

Table 1: Results and z-Scores for Honey Test Material

laboratory number	analyte							
	ciprofloxacin assigned value 19.6 µg/kg				norfloxacin assigned value 19.7 µg/kg			
	result µg/kg	int. std. added or % recovery	LoQ µg/kg	z-score	result µg/kg	int. std. added or % recovery	LoQ µg/kg	z-score
001	22.4		2	0.6	22.6		2	0.7
002	16.8	m	5	-0.7	19.3	m	5	-0.1
003	<LOQ		30		0		5	<b>-4.5</b>
004	19.8	y m	2.2	0.0	18.0	y m	2.2	-0.4
005	22.8	y	5	0.7	24.4	y	5	1.1
006	15.8	y s	0.55	-0.9	19.7	y s	0.60	0.0
007	22	96.8	5	0.5	#			
008	17.4	y	1.0	-0.5	28.5	y	1.0	2.0
009	19.2	84.2	10	-0.1	18.7	87.1	10	-0.2
010	■	124	<10		■	100	<10	
011	17	90	10	-0.6	18	80	10	-0.4
012	17.1	m	9.8	-0.6	16.5	m	6.1	-0.7
013	21.9	81.8	10	0.5	22.5	84.8	10	0.6
014	17.5	y m		-0.5	17.4	y m		-0.5
015	13.6	n		-1.4	#			
016	38		20	<b>4.2</b>	31		20	<b>2.6</b>
017	25.5	30	2	1.4	18.8	40	2	-0.2
018	20	n	5	0.1	#			
019	19.8	m		0.0	21.7	m		0.5
020	28.5	y 105 m	5	2.0	22.2	99 m	5	0.6
021	90	y	5	<b>16.3</b>	#			
022	▲				▲			
023	9.9	y	2	<b>-2.3</b>	20.4	y	2	0.2
024	59	164	20	<b>9.1</b>	<LOQ		20	
025	13.05	y m	3	-1.5	18.60	y m	10	-0.3
026	20	y m	10	0.1	20	y m	10	0.1
027	18.5	y	10	-0.3	15.6	y	10	-0.9
028	12.0		10	-1.8	15.0		10	-1.1
029	14.5	63 m	10.0	-1.2	#			

Some results have been edited for consistency

# = not analysed

s = standard addition used

▲ = 29 µg/kg total (fluoro-)quinolones calculated as norfloxacin

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

Int. Std. = internal standard

m = matrix-based calibration used

■ = total fluoroquinolones 50 µg/kg

Table 2: Additional Quinolones and Fluoroquinolones Reported

laboratory number	residue reported $\geq 4 \mu\text{g}/\text{kg}$	result $\mu\text{g}/\text{kg}$	Int. Std. used or % recovery	LoQ $\mu\text{g}/\text{kg}$
003	marbofloxacin	1000	40	30
008	sarafloxacin	75.6	y	1.0
024	oxolinic acid	30	91	20

Table 3: Assigned Values and Target Standard Deviations

analyte	assigned value, $\mu\text{g}/\text{kg}$				target standard deviation, $\mu\text{g}/\text{kg}$	
	data points $n$	robust mean $\hat{X}$	robust sd $\hat{\sigma}$	uncertainty $u$	derived from	$\sigma_p$
ciprofloxacin	21	19.6	5.1	1.12	Horwitz*	4.32
norfloxacin	17	19.7	2.8	0.67	Horwitz*	4.34

\* = see page 7 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 %
ciprofloxacin	22	26	85
norfloxacin	19	21	90

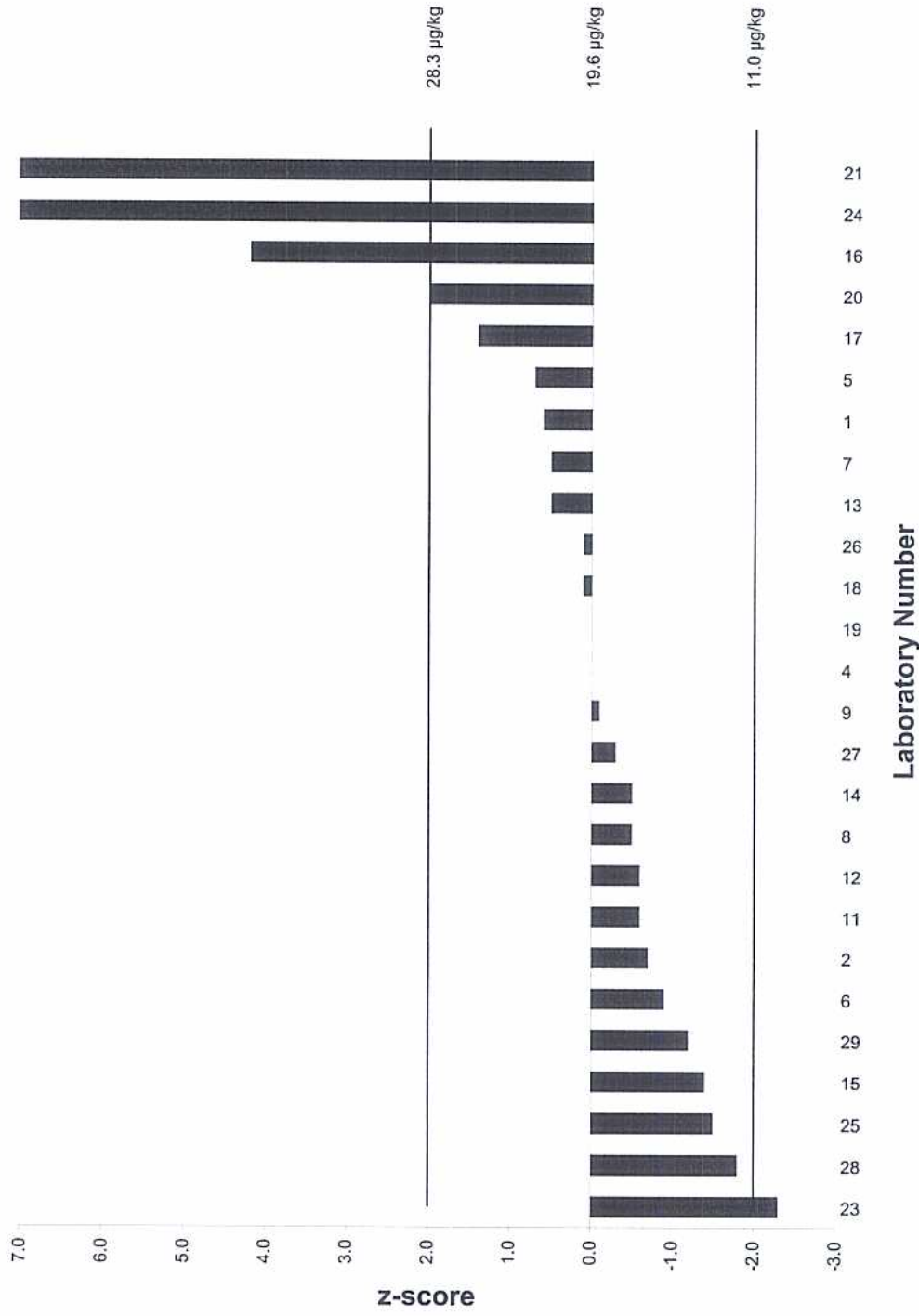


Figure 1: z-Scores for Ciprofloxacin (19.6 µg/kg) in Honey Test Material

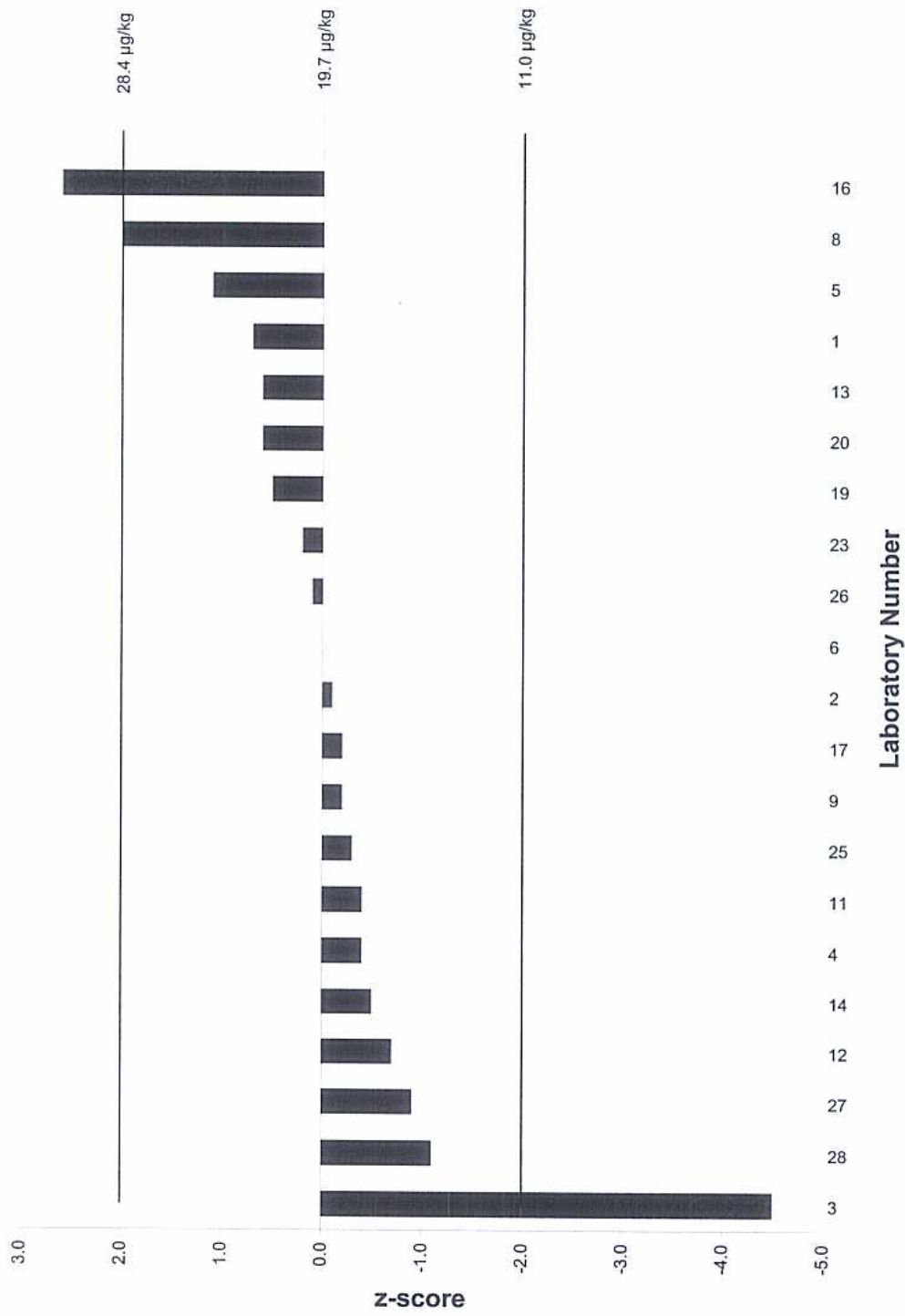


Figure 2: z-Scores for Norfloxacin (19.7 µg/kg) in Honey Test Material

**APPENDIX I: Homogeneity Data for Honey Test Material**

sample identity	analyte			
	ciprofloxacin, µg/kg		norfloxacin, µg/kg	
	replicate 1	replicate 2	replicate 1	replicate 2
1	18.57	16.52	18.89	26.52
2	17.10	22.47	15.78	16.65
3	21.24	17.52	18.22	18.34
4	18.00	21.43	20.26	17.53
5	19.50	17.68	21.85	20.29
6	19.49	22.95	21.70	20.12
7	23.44	25.40	21.16	24.76
8	20.80	20.11	21.90	22.42
9	19.08	21.52	22.86	17.13
10	18.52	17.80	21.22	22.93
mean	19.96		20.53	
<i>n</i>	20		20	
origin of target sd ( $\sigma_p$ )	Horwitz*		Horwitz*	
target sd ( $\sigma_p$ ) as RSD <sub>R</sub> %	22.00		22.00	
abs. target sd ( $\sigma_p$ )	4.391		4.516	
$s_{an}$	2.058		2.453	
$s_{sam}^2$	1.573		1.742	
$\sigma_{all}^2$	1.735		1.835	
<i>critical</i>	7.540		9.529	
$s_{sam}^2 < \text{critical?}$	<b>ACCEPT</b>		<b>ACCEPT</b>	

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