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APPENDICES

APPENDIX A

Cyclic voltammetric results

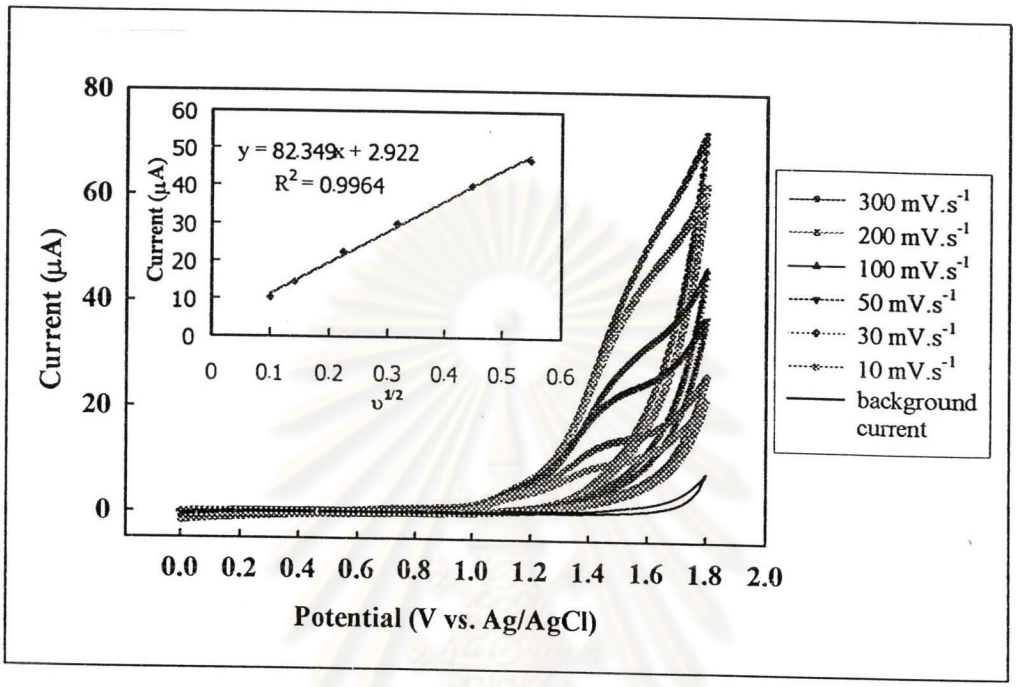

Figure A-1 Cyclic voltammogram of 1 mM tetracycline in 0.1 M phosphate buffer (pH 2) at the Ni-implanted diamond electrode. The scan rate was from 10-300 mV s^{-1}. Background cyclic voltammogram at 50 mVs^{-1} is also shown in this Figure (solid line).

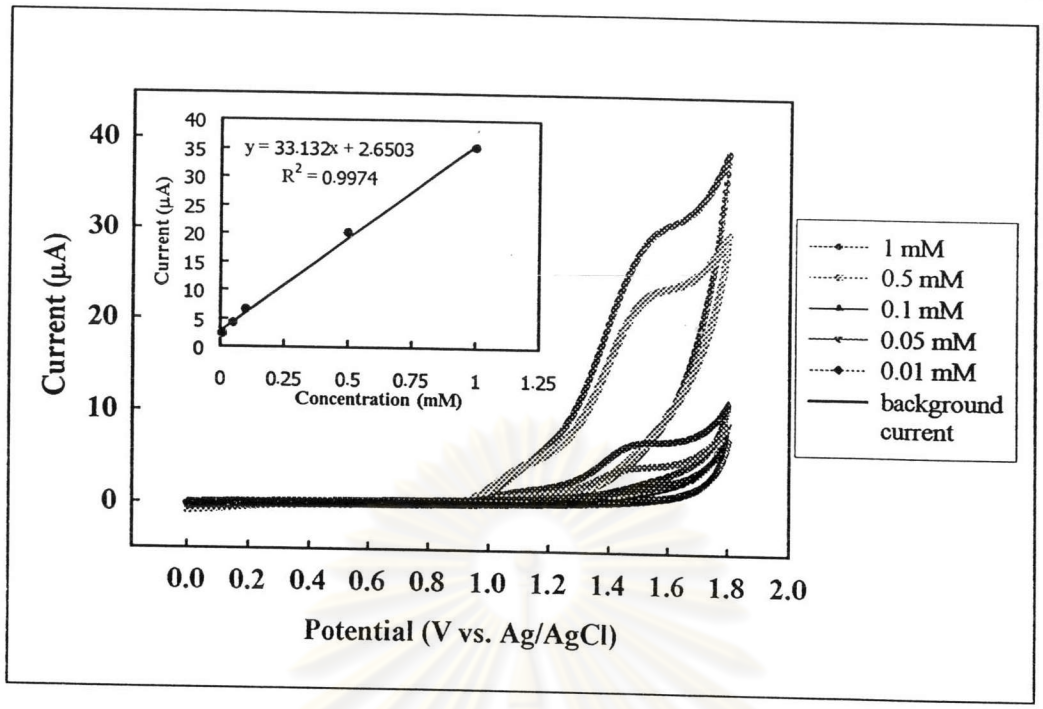

Figure A-2 Cyclic voltammogram of tetracycline (0.01-1 mM) in 0.1 M phosphate buffer (pH 2) at the Ni-implanted diamond electrode. The scan rate was 50 mVs^{-1}. The corresponding calibration curve (inset Figure) and background cyclic voltammogram (solid line) are also shown.

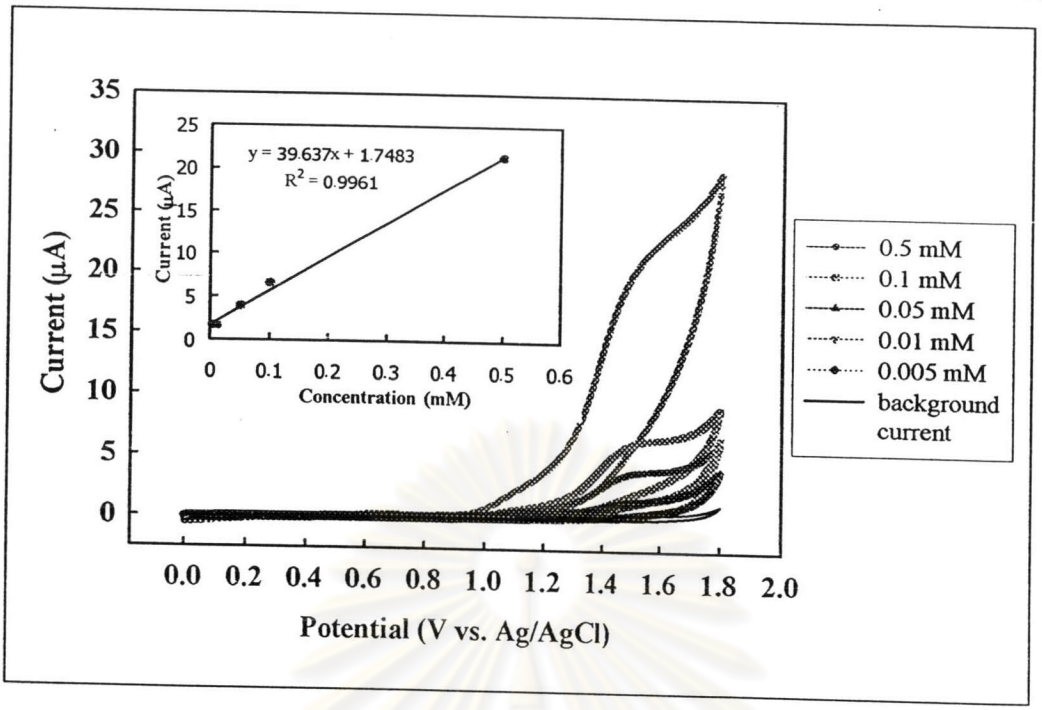

Figure A-3 Cyclic voltammogram of oxytetracycline (0.005-0.5 mM) in 0.1 M phosphate buffer (pH 2) at the Ni-implanted diamond electrode. The scan rate was 50 mVs^{-1}. The corresponding calibration curve (inset Figure) and background cyclic voltammogram (solid line) are also shown.

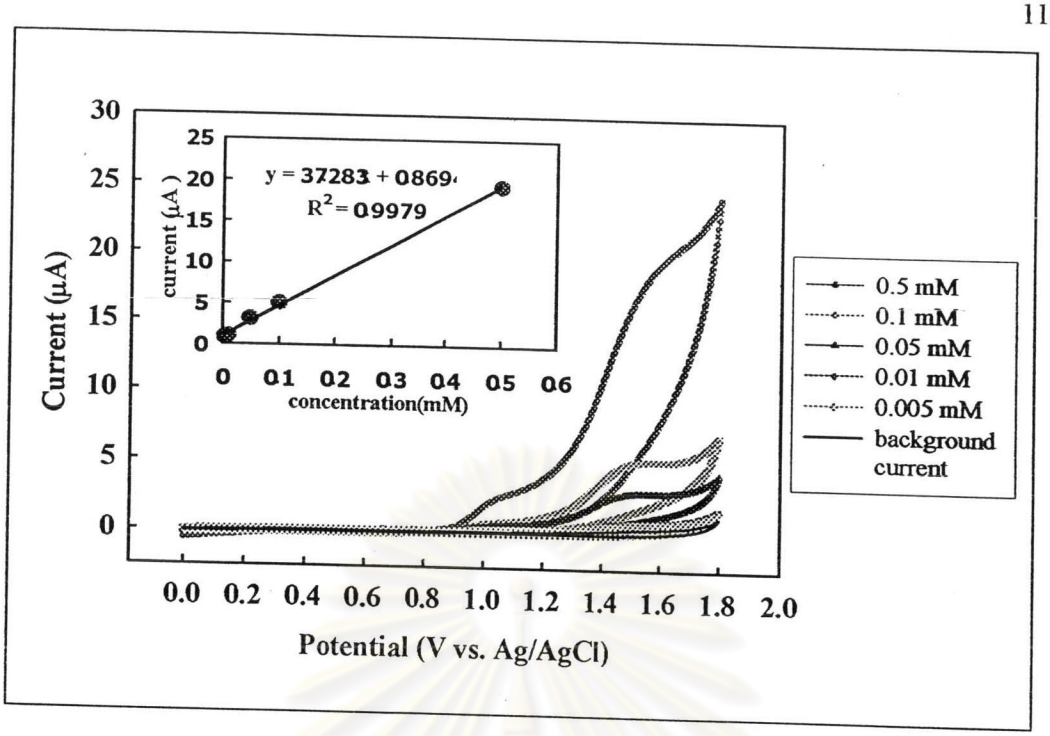

Figure A-4 Cyclic voltammogram of chlortetracycline (0.005-0.5 mM) in 0.1 M phosphate buffer (pH 2) at the Ni-implanted diamond electrode. The scan rate was 50 mVs^{-1}. The corresponding calibration curve (inset Figure) and background cyclic voltammogram (solid line) are also shown.

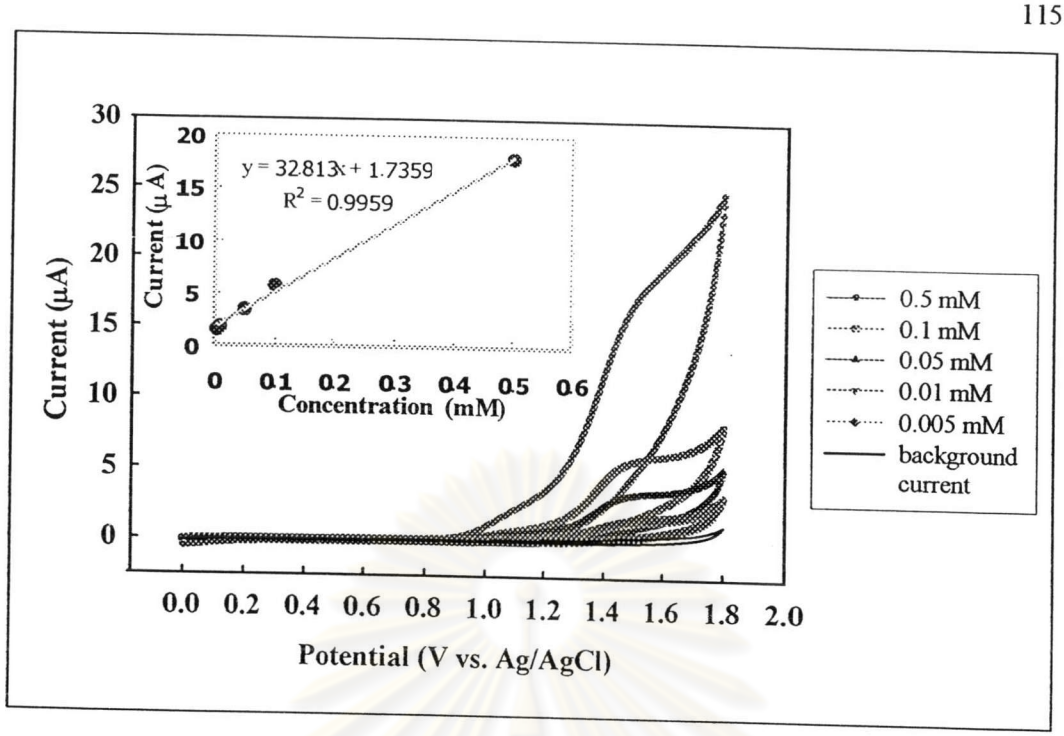

Figure A-5 Cyclic voltammogram of doxycycline hydrochloride (0.005-0.5 mM) in 0.1 M phosphate buffer (pH 2) at the Ni-implanted diamond electrode. The scan rate was 50 mVs^{-1}. The corresponding calibration curve (inset Figure) and background cyclic voltammogram (solid line) are also shown.

APPENDIX B

Flow injection with amperometric detection results

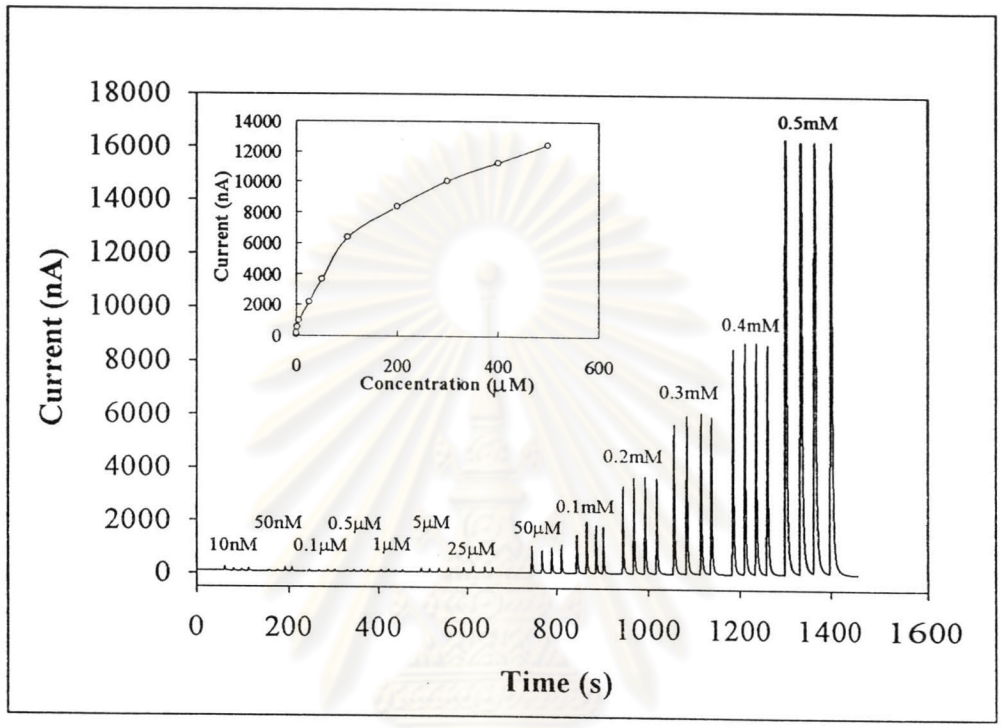

Figure B-1 Flow injection with amperometric detection result of tetracycline (10 mM-0.5 mM) in 0.1 M phosphate buffer (pH 2) with 4 injections at applied potential 1.55 V vs. Ag/AgCl. The flow rate was 1 ml min^{-1}. The corresponding calibration curve is also shown (inset Figure).

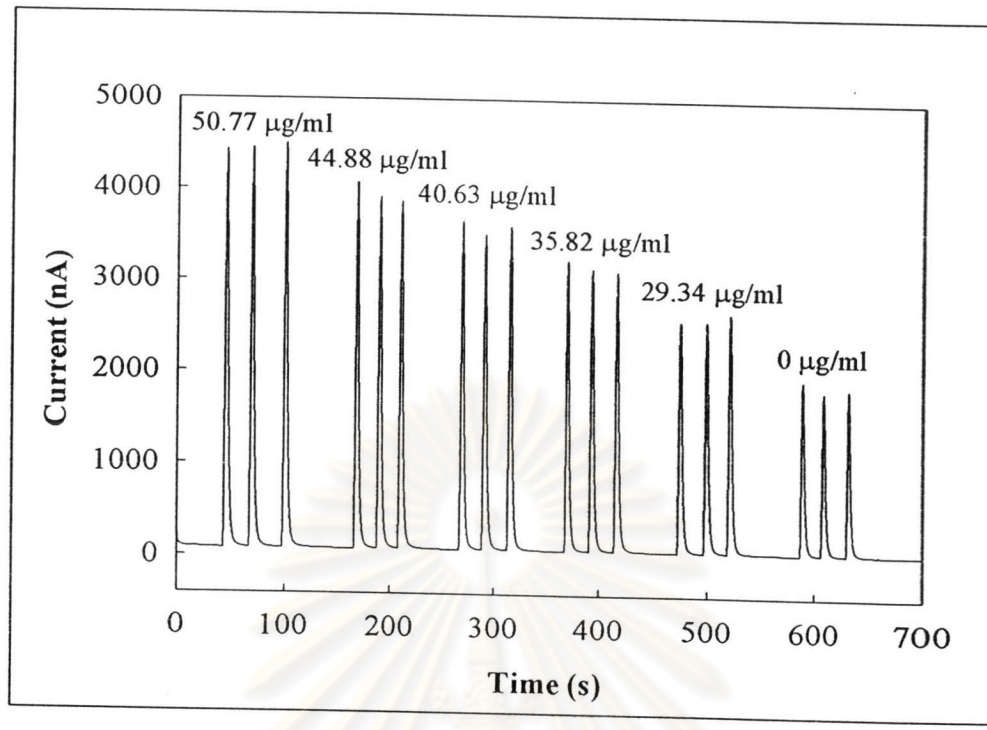

Figure B-2 Flow injection with amperometric detection result of tetracycline hydrochloride capsule in 0.1 M phosphate buffer (pH 2) with 3 injections at applied potential 1.55 V vs. Ag/AgCl. The flow rate was 1 ml min^{-1}.

APPENDIX C

Results form high performance liquid chromatographic with amperometry

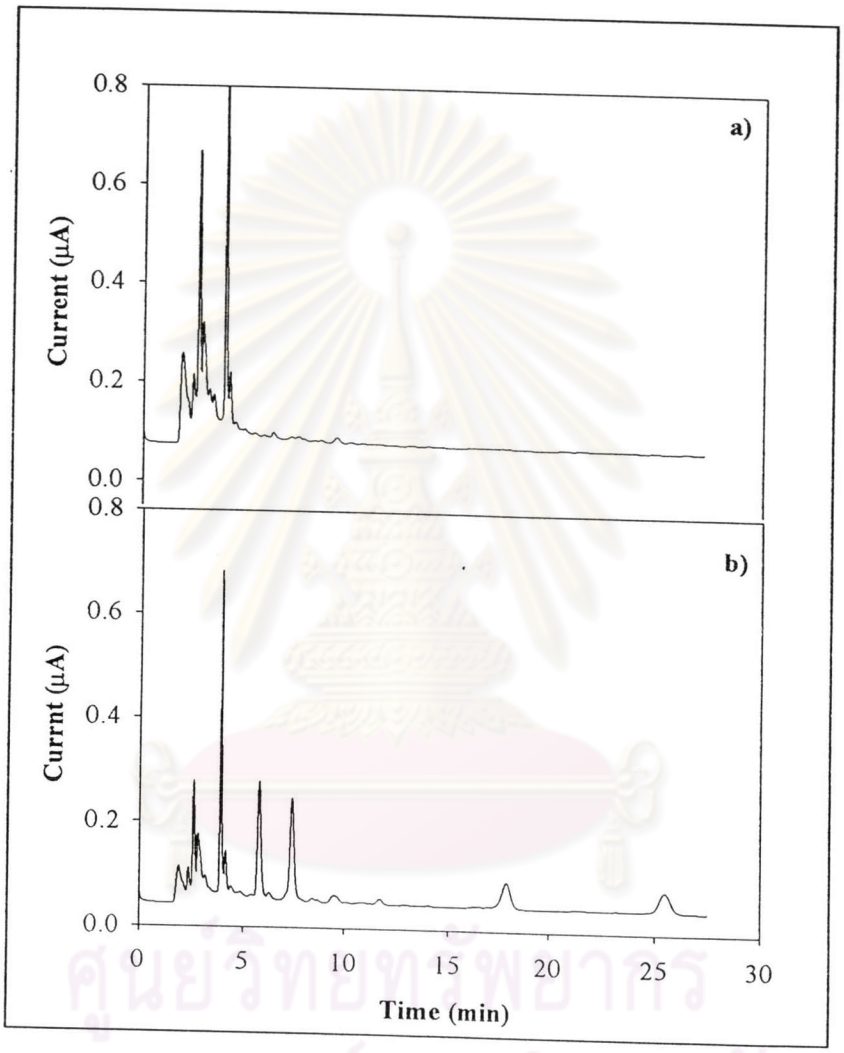

Figure C-1 HPLC chromatograms of blank sample (farming shrimp) a) and spiking mixed standard at the level of 5 mg/kg b)

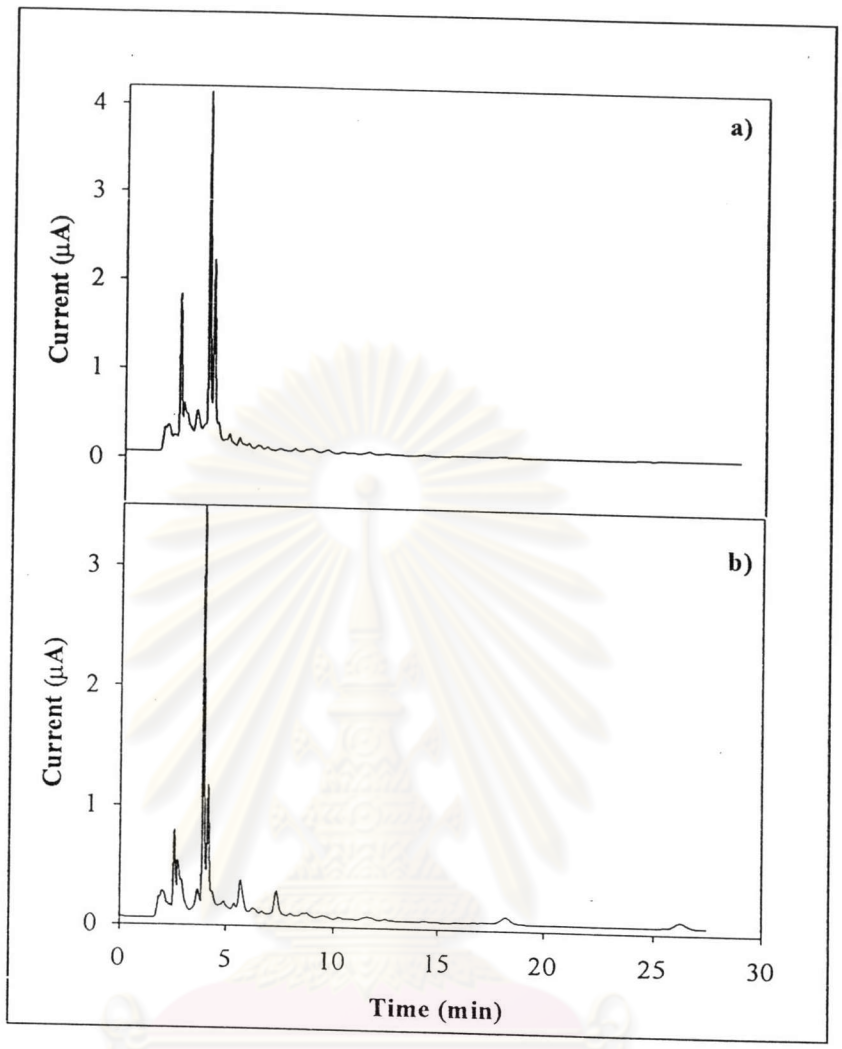

Figure C-2 HPLC chromatograms of blank sample (sea shrimp) a) and spiking mixed standard at the level of 5 mg/kg b)

APPENDIX E

Results form AOAC method

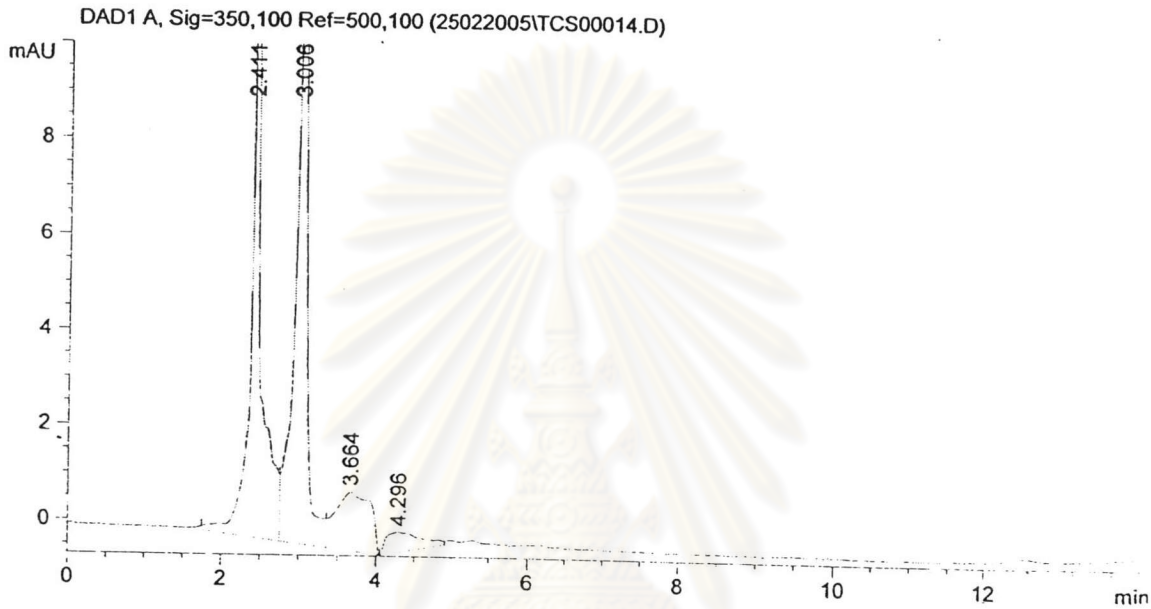

Figure E-1 HPLC chromatograms of blank sample (farming shrimp)

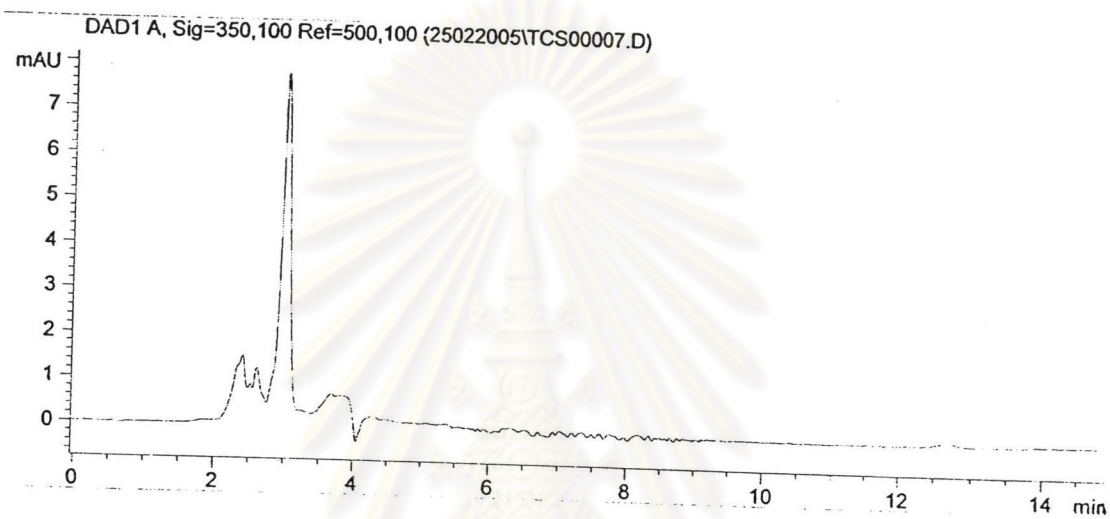

Figure E-2 HPLC chromatograms of blank sample (sea shrimp)

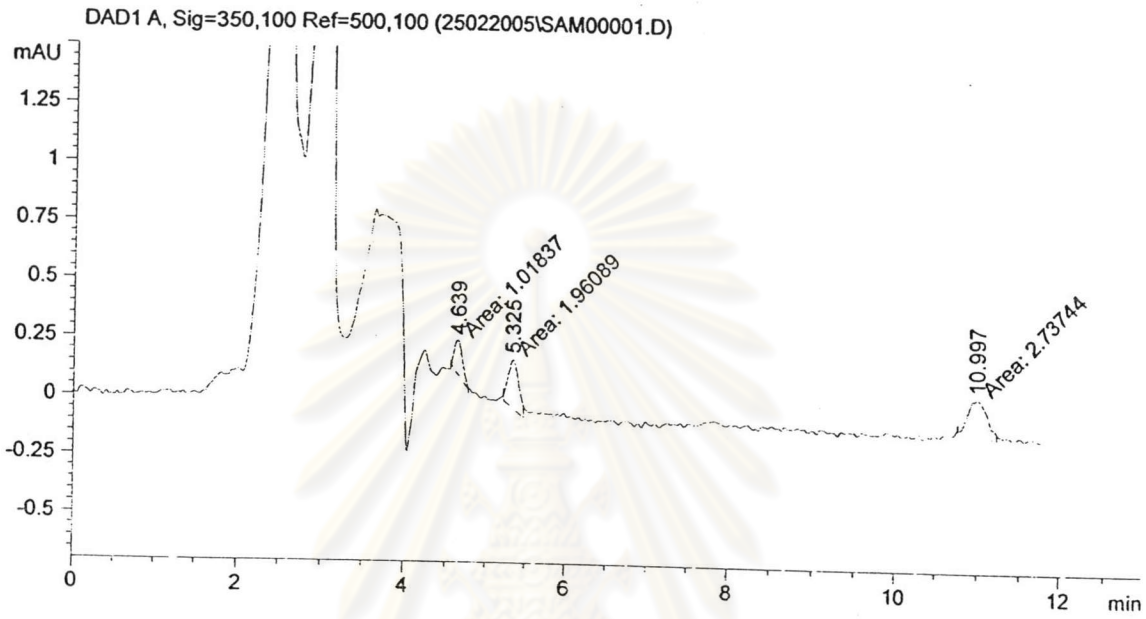

Figure E-3 HPLC chromatograms of spiking sample at the level of 0.5 mg/kg

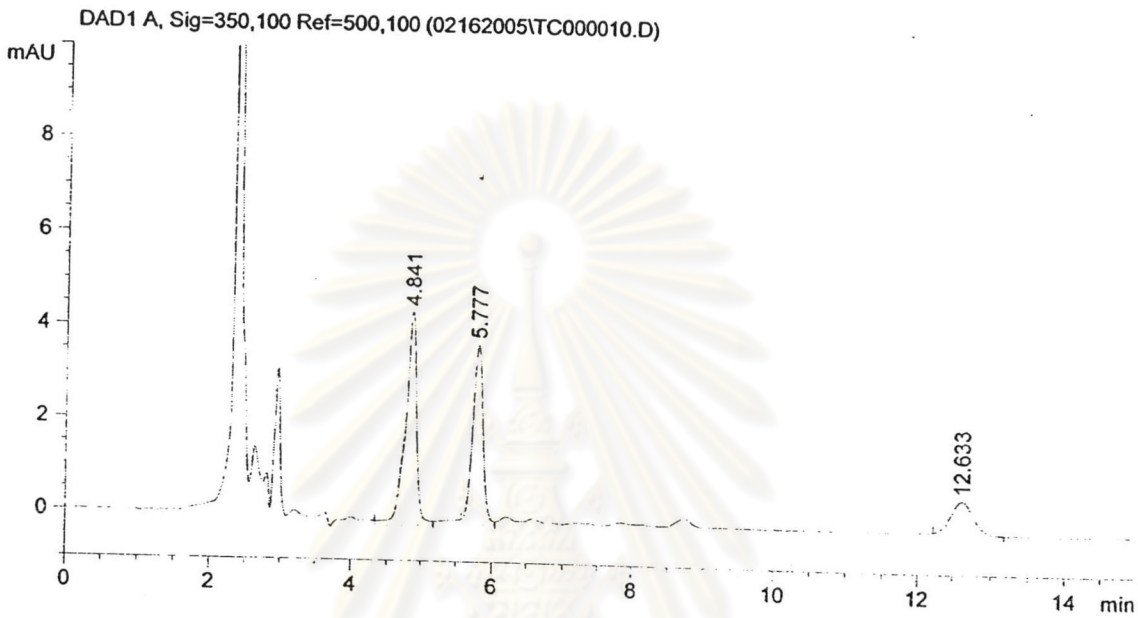

Figure E-4 HPLC chromatograms of spiking sample at the level of 5 mg/kg

APPENDIX D

Results form Laboratory Center for Food and Agricultural Products Co.,Ltd. (LCFA)

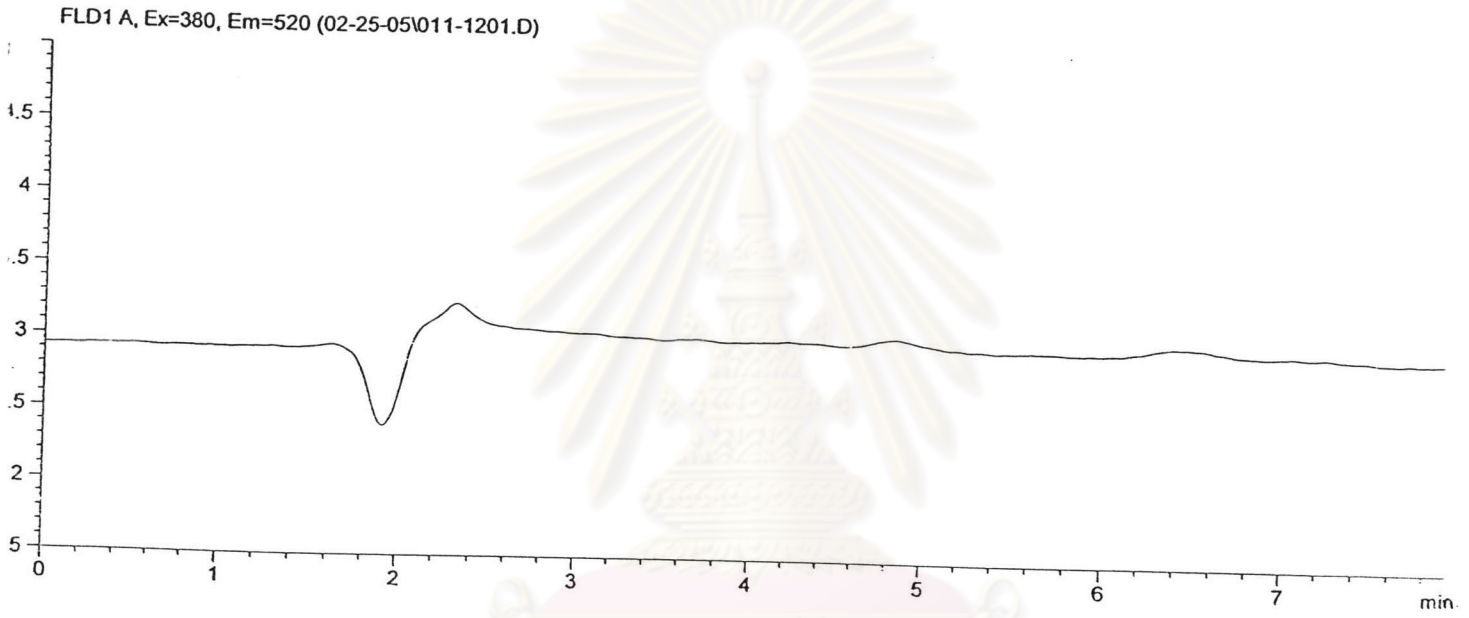

Figure D-1 HPLC chromatograms of shrimp sample 1 (February 17, 2005) of oxytetracycline

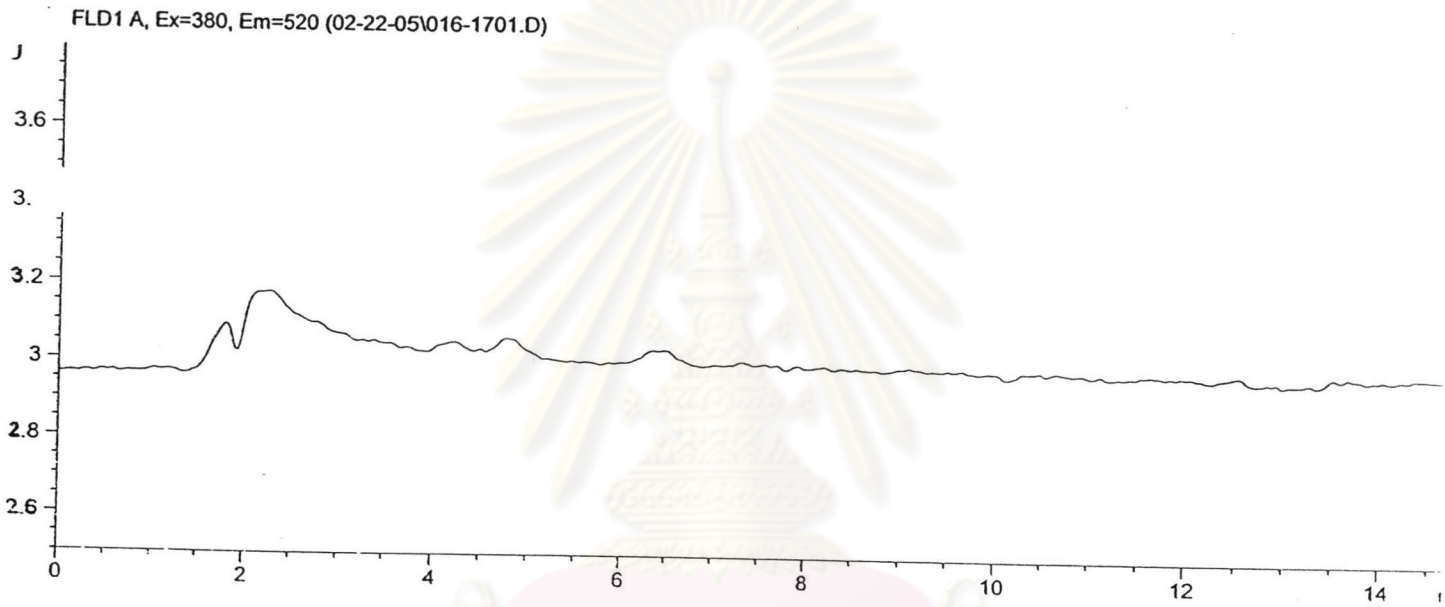

Figure D-2 HPLC chromatograms of shrimp sample 1 (February 17, 2005) of tetracycline

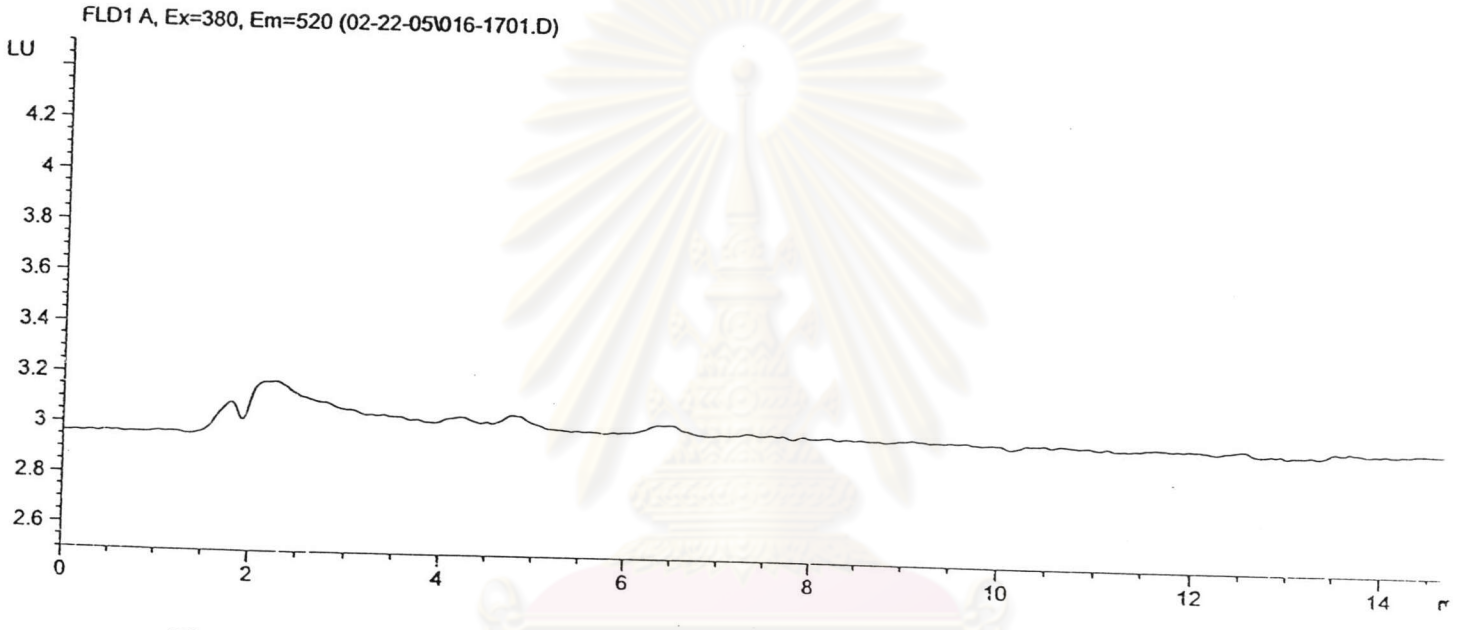

Figure D-3 HPLC chromatograms of shrimp sample 1 (February 17, 2005) of chlortetracycline

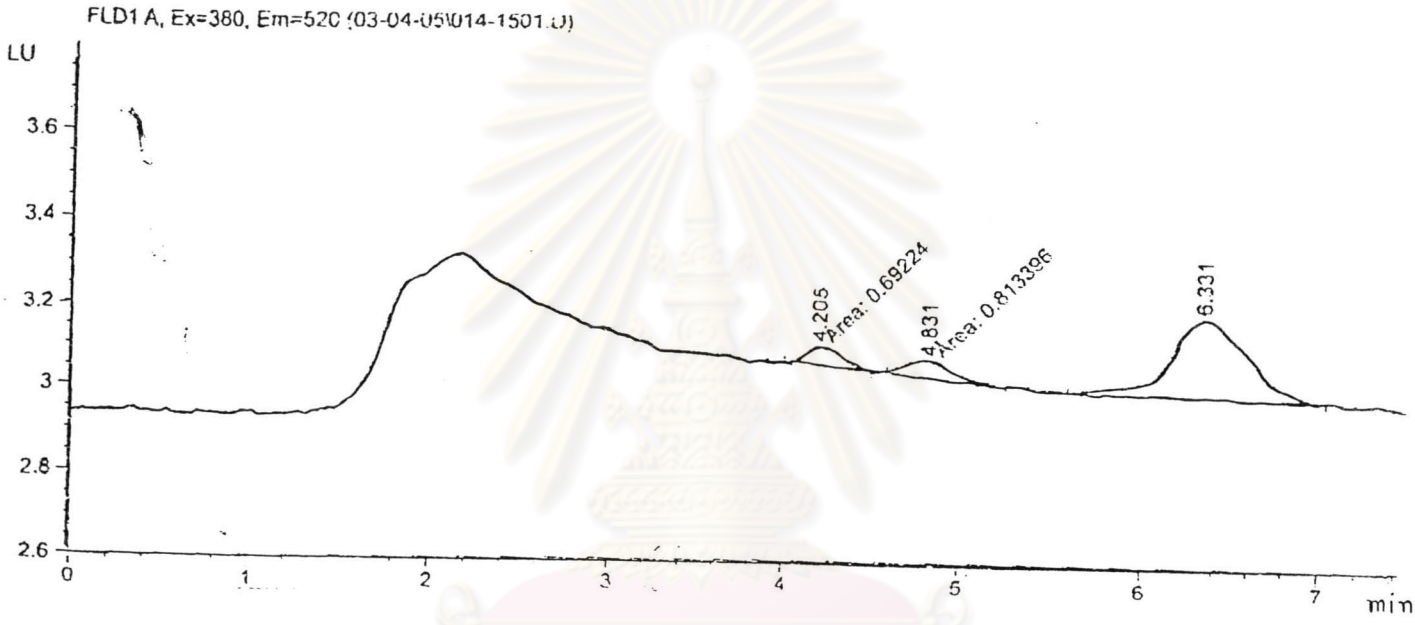

Figure D-4 HPLC chromatograms of shrimp sample 1 (February 28, 2005) of oxytetracycline

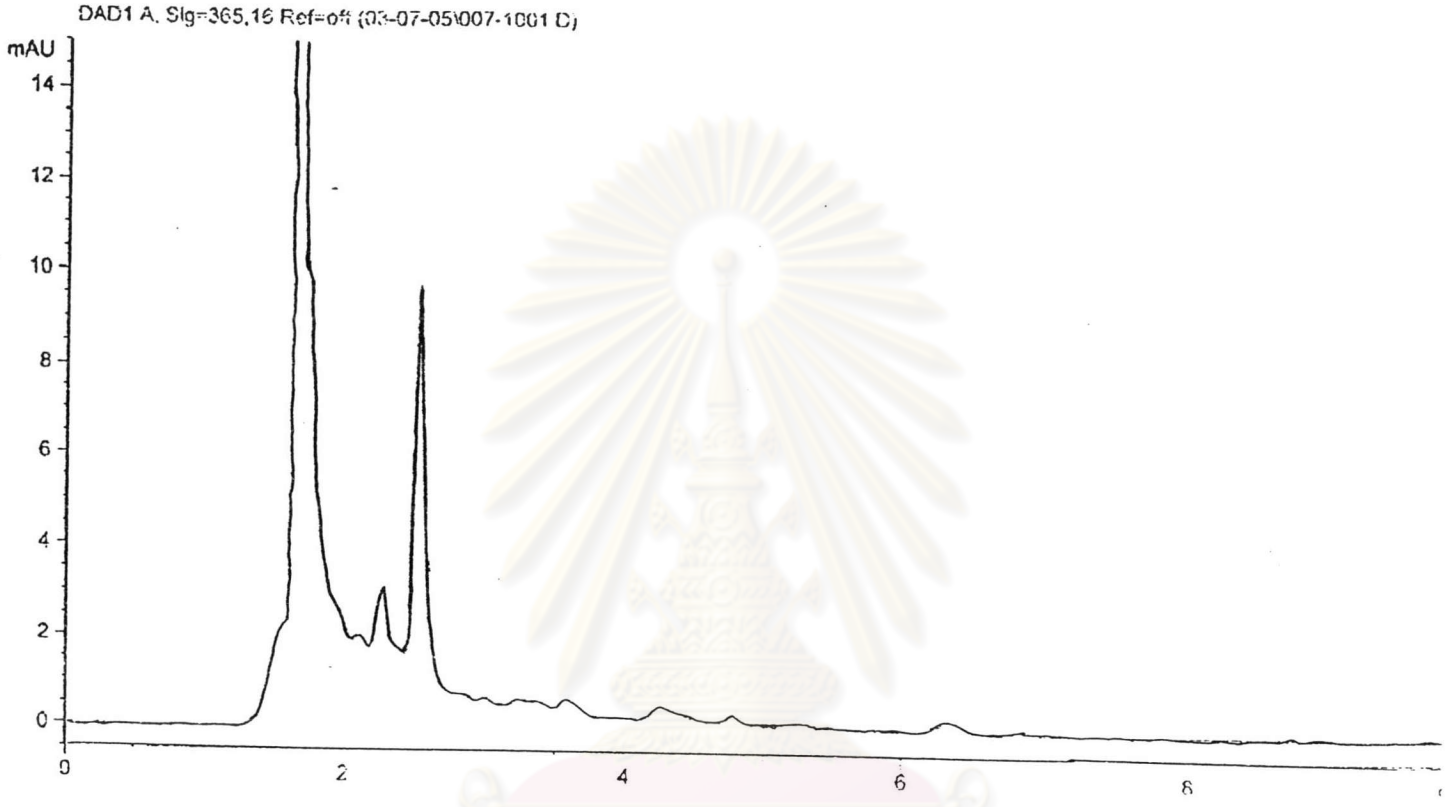

Figure D-5 HPLC chromatograms of shrimp sample 1 (February 28, 2005) of tetracycline

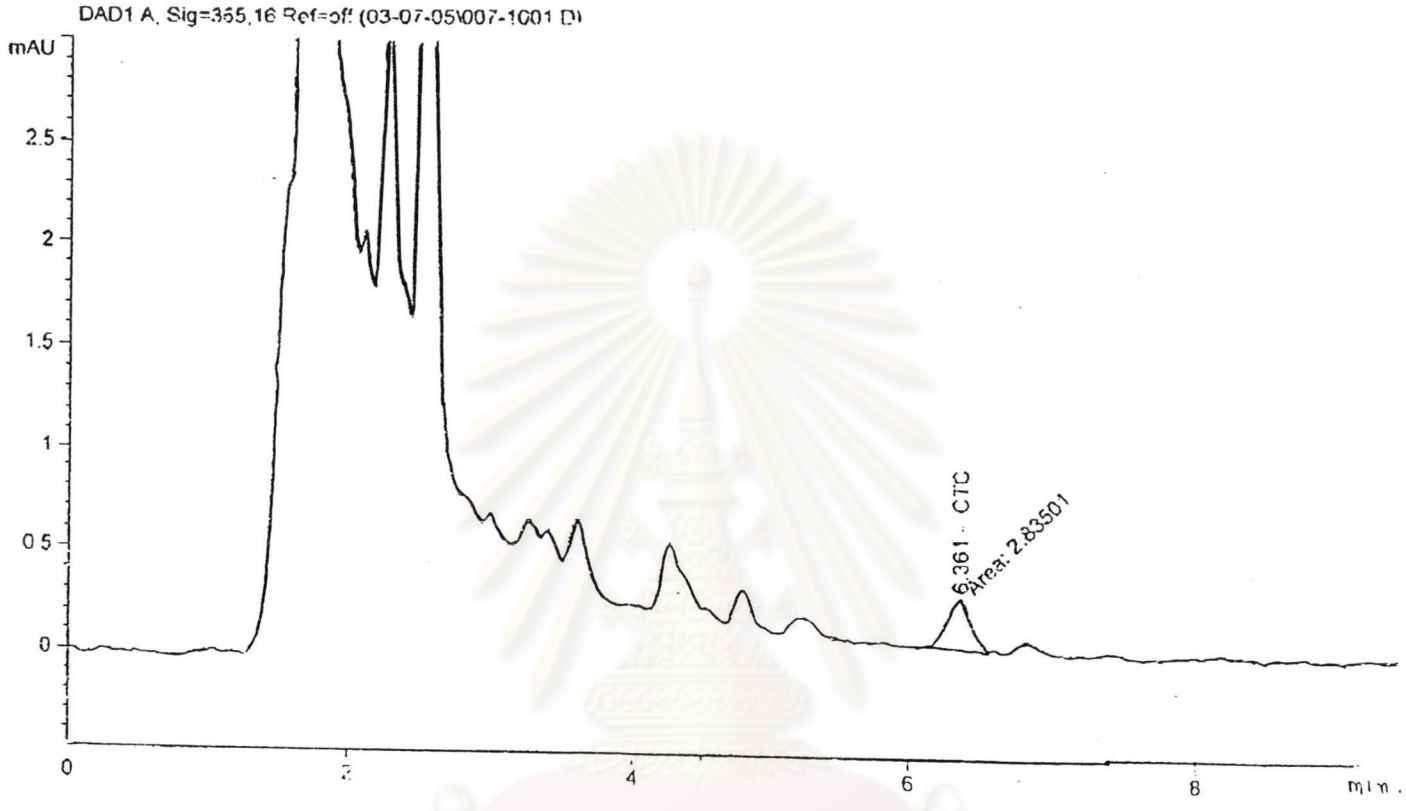

Figure D-6 HPLC chromatograms of shrimp sample 1 (February 28, 2005) of chlortetracycline

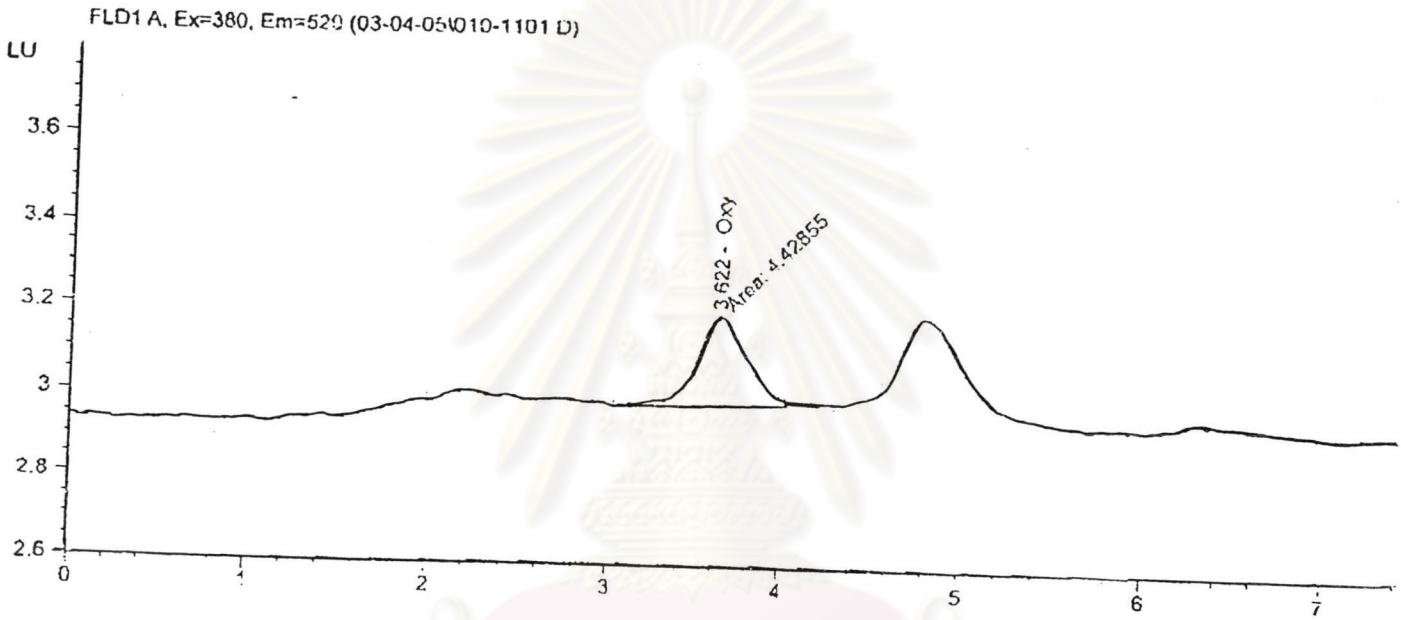

Figure D-7 HPLC chromatograms of shrimp sample spiking oxytetracycline at the level of 0.1 mg/kg

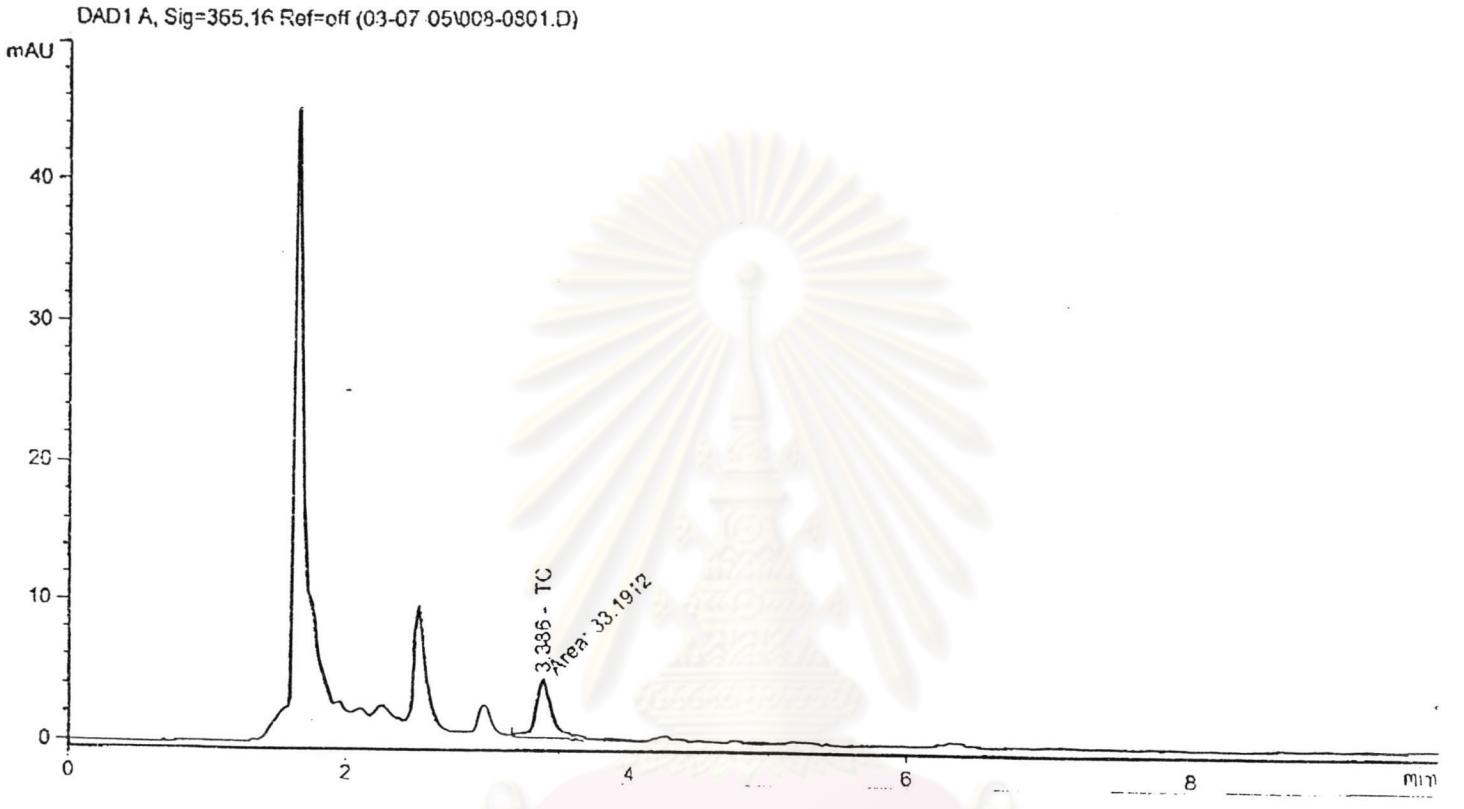

Figure D-8 HPLC chromatograms of shrimp sample spiking tetracycline at the level of 0.1 mg/kg

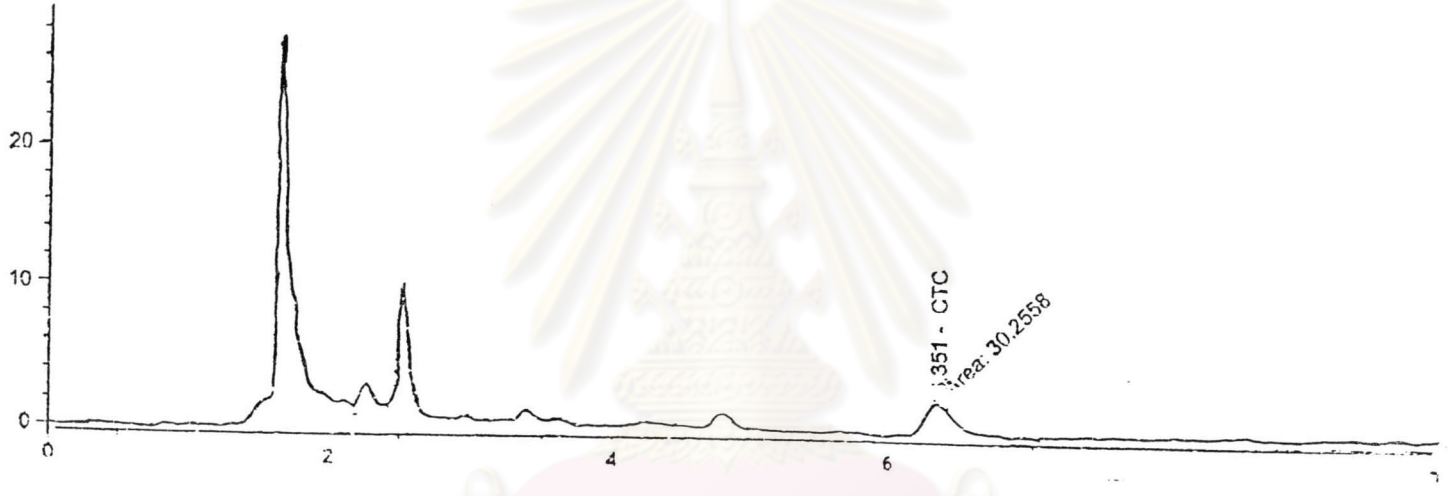

Figure D-9 HPLC chromatograms of shrimp sample spiking chlortetracycline at the level of 0.1 mg/kg

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Report No.: TR 48/01177
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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 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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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 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

On behalf of the LCFA Co., Ltd.

Arom Sangwanich

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

APPENDIX F

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:

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

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

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

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:

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

Linearity (Linear range)

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 way that detection limit have been defined. An example is the lowest analyte concentration that is above the noise level of the system, typically, three time the noise level (S/N = 3). This term is used to describe low analyte concentrations (< 10 μM). 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:

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

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