

Mycotoxins in feed round robin

Samples sent early 2016

- 4 previously analyzed feed samples from the AAFCO mycotoxin PT program in 2014 were sent to 8 participating labs
- Labs were instructed to use the method they are currently using to detect the mycotoxins of interest and provide a brief description of the method

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- 7 labs analyzed by solvent extraction followed by LC/MS/MS analysis
 - 1 lab (lab 8) used ELISA kits
 - Most extraction solvents were acetonitrile/water

Data

- The assigned value was taken from the original results in 2014
- The z-scores were calculated using the new reported data and the original assigned value and Horwitz value
- Z scores > 3 or < -3 are highlighted in red

Aflatoxin B₁

Lab #	201461 Poultry Feed			201462 Swine Feed		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1	24	18.5	1.35	29.9	20	2.25
2	16.68	18.5	-0.45	16.68	20	-0.75455
3	13.3	18.5	-1.27	14.93	20	-1.15227
4	14	18.5	-1.1	12.9	20	-1.61364
5	14	18.5	-1.1	22	20	0.454545
6	14.5	18.5	-0.98	25	20	1.136364
7	11.7	18.5	1.21	9.58	20	-2.36818
8	42.9	18.5	5.98	32	20	2.727273

Aflatoxin B₁

Lab #	201463 Cattle Feed			201464 Dog Food		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1	62.2	40.2	2.488688	42.5	25.98	2.433707
2	43.76	40.2	0.809091	32.99	25.98	1.032705
3	29.72	40.2	-2.38182	27.82	25.98	0.271067
4	28.4	40.2	-2.68182	19.35	25.98	-0.97672
5	36.5	40.2	-0.84091	33	25.98	1.034178
6	47.5	40.2	1.659091	32	25.98	0.886859
7	23.27	40.2	-3.84773	18.35	25.98	-1.12404
8	44.75	40.2	1.034091	36.75	25.98	1.586623

Aflatoxin B₂

Lab #	201461 Poultry Feed			201462 Swine Feed		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1	1.5	1.6	-0.28409	2	1.5	1.515152
2	3.23	1.6	4.630682	2.66	1.5	3.515152
3	1.59	1.6	-0.02841	1.7	1.5	0.606061
4	1.23	1.6	-1.05114	0.98	1.5	-1.57576
5						
6	1.99	1.6	1.107955	2.02	1.5	1.575758
7	2.34	1.6	2.102273	1.326	1.5	-0.52727
8						

Aflatoxin B₂

Lab #	201463 Cattle Feed			201464 Dog Food		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1	2.4	1.7	1.871658	2.9	2.765	0.222039
2	1.85	1.7	0.40107	3.03	2.765	0.435855
3	1.98	1.7	0.748663	3.24	2.765	0.78125
4	1.31	1.7	-1.04278	1.72	2.765	-1.71875
5						
6	2.12	1.7	1.122995	3.68	2.765	1.504934
7	1.67	1.7	-0.08021	3	2.765	0.386513
8						

Deoxynivalenol

Lab #	201461 Poultry Feed			201462 Swine Feed		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1	945.5	1200	-1.36242	691	830	-1.01832
2	1994	1200	4.250535	811	830	-0.13919
3	860	1200	-1.82013	625.5	830	-1.49817
4	362	1200	-4.48608	293	830	-3.93407
5	3635	1200	13.03533	1571	830	5.428571
6	1669	1200	2.510707	803.5	830	-0.19414
7	904	1200	-1.58458	650	830	-1.31868
8	1740	1200	2.890792	1400	830	4.175824

Deoxynivalenol

Lab #	201463 Cattle Feed			201464 Dog Food		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1	2032	2700	-1.79618	1048	1288	-1.21029
2	3706	2700	2.705028	2020	1288	3.691377
3	2032	2700	-1.79618	858	1288	-2.16843
4	785	2700	-5.14923	512	1288	-3.91326
5	2183	2700	-1.39016			
6	2512	2700	-0.50551	1599	1288	1.568331
7	1824	2700	-2.35547	1071	1288	-1.0943
8	2630	2700	-0.18822	1680	1288	1.976803

Ochratoxin A

Lab #	201461 Poultry Feed			201462 Swine Feed		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1	59	44	1.549587	93	85.7	0.388298
2	57.3	44	1.373967	139	85.7	2.835106
3	136	44	9.504132	150	85.7	3.420213
4	25.5	44	-1.91116	50.8	85.7	-1.85638
5	104	44	6.198347	138	85.7	2.781915
6	305	44	26.96281	116	85.7	1.611702
7						
8						

Ochratoxin A

Lab #	201463 Cattle Feed			201464 Dog Food		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1	195	189	0.154639	150	125	0.912409
2	173	189	-0.41237	171	125	1.678832
3	235	189	1.185567	152	125	0.985401
4	194	189	0.128866	101	125	-0.87591
5	260	189	1.829897	343	125	7.956204
6	215	189	0.670103	196	125	2.591241
7						
8						

Fumonisin B₁

		201461 Poultry Feed			201462 Swine Feed		
		Assigned			Assigned		
Lab #		Mean	value	Z score	Mean	value	Z score
1							
2		1192	1300	-0.54027	3937	1500	10.79752
3		1786	1300	2.431216	1911	1500	1.821001
4		822	1300	-2.3912	934	1500	-2.50775
5		1470	1300	0.850425	1132	1500	-1.63048
6		2857	1300	7.788894	1688	1500	0.832964
7							
8	tota l	4400	1300	15.50775	3475	1500	8.750554

Fumonisin B₁

	201463 Cattle Feed			201464 Dog Food		
	Assigned			Assigned		
Lab #	Mean	value	Z score	Mean	value	Z score
1						
2	2236	1400	3.924883	2909	1437	6.761599
3	2047	1400	3.037559	2121	1437	3.141938
4	1044	1400	-1.67136	1315	1437	-0.5604
5	2274	1400	4.103286	1173	1437	-1.21268
6	1657	1400	1.206573	1908	1437	2.163528
7						
8	3530	1400	10	4315	200	18.90216

Fumonisin B2

Lab #	201461 Poultry Feed			201462 Swine Feed		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1						
2	296	400	-1.41689	606	400	2.80654
3	508	400	1.47139	581	400	2.46594
4	212	400	-2.56131	206	400	-2.64305
5	322	400	-1.06267	165	400	-3.20163
6	750	400	4.768392	250	400	-2.0436
7						
8						

Fumonisin B2

Lab #	201463 Cattle Feed			201464 Dog Food		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1						
2	499	270	4.353612	1105	476	7.382629
3	452	270	3.460076	709	476	2.734742
4	229	270	-0.77947	294	476	-2.13615
5	317	270	0.893536	483	476	0.08216
6	323	270	1.007605	519	476	0.504695
7						
8						

Fumonisin B₃

Lab #	201461 Poultry Feed			201462 Swine Feed		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1						
2	131	200	-1.69533	240	200	0.982801
3						
4						
5	131	200	-1.69533	222	200	0.540541
6	449	200	6.117936	224	200	0.589681
7						
8						

Fumonisin B₃

Lab #	201463 Cattle Feed			201464 Dog Food		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1						
2	243	140	3.421927	473	323	2.44898
3						
4						
5	221	140	2.69103	355	323	0.522449
6	205	140	2.159468	341	323	0.293878
7						
8						

Zearalenone

Lab #	201461 Poultry Feed			201462 Swine Feed		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1	261	242	0.395998	204	183.9	0.529505
2	233	242	-0.18758	214	183.9	0.79294
3	206	242	-0.75031	191	183.9	0.187039
4	50.4	242	-3.99333	71.4	183.9	-2.96365
5	131	242	-2.31346	210	183.9	0.687566
6	407	242	3.438933	170	183.9	-0.36617
7						
8	474	242	4.835348	271	183.9	2.294521

Zearalenone

Lab #	201463 Cattle Feed			201464 Dog Food		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1	268	251	0.34413	557	361.7	2.897626
2	343	251	1.862348	361	361.7	-0.01039
3	240	251	-0.22267	458	361.7	1.428783
4	70.5	251	-3.65385	210	361.7	-2.25074
5	135	251	-2.34818	386	361.7	0.360534
6	204	251	-0.95142	485	361.7	1.829377
7						
8	289	251	0.769231	655	361.7	4.351632

T-2

Lab #	201461 Poultry Feed			201462 Swine Feed		
	Assigned			Assigned		
	Mean	value	Z score	Mean	value	Z score
1	191	236.8	-0.97343	43	46.9	-0.37864
2	163	236.8	-1.56854	31.6	46.9	-1.48544
3	152	236.8	-1.80234	32	46.9	-1.4466
4	71	236.8	-3.52391	14.2	46.9	-3.17476
5						
6	112	236.8	-2.6525	48	46.9	0.106796
7						
8						

T-2

	201463 Cattle Feed			201464 Dog Food		
	Assigned			Assigned		
Lab #	Mean	value	Z score	Mean	value	Z score
1	206	244.6	-0.79917	207	254.5	-0.95
2	178	244.6	-1.37888	159	254.5	-1.91
3	175	244.6	-1.44099	145	254.5	-2.19
4	75.4	244.6	-3.50311	69.4	254.5	-3.702
5						
6	205	244.6	-0.81988	228	254.5	-0.53
7						
8						

Reproducibility and Repeatability by Analyte

Mycotoxin Round Robin 2016, Summary Results - R. Sheridan Collaborator

		Pooled STD					(within Labs)
	Grand Mean	of Lab Means	Number of Labs Used	Reproducibility	Repeatability	Reproducibility %RSD	Repeatability %rsd
Aflatoxin B1	27.4	10.3	8	10.2	3.51	37.14%	12.81%
Aflatoxin B2	2.08	0.57	6	0.63	0.41	30.32%	19.79%
Aflatoxin G1†							
Aflatoxin G2†							
Deoxynivalenol	1,359	620	7	642	241	47.24%	17.70%
Ochratoxin A	156	81.6	6	76.4	44.0	48.85%	28.11%
Fumonisin B1	2,172	1,182	6	1,128	424	51.95%	19.52%
Fumonisin B2	441	223	5	258	183	58.42%	41.39%
Fumonisin B3	270	99	3*	114	79	42.34%	29.40%
Zearalenone	282	124	7	120	53.6	42.76%	19.02%
T-2	125	47.6	5	49.2	17.7	39.31%	14.16%

Following principles outlined in: Statistical Manual of the AOAC, W.J. Youden and E.H. Steiner, Pages 78-80

A similar approach is offered in ISO 5725-2, "Accuracy (trueness and precision) of measurement methods and results"

* Only 3 labs with useable data.

† Data mostly ND's and 0's.

Individual Lab repeatability

Mycotoxin Round Robin 2016, Summary Results - R. Sheridan Collaborator

	Repeatability Calculated Within Individual Labs (%rsd)							
	Lab1	Lab2	Lab3	Lab4	Lab5	Lab6	Lab7	Lab8
Aflatoxin B1	1.71%	8.73%	14.26%	11.91%	19.53%	22.92%	7.20%	4.06%
Aflatoxin B2	2.66%	13.69%	13.56%	9.63%		35.40%	5.81%	
Aflatoxin G1 [†]								
Aflatoxin G2 [†]								
Deoxynivalenol	5.99%	13.38%	3.24%	10.88%	16.72%	9.86%	9.86%	15.94%
Ochratoxin A	1.98%	28.87%	11.62%	19.30%	29.85%	35.75%		
Fumonisin B1		5.33%	19.09%	5.81%	22.06%	7.76%		7.76%
Fumonisin B2		31.20%	5.08%	30.68%	64.11%	20.49%		
Fumonisin B3		40.70%			18.89%	25.23%		
Zearalenone	0.51%	18.42%	4.68%	13.28%	18.13%	28.47%		5.79%
T-2	1.76%	6.16%	6.19%	13.80%		19.30%		
Global %rsd by Lab	2.98%	21.89%	11.09%	16.07%	31.25%	24.62%	7.81%	9.54%

Significant f table

Repeatability (%rsd) Significant f Table								
	Lab1	Lab7	Lab8	Lab3	Lab4	Lab2	Lab6	Lab5
Lab1		6.9	10.3	13.9	29.1	54.0	68.3	110.1
Lab7			1.5	2.0	4.2	7.9	9.9	16.0
Lab8				1.4	2.8	5.3	6.7	10.7
Lab3					2.1	3.9	4.9	7.9
Lab4						1.9	2.3	3.8
Lab2							1.3	2.0
Lab6								1.6



Although some Global % rsd and individual lab repeatabilities were high, this is probably due to the fact that not all labs generated results for all analytes and some results were near the limit of quantification for certain analytes.