

Table 1: Results and z-Scores for Turkey Muscle Test Material

laboratory number	analyte			
	doxycycline			
	assigned value 131 µg/kg			
	result µg/kg	Int. Std used? or % recovery	LoQ µg/kg	z-score
001	122	42	100	-0.3
002	63.7	Y M	20	<b>-2.4</b>
003	110	84.5		-0.7
004	143	75.0		0.4
005	160	50	20	1.0
006	104	70	50	-0.9
007	158.40	54	117 •	1.0
008	129	87	15	-0.1
009	94	Y M	10	<b>-1.3</b>
010	143	Y M	0.7	0.4
011	254	M	20	<b>4.3</b>
012	141.1	Y M	5	0.4
013	170	Y	50	1.4
014	142	Y	25	0.4
015	66	97	20	<b>-2.3</b>
016	100	57	200	-1.1
017	68	Y	10	<b>-2.2</b>
018	105	65	70	-0.9
019	122.6	Y M	10	-0.3
020	116.71	46	50	-0.5
021	107	Y	100	-0.8
022	191	Y	25	<b>2.1</b>
023	† 145.58	N	100	0.5
024	59.33	Y M	24	<b>-2.5</b>
025	#			
026	#			
027	91.4	N S	40	-1.4

z-scores outside the satisfactory range, i.e.  $|z| > 2$ , are shown in **bold**

M = matrix-based calibration curve used

S = Standard addition used

† = additional residues reported > 50 µg/kg see -Table 2

• = CC $\alpha$

# = not analysed

Table 1 (continued): Results and z-Scores for Turkey Muscle Test Material

laboratory number	analyte			
	doxycycline assigned value 131 µg/kg			
	result µg/kg	Int. Std used? or % recovery	LoQ µg/kg	z-score
028	147	66	50	0.6
029	190	72	40	<b>2.1</b>
030	111.37	99.35 M	40	-0.7
031	#			
032	128.00	Y	20.00	-0.1
033	#			
034	143	N M	50	0.4
035	87	N 61	20	-1.5
036	143.1	Y	10	0.4
037	73.14	N	50	-2.0
038	#			
039	103	Y	10	-1.0
040	#			
041	#			
042	185	N M	50	1.9
043	123.6	N M	31	-0.3
044	182.05	N, 105.2	20	1.8
045	160	Y	20	1.0
046	120	N; 68	5	-0.4
047	180	91	5	1.7
048	112	74.72	100	-0.7
049	† #			
050	149.4	N M	50	0.7
051	66.1	Y	5	<b>-2.3</b>
052	190.50	N ▲	0.10	<b>2.1</b>
053	† #			
054	#			

z-scores outside the satisfactory range, i.e.  $|z| > 2$ , are shown in **bold**

M = matrix-based calibration curve used

# = not analysed

† = additional residues reported > 50 µg/kg see -Table 2

▲ = result not corrected for recovery (% recovery is 87.1%)

Table 1 (continued): Results and z-Scores for Turkey Muscle Test Material

laboratory number	analyte			
	doxycycline			
	assigned value 131 µg/kg			
	result µg/kg	Int. Std used? or % recovery	LoQ µg/kg	z-score
055	164	79	50	1.2
056	134.5	R	10	0.1
057	† #			
058	#			
059	133.50	86.10 M	50	0.1
060	146	70	30	0.5
061	129.0	64.6	19	-0.1
062	130.9	95	20	0.0
063	107.0	N	1.0	-0.8

R = result corrected for recovery

† = additional residues reported > 50 µg/kg see -Table 2

# = not analysed

M = matrix-based calibration curve used

Table 2: Additional Tetracyclines Reported

laboratory number	analyte reported >50 µg/kg	result µg/kg	int. std. added or % recovery	LoQ µg/kg
023	chlortetracycline	136.55	N	100
049	chlortetracycline	3045.8	80.0	200.0
049	oxytetracycline	2722.5	82.0	100.0
049	tetracycline	1030.0	82.0	100.0
053	chlortetracycline	70.38	80	
053	oxytetracycline	77.05	75	
053	tetracycline	83.71	83	
057	chlortetracycline	446	103	21.9
057	tetracycline	62	55	15.4

Table 3: Assigned Values and Target Standard Deviations

analyte	assigned value				target standard deviation	
	data points <i>n</i>	robust mean $\hat{X}$ , µg/kg	robust sd $\hat{\sigma}$	uncertainty <i>u</i>	derived from	$\sigma_p$ , µg/kg
doxycycline	46	131	37.1	5.47	Horwitz*	28.41

\* = see page 7 & 8 for appropriate form of the Horwitz equation

Table 4: Number and Percentage of Satisfactory z-Scores

analyte	number of satisfactory scores $ z  \leq 2$	total number of scores	satisfactory %
doxycycline	42	51	82

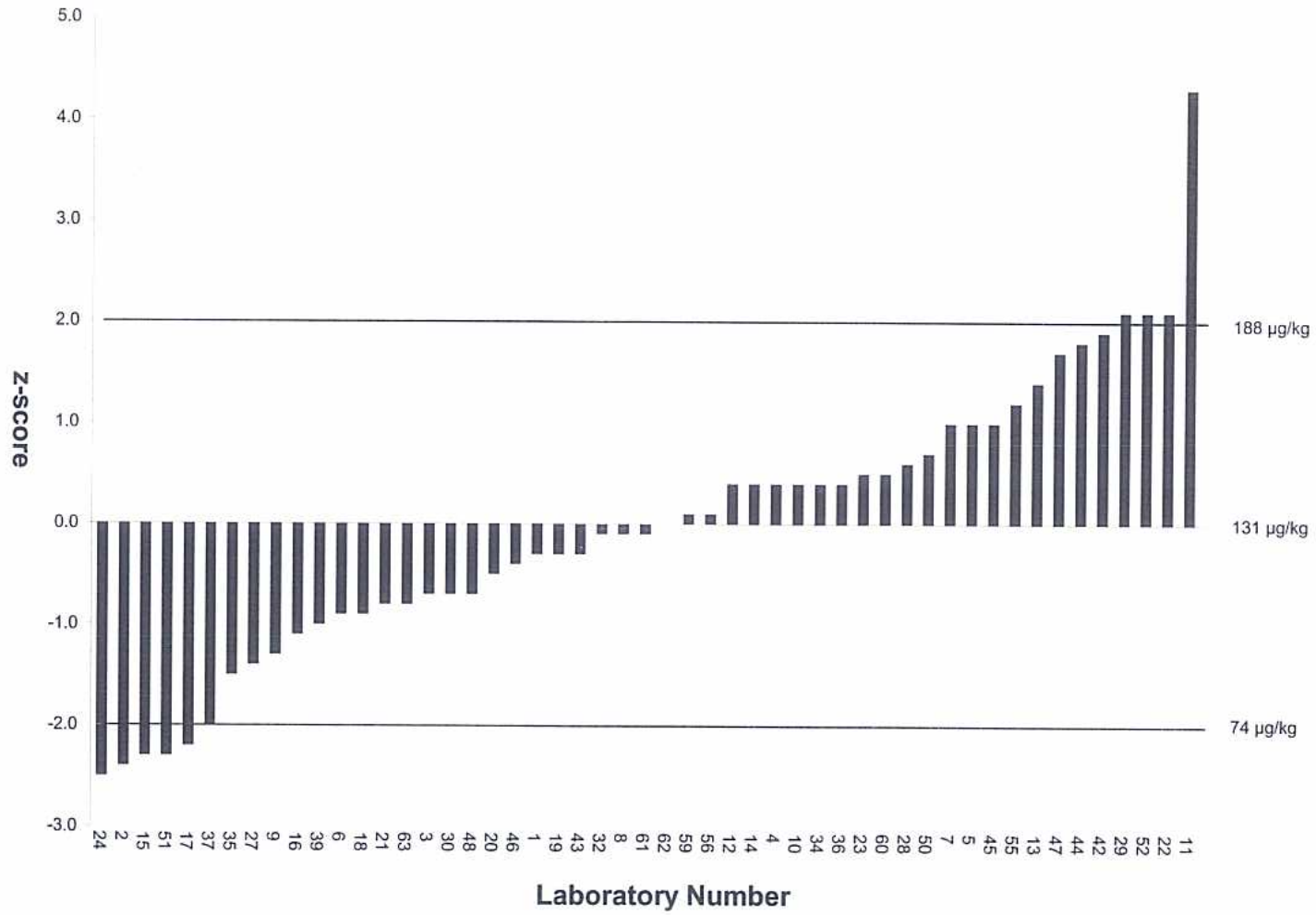


Figure 1: z-Scores for Doxycycline (131 µg/kg) in Turkey Muscle Test Material

**APPENDIX I: Homogeneity Data for Turkey Muscle Test Material**

sample identity	analyte	
	doxycycline	
	µg/kg	
	replicate 1	replicate 2
1	159.4	181.5
2	165.8	164.0
3	147.9	155.1
4	151.8	144.2
5	148.5	184.1
6	184.6	154.3
7	165.0	126.1
8	154.3	165.5
9	178.1	165.4
10	151.6	148.3
mean	159.8	
<i>n</i>	20	
origin of target sd ( $\sigma_p$ )	Horwitz*	
$\sigma_p$ as RSD%	21.08	
abs. target sd ( $\sigma_p$ )	33.68	
$s_{an}$	15.16	
$s_{sam}^2$	0.00	
$\sigma_{all}^2$	102.11	
critical	424.14	
$s_{sam}^2 < \text{critical?}$	<b>ACCEPT</b>	

**APPENDIX II: Stability Data for Turkey Muscle Test Material**

sample identity	analyte	
	Set A <i>control</i>	Set B <i>experimental</i>
	doxycycline µg/kg	
1	174	150
2	147	167
3	149	165
4	153	123
5	129	158
<i>n, n</i>	5	5
means	150	153
difference	-2	

t-Test: Two-Sample Assuming Equal Variances

	Variable 1	Variable 2
Mean	150.4	152.6
Variance	258.8	318.3
Observations	5	5
Pooled Variance	288.55	
Hypothesized Mean Difference	0	
df	8	
t Stat	-0.20	
P(T<=t) two-tail	0.84	
t Critical two-tail	2.31	

Is there a statistical difference between the two data sets?

P(T<=t) two-tail > t Critical two-tail ? No

Test material is Stable

Does this difference affect participants' z-scores?

difference > 0.1  $\sigma_p$ ? not applicable