

Matrix Study 10/09/15 Marco Troiani Savino Sguera

The following codes are used for subsequent expedience:

Initial Extraction Kits

Code **α** – PerkinElmer Initial Extraction: **N9306901**

MgSO <sub>4</sub>	4g
PSA	50 mg
C <sub>18</sub>	50 mg
GCB	0 mg

Code **β** – Restek Initial Extraction: **#26238**

MgSO <sub>4</sub>	6g
NaCH <sub>3</sub> CO <sub>2</sub>	1.5g
NaH <sub>2</sub> C <sub>4</sub> H <sub>7</sub> O <sub>2</sub>	0g
Na <sub>2</sub> H <sub>2</sub> C <sub>4</sub> H <sub>7</sub> O <sub>2</sub> +1.5H <sub>2</sub> O	0g
Na <sub>2</sub> C <sub>10</sub> H <sub>8</sub> O <sub>2</sub> +2H <sub>2</sub> O	0g
NaCl	0g

Code **γ** – UCT Initial Extraction: **ECQUEU75OCT-MP**

MgSO <sub>4</sub>	4g
NaCH <sub>3</sub> CO <sub>2</sub>	0g
Na <sub>2</sub> C <sub>10</sub> H <sub>8</sub> O <sub>2</sub>	0g
Na <sub>2</sub> H <sub>2</sub> C <sub>4</sub> H <sub>7</sub> O <sub>2</sub> +1.5H <sub>2</sub> O	0.5g
Na <sub>2</sub> C <sub>10</sub> H <sub>8</sub> O <sub>2</sub> +2H <sub>2</sub> O	1g
NaCl	1g

Clean-Up Extraction Kits

Code **δ** – Waters Clean-Up Extraction: **186004830**

MgSO <sub>4</sub>	150 mg
PSA	50 mg
C <sub>18</sub>	50 mg
GCB	0 mg

Code **ε** – Restek Clean-Up Extraction: **#26123**

MgSO <sub>4</sub>	150 mg
PSA	50 mg
C <sub>18</sub>	0 mg
GCB	50 mg

Code **ζ** – PerkinElmer Clean-Up: PerkinElmer Clean-Up Extraction: **N9306923**

MgSO <sub>4</sub>	900 mg
PSA	150 mg
C <sub>18</sub>	200 mg
GCB	0 mg

Experiment Sample List 01: Prepare the following samples using the prescribed combinations of extraction kits.

Sample A – β:ε

Sample B – α:(δ + 7.5 mg GCB)

Sample C – β:(δ + 7.5 mg GCB)

Sample D – α:ε

Sample E – α:(δ + 50.0 mg GCB)

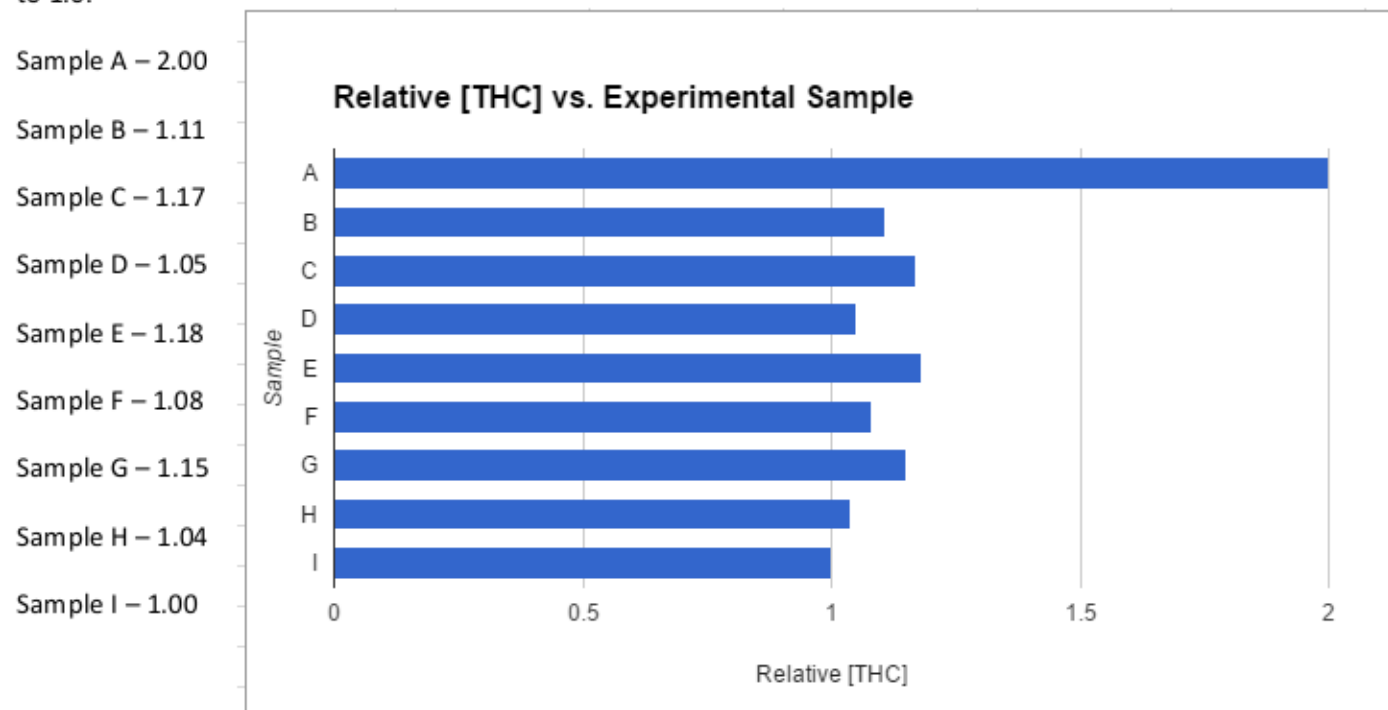
Sample F – β:(δ + 50.0 mg GCB)

Sample G – γ:ε

Sample H – γ:(δ + 7.5 mg GCB)

Sample I – γ:(δ + 50.0 mg GCB)

Experimental Results 01: Relative THC concentration in extract, highest value set to 2.0, lowest value set to 1.0.



Sample ID	Sample Type	Concentration (ppb)	Stock Volume Used (µl)
Cal-010	Standard	10	4
Cal-020	Standard	20	8
Cal-050	Standard	50	20
Cal-100	Standard	100	40
Cal-250	Standard	250	100
Cal-MB	Matrix Blank	0	0
Cal-Val	Analyte	75	30

Matrix Study 10/28/15 Marco Troiani

The following extraction procedure is to be used for the following study:

Initial Extraction: PerkinElmer Initial Extraction: **N9306901**  
 MgSO<sub>4</sub> – 4 g; NaH<sub>2</sub>C<sub>4</sub>H<sub>7</sub>O<sub>2</sub> – 1 g; Na<sub>2</sub>H<sub>2</sub>C<sub>4</sub>H<sub>7</sub>O<sub>2</sub>+1.5H<sub>2</sub>O – 0.5 g; NaCl – 1 g

Clean-Up Extraction: Waters Clean-Up Extraction: **186004830**  
 MgSO<sub>4</sub> – 150 mg; PSA – 50 mg; C<sub>18</sub> – 50 mg

NOTE: 7 mg of Graphitized Carbon, Black (GCB) is to be added to each Clean-Up Extraction tube

Experimental Sample List:

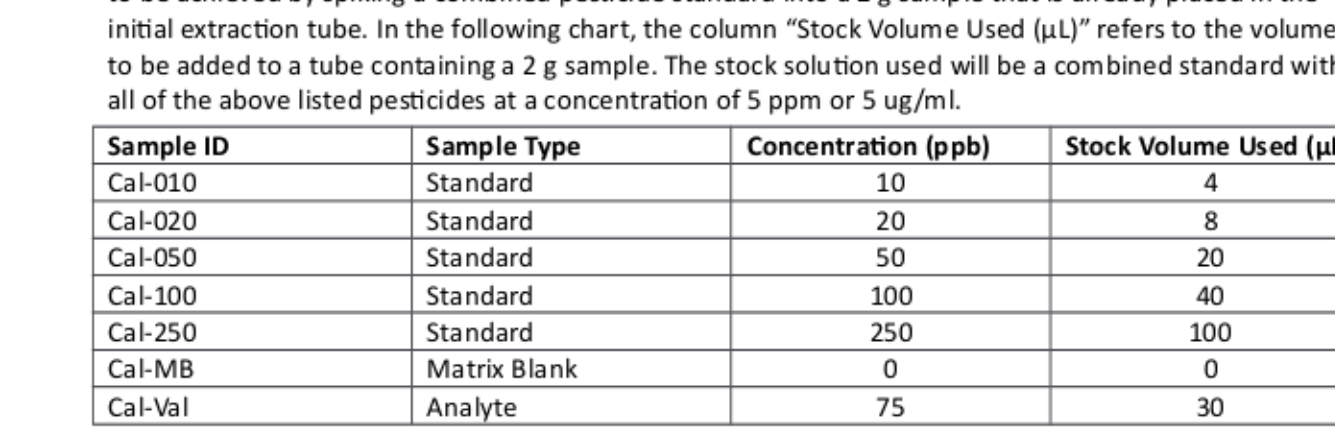
The pesticides required for this experiment are as follows with tolerance limits in ppm and ppb:

Pesticide	Limit ppm	Limit ppb
Abamectin	0.05	50
Acequinocyl	4	4000
Bifenazate	15	1500
Bifenthrin	0.05	50
Captan	0.05	50
Cyfluthrin and b-Cyfluthrin	4	4000
Cypermethrin	0.05	50
Dimethomorph	60	60000
Etoazolo	7	7000
Fenhexamid	30	30000
Flonicamid	7	7000
Fludioxonil	0.02	20
Imidacloprid	0.05	50
Myoclobutani	9	9000
Pentachloronitrobenzene	0.02	20
Piperonyl Butoxide	2	2000
Pyrethrin	1.7	1700
Spinetoram	1.7	1700
Spinosad	1.7	1700
Spirotetramat	10	10000
Thiamethoxam	0.02	20
Trifloxystrobin	11	11000

Each sample is to be prepared according to the following chart. The final concentration in the sample is to be achieved by spiking a combined pesticide standard into a 2 g sample that is already placed in the initial extraction tube. In the following chart, the column "Stock Volume Used (µl)" refers to the volume to be added to a tube containing a 2 g sample. The stock solution used will be a combined standard with all of the above listed pesticides at a concentration of 5 ppm or 5 µg/ml.

Sample ID	Sample Type	Concentration (ppb)	Stock Volume Used (µl)
Cal-010	Standard	10	4
Cal-020	Standard	20	8
Cal-050	Standard	50	20
Cal-100	Standard	100	40
Cal-250	Standard	250	100
Cal-MB	Matrix Blank	0	0
Cal-Val	Analyte	75	30

Quantify Compound Summary Report



Experiment Sample List 02: Prepare the following samples using the prescribed combination of extraction kits. Spike the 2 g Cannabis plant sample with the 22 required Nevada State pesticide monitoring program pesticides so that 2 µg of each pesticide is introduced to the 2 g sample.

Sample J – β:ε

Sample K – β:(δ + 7.5 mg GCB)

Sample L – β:(δ + 50.0 mg GCB)

Sample M – γ:ε

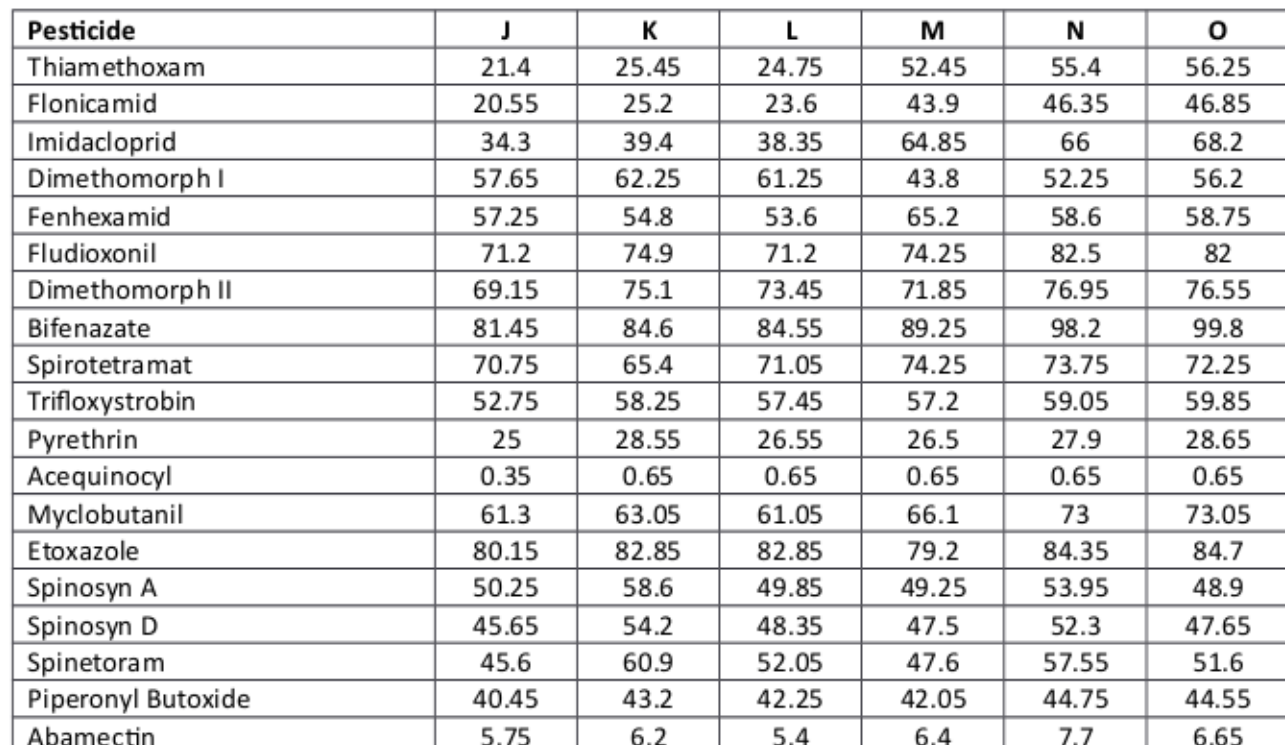
Sample N – γ:(δ + 7.5 mg GCB)

Sample O – γ:(δ + 50.0 mg GCB)

Experimental Results 02: The experimental results are as follows

Pesticide	J	K	L	M	N	O
Thiamethoxam	42.8	50.9	49.5	104.9	110.8	112.5
Fonicamid	41.1	50.4	47.2	87.8	92.7	93.7
Imidacloprid	68.6	78.8	76.7	129.7	132	136.4
Dimethomorph I	115.3	124.5	122.5	87.6	104.5	112.4
Fenhexamid	114.5	109.6	107.2	130.4	117.2	117.5
Fludioxonil	142.4	149.8	142.4	148.5	165	164
Dimethomorph II	138.3	150.2	146.9	143.7	153.9	153.1
Bifenazate	162.9	169.2	169.1	178.5	196.4	199.6
Spirotetramat	141.5	130.8	142.1	148.5	147.5	144.5
Trifloxystrobin	105.5	116.5	114.9	114.4	118.1	119.7
Pyrethrin	50	57.1	53.1	53	55.8	57.3
Acequinocyl	0.7	1.3	1.3	1.3	1.3	1.3
Myoclobutani	122.6	126.1	122.1	132.2	146	146.1
Etoazolo	160.3	165.7	165.7	158.4	168.7	169.4
Spinosyn A	100.5	117.2	99.7	98.5	107.9	97.8
Spinosyn D	91.3	108.4	96.7	95	104.6	95.3
Spinetoram	91.2	121.8	104.1	95.2	115.1	103.2
Piperonyl Butoxide	80.9	86.4	84.5	84.1	89.5	89.1
Abamectin	11.5	12.4	10.8	12.8	15.4	13.3

Results as a % Recovery from total spike (200 ppb)



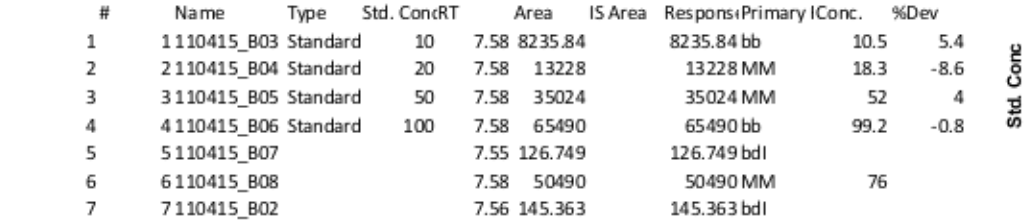
Bar Graph representing the % Recoveries of various pesticides from matrix according to extraction letter code J-O

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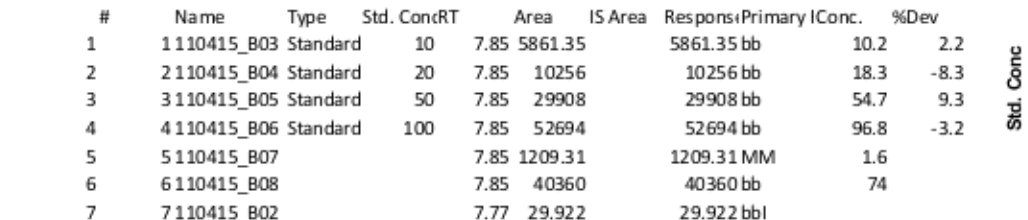
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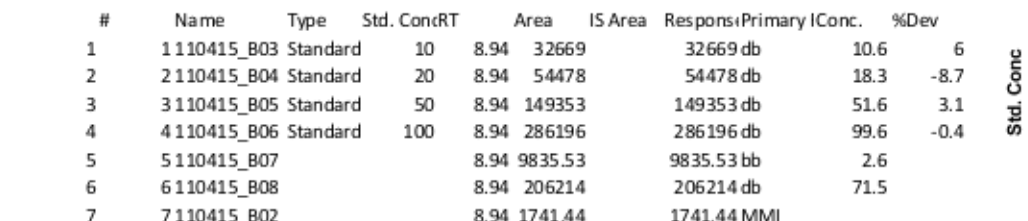
Compound 8: Bifenazate 101.333333



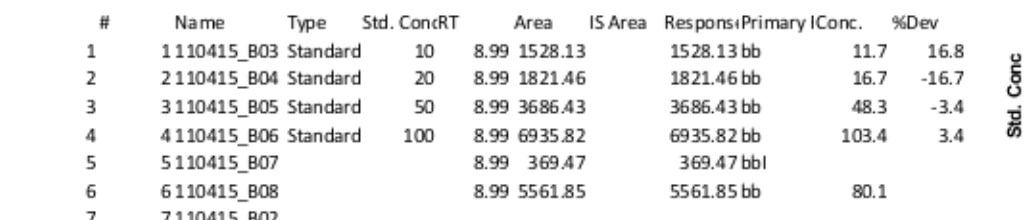
Compound 9: Spirotetramat 98.66666667



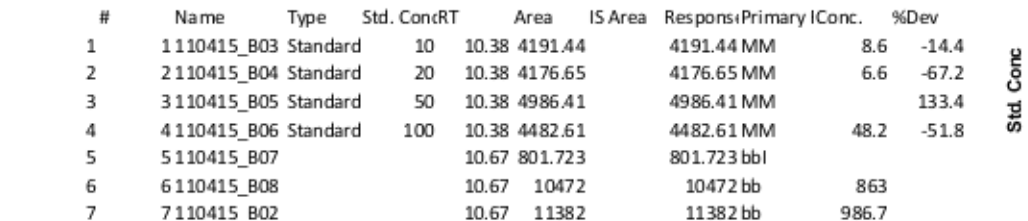
Compound 10: Trifloxystrobin 95.33333333



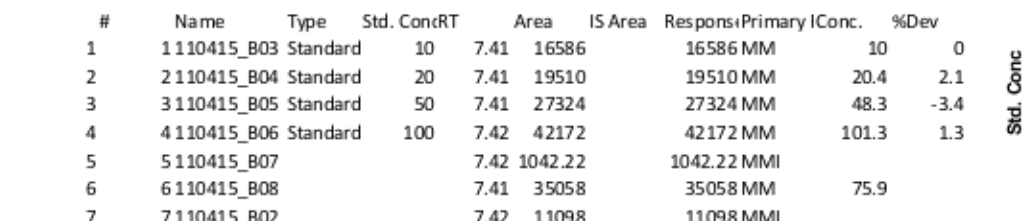
Compound 11: Pyrethrin 106.8



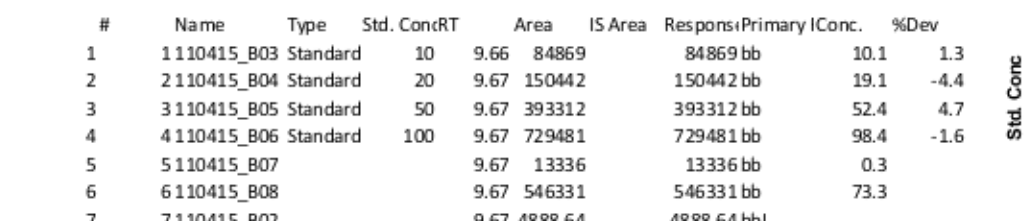
Compound 12: Acequinocyl 1150.666667



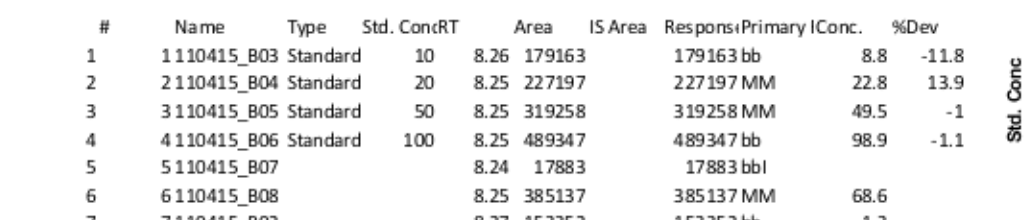
Compound 13: Myclobutanol 101.2



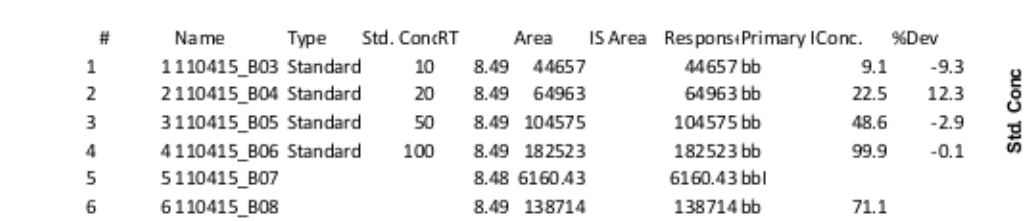
Compound 14: Etoazolo 97.73333333



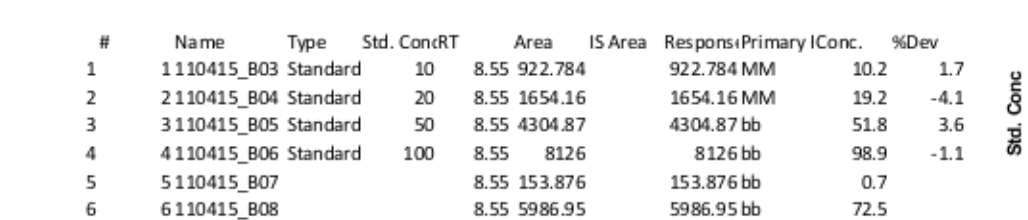
Compound 15: Spinosyn A 91.46666667



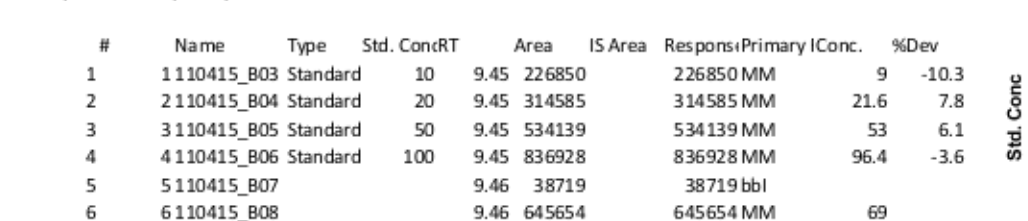
Compound 16: Spirotetramat 94.8



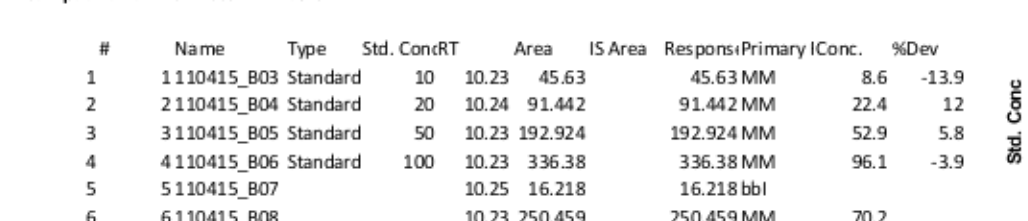
Compound 17: Spinetoram 96.66666667



Compound 18: Piperonyl Butoxide 93.6



Compound 19: Abamectin 99.6



Compound 20: Fludioxonil 92.4



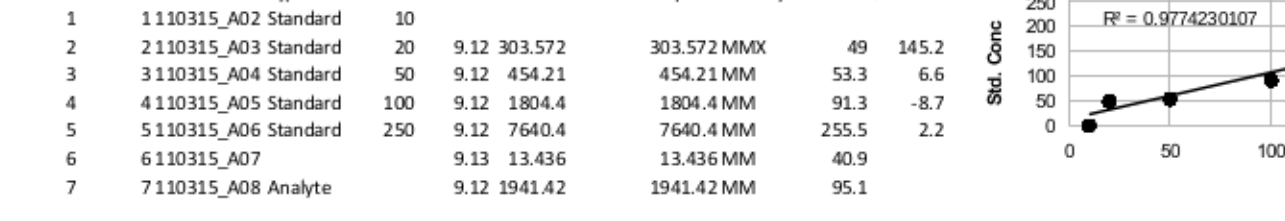
Compound 21: Dimethomorph 100.13333333



Quantify Compound Summary Report

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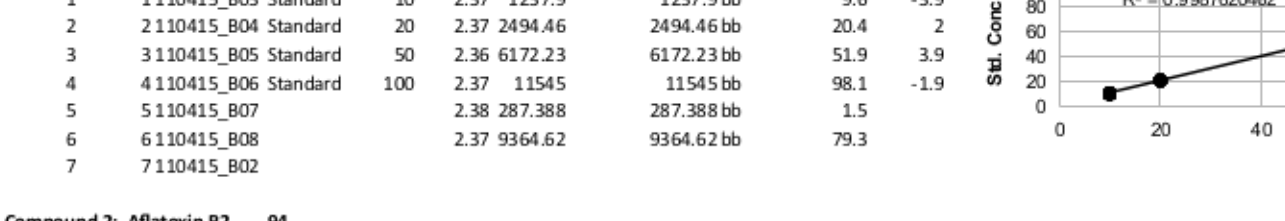
Compound 1: PC 126.4



Quantify Compound Summary Report

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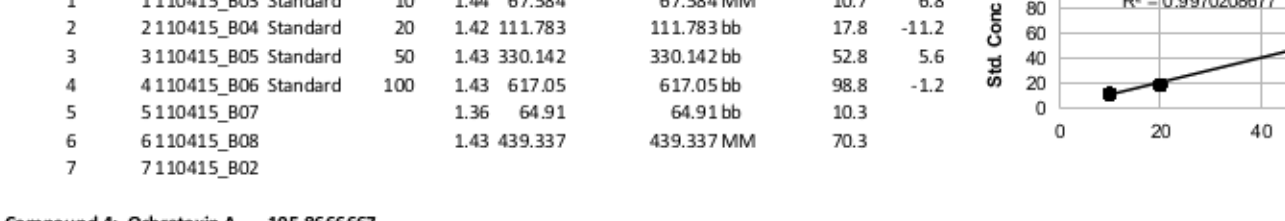
Compound 1: Aftainin B1 105.7333333



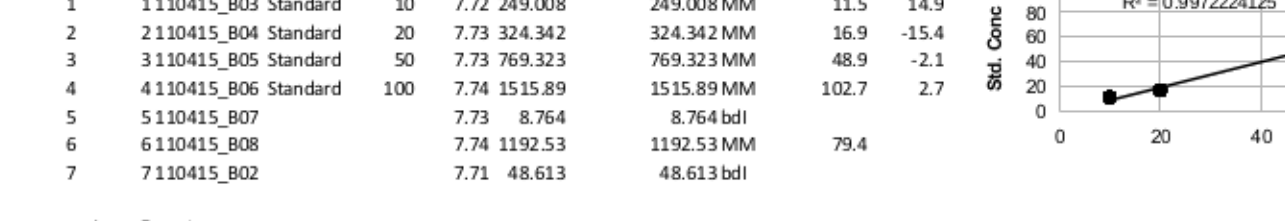
Compound 2: Aftainin B2 94



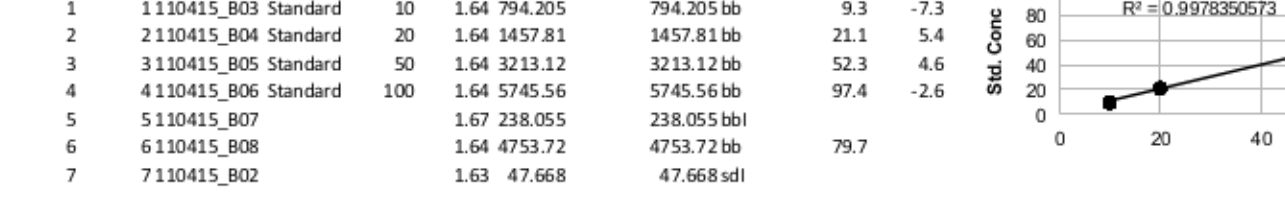
Compound 3: Aftainin G2 93.73333333



Compound 4: Odrabrin A 105.86666667



Compound 5: Aftainin G1 106.26666667



Extraction and Calibration Efficiency of Pesticides and Mycotoxins in Cannabis Flower  
 ACS Conference 2016, Philadelphia  
 Marco Troiani  
 Savino Sguera

All of the data displayed here is taken from experiments performed in October 2015 at DB Labs in Las Vegas, Nevada by Marco Troiani, Savino Sguera, and Chris Kowalczyk. Pesticide analysis was performed with a Waters UHPLC-MS-MS.