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APPENDICES

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APPENDIX A: Voltammetric Study

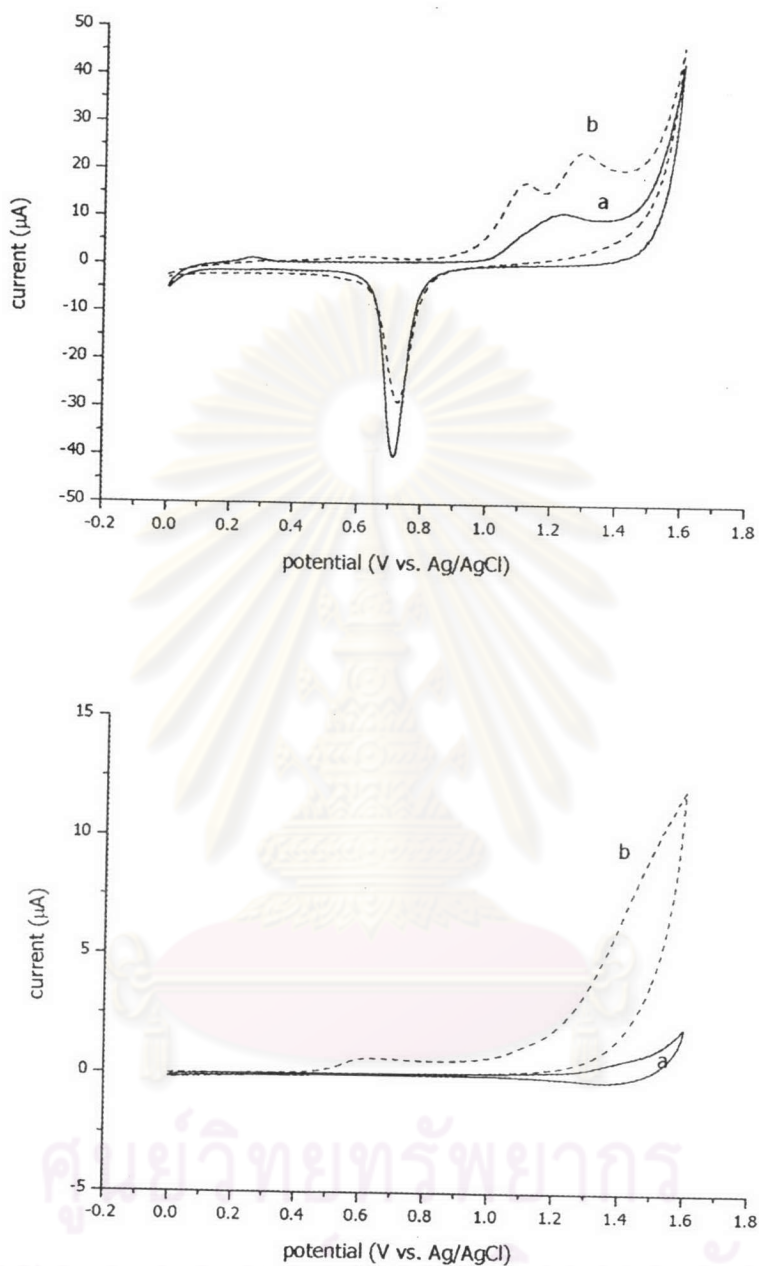


Figure A-1 Cyclic voltammograms for 1 mM oxytetracycline (line b) in 0.01 M phosphate buffer (pH 2.5) – acetonitrile (80:20, v/v) at (A) Au electrode and (B) anodized BDD electrode. The sweep rate was 100 mV/s. Background voltammograms are also shown in the figure (line a).

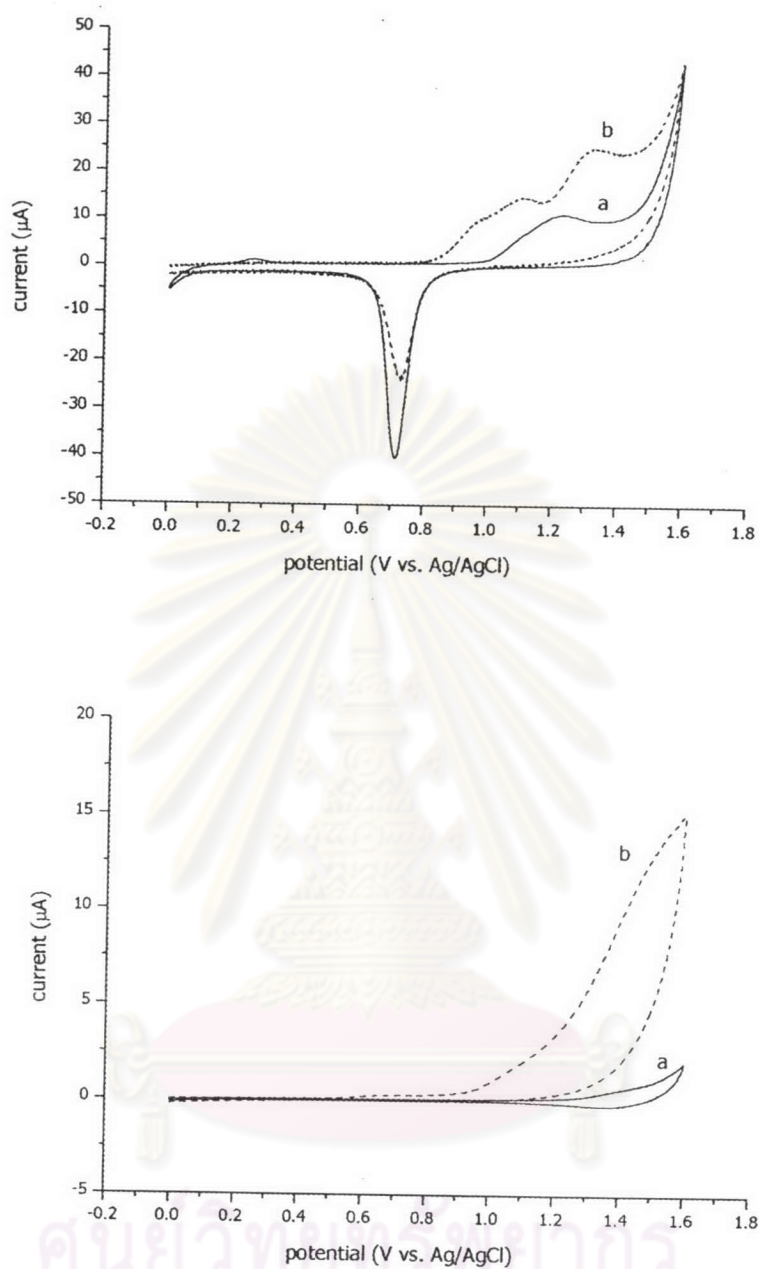


Figure A-2 Cyclic voltammograms for 1 mM chlortetracycline (line b) in 0.01 M phosphate buffer (pH 2.5) – acetonitrile (80:20, v/v) at (A) Au electrode and (B) anodized BDD electrode. The sweep rate was 100 mV/s. Background voltammograms are also shown in the figure (line a).

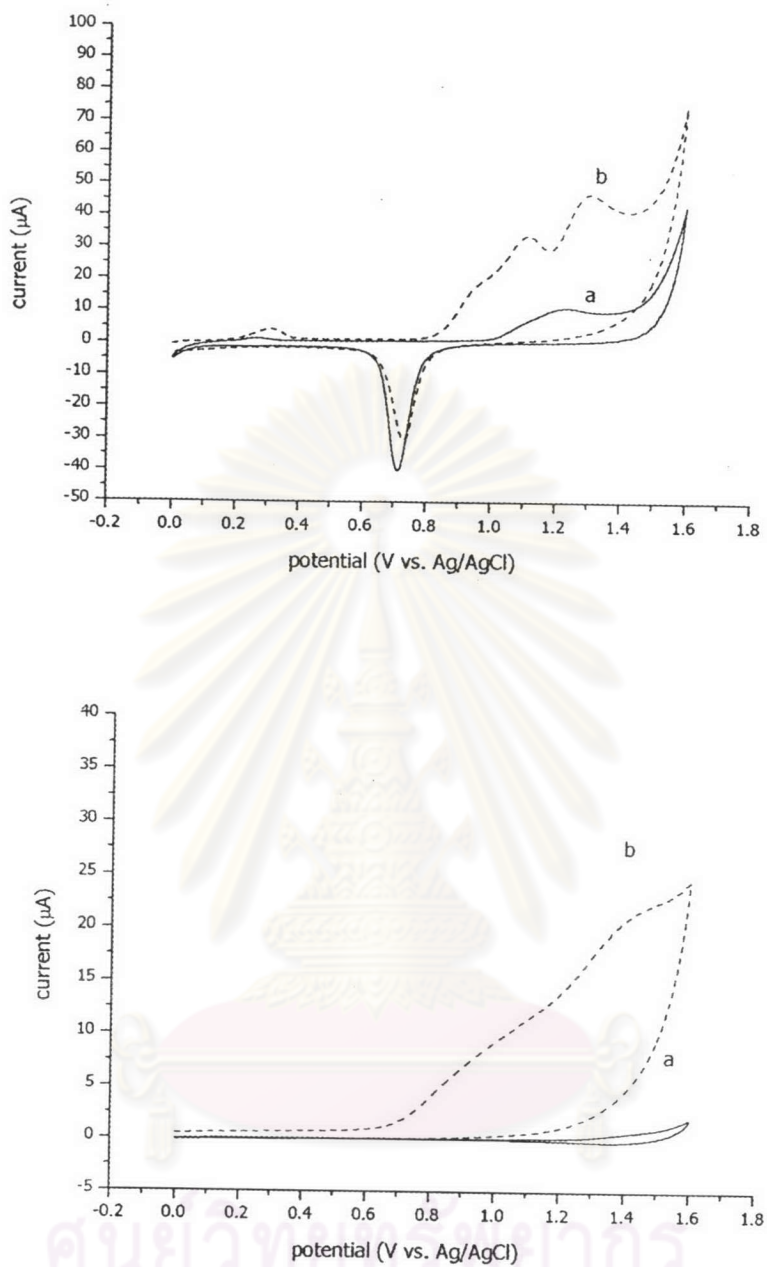


Figure A-3 Cyclic voltammograms for 1 mM doxycycline (line b) in 0.01 M phosphate buffer (pH 2.5) – acetonitrile (80:20, v/v) at (A) Au electrode and (B) anodized BDD electrode. The sweep rate was 100 mV/s. Background voltammograms are also shown in the figure (line a).

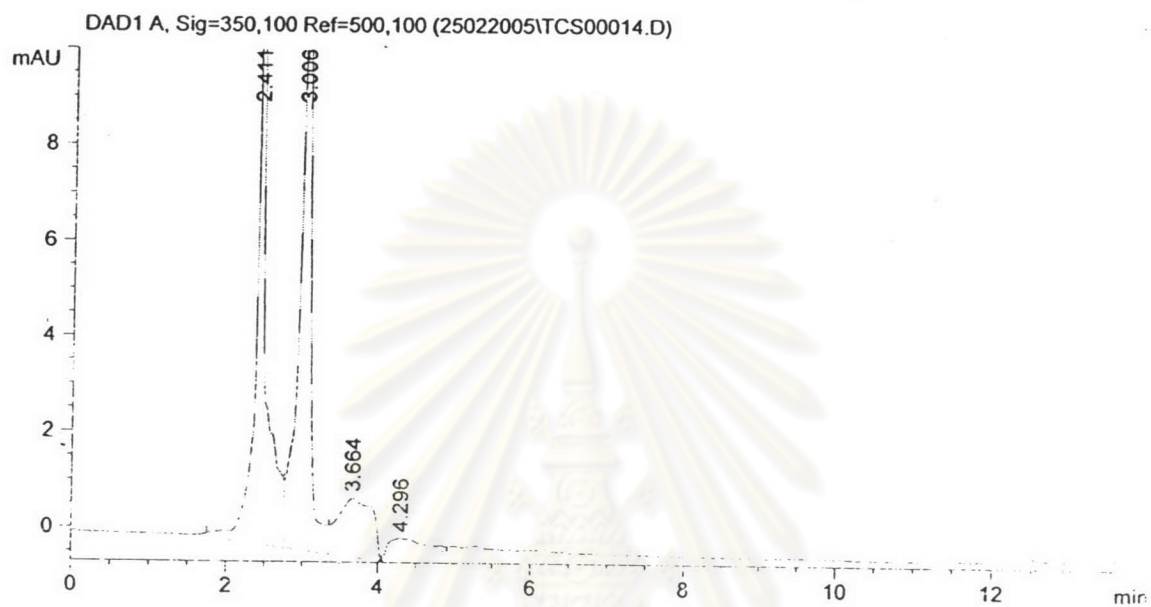
APPENDIX B: Results of AOAC Official Method

Figure B-1 HPLC chromatogram of blank sample (farming shrimp) using AOAC official method.

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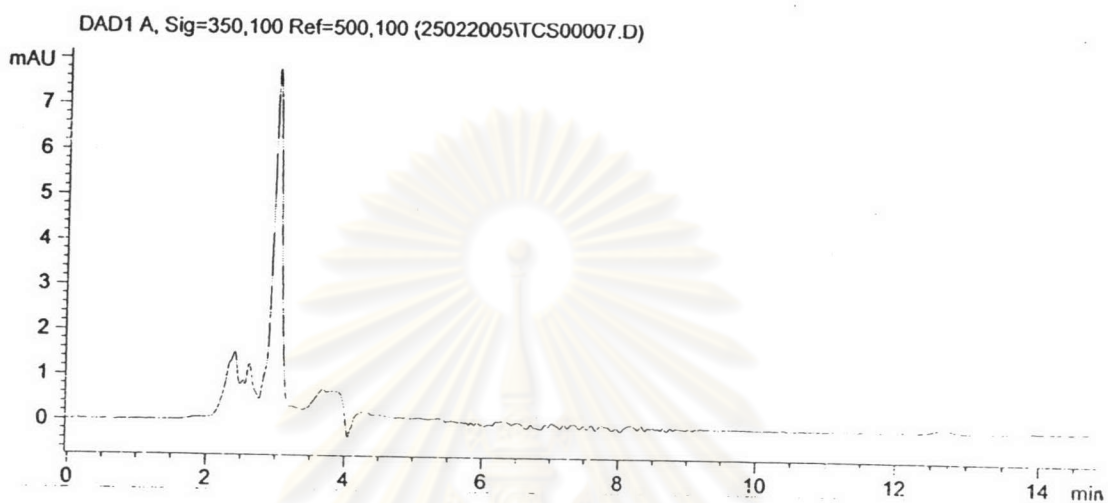


Figure B-2 HPLC chromatogram of blank sample (sea shrimp) using AOAC official method.

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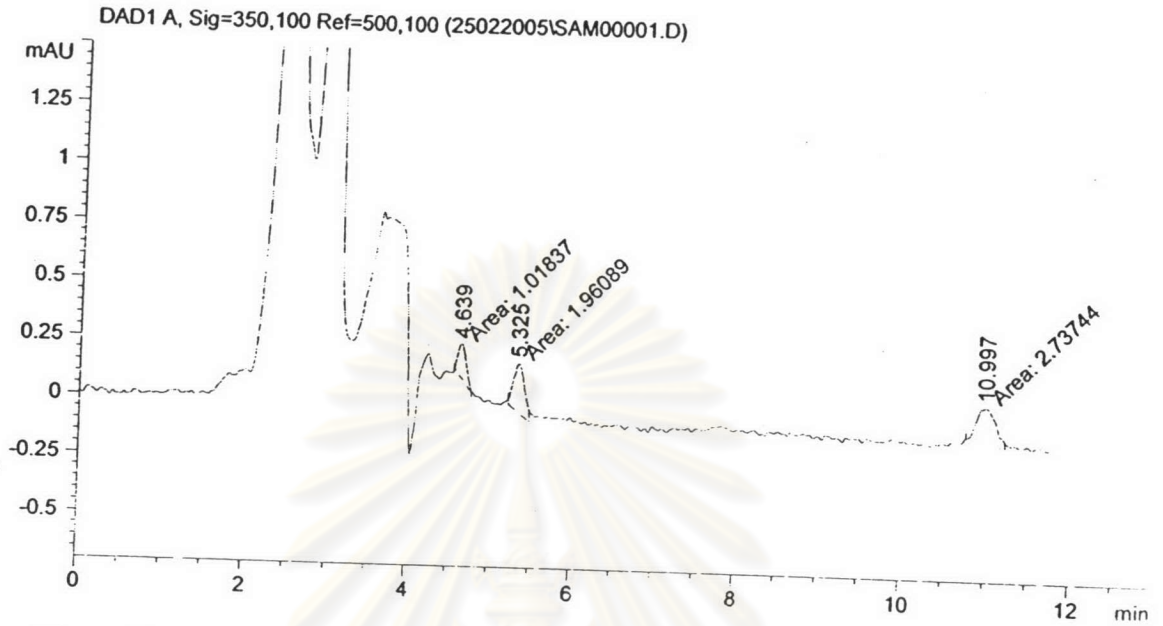


Figure B-3 HPLC chromatogram of sample spiked with mixed standard 0.5 mg/kg using AOAC official method.

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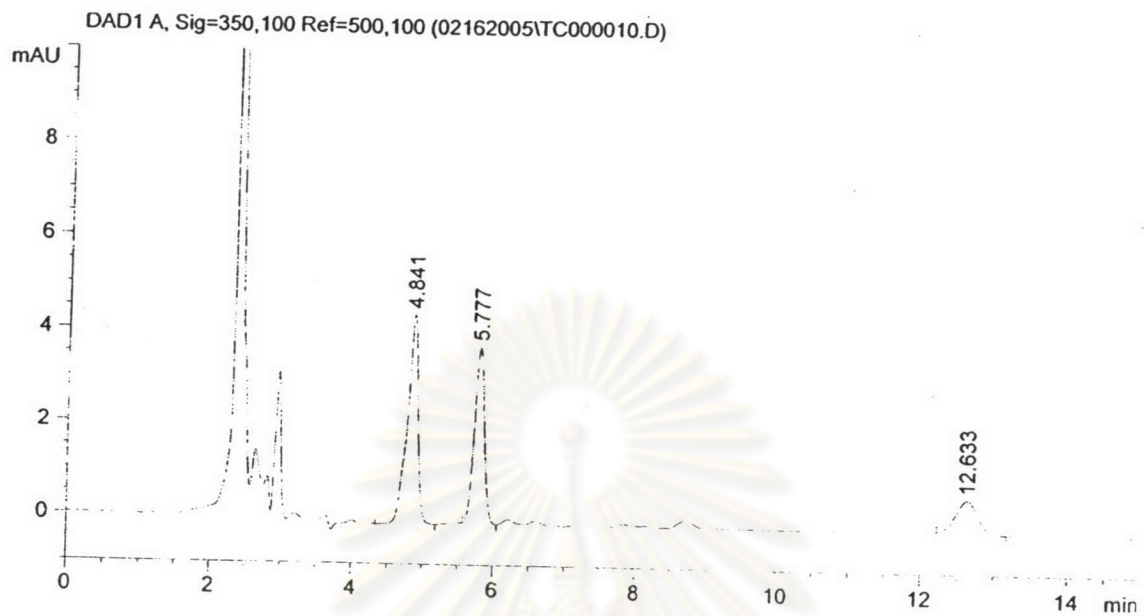


Figure B-4 HPLC chromatogram of sample spiked with mixed standard 1.0 mg/kg using AOAC official method.

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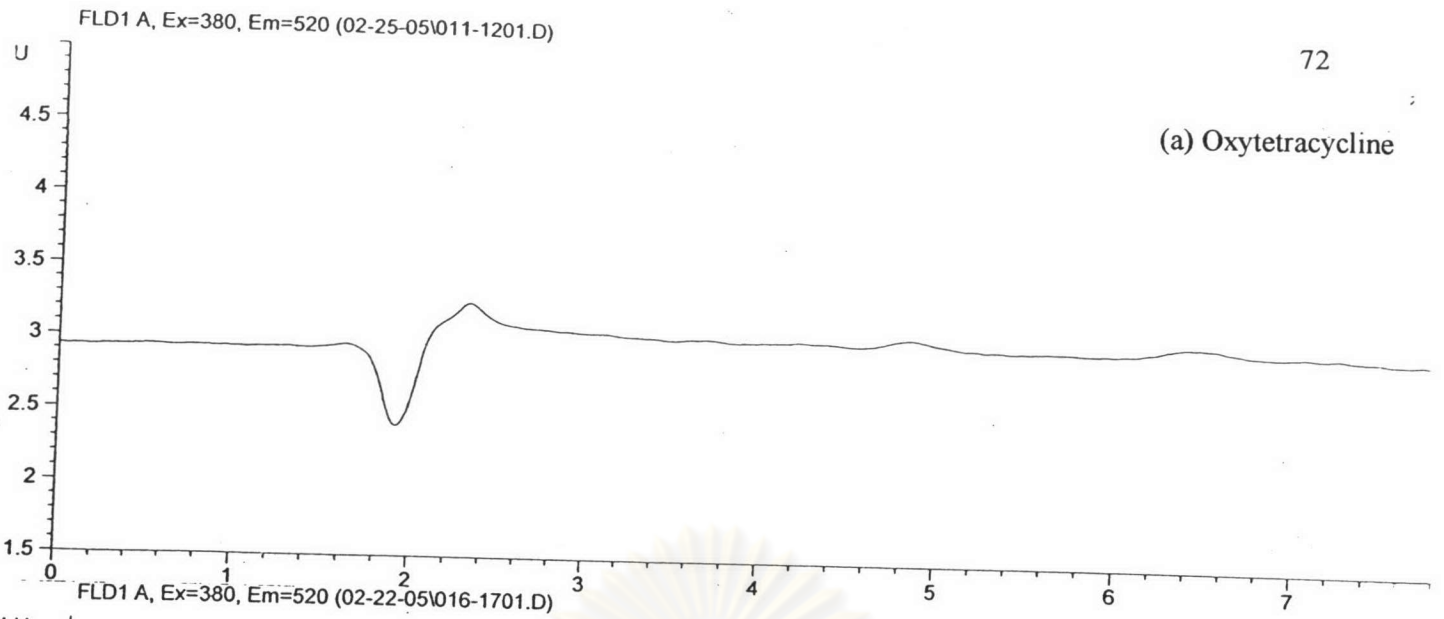
APPENDIX C

Results of Laboratory Center of Food and Agricultural Products Co., Ltd.

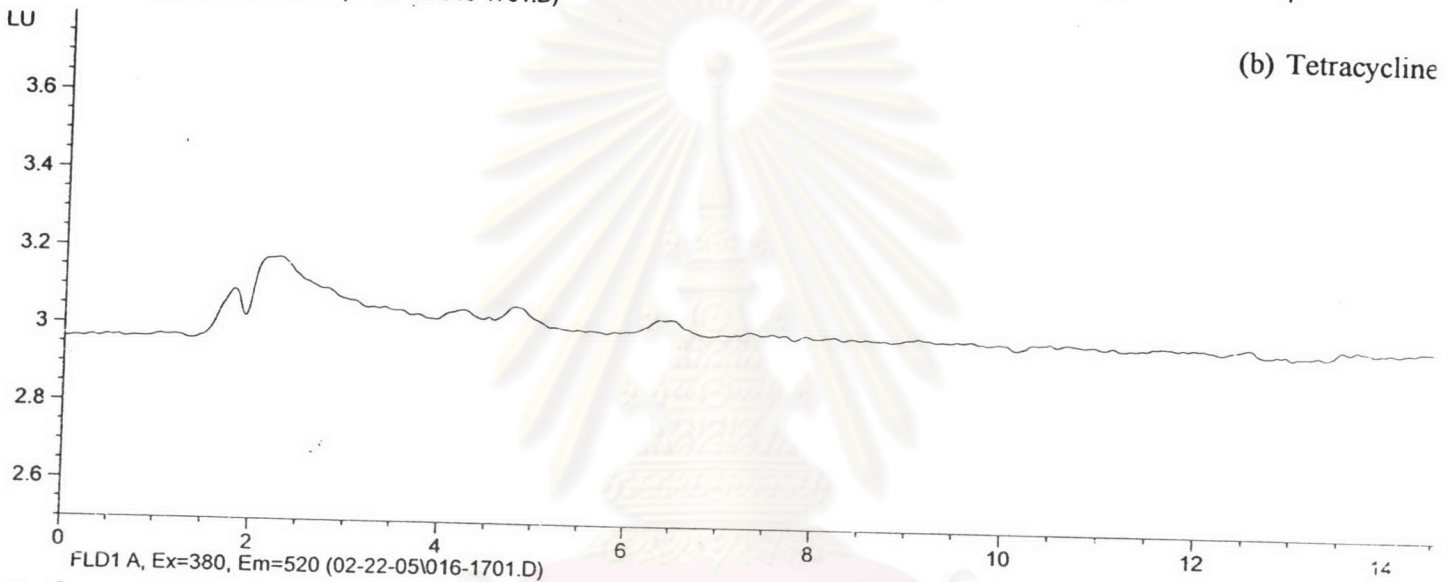
(LCFA)

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(a) Oxytetracycline



(b) Tetracycline



(c) Chlortetracycl



Figure C-1 HPLC chromatograms of Shrimp Sample 1 (February 17, 2005, farming shrimp) (a) oxytetracycline, (b) tetracycline and (c) chlortetracycline.

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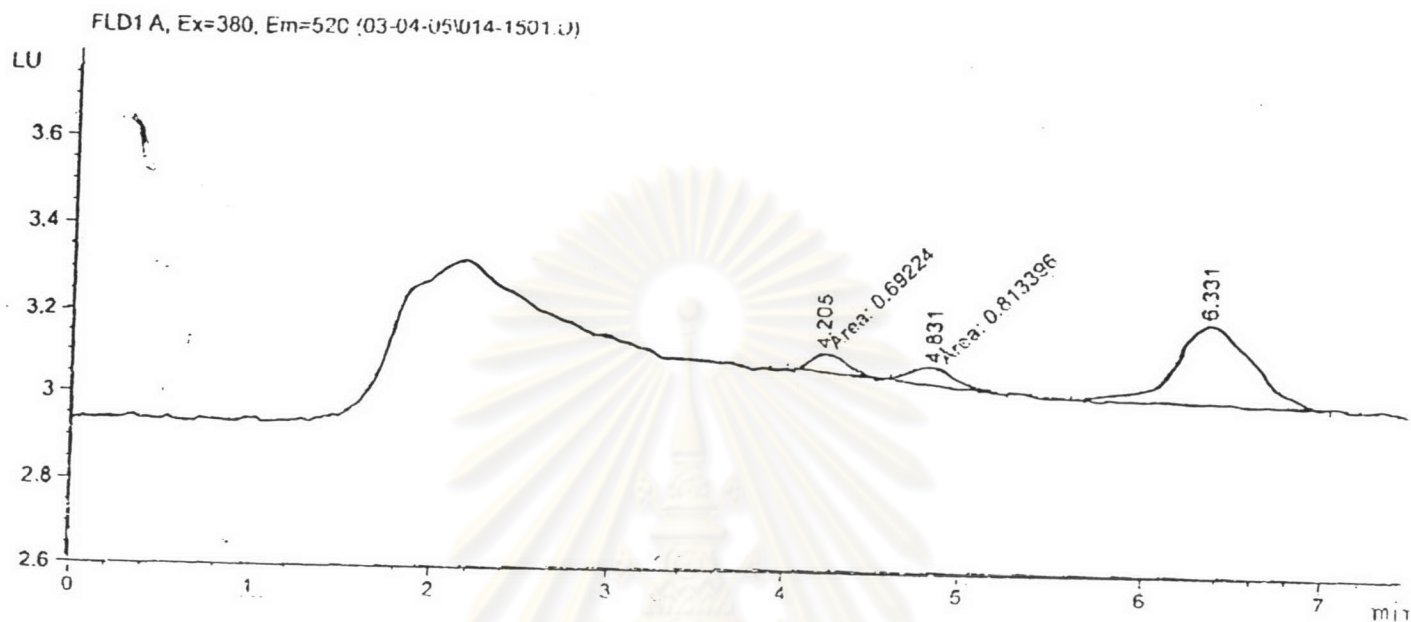


Figure C-2 HPLC chromatograms of Shrimp Sample 1 (February 28, 2005, sea shrimp) of oxyteracycline ($t_R = 3.635$ min)

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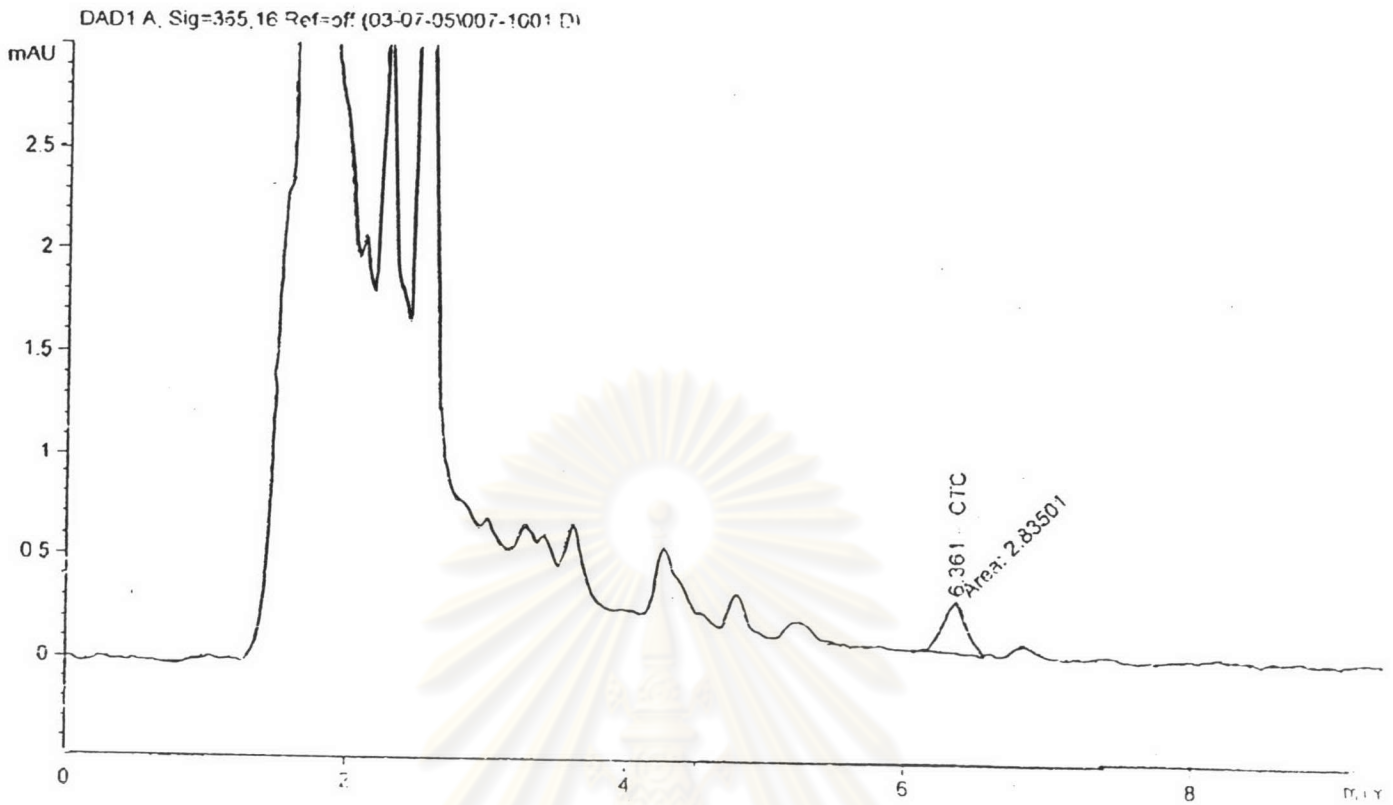


Figure C-3 HPLC chromatograms of Shrimp Sample 1 (February 28, 2005, sea shrimp) of chlortetracycline ($t_R = 6.361$ min); Found chlortetracycline = 0.07 mg/kg

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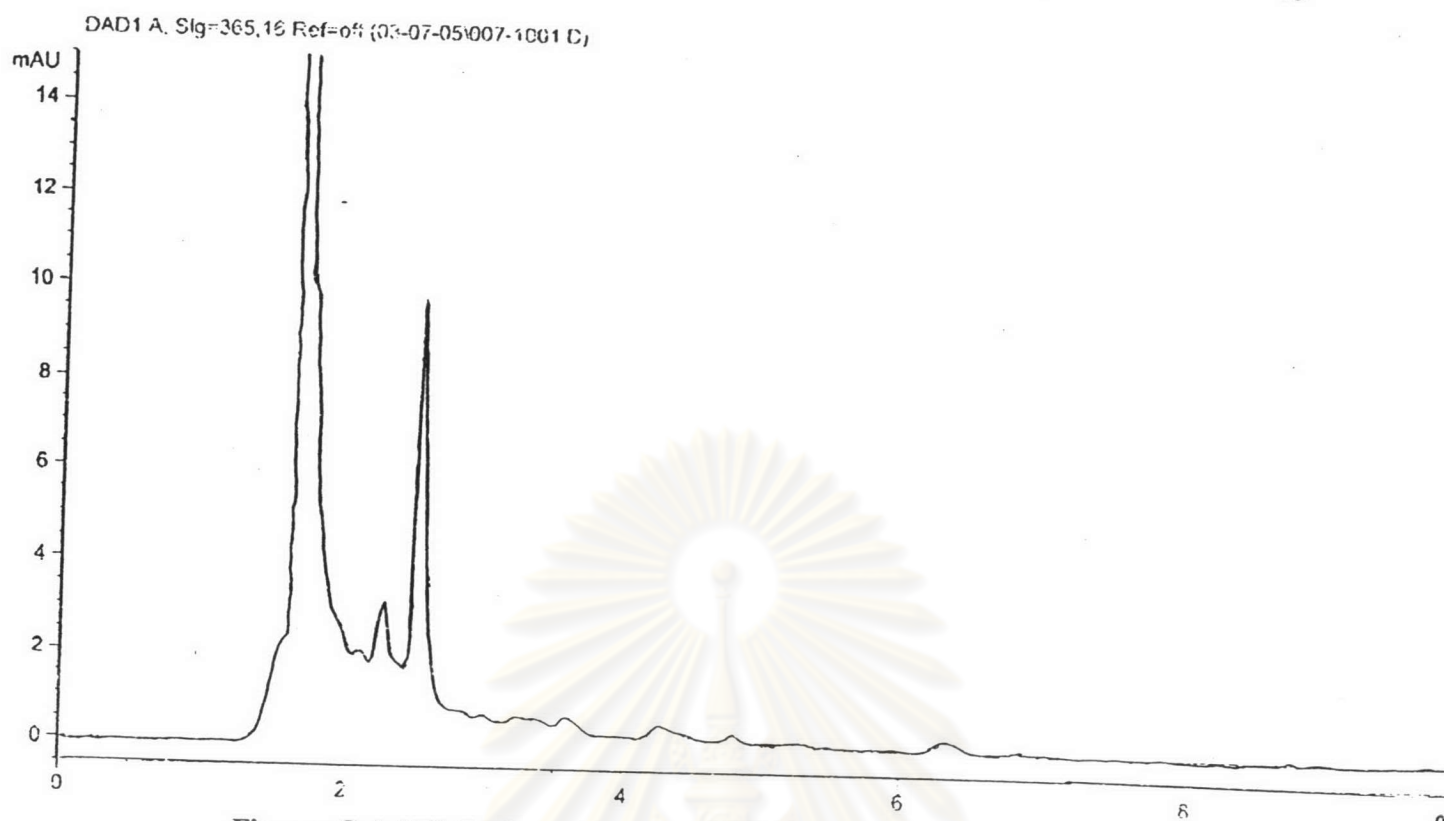


Figure C-4 HPLC chromatograms of Shrimp Sample 1 (February 28, 2005, sea shrimp) of tetracycline ($t_R = 3.401$ min)

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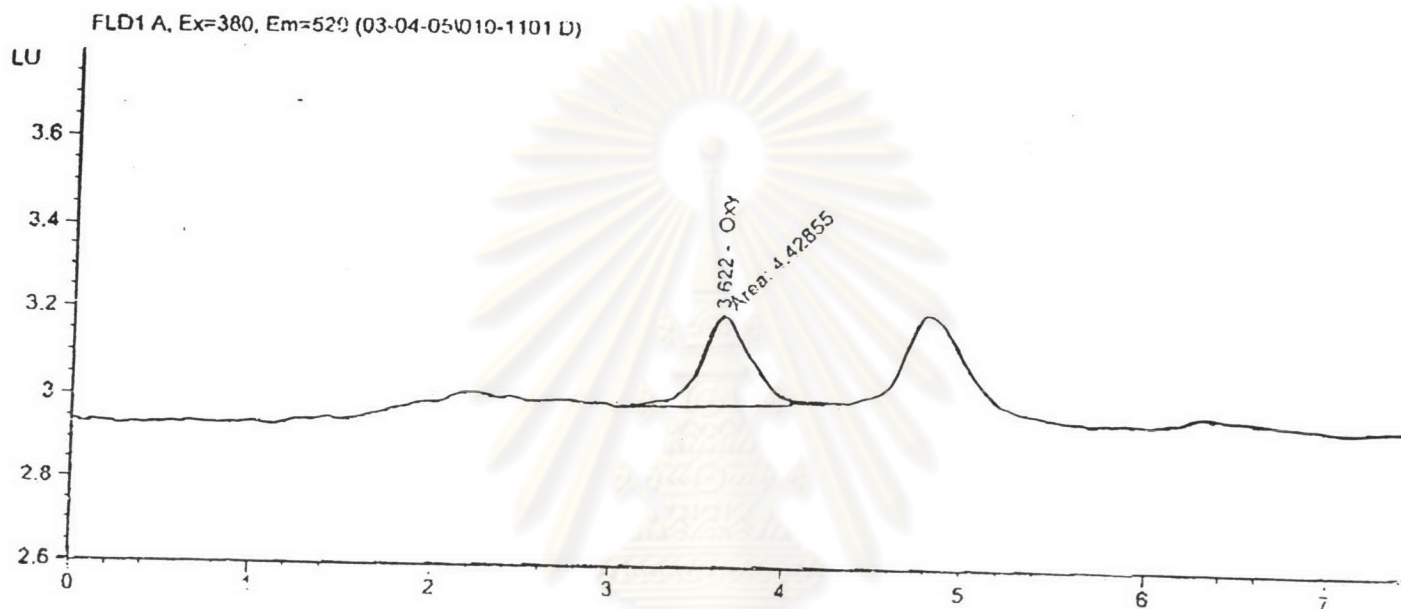


Figure C-5 HPLC chromatograms of Shrimp Sample 1 (February 28, 2005, sea shrimp) spiking oxytetracycline at the level 0.06 mg/kg ($t_R = 4.429$ min). %Recovery = 75%.

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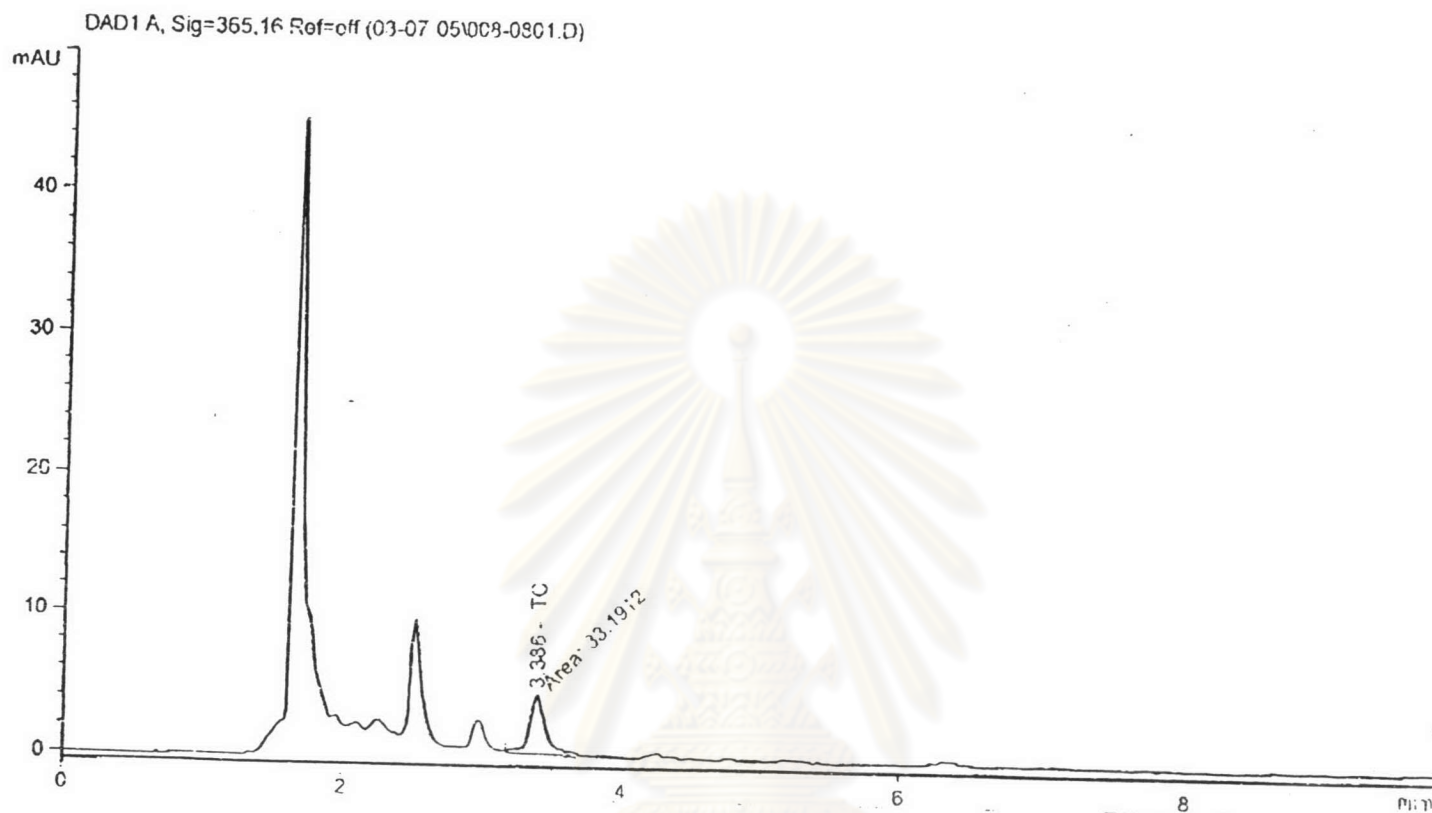


Figure C-6 HPLC chromatograms of Shrimp Sample 1 (February 28, 2005, sea shrimp) spiking tetracycline at the level 0.08 mg/kg ($t_R = 3.386$ min). %Recovery = 74%.

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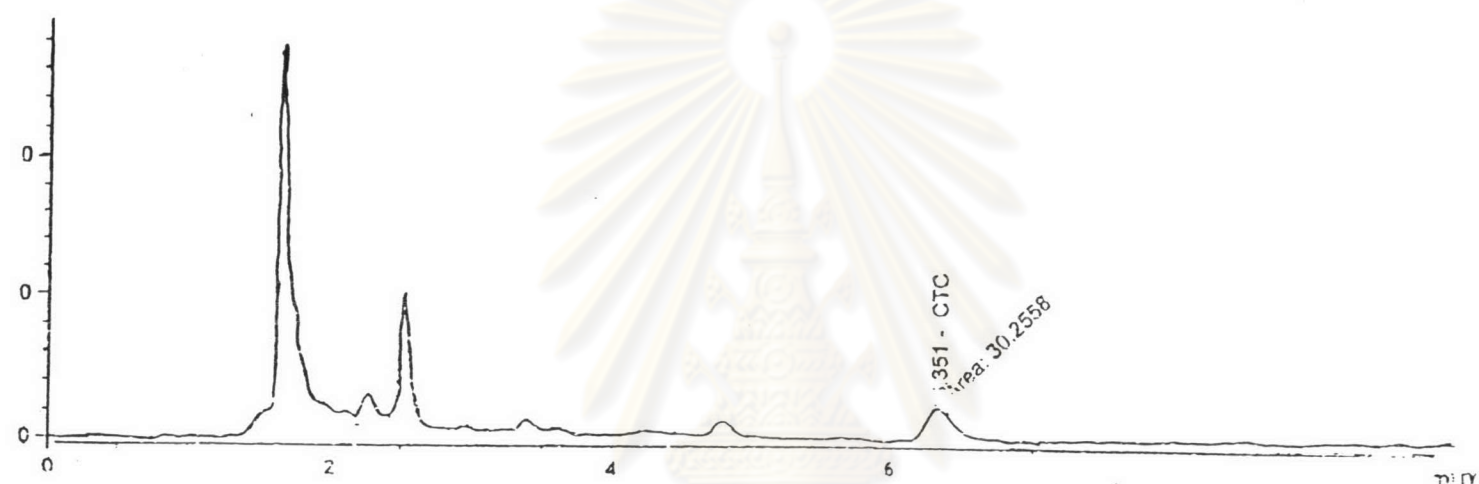


Figure C-7 HPLC chromatograms of Shrimp Sample 1 (February 28, 2005, sea shrimp) spiking chlortetracycline at the level 0.1 mg/kg ($t_R = 6.365$ min). %Recovery = 72%.

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Laboratory Center for Food and Agricultural Products Co., Ltd. : LCFA

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Issue Date: March 08, 2005

Report No.: TR 48/01508

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TEST REPORT

Customer Name and Address	Thiraporn Charoenraks Department of Chemistry, Faculty of Science, Chulalongkorn University, Patumwan, Bangkok 10330
Sample Description	Shrimp Sample 1
Sample Code	48/01045
Sample Characteristic and Condition	The samples were packed in a zip lock plastic bag and kept frozen. Quantity: one bag, weighing 60 g, in good condition when received.
Received Date	February 28, 2005
Test Date	February 28-March 06, 2005

Analysis Results

Test items	Test Results	Units	Reference Methods
Chlortetracycline	0.07	mg/kg	In house method by HPLC
Oxytetracycline	Not Detected	mg/kg	In house method based on AOAC (2000), 995.09
Tetracycline	Not Detected	mg/kg	In house method by HPLC

Note LOQ (Limit of quantification) for Oxytetracycline = 0.05 mg/kg

ศูนย์วิทยทรัพยากร

On behalf of the LCFA Co., Ltd.

จุฬาลงกรณ์มหาวิทยาลัย

Arom Sangwanich

(Mrs. Arom Sangwanich)
Director,

Laboratory Service Bangkok Office

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Issue Date: March 01, 2005
 Report No.: TR 48/01177
 Page: 1 of 1

TEST REPORT

Customer Name and Address	Thiraporn Charoenraks Department of Chemistry, Faculty of Science, Chulalongkorn University, Patumwan, Bangkok 10330
Sample Description	Shrimp Sample 1
Sample Code	48/01045
Sample Characteristic and Condition	The samples were packed in a zip lock plastic bag and kept frozen. Quantity: one bag, weighing 60 g, in good condition when received.
Received Date	February 17, 2005
Test Date	February 22-28, 2005

Analysis Results

Test items	Test Results	Units	Reference Methods
Chlortetracycline	Not Detected	mg/kg	In house method based on AOAC (2000), 995.09
Oxytetracycline	Not Detected	mg/kg	
Tetracycline	Not Detected	mg/kg	

Note LOQ (Limit of quantification) for Oxyletracycline = 0.05 mg/kg

On behalf of the LCFA Co., Ltd.

Arom Sangwanich

(Mrs. Arom Sangwanich)
 Director,
 Laboratory Service Bangkok Office

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APPENDIX D

Description of Analytical Performance Characteristics

Accuracy

Accuracy denotes that closeness of a measurement or set of measurements to the accepted value. Accuracy is normally reported in terms of error. Error is the difference between the accepted and measured values. There are several ways and units in which the accuracy can be expressed. Recovery is a term often used to describe accuracy, the equation for recovery is:

$$\% \text{Recovery} = \frac{\text{Measured value}}{\text{True value}} \times 100$$

Relative error is the another term that can be expressing the accuracy. The equation is shown below:

$$\% \text{error} = \frac{(\text{Measured value} - \text{True value})}{\text{True value}} \times 100$$

Precision

Precision refers to the agreement between values in a set of data that have been carried out in exactly the same mode. It is a measure of the reproducibility of the analysis. Precision of the results can be ascertained through the use of replicate measurements. There are several popular ways to express the precision of data. Multiple injections of a homogeneous sample and calculation of the relative standard deviation (%RSD) do it. The equation for % RSD is shown below:

$$\% \text{RSD} = \frac{\text{standard deviation}}{\text{Mean}} \times 100$$

Linearity

A linearity is the range where the analyte response is linearly proportional to concentration. The working sample concentration and samples tested for accuracy should be in the linear range.

Sensitivity

Sensitivity is the change in the analytical response divided by the corresponding change in the concentration of a standard (calibration) curve, i.e. the slope of the analytical calibration.

Limit of Detection (LOD)

The detection limit of a method is the lowest analyte concentration that can be determined to be different from an analyte blank. There are numerous ways that detection limit has been defined. An example is the lowest analyte concentration that above the noise level of the system, typically, three time the noise level ($S/N = 3$). This term is used to describe low analyte concentration. For high analyte concentrations, the detection limit is defined as the lowest concentration that provides a signal to background ratio S/B of three. The equation of S/B ratio is shown below:

$$S/B \text{ ratio} = \frac{(\text{total signal} - \text{blank signal})}{\text{blank signal}}$$

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จุฬาลงกรณ์มหาวิทยาลัย

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