Application Work with 6460 QQQ

USDA Sample Analyses

Agilent G6460A QQQ

LC/MS Demo Data

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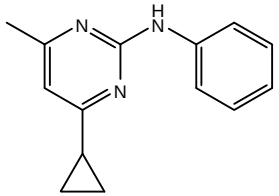
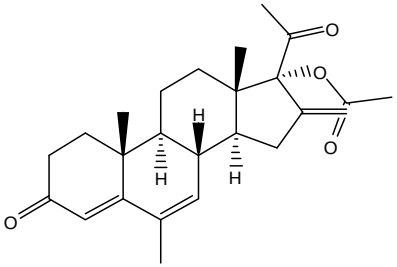
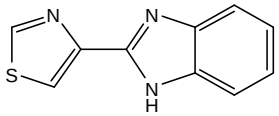
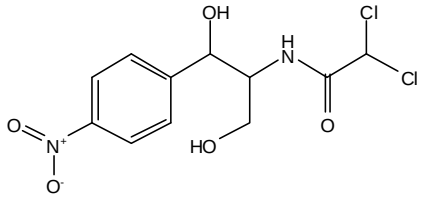
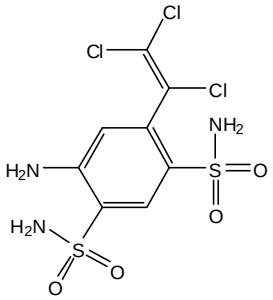
Analyte Information (Positive Polarity)

Analyte Formula CAS No. MW (monoisotopic)	**Structure**	**ESI**	**MS/MS**
Cyprodinil $C_{14}H_{15}N_3$ 121552-61-2 225.127		positive	226 → 108 226 → 118
Melengestrol acetate $C_{25}H_{32}O_4$ 2919-66-6 396.230		positive	397 → 337 397 → 279
Thiabendazole $C_{10}H_7N_3S$ 148-79-8 201.036		positive	202 → 175 202 → 131

Analyte Information (Negative Polarity)

Analyte Formula CAS No. MW (monoisotopic)	**Structure**	**ESI**	**MS/MS**
Chloramphenicol $C_{11}H_{12}Cl_2N_2O_5$ 56-75-7 322.012		negative	321 → 152 321 → 257
Clorsulon $C_8H_8Cl_3N_3O_4S_2$ 60200-06-8 378.902		negative	378 → 342 378 → 242

Requirements for Sample Analyses:

- Demonstration of +/- ESI switching – 10 transitions
  - 2 transitions per analyte
  - 5 ms dwell time
  - 5 replicates per sample (kidney juice vs water spike)

- Results for 5 tested analytes: Peak areas, RSD%, S/N, points per peak req'd

Method Conditions (LC):

Column temperature:	55 ºC
Flow rate:	0.5 mL/min
Mobile phase A:	0.1% formic acid in water
Mobile phase B:	0.1% formic acid in acetonitrile
Column:	Agilent Zorbax Eclipse Plus C18, 2.1 x 50 mm, 1.8 µm
Guard column:	None
Injection volume:	5 µL

Gradient timetable:	**Time**	**%B**
1	0.00	5.0
2	2.00	90.0
3	3.75	90.0
4	3.80	5.0

Analysis time: 5 min, post time (eql.): 0 min, overall run time: 5 min

Method Conditions (6460 QQQ):

Source Conditions:

- Nebuliser Pressure – 45 psi
- Nozzle Voltage – 250 V (+ve)/1500 V (-ve)
- Sheath Gas Flow – 11 L/min
- Sheath Gas Temperature – 380 degrees
- Drying Gas Flow – 4 L/min
- Drying Gas Temperature – 200 degrees
- Capillary Voltage – 2500 V (positive & negative polarities)

Method Conditions (6460 QQQ):

Compound Name	ISTD?	Prec Ion	MS1 Res	Prod Ion	MS2 Res	Dwell	Frag (V)	CE (V)
Melengestrol acetate	☐	397	Wide	337	Wide	5	130	10
Melengestrol acetate	☐	397	Wide	279	Wide	5	130	10
Cyprodinil	☐	226	Wide	118	Wide	5	170	35
Cyprodinil	☐	226	Wide	108	Wide	5	170	30
Thiabendazole	☐	202	Wide	175	Wide	5	170	30
Thiabendazole	☐	202	Wide	131	Wide	5	170	35
Clorsulon	☐	377.8	Wide	342	Wide	5	110	6
Clorsulon	☐	377.8	Wide	242	Wide	5	110	18
Chloramphenicol	☐	321	Wide	257	Wide	5	130	3
Chloramphenicol	☐	321	Wide	152	Wide	5	130	14

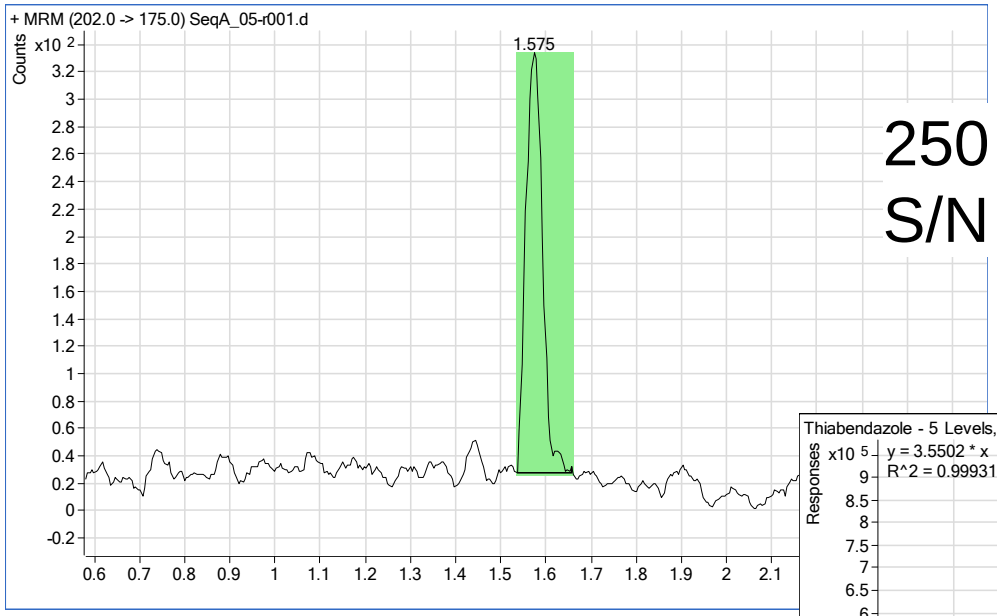
Calibration and LOD Data:

The Calibration Curves in this section were created by spiking the mixed analytes into Tap Water (potable) to give a matrix effect.

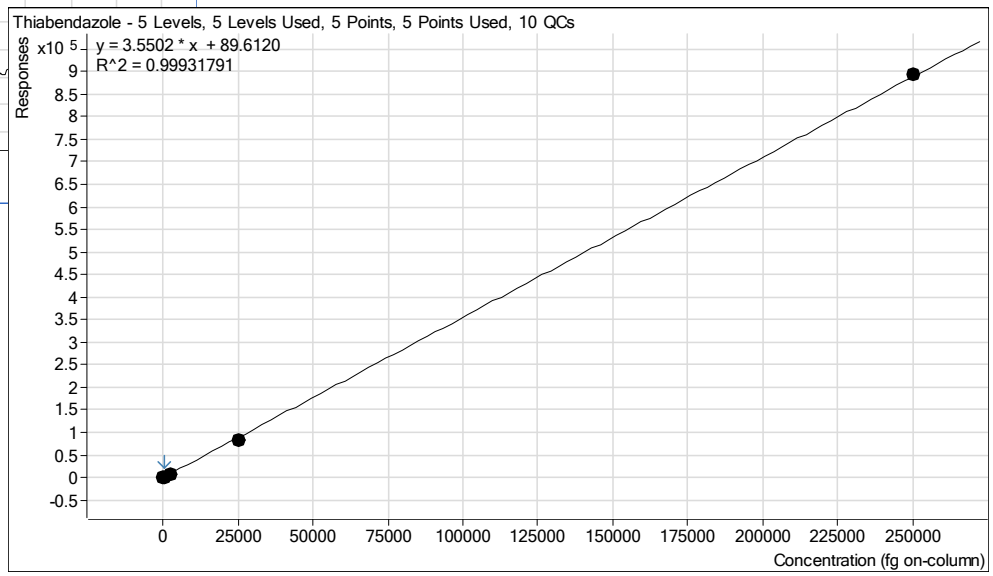
Matrix Samples:

Other slides labeled Matrix Samples describe sample analyses undertaken on matrix samples supplied by the USDA. These were analysed as supplied.

Limit of detection & Calibration Data
Thiabendazole (anti-fungicide)



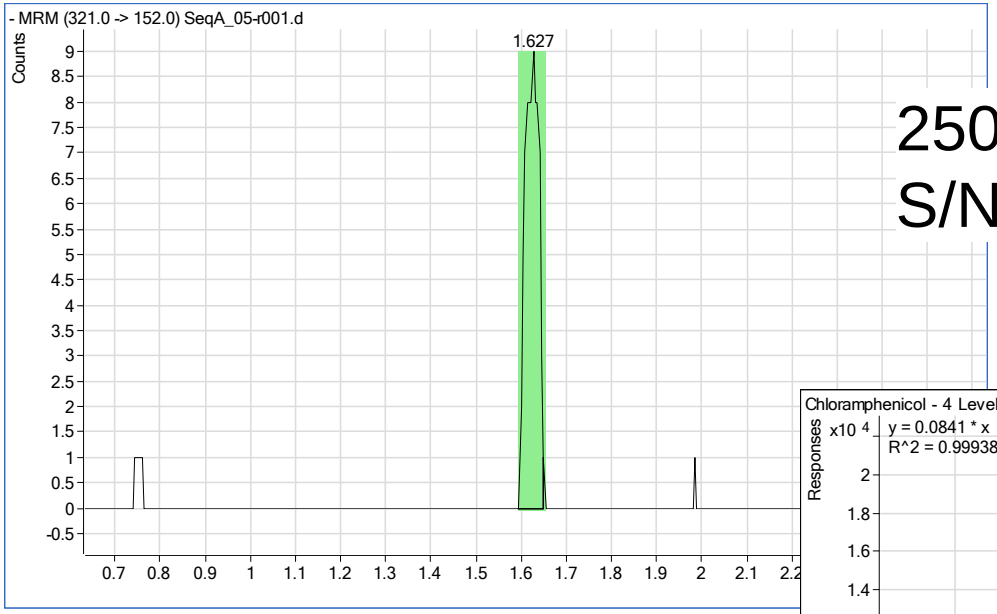
250 fg on-column
S/N = 14



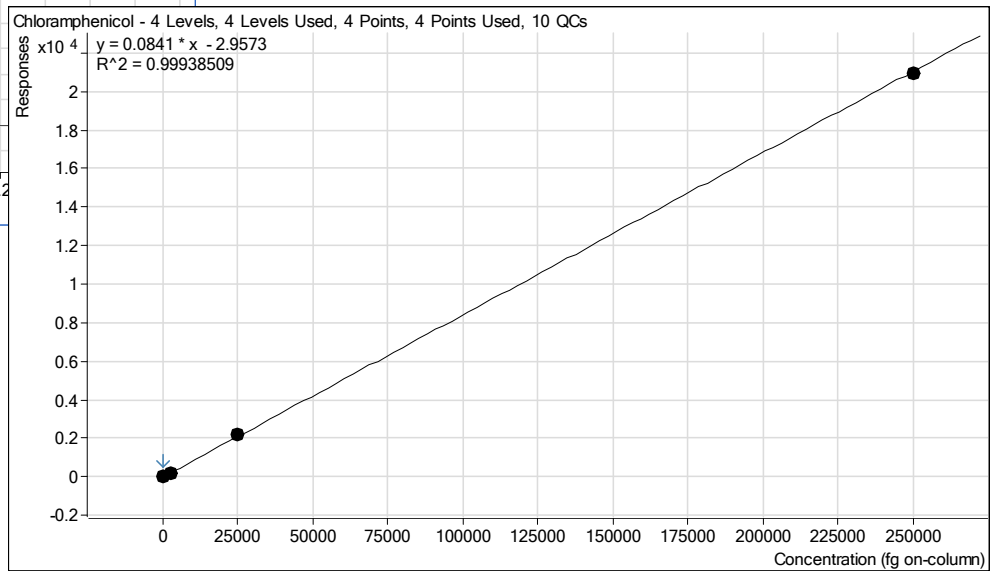
Correlation = 0.9993
Linearity up to 250 pg on-column

5 ms dwell + polarity switching

Limit of detection & Calibration Data Chloramphenicol (antibiotic)



250 fg on-column
S/N =10

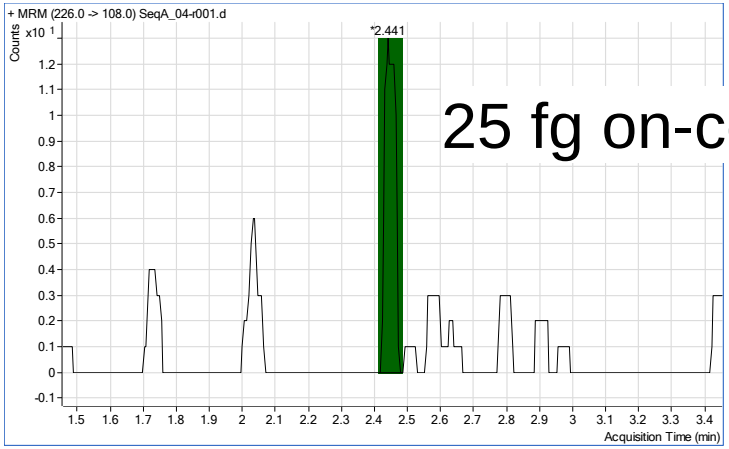


Correlation = 0.9994
Linearity up to 250 pg on-column

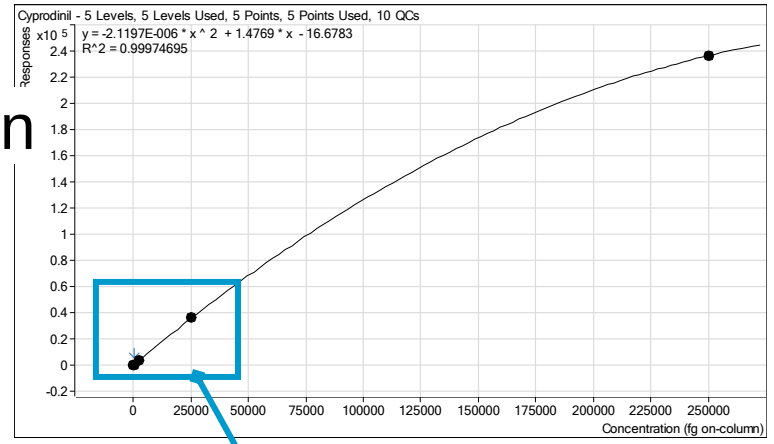
5ms dwell + polarity switching

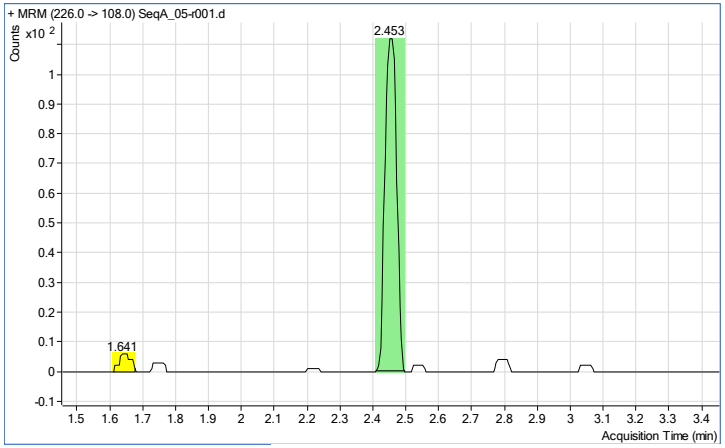
Limit of detection & Calibration Data
Cyprodinil (anti-fungicide)

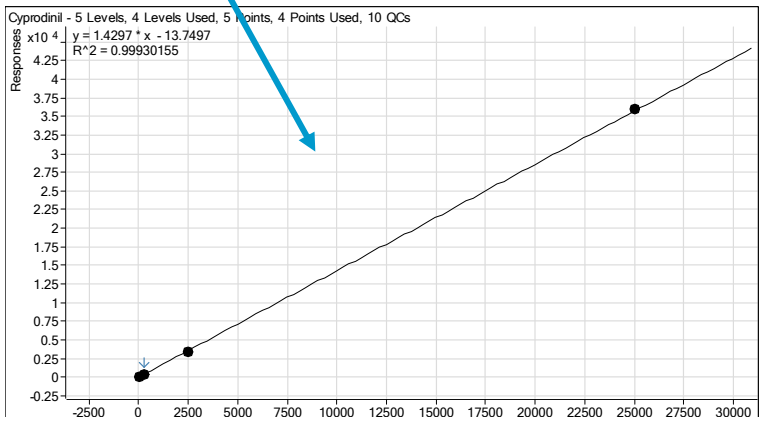
5ms dwell + polarity switching



25 fg on-column



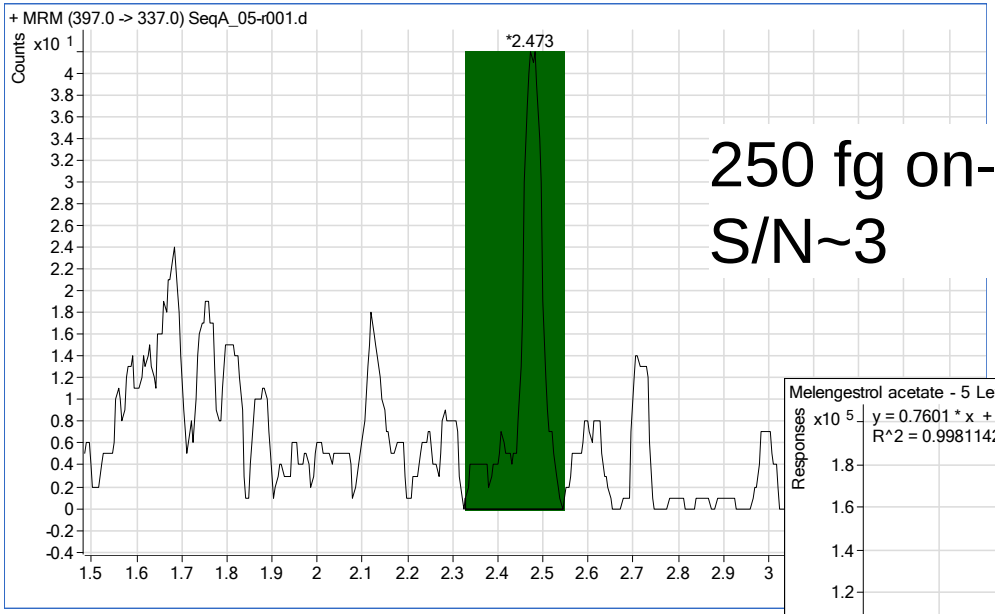




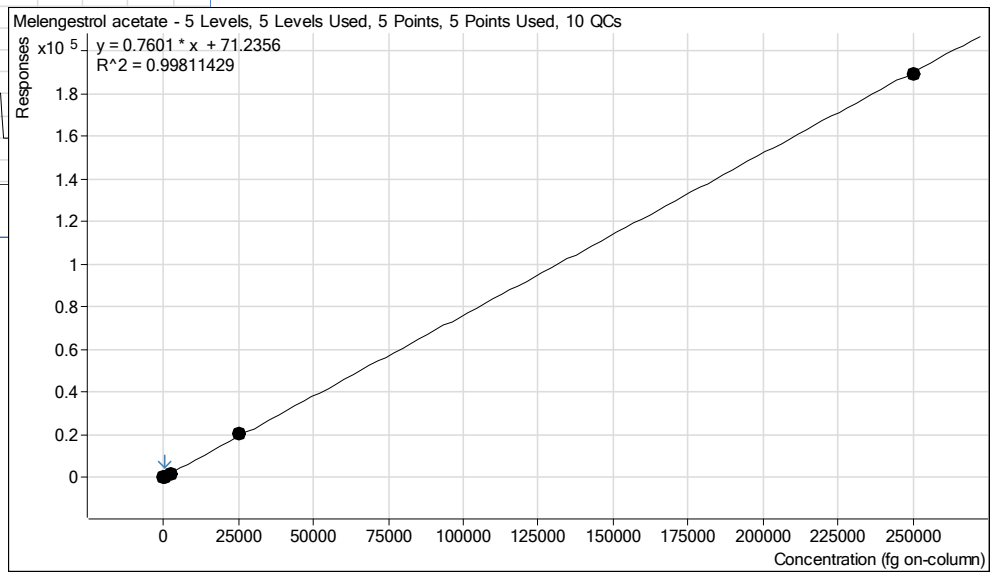
250 fg on-column
S/N=23

Correlation = 0.9993
Linearity up to 25 pg on-column

Limit of detection & Calibration Data
Melengestrol acetate (Veterinary Medicine/Steroid)



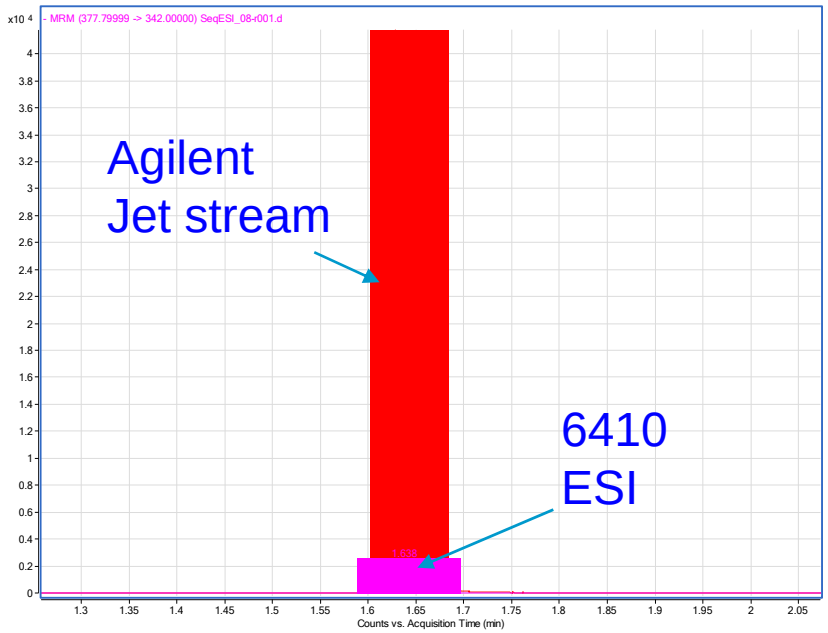
250 fg on-column
S/N~3



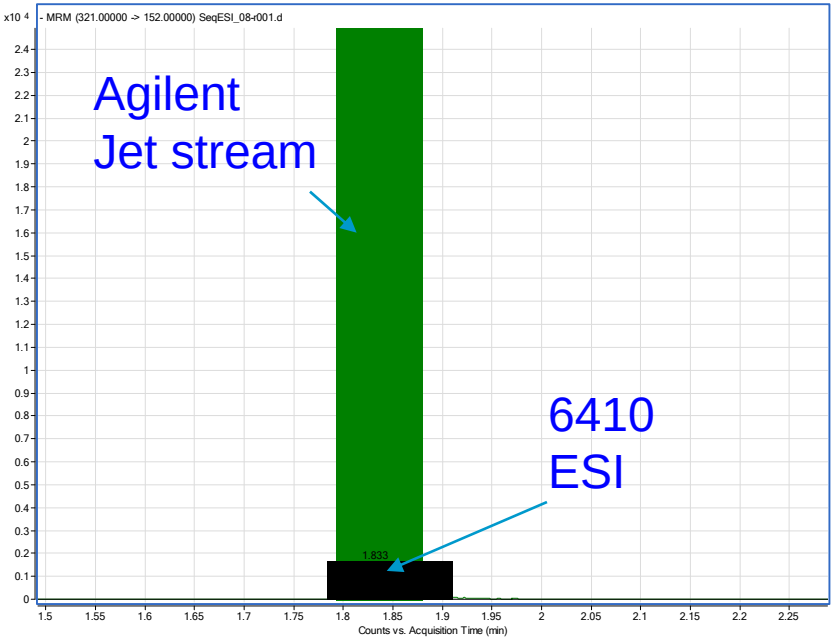
Correlation = 0.9981
Linearity up to 250 pg on-column

5ms dwell + polarity switching

Agilent 6410 ESI vs 6460 Agilent ESI Jetstream



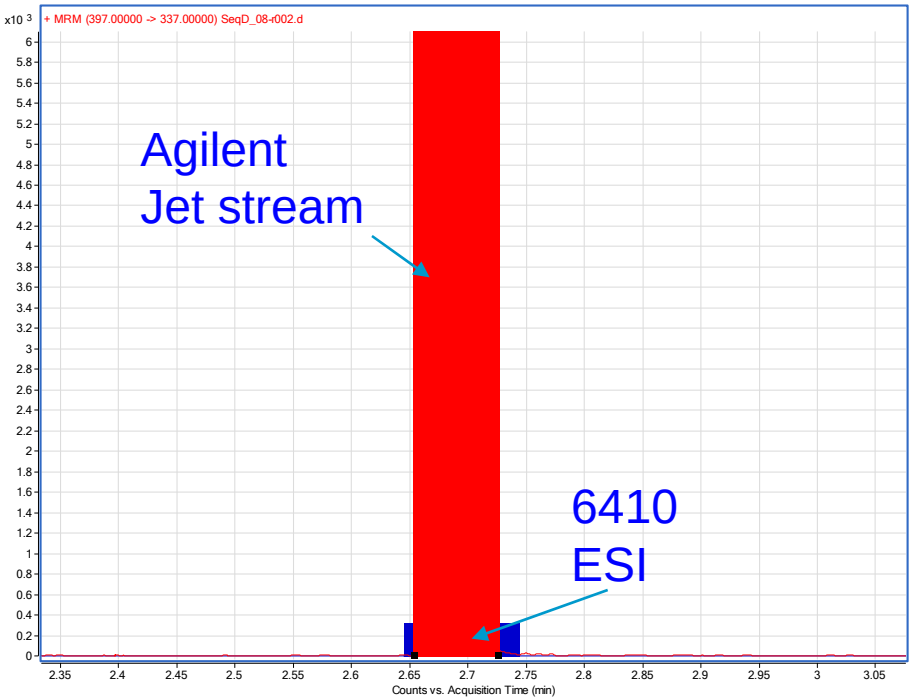
Clorsulon
50 ppb



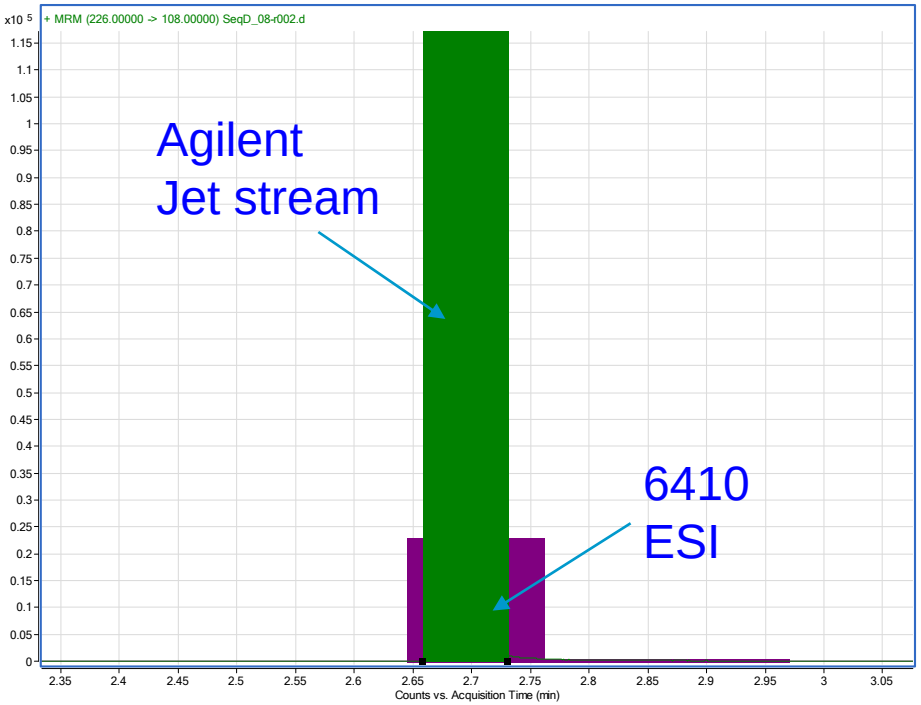
Chloramphenicol
50 ppb

Negative ions
~10x enhanced

Agilent 6410 ESI vs 6460 Agilent ESI Jetstream



Melengestrol
acetate
50 ppb



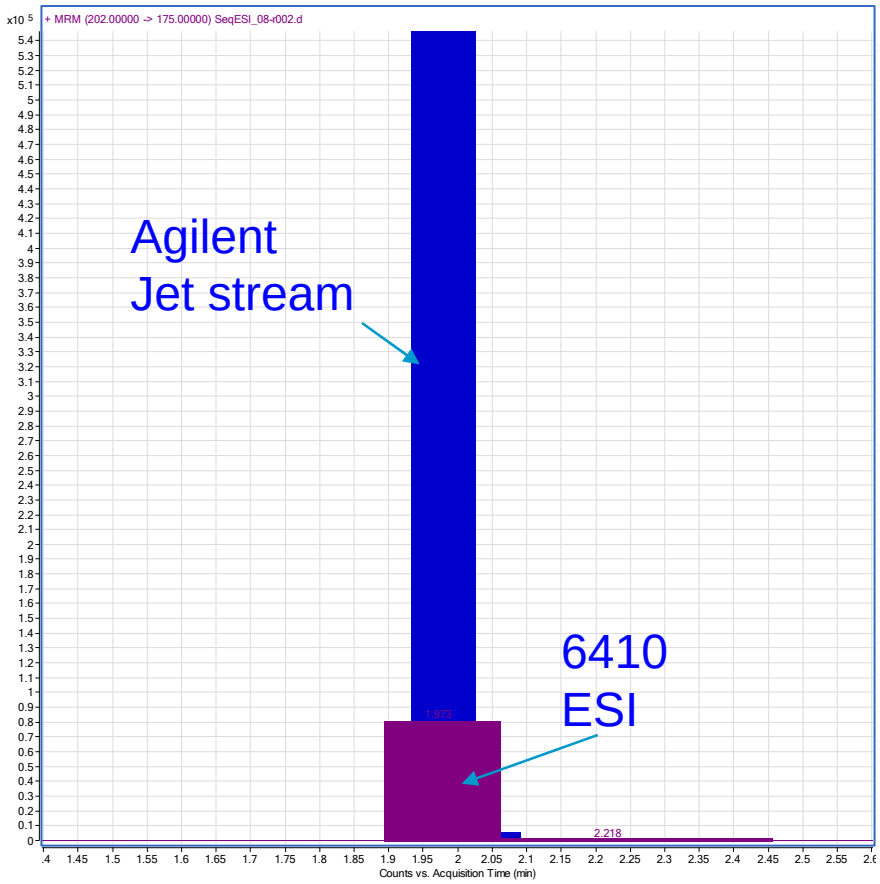
Cyprodinil
50 ppb

Positive ions
~4-10x enhanced

Agilent 6410 ESI vs 6460 Agilent ESI Jetstream

Thiabendazole
50 ppb

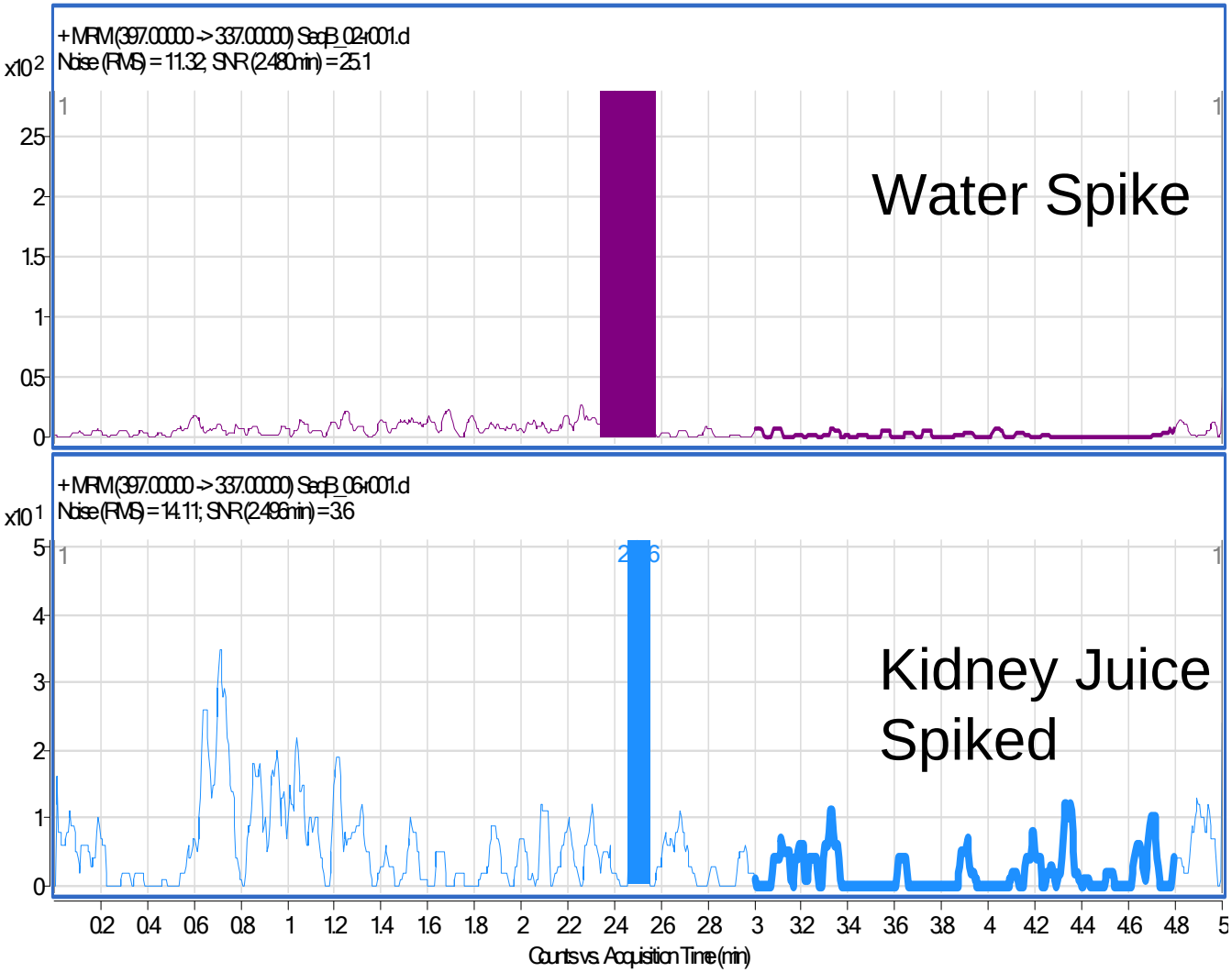
Positive ionisation



Thiabendazole
~4x enhanced

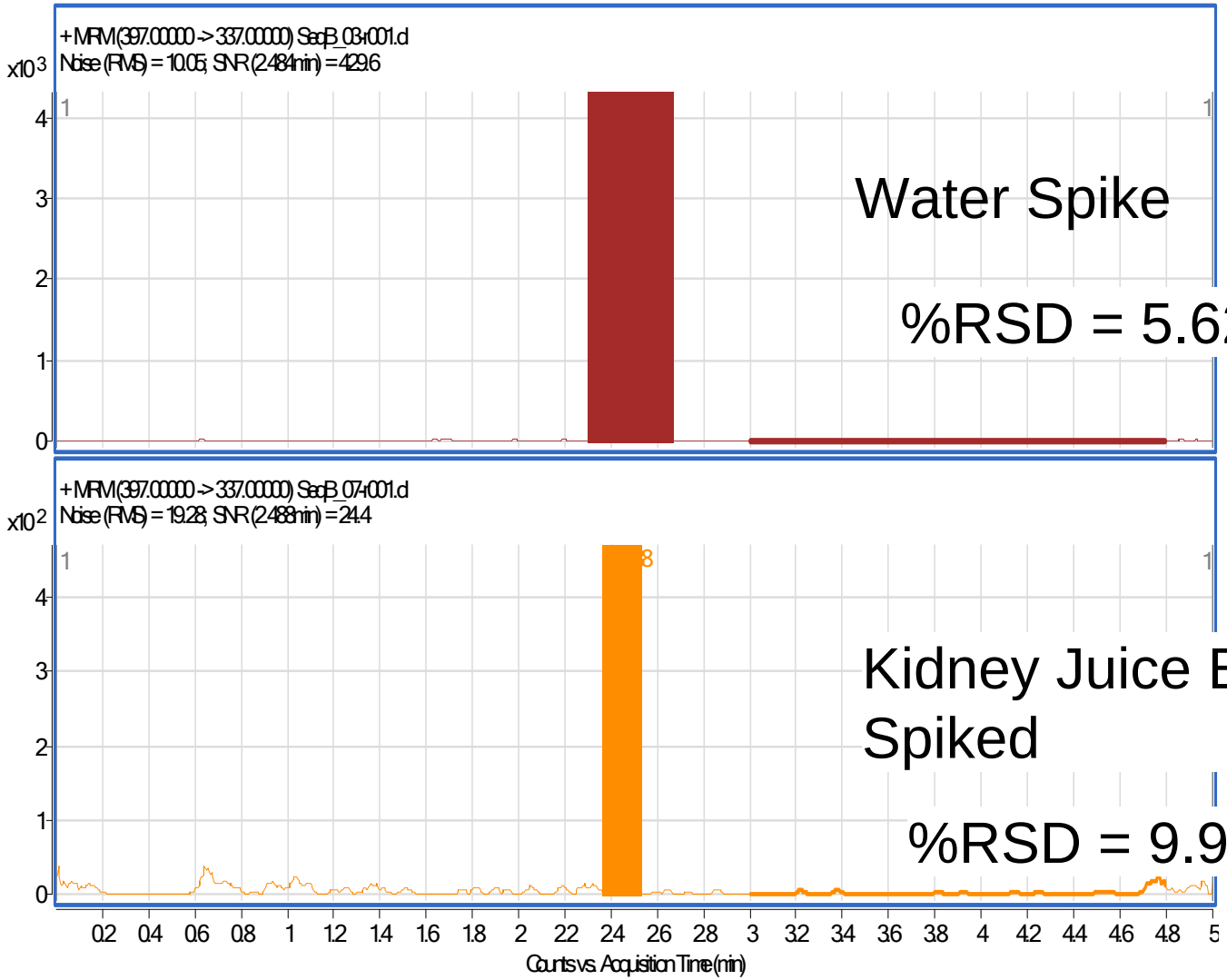
Agilent Technologies

Matrix Samples – Melengestrol acetate (5 ppb)

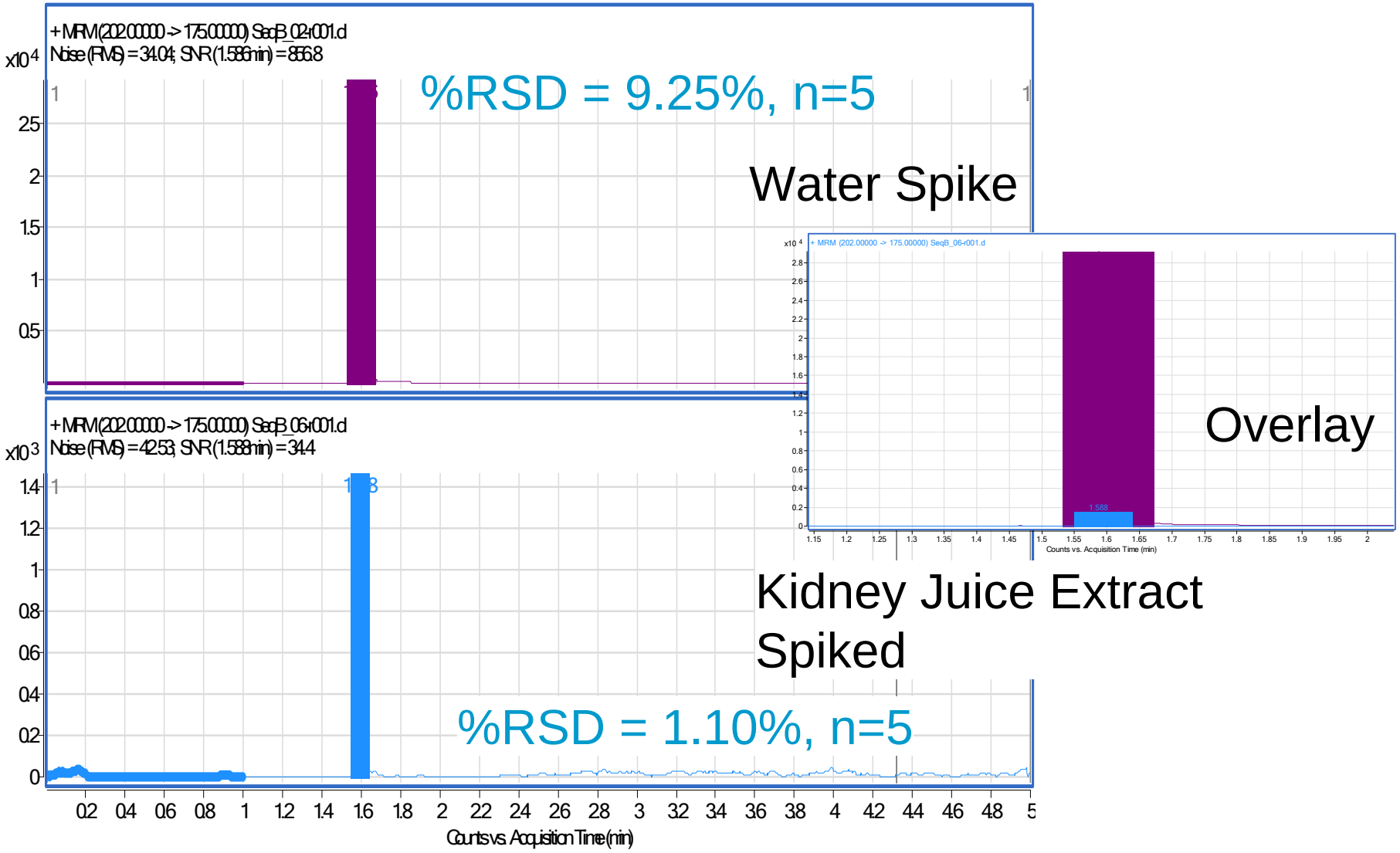


S/N calculation:
5 x RMS

Matrix Samples – Melengestrol acetate (50 ppb)

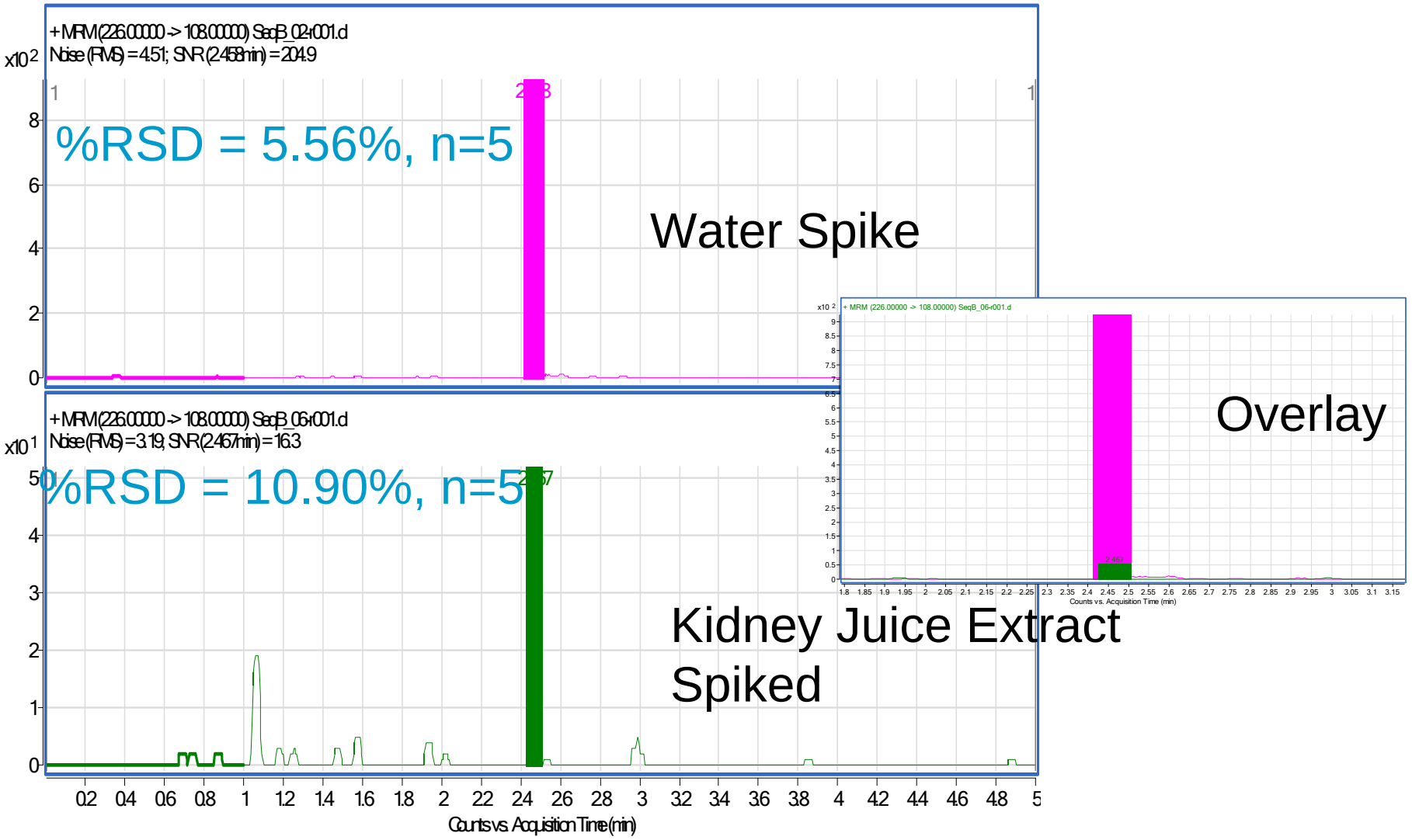


Matrix Samples – Thiabendazole (5 ppb)

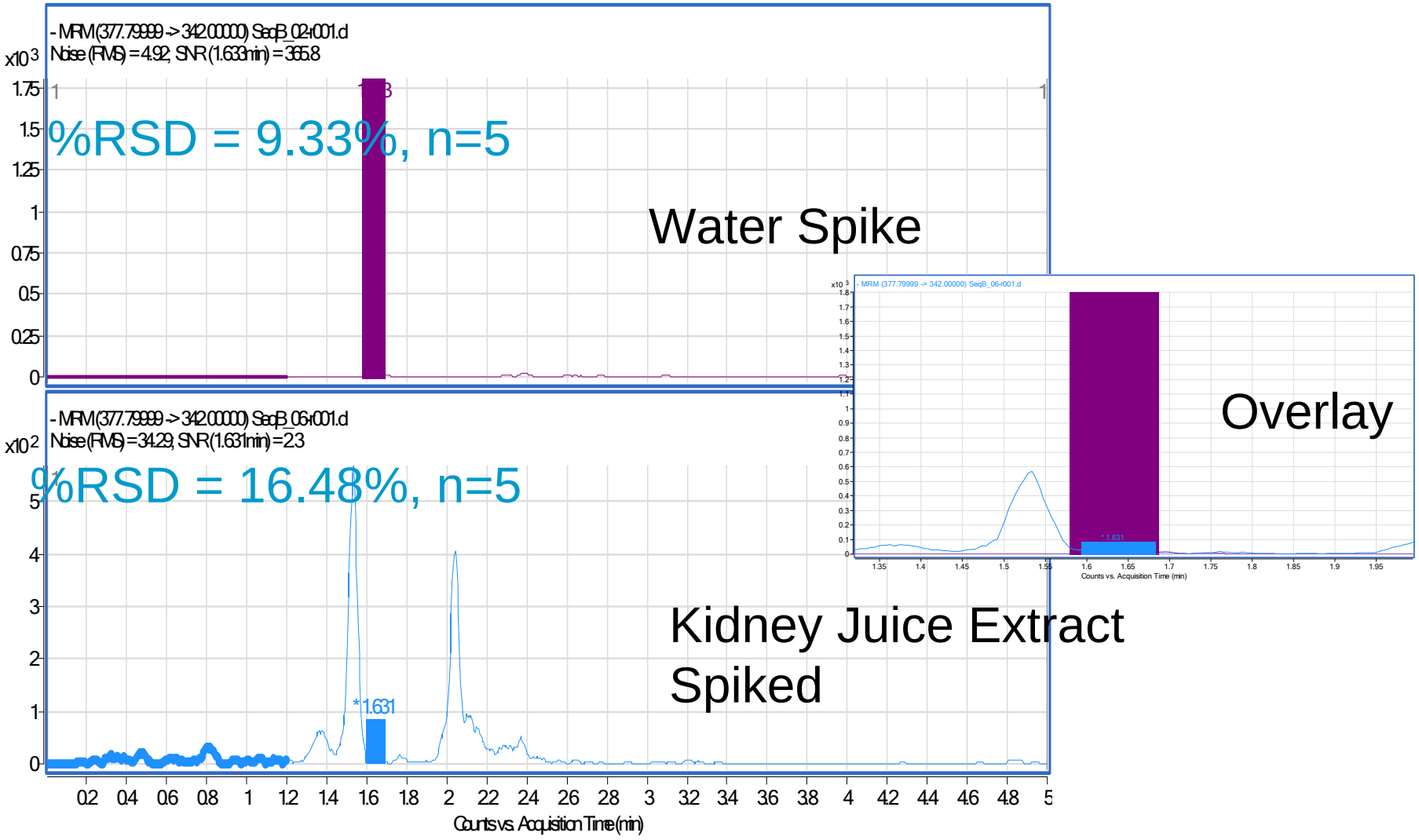


Agilent Technologies

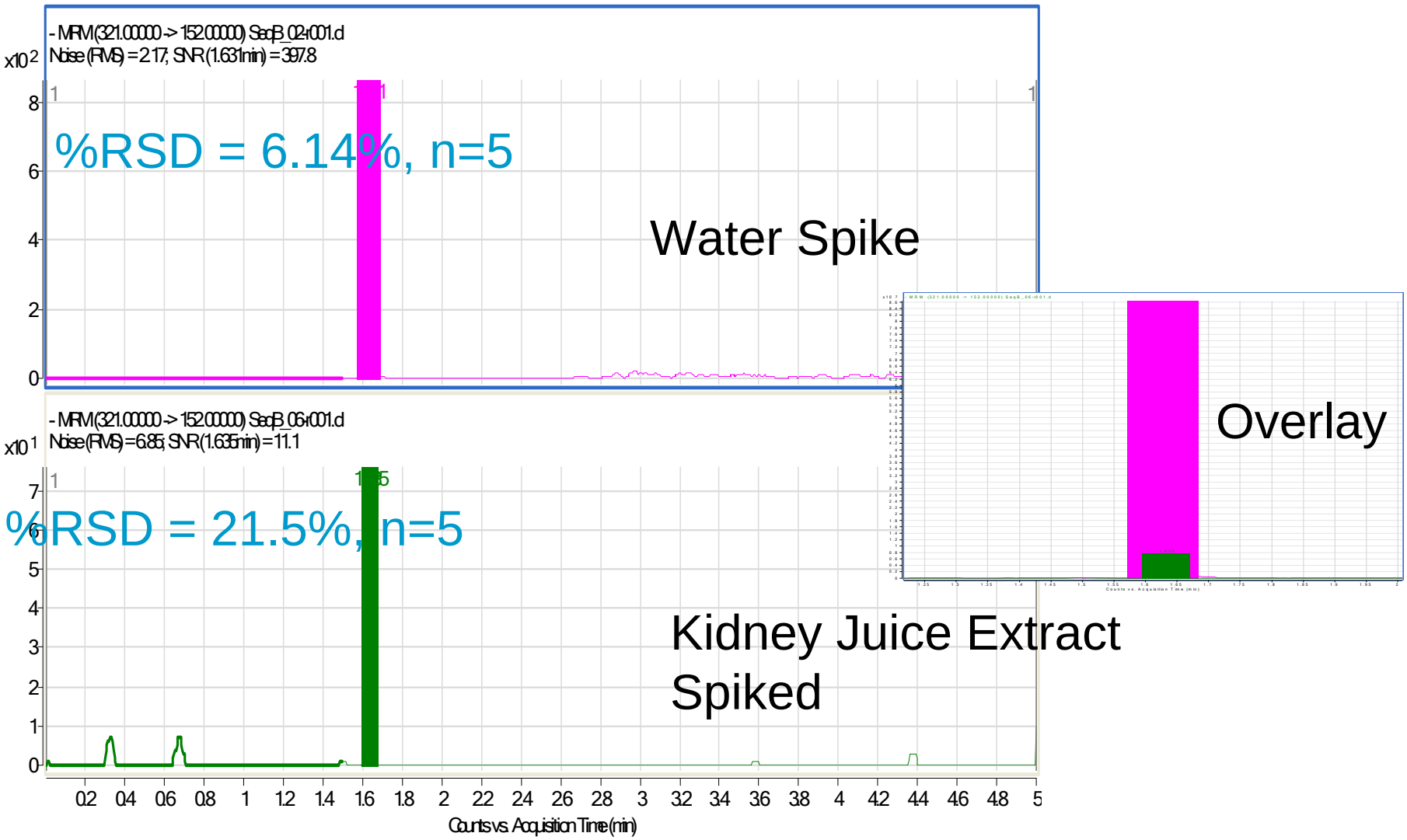
Matrix Samples – Cyprodinil (5 ppb)



Matrix Samples – Chlorsulon (5 ppb)

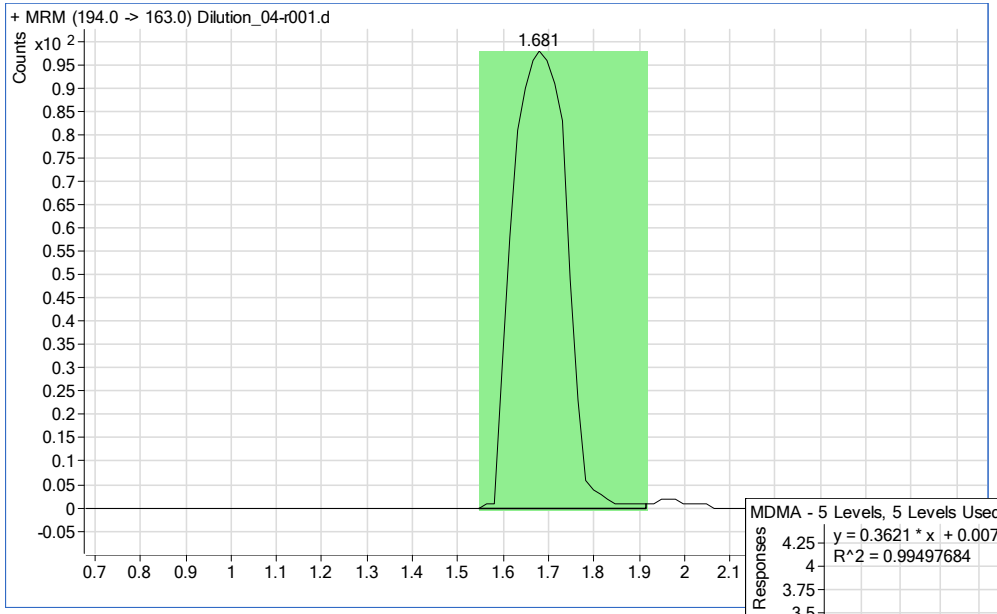


Matrix Samples – Chloramphenicol (5 ppb)



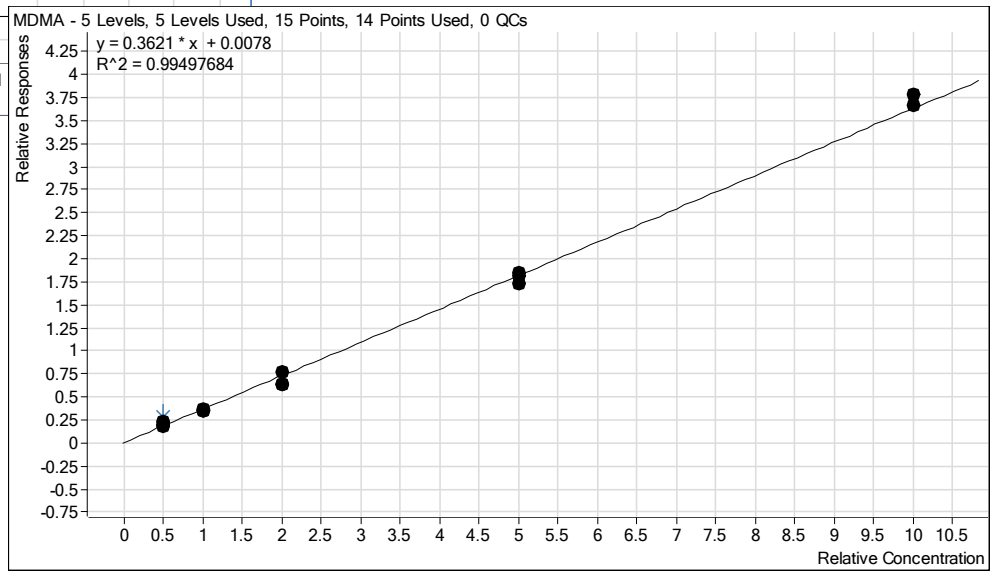
Agilent Technologies

Other Applications MDMA in Urine

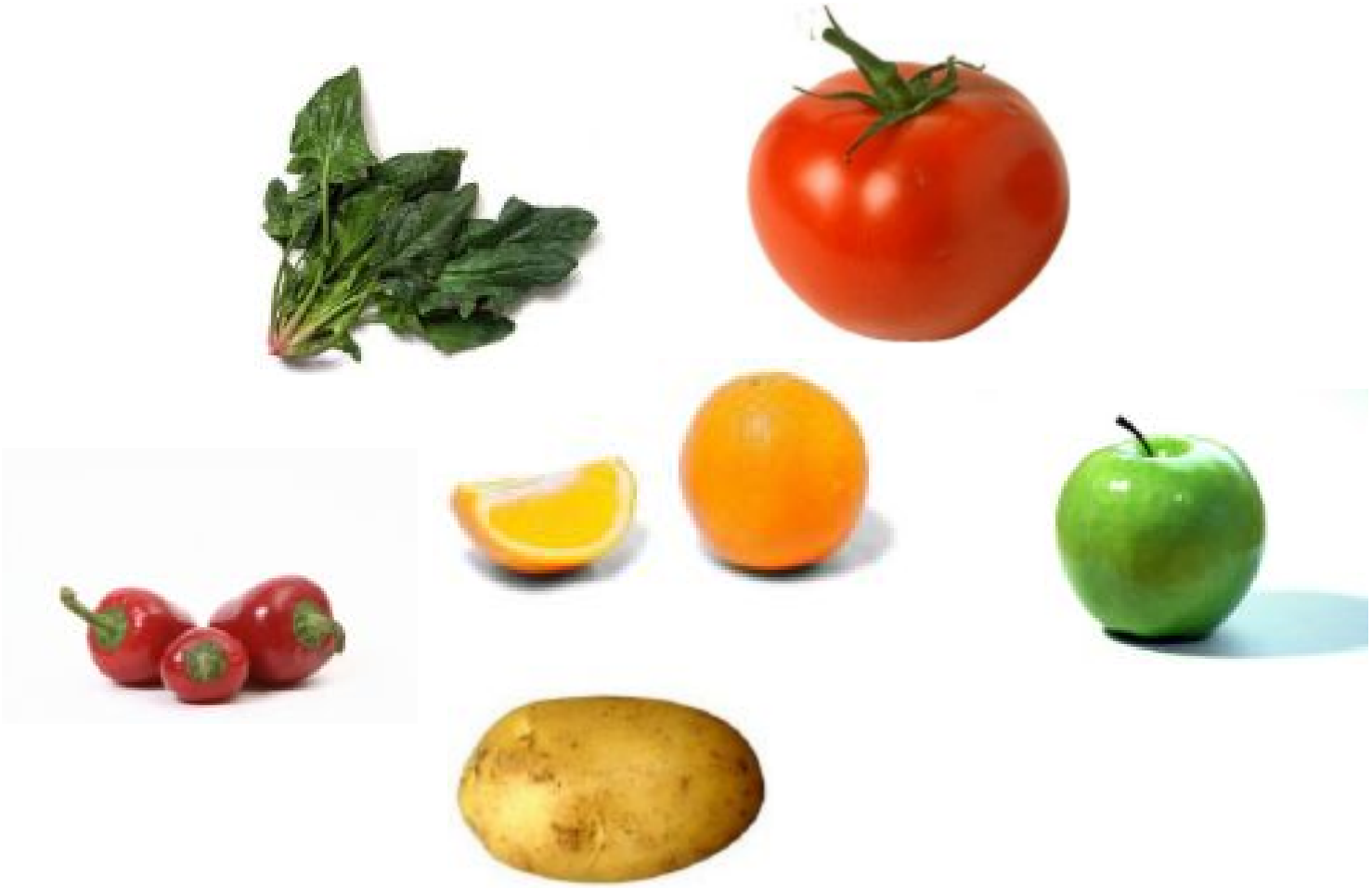


50 fg on-column

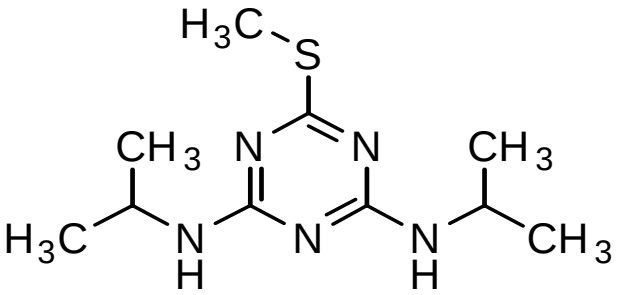
Concentration range
Up to 1 ng on column
RSDs < 12% (n=3)



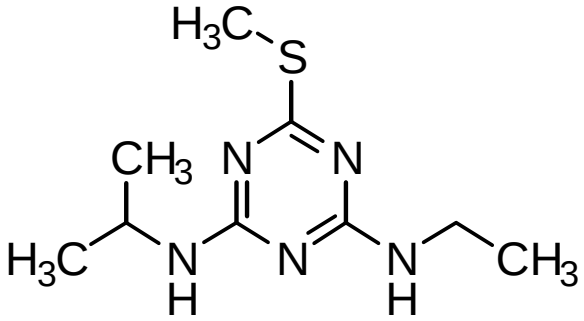
Matrix Diversities for the Study



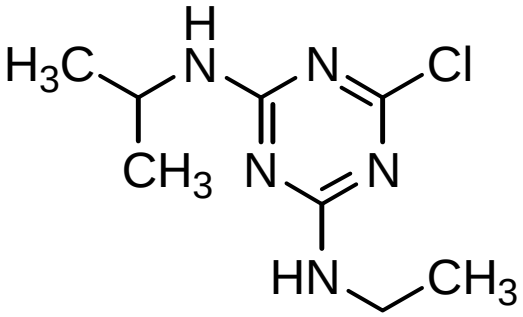
Group 1 compounds- Triazines



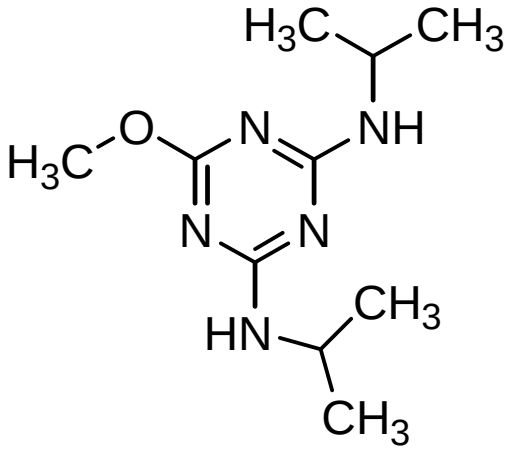
Prometryn



Ametryn

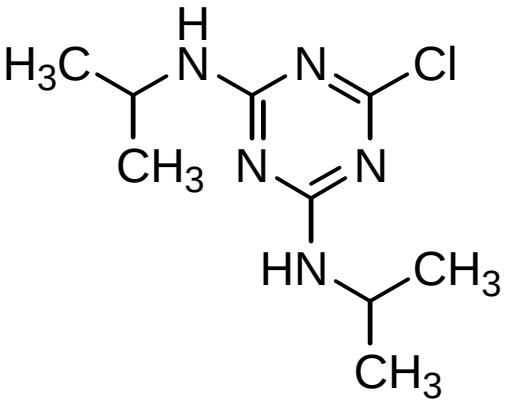


Triazine

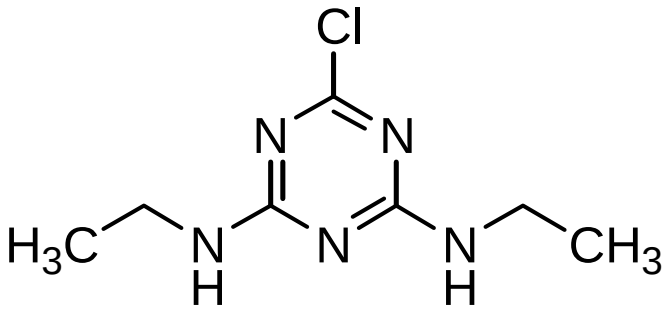


Prometon

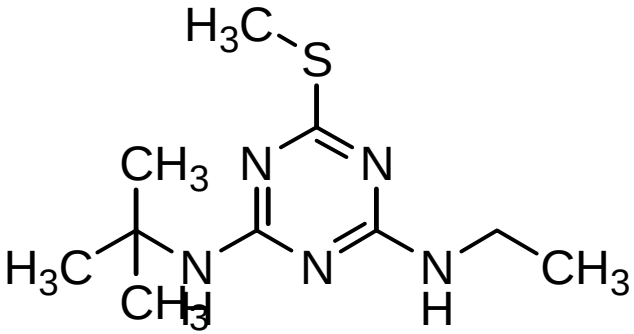
Group 1 compounds- Trazines



Propazine

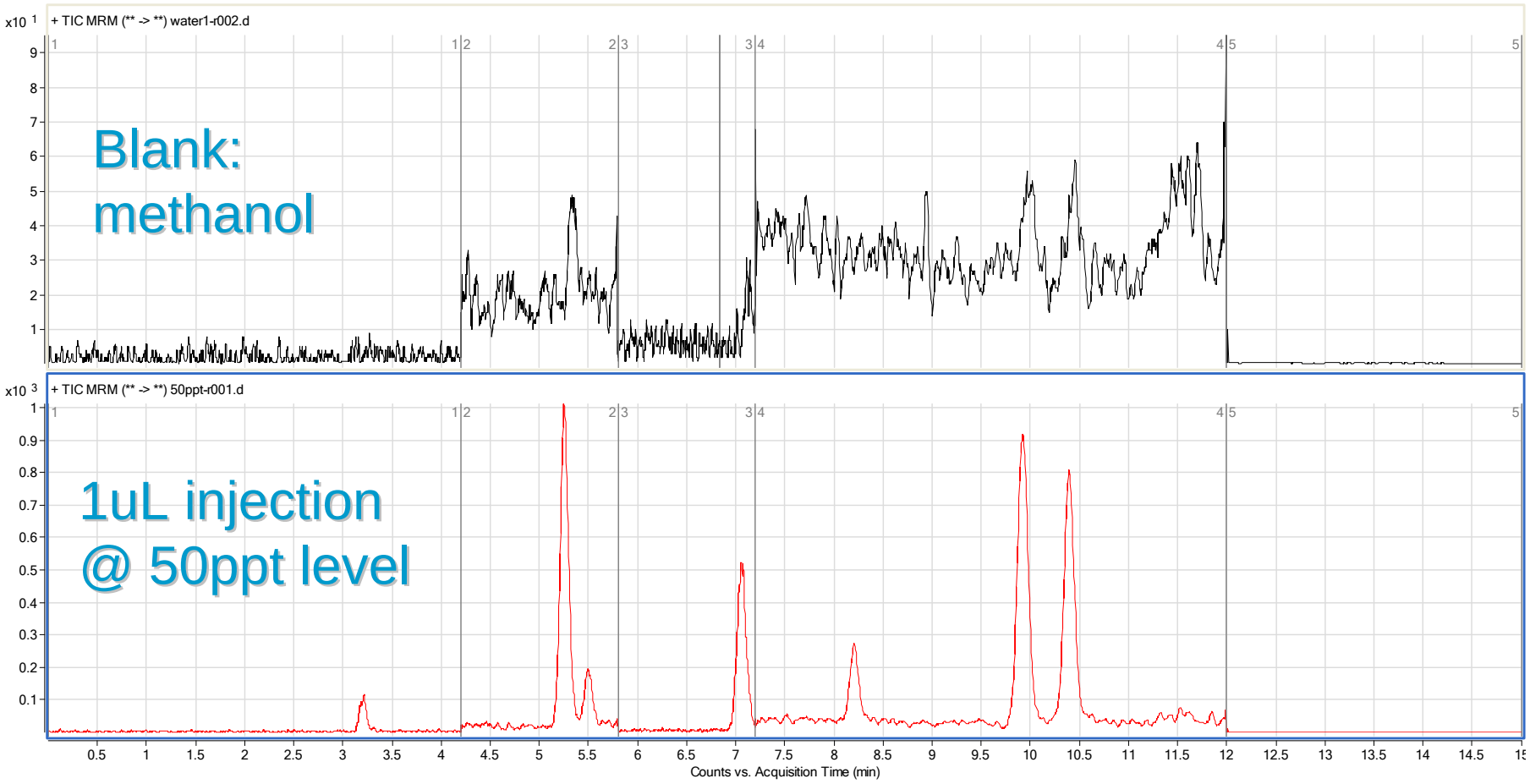


Simazine

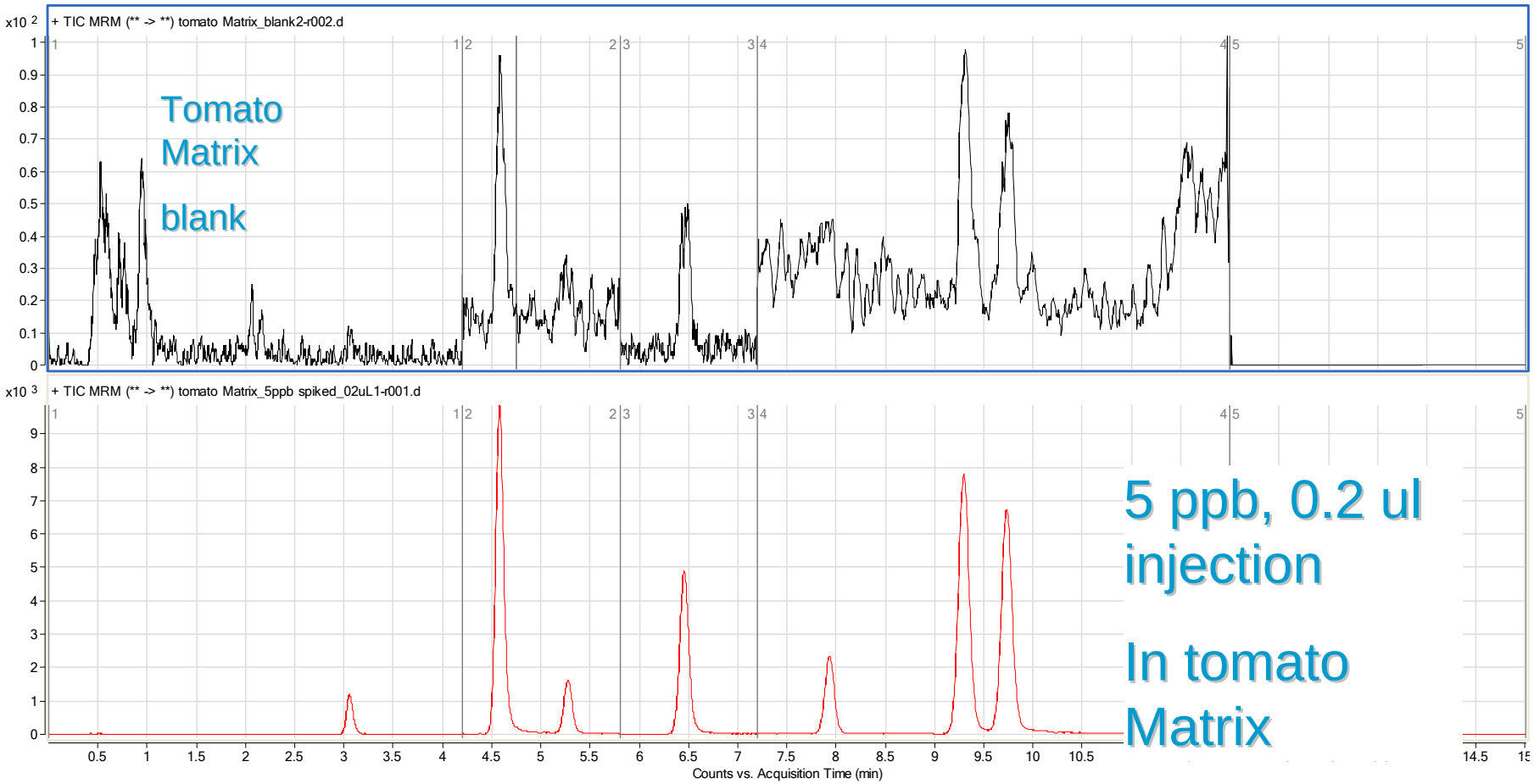


Terbutryn

Sensitivity for the Trazines Stds



Results _ Sensitivity in Tomato Matrix



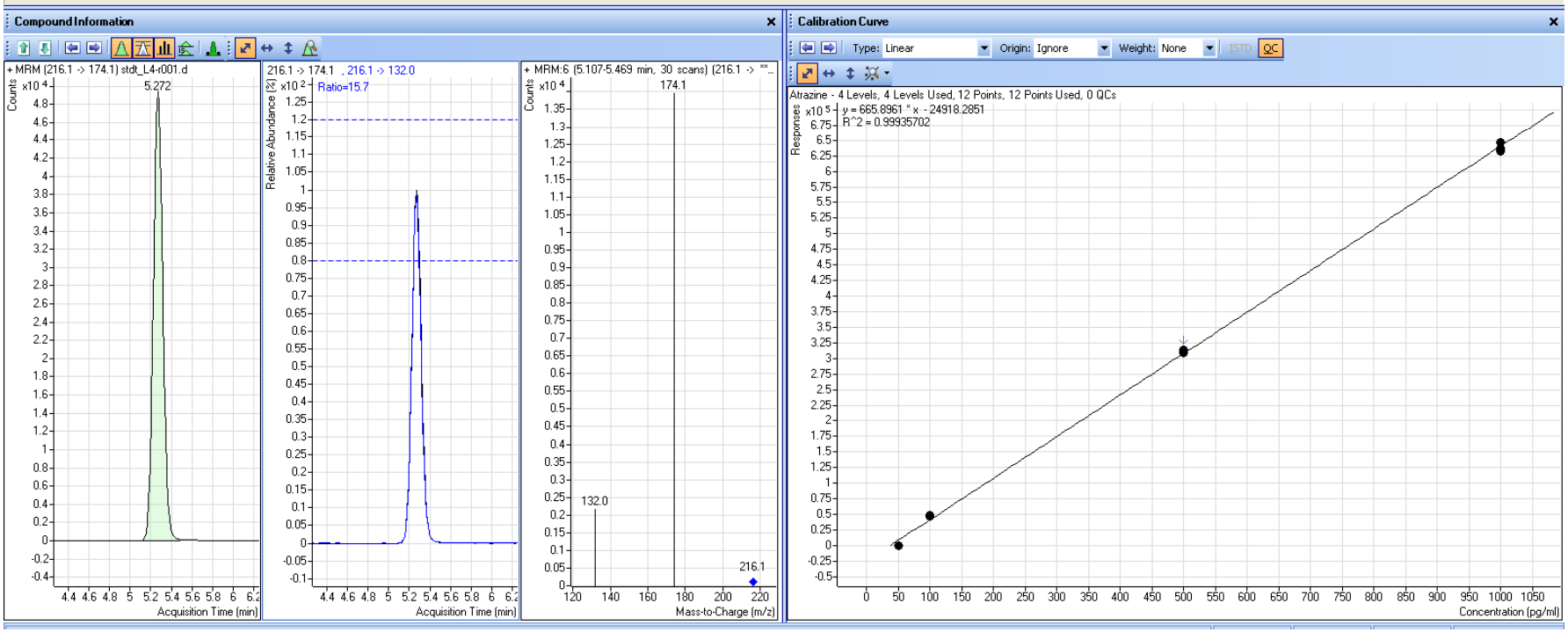
Linearity – Stds in methanol

		Sample					Atrazine...	Atrazine Results						Qualifier (216.1 -...		
		Name	Data File	Type	Level	Acq. Date-Time	Exp. Conc.	RT	Resp.	S/N	MI	Calc. Conc.	Final Conc.	Accuracy	Ratio	S/N	MI
		blank1	water1-r001.d	Blank		4/29/2008 7:17 PM		5.592	364	3.16		37.9673	37.9673		22.1	1.11	
		blank1	water1-r002.d	Blank		4/29/2008 7:38 PM		5.534	248	3.33		37.7935	37.7935				
		blank1	water1-r003.d	Blank		4/29/2008 9:00 PM		5.357	320	5.46		37.9011	37.9011			0.59	
		std1	stdt_L1-r001.d	Cal	L1	4/30/2008 1:29 AM	50.0000	5.288	203	3.00		37.7252	37.7252	75.5		0.90	
		std1	stdt_L1-r002.d	Cal	L1	4/30/2008 1:50 AM	50.0000	5.261	173	2.00		37.6810	37.6810	75.4	28.7	0.60	
		std1	stdt_L1-r003.d	Cal	L1	4/30/2008 2:10 AM	50.0000	5.279	259	4.86		37.8092	37.8092	75.6	54.1	1.52	
		std2	stdt_L2-r001.d	Cal	L2	4/30/2008 2:31 AM	100.0000	5.271	48140	201.95		109.7149	109.7149	109.7	16.5	103.88	
		std2	stdt_L2-r002.d	Cal	L2	4/30/2008 2:52 AM	100.0000	5.271	47721	201.48		109.0845	109.0845	109.1	17.0	62.60	
		std2	stdt_L2-r003.d	Cal	L2	4/30/2008 3:12 AM	100.0000	5.270	47640	227.79		108.9632	108.9632	109.0	16.6	80.46	
		std4	stdt_L4-r001.d	Cal	L4	4/30/2008 4:35 AM	500.0000	5.272	314166	289.63		509.2149	509.2149	101.8	15.7	353.92	
		std4	stdt_L4-r002.d	Cal	L4	4/30/2008 4:56 AM	500.0000	5.262	309734	286.42		502.5594	502.5594	100.5	16.1	209.52	
		std4	stdt_L4-r003.d	Cal	L4	4/30/2008 5:16 AM	500.0000	5.268	313453	337.13		508.1436	508.1436	101.6	15.9	299.06	
		std1	stdt_L5-r001.d	Cal	L5	4/30/2008 5:37 AM	1000.0000	5.267	646203	317.32		1007.8463	1007.8463	100.8	15.7	192.97	
		std1	stdt_L5-r002.d	Cal	L5	4/30/2008 5:58 AM	1000.0000	5.267	632640	441.30		987.4787	987.4787	98.7	15.8	190.67	
		std1	stdt_L5-r003.d	Cal	L5	4/30/2008 6:18 AM	1000.0000	5.265	636835	295.30		993.7790	993.7790	99.4	16.0	318.74	

Atrazine:

R^2=0.999

50 ppt – 1 ppb



Linearity in Tomato Matrix

Sample						Atrazine...	Atrazine Results							Qualifier (216.1...		
	Name	Data File	Type	Level	Acq. Date-Time	Exp. Conc.	RT	Resp.	S/N	MI	Calc. Conc.	Final Conc.	Accuracy	Ratio	S/N	MI
	blank1	blank1-r001.d	Blank		4/29/2008 7:58 PM		5.479	424	3.87		0.0000	0.0000		14.5	0.59	
	blank1	blank1-r002.d	Blank		4/29/2008 8:19 PM		5.444	213	2.24		0.0000	0.0000		56.3	0.85	
	blank1	blank1-r003.d	Blank		4/29/2008 8:40 PM		5.416	314	3.42		0.0000	0.0000		17.0	0.84	
	50ppt1	50ppt_L11-r001.d	Cal	L1	4/29/2008 9:21 PM	50.0000	5.343	994	11.09		43.2087	43.2087	86.4	15.5	1.49	
	50ppt1	50ppt_L11-r002.d	Cal	L1	4/29/2008 9:42 PM	50.0000	5.289	1074	9.26		50.3409	50.3409	100.7	15.3	2.10	
	50ppt1	50ppt_L11-r003.d	Cal	L1	4/29/2008 10:02 PM	50.0000	5.286	974	11.48		41.4176	41.4176	82.8	19.9	2.86	
	100ppt1	100ppt_L21-r001.d	Cal	L2	4/29/2008 10:23 PM	100.0000	5.270	1571	16.56		95.0323	95.0323	95.0	19.3	2.73	
	100ppt1	100ppt_L21-r002.d	Cal	L2	4/29/2008 10:44 PM	100.0000	5.279	1619	14.44		99.3863	99.3863	99.4	23.1	5.23	
	100ppt1	100ppt_L21-r003.d	Cal	L2	4/29/2008 11:04 PM	100.0000	5.285	1652	16.95		102.3083	102.3083	102.3	24.7	5.05	
	200ppt1	200ppt_L31-r001.d	Cal	L3	4/29/2008 11:25 PM	200.0000	5.282	2769	27.01		202.7863	202.7863	101.4	21.7	8.04	
	200ppt1	200ppt_L31-r002.d	Cal	L3	4/29/2008 11:46 PM	200.0000	5.283	2876	26.34		212.3199	212.3199	106.2	21.9	6.03	
	200ppt1	200ppt_L31-r003.d	Cal	L3	4/30/2008 12:06 AM	200.0000	5.275	2870	24.86		211.8072	211.8072	105.9	21.3	5.29	
	500ppt1	500ppt_L41-r001.d	Cal	L4	4/30/2008 12:27 AM	500.0000	5.282	5849	56.85		479.6036	479.6036	95.9	18.7	15.92	
	500ppt1	500ppt_L41-r002.d	Cal	L4	4/30/2008 12:48 AM	500.0000	5.283	6255	74.19		516.0766	516.0766	103.2	16.7	10.98	
	500ppt1	500ppt_L41-r003.d	Cal	L4	4/30/2008 1:08 AM	500.0000	5.284	6028	52.97		495.7123	495.7123	99.1	17.1	11.41	

Atrazine