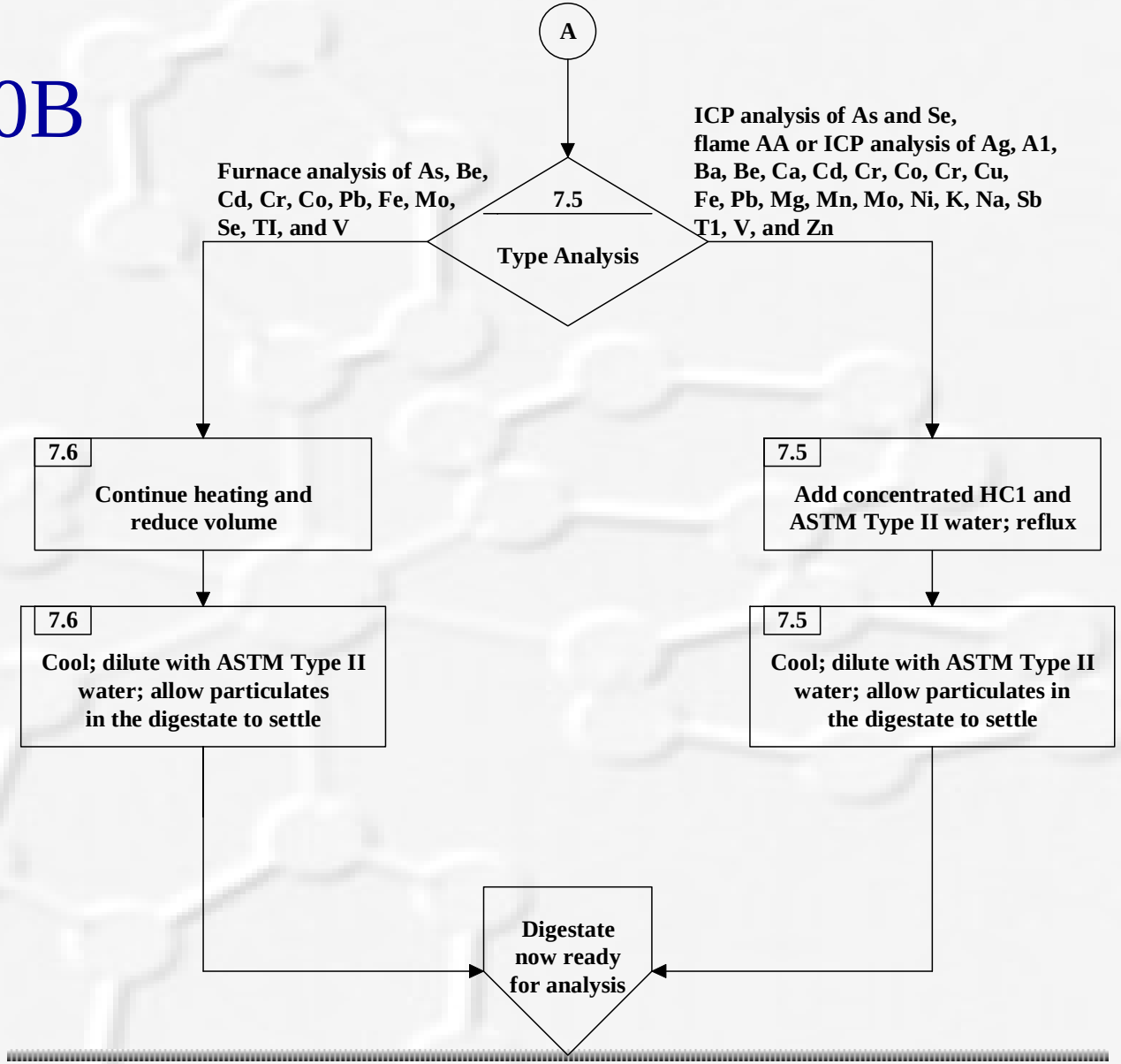
Method 3050B Procedure



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Method 3050B Options

NOTE: Section 7.5 may be used to improve the solubilities and recoveries of antimony, barium, lead, and silver when necessary. These steps are optional and are not required on a routine basis.

7.5 Add 2.5 mL conc. HNO and 10 mL conc. HCl to a 1-2 g sample (wet weight) or 1 g sample (dry weight) and cover with a watchglass or vapor recovery device. Place the sample on/in the heating source and reflux for 15 minutes.

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Method 3050B Options

7.5.1 Filter the digestate and collect filtrate in a 100-mL volumetric flask. Wash the filter paper, while still in the funnel, with no more than 5 mL of hot (~95°C) HCl, then with 20 mL of hot (~95°C) reagent water.
Collect washings in the same 100-mL volumetric flask.

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Method 3050B Options

7.5.2 Remove the filter and residue from the funnel, and place them back in the vessel. Add 5 mL of conc. HCl, place the vessel back on the heating source, and heat at 95°C ± 5°C until the filter paper dissolves. Remove the vessel from the heating source and wash the cover and sides with reagent water. Filter the residue and collect the filtrate in the same 100-mL volumetric flask. Allow filtrate to cool, then dilute to volume.

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Method 3050B Options

7.5.3 If a precipitate forms on the bottom of a flask, add up to 10 mL of concentrated HCl to dissolve the precipitate. After precipitate is dissolved, dilute to volume with reagent water. Analyze by FLAA or ICP-AES.

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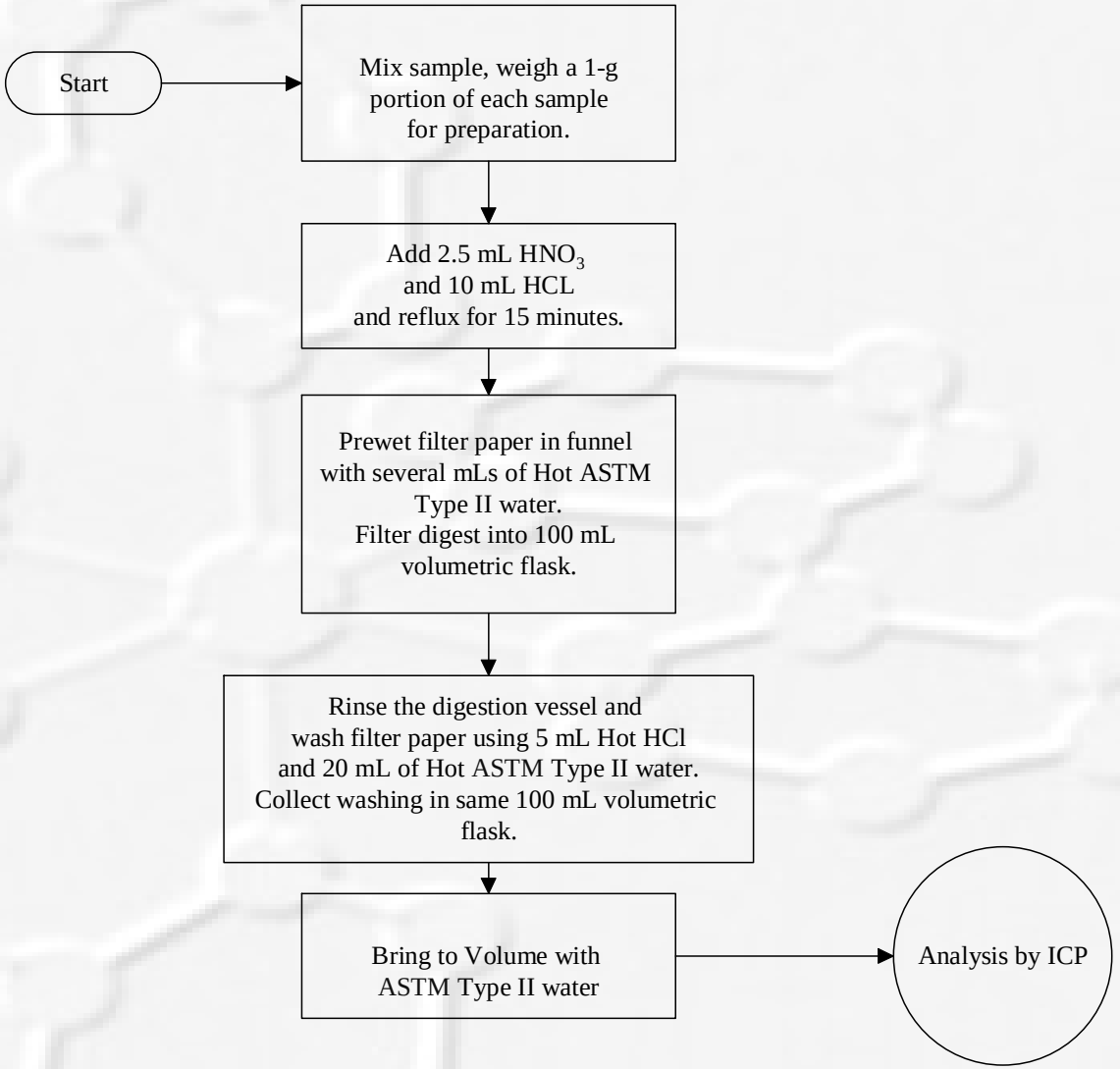
New Procedure



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New Procedure

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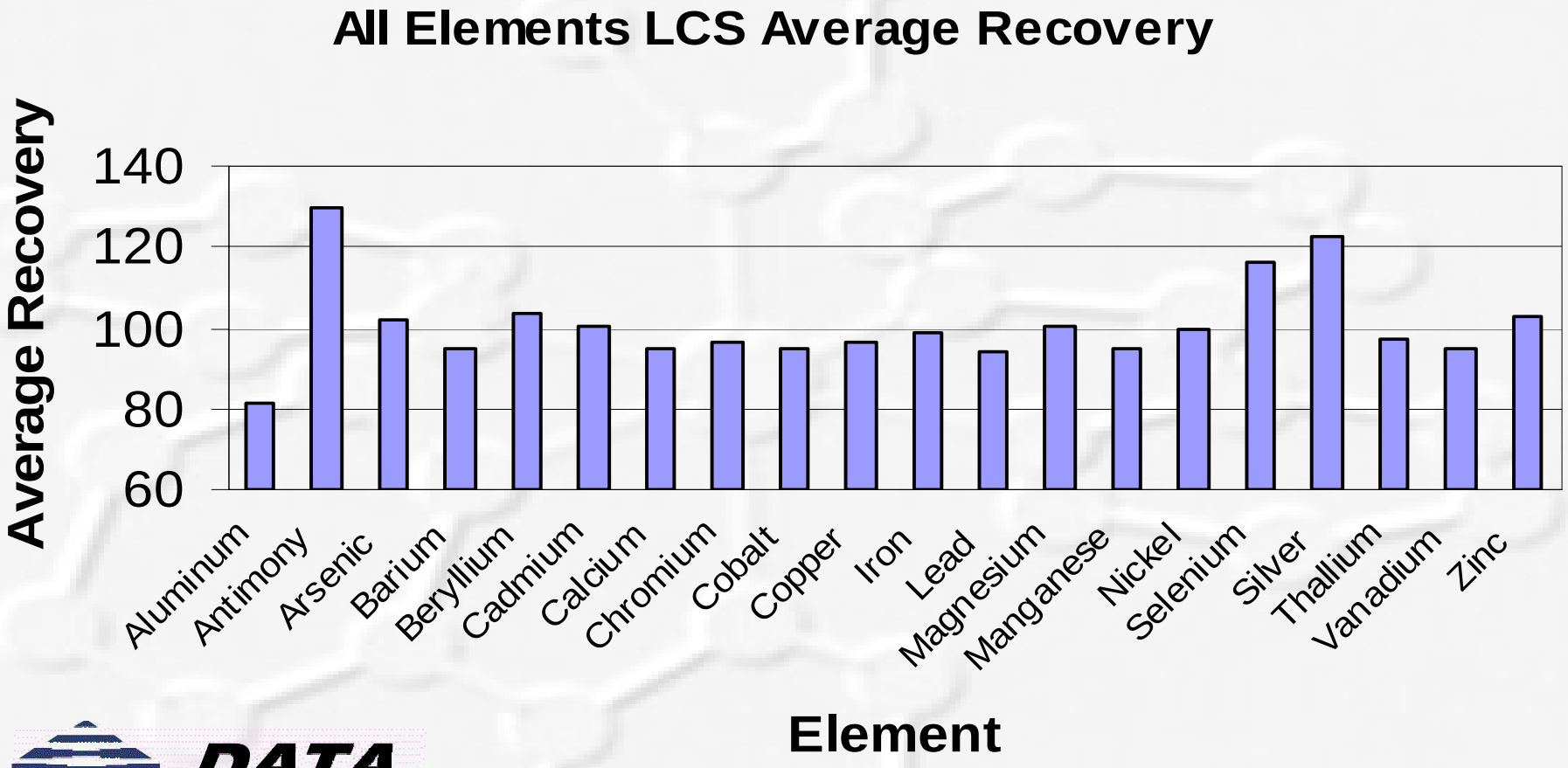
Change in Performance – All Elements



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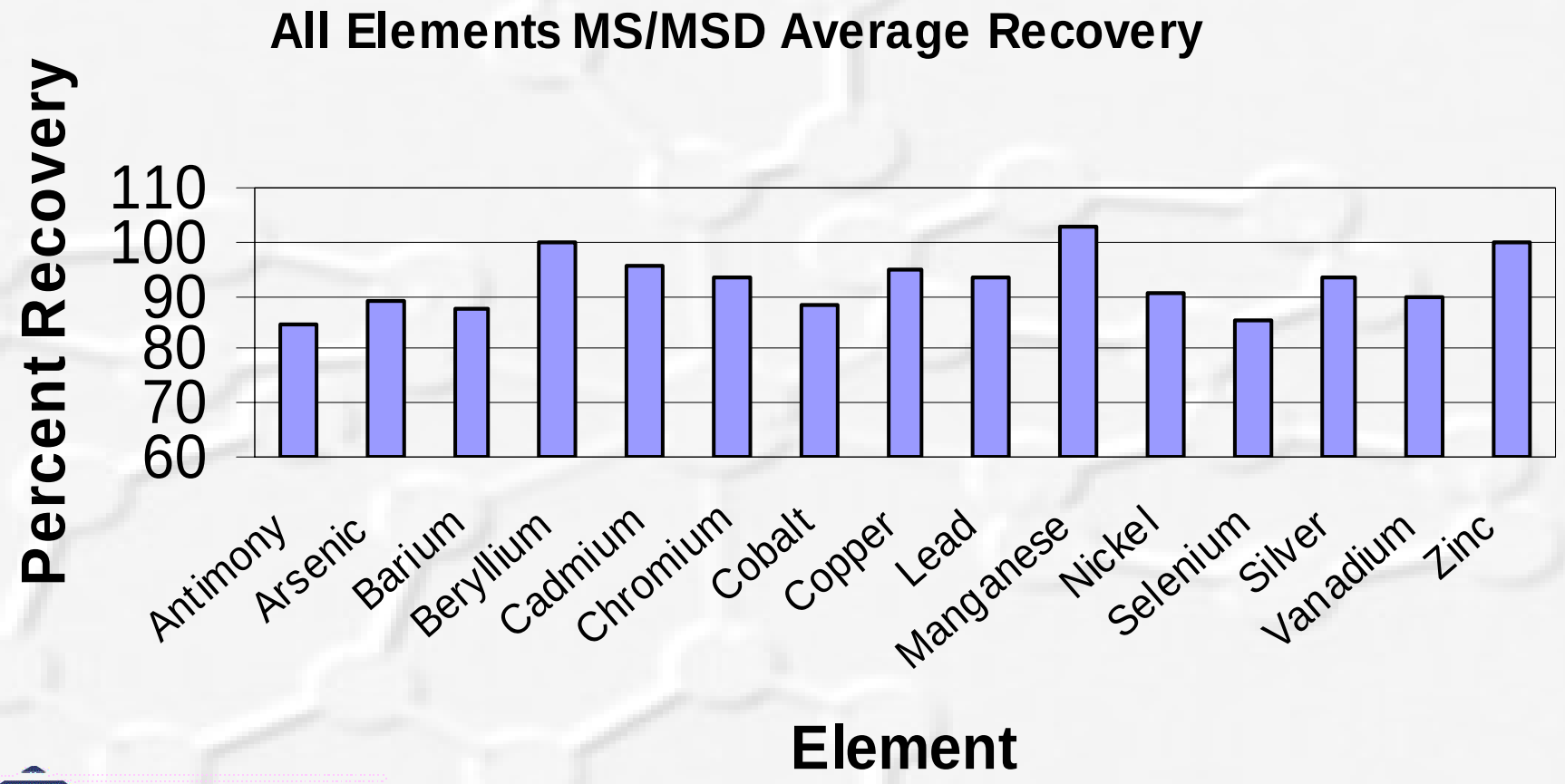
Change in Performance – All Elements



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Conclusions

Everything should be made as simple as possible, but not simpler.

--- *Albert Einstein*

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Conclusions

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Conclusions

A new scientific truth does not triumph
by convincing its opponents and making them see the light,
but rather because its opponents eventually die,
and a new generation grows up that is familiar with it.
--- Max Planck

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Thank You

Questions???

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