

## OXYTETRACYCLINE

First draft prepared by  
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### ADDENDUM

to the Oxytetracycline residue monograph prepared by  
the 45th meeting of the Committee and published in  
FAO Food and Nutrition Paper 41/8, Rome 1996

The Committee, at its 45th meeting, stated that a validated analytical method for the determination of oxytetracycline residues in prawn tissue was required for evaluation at the 47th meeting of the Committee before a permanent MRL could be assigned to oxytetracycline in prawn.

A method for the quantification of oxytetracycline in giant prawn was submitted for evaluation at the present meeting by the Department of Medical Sciences, Ministry of Public Health, Thailand. The method has a limit of detection (LOD) of 10 µg/kg and a limit of quantification (LOQ) of 50 µg/kg with a coefficient of variation of 21 % at LOQ. The supporting validation data is summarised below.

The oxytetracycline method is based on the Oka method for tetracyclines which is accepted as the benchmark method for this purpose (Oka et al, 1985).

The method for prawn, which is required to monitor a proposed MRL of 100 µg/kg, has been tested in two laboratories utilising slightly different validation regimes. The first laboratory used fortification levels 50, 100 and 200 µg/kg whereas the second laboratory chose fortification levels of 100, 200 and 400 µg/kg. The first laboratory used two separate analysts to produce data on six samples each analysed twice, at the 50 µg/kg fortification level. The results are shown in Table 1.

Table 1. Analytical Data for Recovery of Oxytetracycline from Prawn Meat at 50% of the MRL (50 µg/kg)

	% Recovery of Oxytetracycline	
	Analyst 1	Analyst 2
Run 1	52, 55, 67, 60, 77, 47	91, 95, 84, 84, 87, 96
Run 2	96, 119, 110, 80, 84, 82	89, 96, 90, 98, 89, 88
Mean	78	91
SD	23	4.7
%CV	29	5.2

This data gives a mean recovery of 84 % with a %CV of 21 % at the LOQ which is 50 % of the MRL.

In the second laboratory one analyst conducted six recoveries at fortification levels of 100, 200 and 400 µg/kg, each being analysed twice. Results are summarised in Table 2. It should be noted that data for the 50 µg/kg level presented for Laboratory 1 is the same data obtained by two separate analysts presented in Table 1.

**Table 2. Analytical Data for Mean Recovery of Oxytetracycline from Prawn Meat at Different Fortification Levels at or near the MRL by Two Different Laboratories**

Run	Fortification Level ( $\mu\text{g/kg}$ )	Laboratory 1		Laboratory 2	
		% Mean Recovery (n=6)	% CV	% Mean Recovery (n=6)	% CV
1	50	60	18	NM	NM
	100	89	9.2	97	5.8
	200	86	10.7	88	4.8
	400	NM	NM	92	12.1
2	50	95	17.1	NM	NM
	100	93	5.4	82	14.3
	200	85	4.6	78	18.1
	400	NM	NM	88	11.4
3	50	89	5.8	NM	NM
	100	92	2.7	NM	NM
	200	91	9.9	NM	NM
	400	NM	NM	NM	NM
4	50	91	5.6	NM	NM
	100	89	1.6	NM	NM
	200	85	2.5	NM	NM
	400	NM	NM	NM	NM

NM = Not Measured

### APPRAISAL

A method for the quantitative determination of oxytetracycline in giant prawn was submitted for consideration. The analytical method submitted is Method 995.09 AOAC International, for analysis of residues of chlortetracycline, oxytetracycline and tetracycline in bovine and porcine muscle and kidney. This method was evaluated for use in prawn muscle.

The performance testing results for oxytetracycline in prawn muscle included data from two laboratories and three analysts. The results indicate that the recovery of oxytetracycline from giant prawn muscle at the temporary MRL established at the forty-fifth meeting of the Committee (100  $\mu\text{g/kg}$ ) was 82-97 %. Recovery at 50  $\mu\text{g/kg}$  was 60-91 %. The coefficient of variability for analyst repeatability was acceptable.

These values are similar to recovery reports of other studies in different tissues.

Considering that the requested analytical method has been provided and that it is acceptable, the Committee recommended an MRL of 100  $\mu\text{g/kg}$  for oxytetracycline residues in giant prawn muscle.

### REFERENCE

Department of Medical Sciences, Ministry of Public Health, (1996). Validation of the analytical method of oxytetracycline in giant prawn, Thailand, May 1996.