

## Seafood/fish samples results report:

The Pesticide Section of Laboratory Services received seven samples between 8/20/2007 and 8/30/2007. The samples were logged in according to protocol. They were assigned the following identifying numbers:

08-Pr-315 for ASP-001  
08-Pr-316 for ASP-002  
08-Pr-317 for ASP-003  
08-Pr-318 for ASP-004  
08-Pr-319 for ASP-005  
08-Pr-366 for ASP-006  
08-Pr-395 for ASP-007

The samples were from various sources of seafood/fish as seen here:

Sample #	Type
315	Catfish
316	Basa
317	Tilapia
318	Tilapia
319	Tilapia
366	Tilapia
395	Shrimp

The requested analysis for these samples was Ciprofloxacin, Enrofloxacin, Chloramphenicol, Malachite Green, & Crystal Violet. Because the Lab had no methods for these compounds, we had to develop and validate both the extraction & HPLC/3D ion trap methods for each compound. During development, we determined that our quantitation limit (LOQ) was 20ppb for all of the compounds on the 3D ion trap.

We then analyzed the samples and found that none of the samples had residues for any of the requested analyses that were above our 20ppb limit of quantitation. We did a second analysis on the samples, confirmed the same results, and reported the results on 9/28/2007.

We were asked to confirm our results, so we sent aliquots of the samples to Mississippi State Chemical Lab (MSCL) on 10/15/07. MSCL agreed to test our samples free of charge for all of the components except Chloramphenicol. We received the results back on 12/4/2007. The attached Table 1 shows our results compared to the MSCL results. It shows that no residues were found by MSCL above 20ppb. This confirms our results. Now, MSCL did find some residues below this limit, but we could not see below 20ppb, because 20ppb is the lowest obtainable level we can achieve with our current instrumentation.

We researched and found that according to Federal code 21 CFR (556 & others) that these compounds have a zero tolerance in seafood or farm raised fish products. According to the FDA, the seafood is considered adulterated if any of the compounds are present. The FDA says that a practical level for the "action level" is "what you can see and confirm". This level can vary from lab to lab and state to state. I have attached some action limits that I have obtained from the FDA and other states in Table 2. Most other agencies have other equipment that allows them to get lower limits. If we wish to get to the same level as the other agencies, we need to purchase an HPLC/MSMS Triple quadrupole, otherwise we will not be able to go below our current level of 20ppb.

**Table 1:**

# Results Comparison on Fish Samples

## ODAFF vs MSCL (Mississippi State Chemical Lab)

### Limit of Quantitation

	ODAFF	MSCL
Malachite Green	20 ppb	0.5 ppb
Crystal Violet	20 ppb	0.5 ppb
Ciprofloxacin	20 ppb	1 ppb
Enrofloxacin	20 ppb	1 ppb

### 08-Pr-315

	ODAFF	MSCL
Malachite Green	ND greater than 20ppb	ND greater than 0.5ppb
Crystal Violet	ND greater than 20ppb	ND greater than 0.5ppb
Ciprofloxacin	ND greater than 20ppb	13.9ppb
Enrofloxacin	ND greater than 20ppb	6.31ppb

### 08-Pr-316

	ODAFF	MSCL
Malachite Green	ND greater than 20ppb	ND greater than 0.5ppb
Crystal Violet	ND greater than 20ppb	ND greater than 0.5ppb
Ciprofloxacin	ND greater than 20ppb	1.59ppb
Enrofloxacin	ND greater than 20ppb	3.09ppb

### 08-Pr-317

	ODAFF	MSCL
Malachite Green	ND greater than 20ppb	ND greater than 0.5ppb
Crystal Violet	ND greater than 20ppb	ND greater than 0.5ppb
Ciprofloxacin	ND greater than 20ppb	ND greater than 1.0ppb
Enrofloxacin	ND greater than 20ppb	ND greater than 1.0ppb

### 08-Pr-318

	ODAFF	MSCL
Malachite Green	ND greater than 20ppb	ND greater than 0.5ppb
Crystal Violet	ND greater than 20ppb	ND greater than 0.5ppb
Ciprofloxacin	ND greater than 20ppb	ND greater than 1.0ppb
Enrofloxacin	ND greater than 20ppb	1.37ppb

### 08-Pr-319

	ODAFF	MSCL
Malachite Green	ND greater than 20ppb	ND greater than 0.5ppb
Crystal Violet	ND greater than 20ppb	ND greater than 0.5ppb
Ciprofloxacin	ND greater than 20ppb	ND greater than 1.0ppb
Enrofloxacin	ND greater than 20ppb	ND greater than 1.0ppb

### 08-Pr-366

	ODAFF	MSCL
Malachite Green	ND greater than 20ppb	ND greater than 0.5ppb
Crystal Violet	ND greater than 20ppb	ND greater than 0.5ppb
Ciprofloxacin	ND greater than 20ppb	ND greater than 1.0ppb
Enrofloxacin	ND greater than 20ppb	ND greater than 1.0ppb

### 08-Pr-395

	ODAFF	MSCL
Malachite Green	ND greater than 20ppb	ND greater than 0.5ppb
Crystal Violet	ND greater than 20ppb	ND greater than 0.5ppb
Ciprofloxacin	ND greater than 20ppb	ND greater than 1.0ppb
Enrofloxacin	ND greater than 20ppb	ND greater than 1.0ppb

ND = None Detected

**Table 2:**

## Action Levels from Various States

Action levels are based on LOQ

### **FDA Arkansas (Contact: Andrew Fong)**

Ciprofloxacin	5ppb
Enrofloxacin	5ppb
Chloramphenicol	0.3ppb
Malachite Green	1ppb
Crystal Violet	1ppb

### **Tennessee (Contact: Douglas Hite)**

Ciprofloxacin	5ppb
Enrofloxacin	5ppb
Chloramphenicol	5ppb
Malachite Green	5ppb
Crystal Violet	5ppb

### **Alabama (Contact: Joe Basile)**

Ciprofloxacin	1ppb
Enrofloxacin	1ppb
Chloramphenicol	0.3ppb
Malachite Green	1ppb
Crystal Violet	1ppb

### **Mississippi (Contact: Gale Hagood)**

Ciprofloxacin	1ppb
Enrofloxacin	1ppb
Chloramphenicol	5ppb
Malachite Green	0.5ppb
Crystal Violet	0.5ppb

### **ODAFF (Current LOQ)**

Ciprofloxacin	20ppb
Enrofloxacin	20ppb
Chloramphenicol	20ppb
Malachite Green	20ppb
Crystal Violet	20ppb