REPORT

Canadian Goose, Duck and Swan Samples: Determination of White Phosphorus Residues

Study Director:	R E Butler
Test Facility:	**Harlan Laboratories Ltd.** Shardlow Business Park Shardlow Derbyshire DE72 2GD UK
Sponsor:	**Rhodia UK Limited** Trinity Street PO Box 80 Oldbury West Midlands B69 4LN UNITED KINGDOM
Harlan Study Number:	**41201796**
Study Completion Date:	18 September 2012

CONTENTS

STUDY DIRECTOR AUTHENTICATION

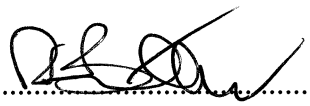
Harlan Laboratories Ltd., Shardlow Business Park, Shardlow, Derbyshire, DE72 2GD, UK

Harlan Study Number:	41201796
Study Title:	Canadian Goose, Duck and Swan Samples: Determination of White Phosphorus Residues

This study was conducted in a facility operating to Good Laboratory Practice (GLP) within the national GLP monitoring program, but the study report has not been audited by the Quality Assurance Unit. A formal claim of GLP compliance cannot therefore be made.

I the undersigned, hereby declare that this report accurately reflects the original data generated in the study.

Study Director: R E Butler



Date: 18 SEP 2012

SUMMARY

The white phosphorus residues of Canadian Goose, Duck and Swan Samples have been determined.

The amount of white phosphorus present (mg/kg) in the tissue samples and the limit of detection (mg/kg) for each sample are shown in the following table:

Tissue Sample	White Phosphorus Residue (mg/kg of tissue)	Limit of Detection (LOD) (mg/kg of tissue)
Swan fat	<LOD	4.53×10^{-3}
Swan kidney	<LOD	4.73×10^{-3}
Swan muscle	<LOD	4.18×10^{-3}
Swan liver	<LOD	4.18×10^{-3}
Swan intestine	<LOD	4.78×10^{-3}
Swan gizzard	2.28×10^{-2}	1.38×10^{-2}
Goose fat	7.85×10^{-2}	1.60×10^{-2}
Goose kidney	<LOD	1.51×10^{-2}
Goose muscle	0.284	1.46×10^{-2}
Goose liver	<LOD	1.98×10^{-2}
Goose intestine	0.155	1.40×10^{-2}
Goose gizzard	1.61×10^{-2}	1.39×10^{-2}
Duck fat	3.38	2.30×10^{-3}
Duck kidney	6.91×10^{-2}	4.93×10^{-3}
Duck muscle	3.89×10^{-2}	2.20×10^{-3}
Duck liver	<LOD	3.40×10^{-3}
Duck intestine	0.160	3.54×10^{-3}
Duck gizzard	4.01×10^{-2}	2.31×10^{-3}

The amount of white phosphorus present (mg) in the gizzard and intestine contents and the corresponding limits of detection (mg) are shown in the following table:

Contents Sample	White Phosphorus Residue (mg)	Limit of Detection (LOD) (mg)
Swan Intestine contents	7.91×10^{-3}	4.87×10^{-5}
Swan Gizzard contents	0.308	1.42×10^{-4}
Goose Intestine contents	3.68×10^{-3}	1.61×10^{-4}
Goose Gizzard contents	14.3	2.37×10^{-5}
Duck Intestine contents	6.02×10^{-4}	2.37×10^{-5}
Duck Gizzard contents	0.349	2.37×10^{-5}

GENERAL INFORMATION

Schedule

Study initiation date:	19 April 2012
Experimental Starting Date:	14 June 2012
Experimental Completion Date:	27 June 2012

Archiving

Unless instructed otherwise by the Sponsor, all original data and the final report will be retained in the Harlan Laboratories Ltd, Shardlow, UK archives for five years after which instructions will be sought as to further retention or disposal. Further retention or return of the data will be chargeable to the Sponsor.

1 INTRODUCTION AND PURPOSE

The purpose of this study is to determine the white phosphorus residues in Canadian Goose, Duck and Swan Samples.

The procedure was based on Johnston, JJ, Goldade, DA, Kohler DJ, Cummings, JL (2000). Determination of white phosphorus residues in ducks: an atomic emission detection/compound-independent calibration-based method of generating residue data for risk assessment and environmental monitoring. Environ Sci Technol 34(9):1856-1861.

2 TEST ITEMS AND ANALYTICAL STANDARD SOLUTION

2.1 Test Items

The integrity of the supplied data relating to the identity of the test items is the responsibility of the Sponsor.

Sponsor's identification	:	Swan Fat
Label	:	S26-B0406-04-12 / 1 Sample Unspecified / Fat
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Swan Kidney
Label	:	26-B0406-04-12 / 1 Kidney
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Swan Muscle
Label	:	26-B0406-04-12 / 1 Muscle
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Swan Liver
Label	:	26-B0406-04-12 / 1 Liver
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Swan Intestine
Label	:	26-B0406-04-12 / 1 Intestine Unspec
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Swan Gizzard
Label	:	26-B0406-04-12 / 1 Gizzard
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Goose Muscle
Label	:	26-B0537-01-12 / Goose Muscle
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Goose Fat
Label	:	26-B0537-01-12 / Goose Tissue Unspecified / Fat
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Goose Intestine
Label	:	26-B0537-01-12 / Goose Intestine Unspec - 2
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Goose Liver
Label	:	26-B0537-01-12 / Goose Liver
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Goose Kidney
Label	:	26-B0537-01-12 / Goose Kidney
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Goose Gizzard
Label	:	26-B0537-01-12 / Goose Gizzard
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Duck Fat
Label	:	26-B0537-01-12 / Duck Tissue Unspecified / Fat
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Duck Muscle
Label	:	26-B0537-01-12 / Duck Muscle
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Duck Liver
Label	:	26-B0537-01-12 / Duck Liver
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Duck Kidney
Label	:	26-B0537-01-12 / Duck Kidney
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Duck Gizzard
Label	:	26-B0537-01-12 / Duck Gizzard
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

Sponsor's identification	:	Duck Intestine
Label	:	26-B0537-01-12 / Duck Intestine Unspec - 3
Date received	:	12 June 2012
Expiry date	:	not available
Storage conditions	:	frozen at approximately -20°C, in the dark

2.2 Analytical Standard Solution

The integrity of the supplied data relating to the identity, purity and stability of the phosphorus analytical standard solution is responsibility of the Sponsor. The preparation of the standard solution was performed at the Sponsor's facilities at Oldbury (UK) and this procedure was witnessed and documented by a member of Harlan Laboratories Ltd. staff with management responsibilities to maintain study integrity.

Sponsor's identification	:	Technical Yellow Phosphorus Certificate #538 Purity 99.83%
Standard solution concentration	:	1.073×10^3 mg/L in iso-octane
Suppliers reference	:	753JD113
Date received at test facility	:	12 June 2012
Expiry date	:	12 July 2012
Storage conditions	:	approximately -20°C, in the dark

3 DETERMINATION OF WHITE PHOSPHORUS RESIDUES

3.1 Principle of the Test

The determination was carried out using a procedure based on Johnston, JJ, Goldade, DA, Kohler DJ, Cummings, JL (2000), Determination of white phosphorus residues in ducks: an atomic emission detection/compound-independent calibration-based method of generating residue data for risk assessment and environmental monitoring. Environ Sci Technol 34(9):1856-1861.

3.2 Performance of the Test

3.2.1 Standard Solution Preparation

The stock standard solution of white phosphorus (see section 2.2) was prepared as follows:

A white phosphorus pellet, stored in deionized water, was dried using acetone and nitrogen prior to weighing (0.1073 g) and then diluted to a volume of 100 mL with iso-octane.

At the test facilities in Shardlow, the stock standard solution was ultrasonicated to fully dissolve the pellet of white phosphorus and homogenize the solution.

3.2.2 Sample Preparation

The samples were defrosted by placing the containers in a 20 °C nominal temperature water bath for a minimum of 1 hour. The swan and goose gizzards samples required considerably longer defrosting periods due to their volume and therefore they were stored in a fridge (approximately 4 °C) overnight prior to dissecting.

Degassed water was prepared by boiling purified water vigorously and then purging with nitrogen as it cooled.

Gizzard Contents

Each gizzard was dissected longitudinally between the crushing plates to expose its contents, which were transferred to a glass jar. The exposed gizzard was then quantitatively rinsed using 60 mL of degassed purified water, collecting the rinses in the same glass jar.

Intestine Contents

Each intestine was dissected longitudinally and its contents transferred to a glass jar. The remaining tissue was then quantitatively rinsed using 60 mL of degassed purified water, collecting the rinses in the same glass jar.

Tissue Samples

For each bird, the fat, kidney, muscle, liver, intestine and gizzard tissues were cut into small sections and a portion was weighed in a glass jar (see following table).

Table 3.1

Tissue Sample	Mass of Tissue (g)
Swan fat	5.3762
Swan kidney	5.1488
Swan muscle	5.8313
Swan liver	5.8245
Swan intestine	5.0950
Swan gizzard	5.1408
Goose fat	5.0509
Goose kidney	5.3389
Goose muscle	5.5291
Goose liver	5.3543
Goose intestine	5.0639
Goose gizzard	5.1113
Duck fat	5.1493
Duck kidney	5.0010
Duck muscle	5.3782
Duck liver	5.2260
Duck intestine	3.3431
Duck gizzard	5.1264

20 mL of degassed purified water was added to each tissue sample and then macerated for at least 1 minute. Degassed purified water (10 mL) was used to quantitatively rinse the macerator blades, adding the rinsings to the sample.

Extraction of Samples for Analysis

10 mL of iso-octane was added to the tissue samples (suspended in 30 mL of degassed purified water) and 20 mL of iso-octane was added to the intestine and gizzard contents (suspended in 60 mL of degassed purified water). The headspace of all the glass jars were filled with nitrogen before shaking the samples on a horizontal flat bed shaker at approximately 150 rpm for 18 hours. During this period, the samples were kept at ambient temperature and protected from light.

The samples were allowed to stand for approximately 1 hour at room temperature prior to decanting into glass centrifuge tubes and were centrifuged at 2500 rpm for 15 minutes. The iso-octane extracts were then removed to clean glass vessels and an aliquot taken for analysis in amber vials.

Additional procedural steps were taken to provide adequate supernatants for analysis as follows:

- Swan kidney and liver: Samples were centrifuged for a further 30 minutes at 2500 rpm.
- Goose liver: Samples were centrifuged for a further 30 minutes at 2500 rpm. Still insufficient supernatant was obtained and so a further 5 mL of iso-octane was added to the whole sample. This was shaken for 1 minute and centrifuged for 15 minutes at 2500 rpm.
- Goose gizzard tissue: Samples were centrifuged for a further 30 minutes at 2500 rpm.
- Duck muscle, fat and gizzard tissue: Samples were centrifuged for a further 60 minutes at 2500 rpm.
- Duck liver: The sample was centrifuged for a further 60 minutes at 2500 rpm. Still insufficient supernatant was obtained and so a further 5 mL of iso-octane was added to the whole sample. This was shaken for 1 minute and centrifuged for 15 minutes at 2500 rpm.

Sample Blank

A sample blank was prepared by shaking a mixture of 30 mL of degassed purified water with 10 mL of iso-octane as detailed for the samples.

3.2.3 Analysis

Samples

Preliminary analysis of the samples showed that the following dilutions were required:

Sample:		Dilution factor:
Swan intestine content	:	x 10
Swan gizzard content	:	x 1000
Goose gizzard content	:	x 5000
Duck gizzard content	:	x 100
Duck fat	:	x 20

The dilutions were performed in duplicate using iso-octane. The remaining samples were analyzed undiluted.

The iso-octane extracts were placed in a freezer and stored at approximately -20 °C, in the dark.

Sample blank

Iso-octane extract of the sample blank mixture.

Standards

Dilutions of the stock standard solution of white phosphorus were prepared in iso-octane to cover a nominal concentration range of 0.005 to 0.25 mg/L.

Standard blank

Iso-octane.

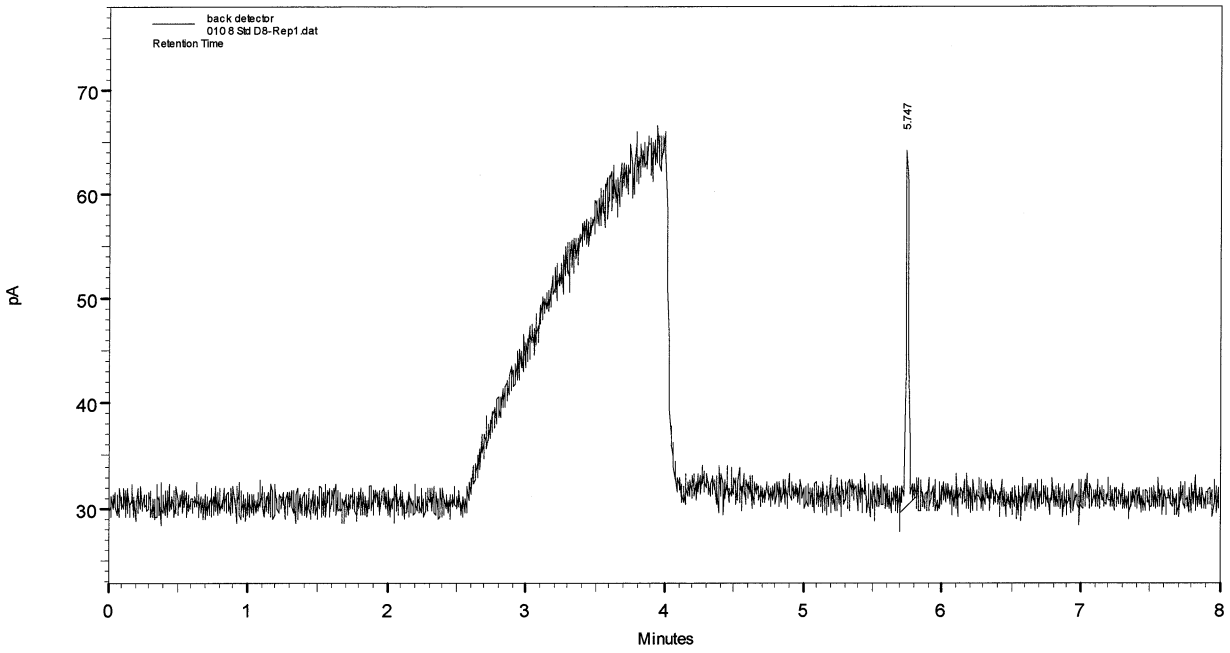
Analysis

The concentration of white phosphorus in the standard and sample solutions was analyzed by gas chromatography (GC) with flame photometric detection (FPD) using the following conditions:

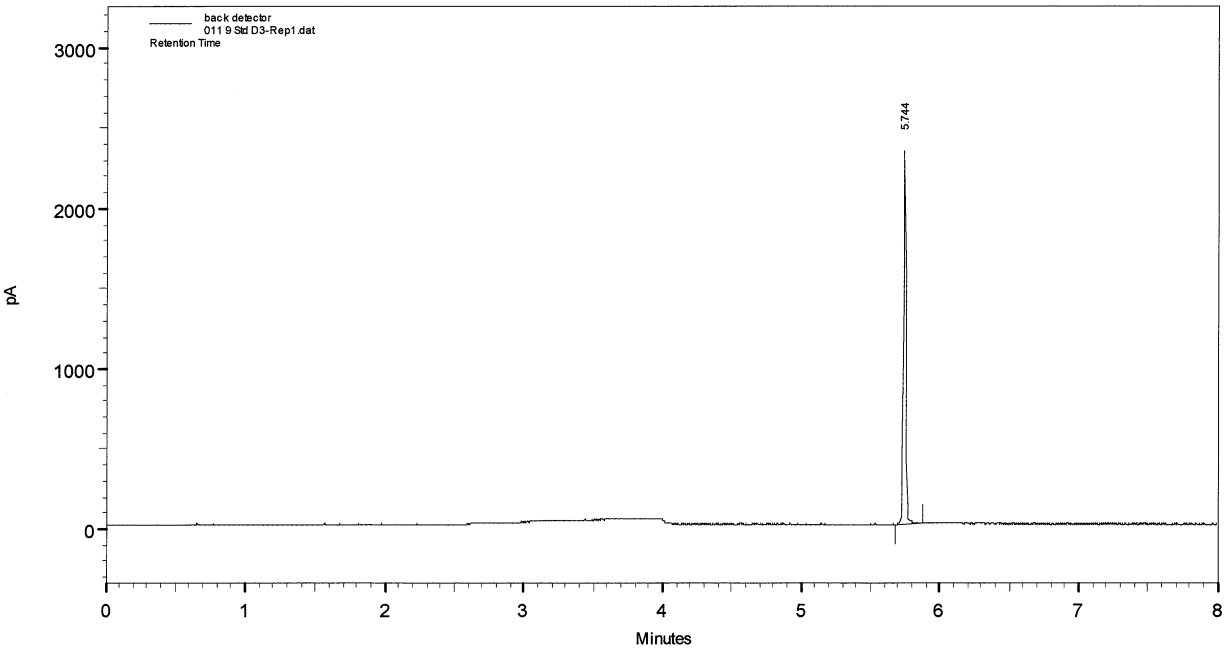
GC System	:	Agilent Technologies 6890, incorporating workstation
Column	:	DB-1 or TRB-1 (30 m x 0.25 mm id x 0.25 µm film)
Oven temperature program	:	Initial 40 °C for 0.5 minutes Rate 20 °C/minute Final 150 °C for 2 minutes
Injection temperature	:	250 °C
FPD mode	:	Phosphorus
FPD temperature	:	250 °C
Injection volume	:	2 µL
Injection mode	:	Splitless (purge on at 0.5 minute)
Carrier gas	:	Nitrogen
Flow rate	:	0.7 mL/minute
Pressure	:	8.1 psi (constant pressure)
Retention time	:	~6 minutes

Typical Chromatography

Standard Solution 5.37 x 10^{-3} mg/L

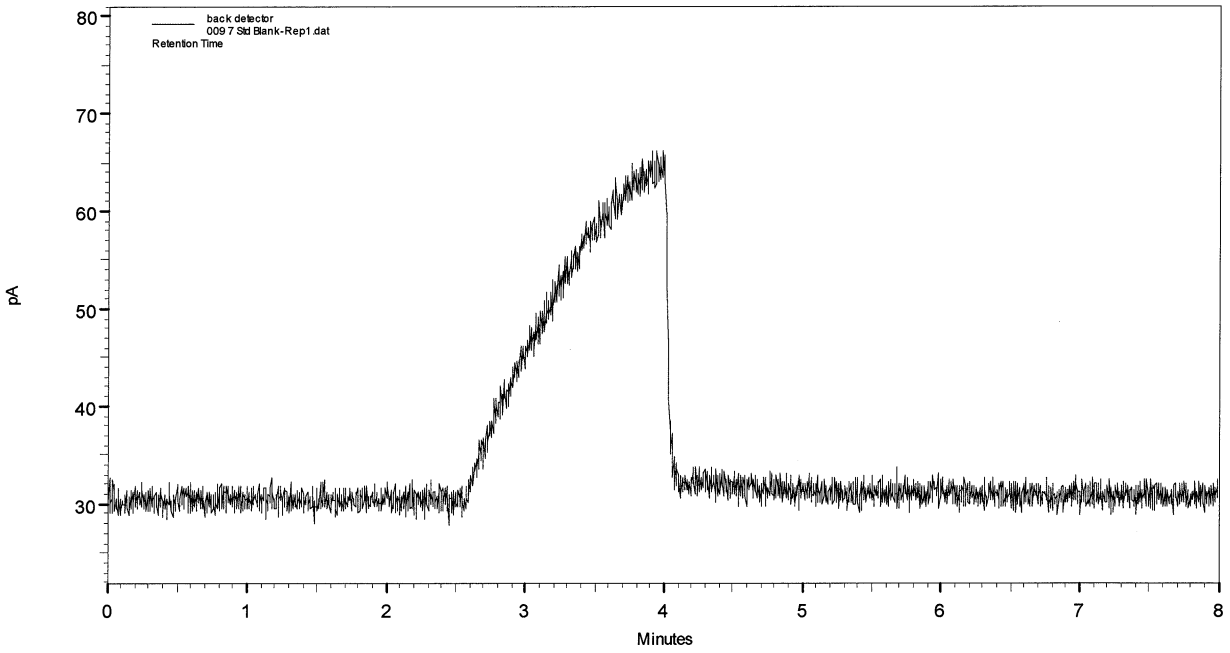


Standard Solution 0.269 mg/L

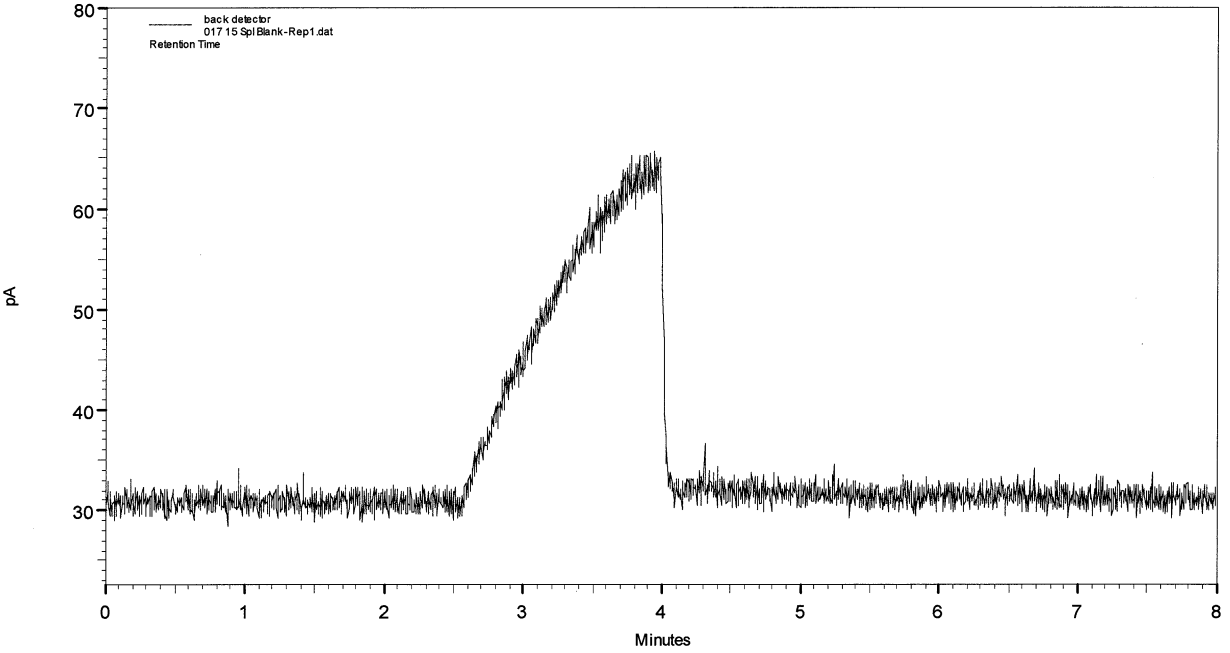


Typical Chromatography

Solvent Blank

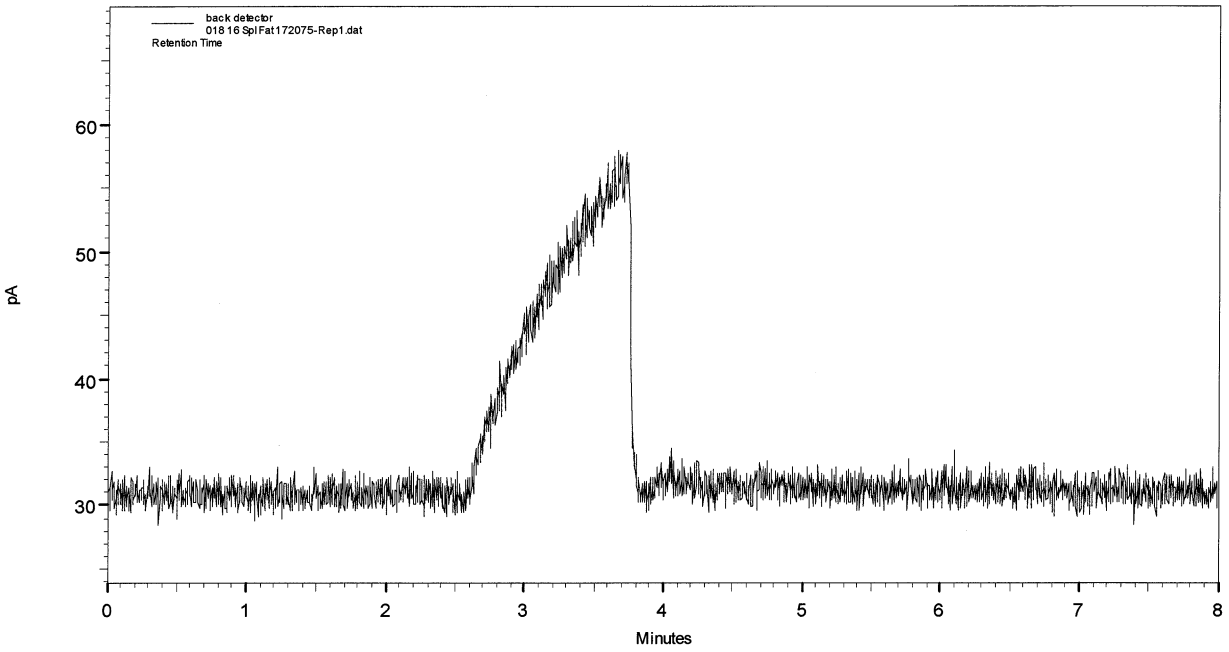


Sample Blank

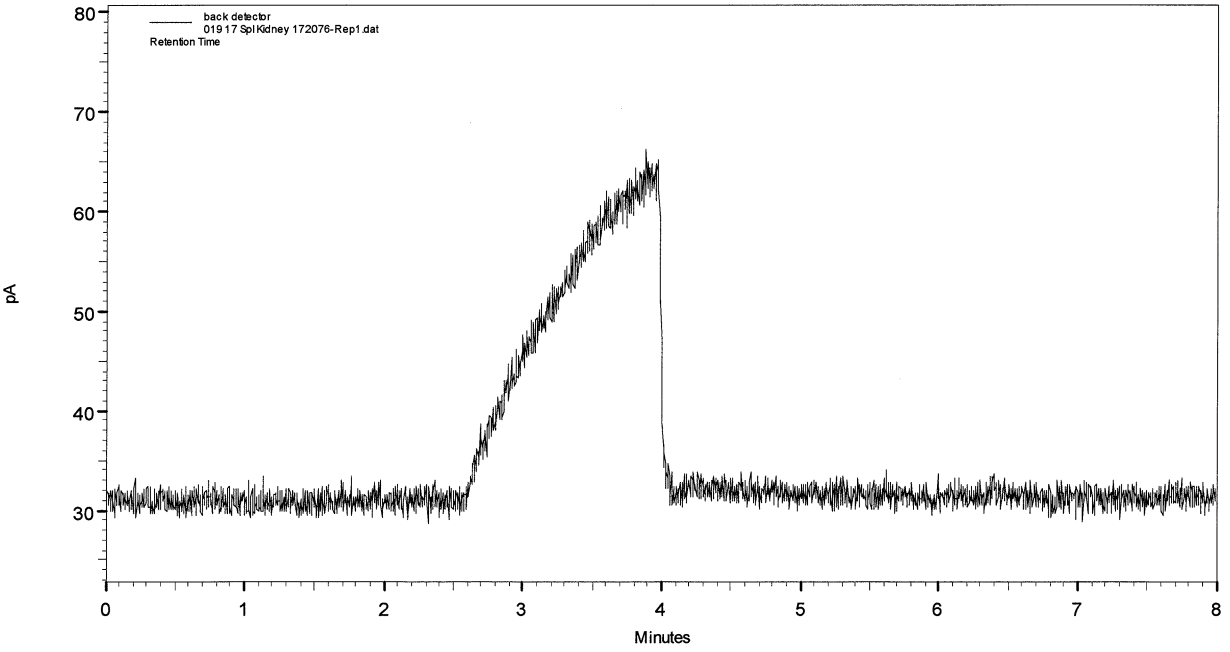


Typical Chromatography

Swan Fat

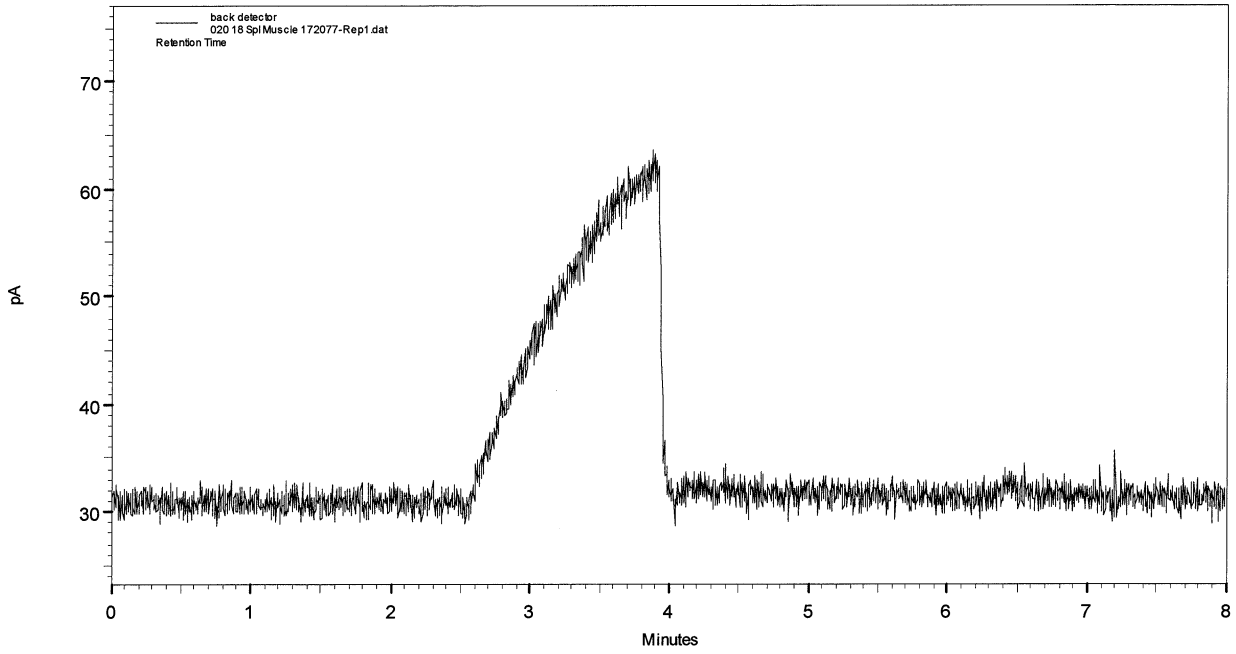


Swan Kidney

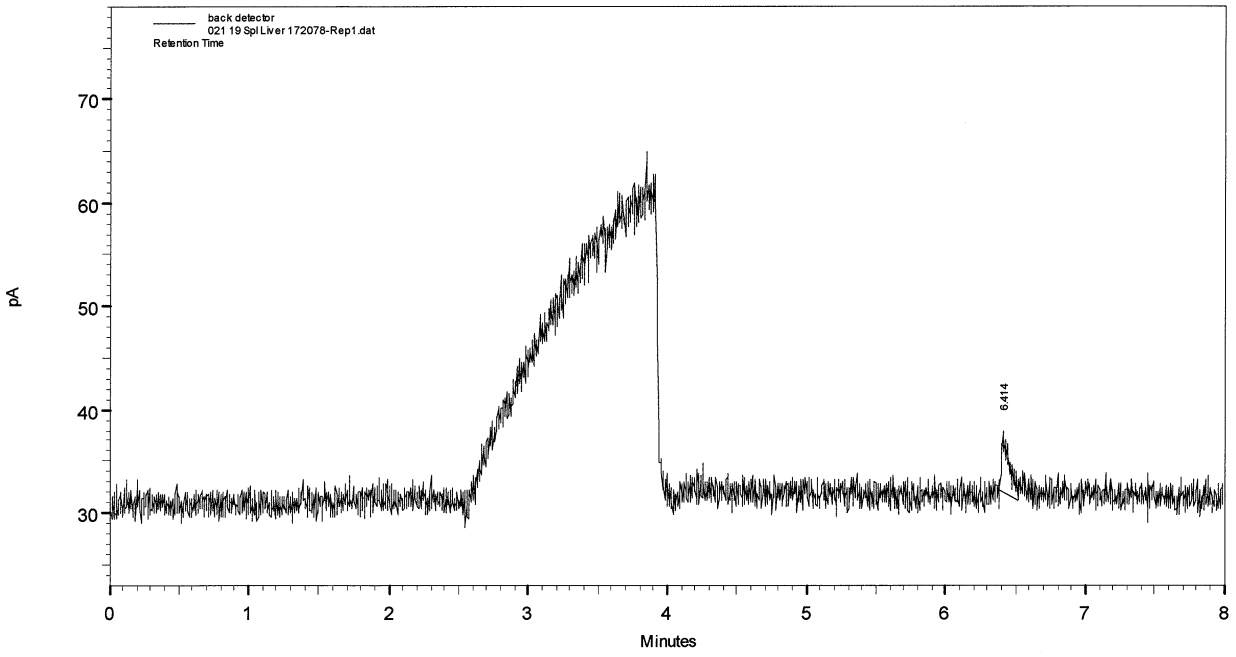


Typical Chromatography

Swan Muscle

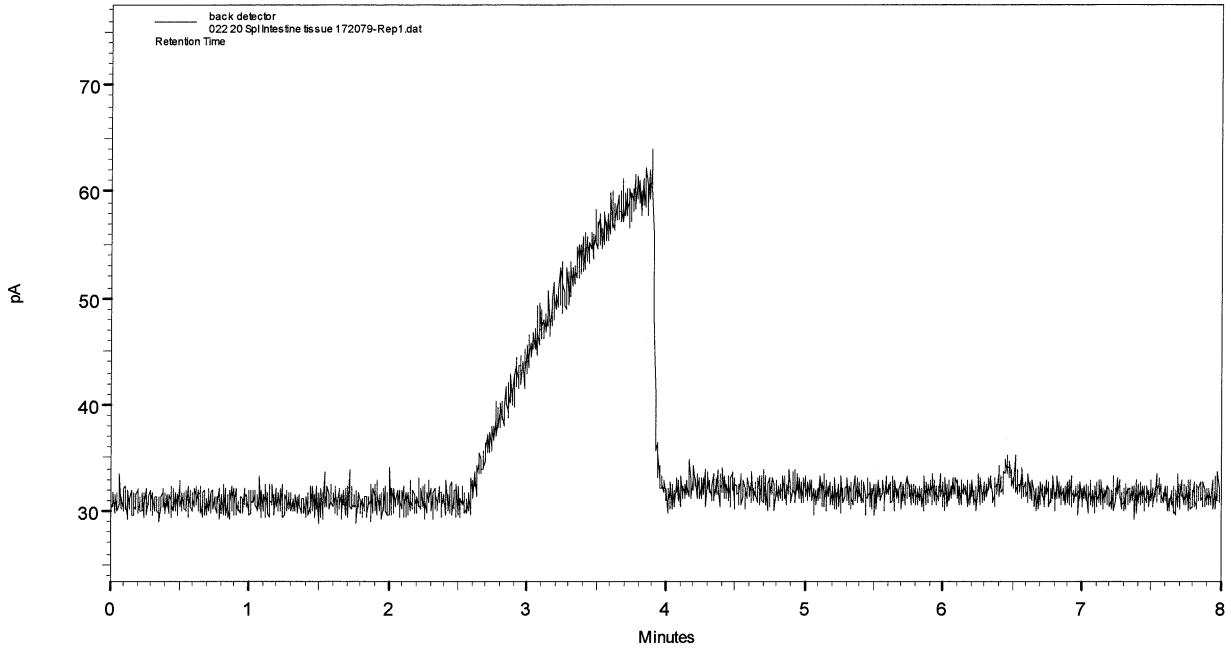


Swan Liver

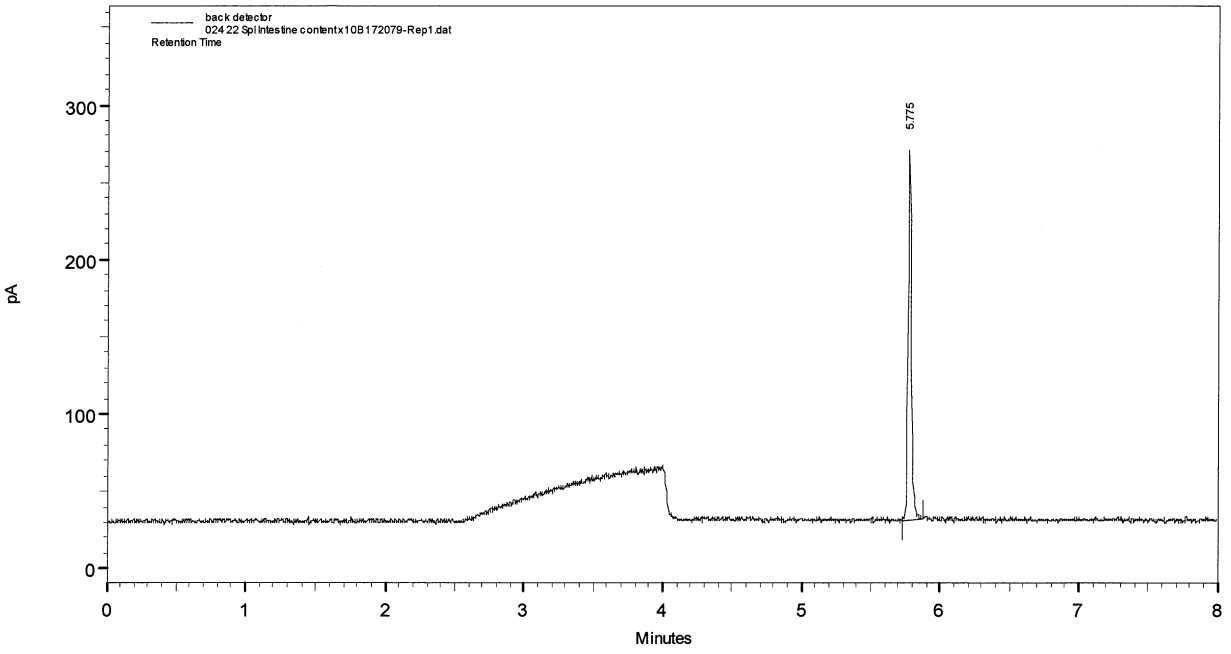


Typical Chromatography

Swan Intestine Tissue

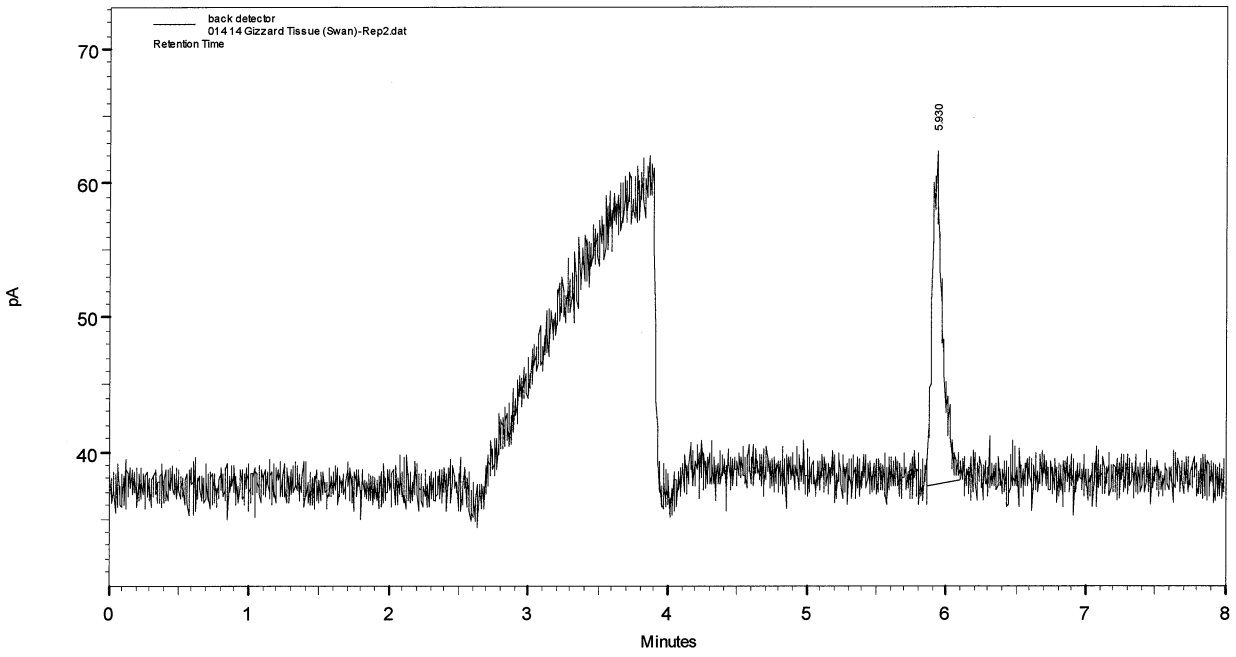


Swan Intestine Contents (x 10 dilution)

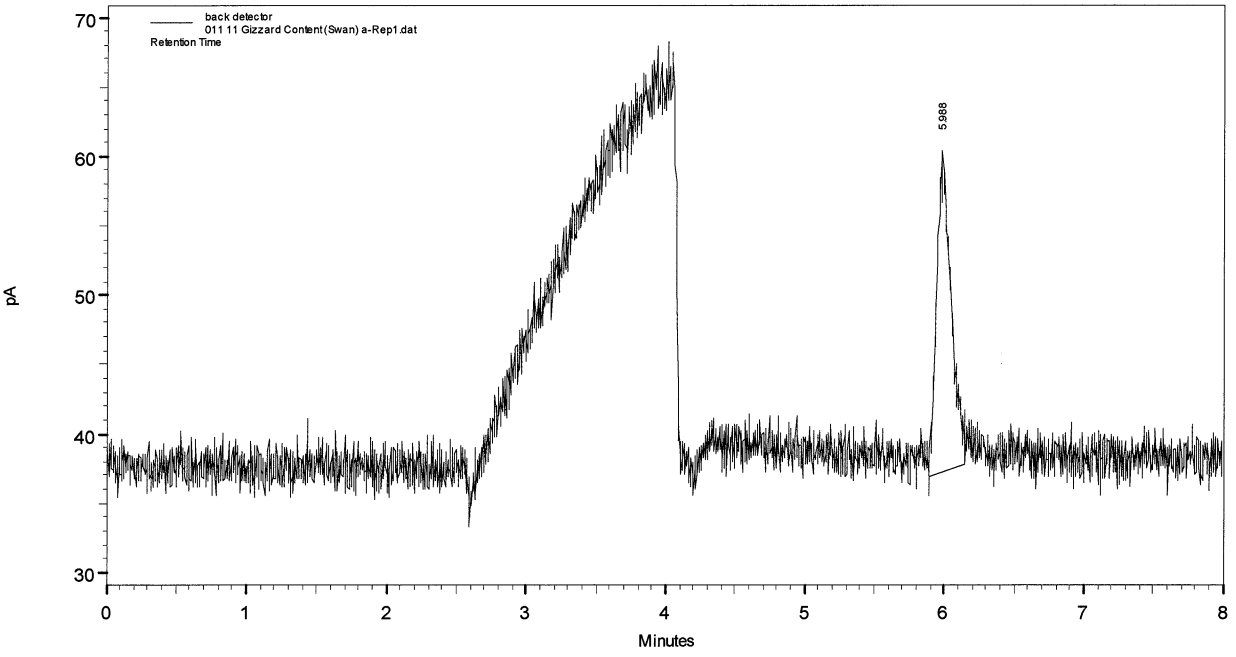


Typical Chromatography

Swan Gizzard Tissue

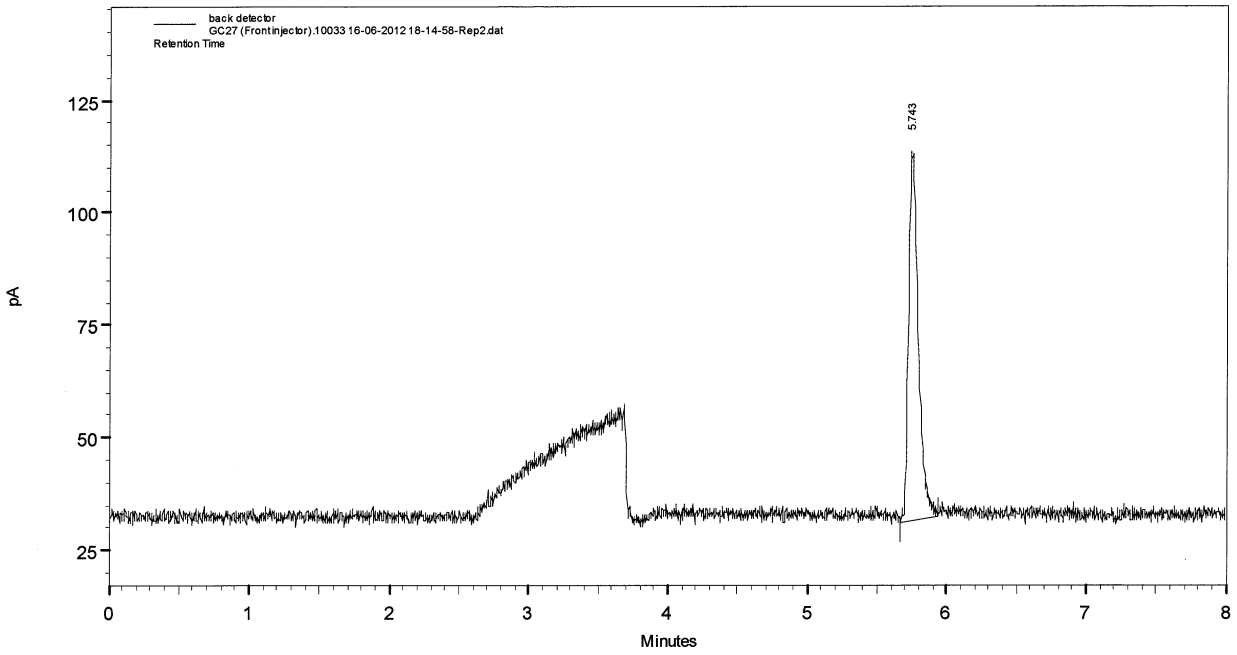


Swan Gizzard Contents (x 1000 dilution)

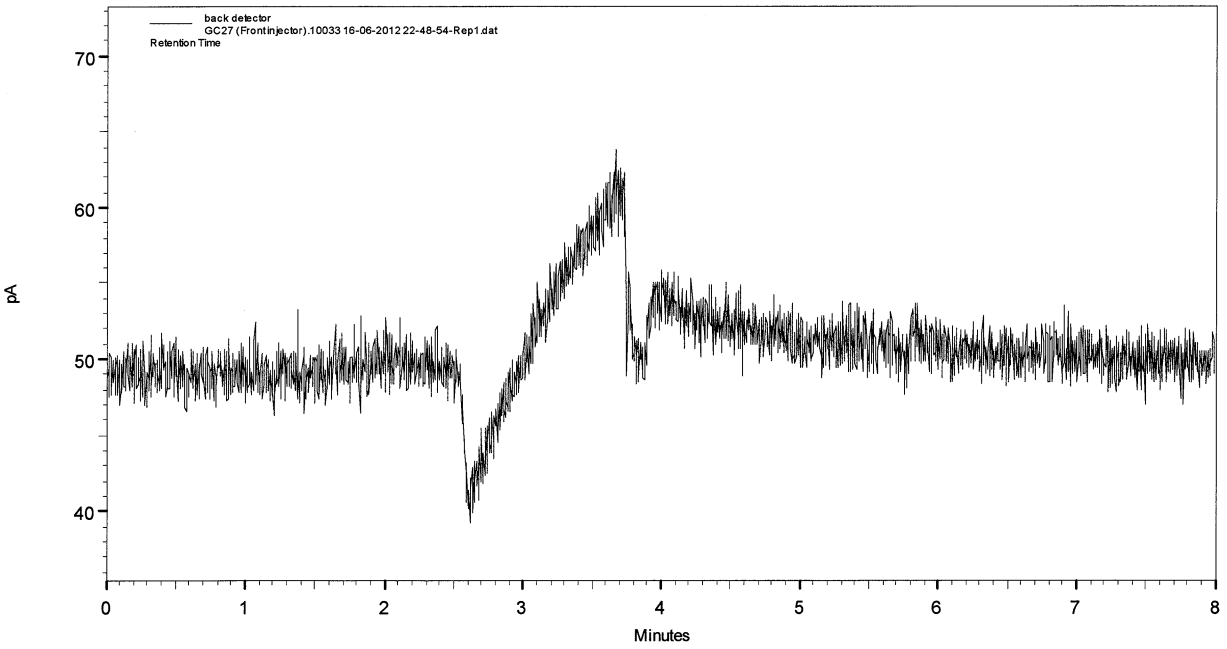


Typical Chromatography

Goose Fat

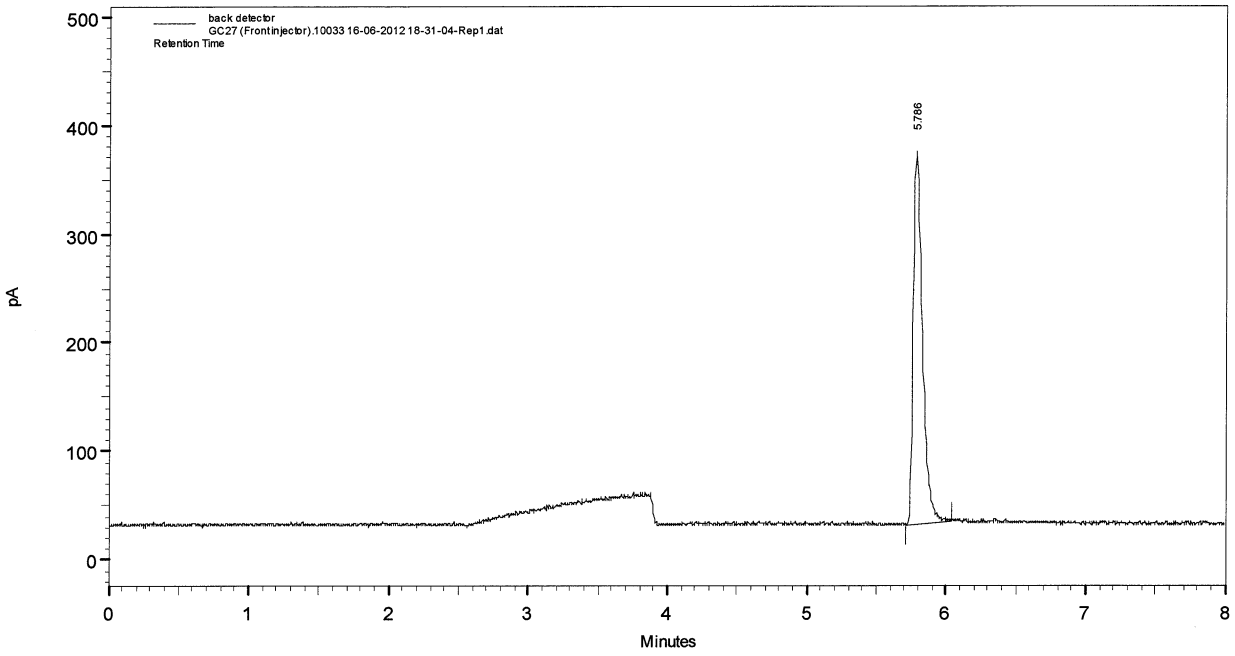


Goose Kidney

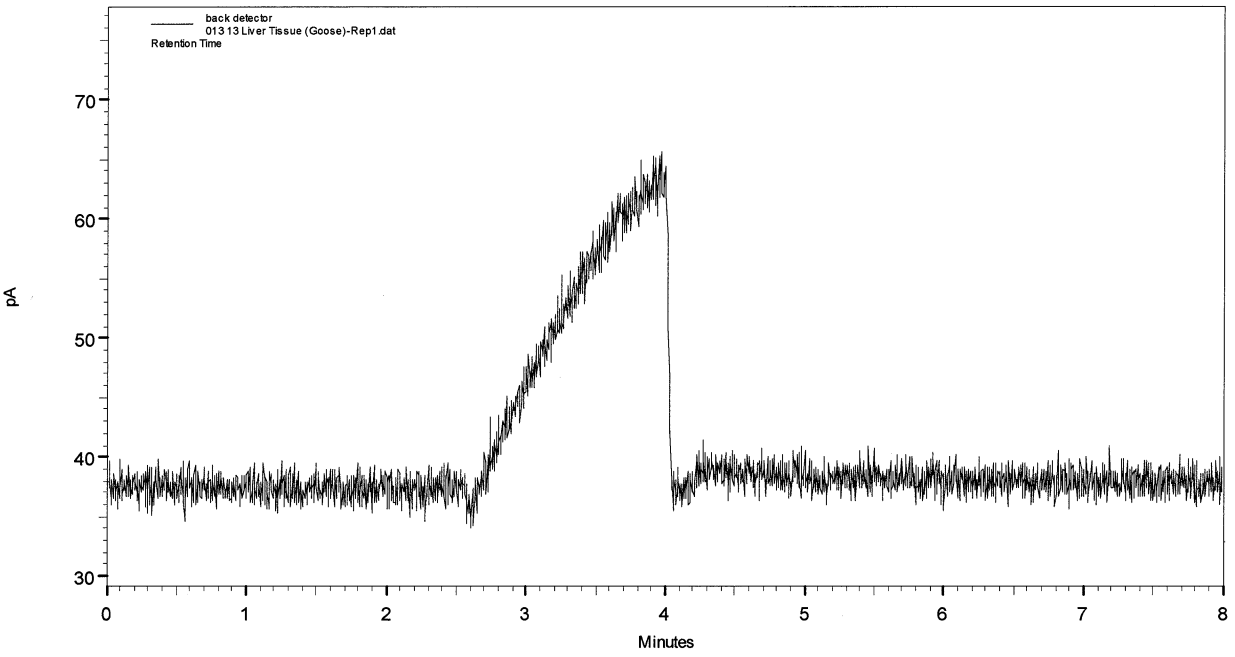


Typical Chromatography

Goose Muscle

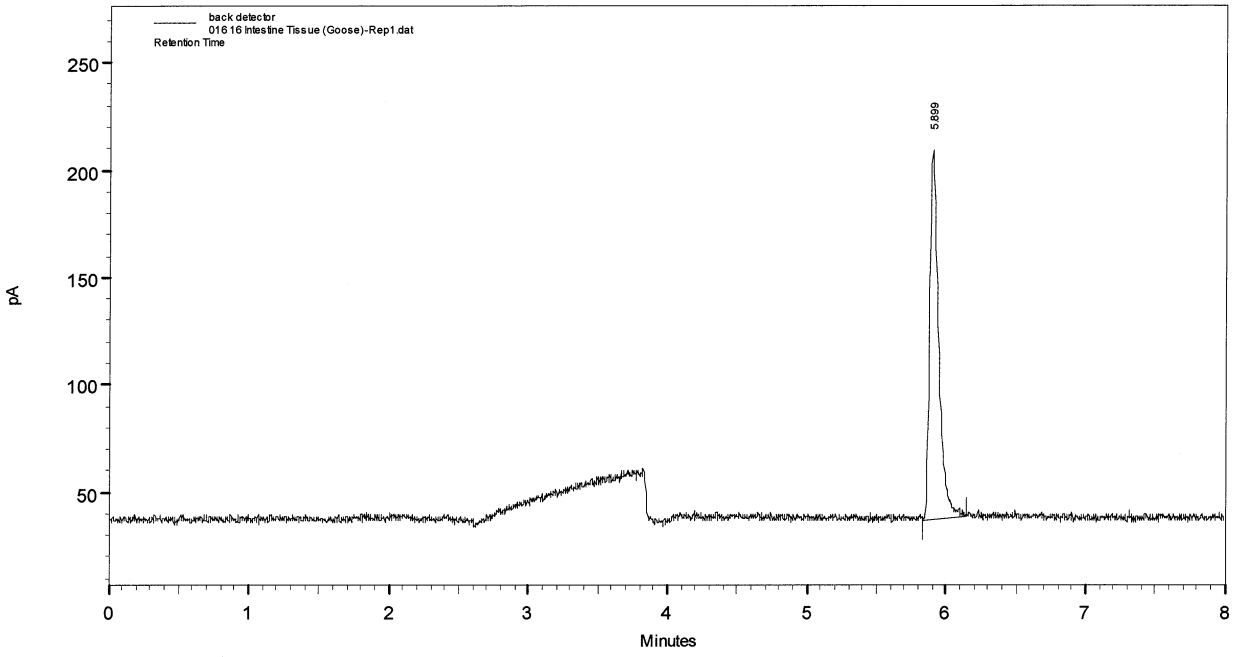


Goose Liver

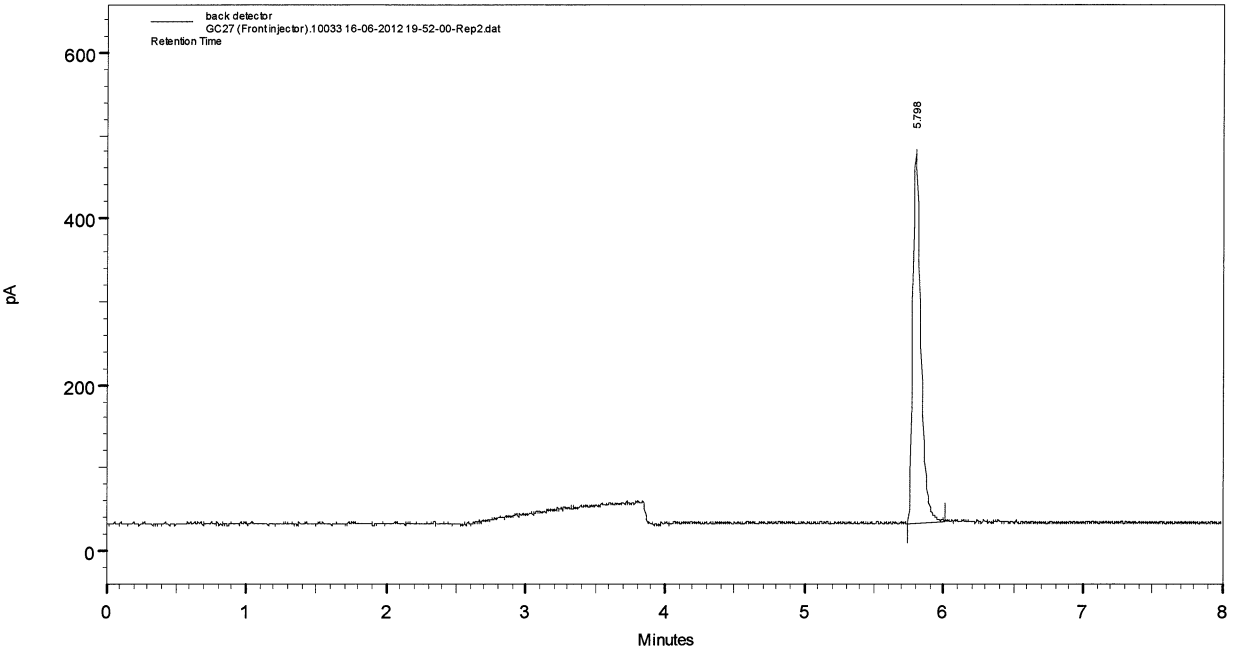


Typical Chromatography

Goose Intestine Tissue

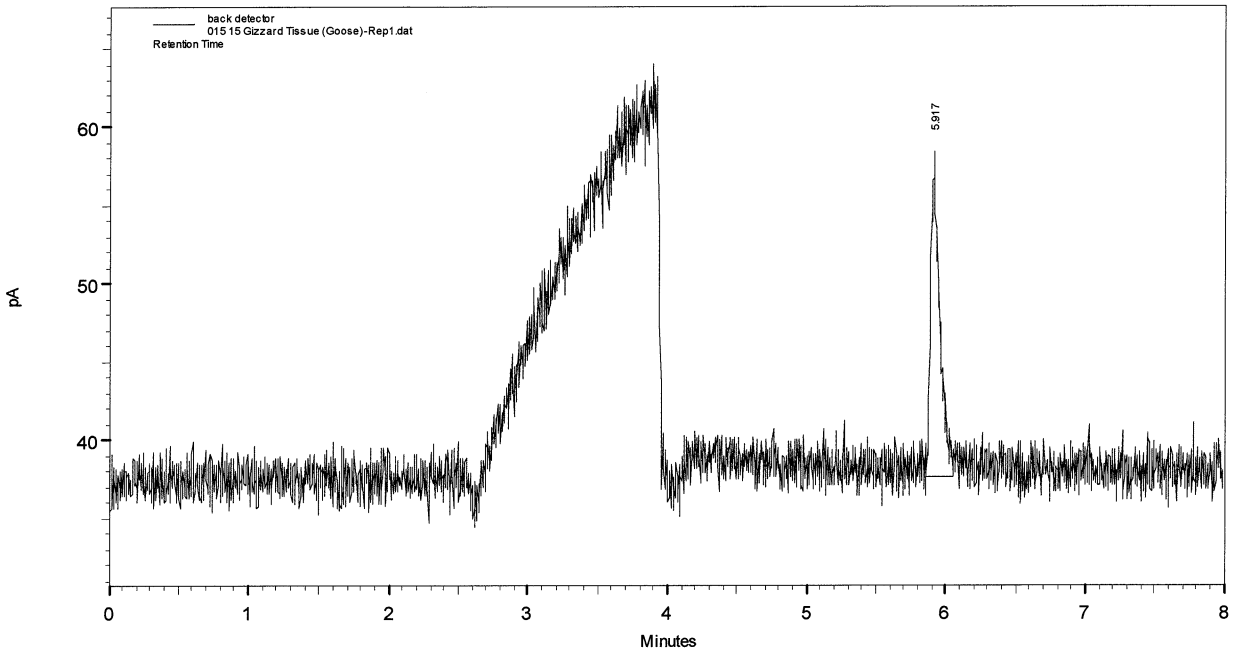


Goose Intestine Contents

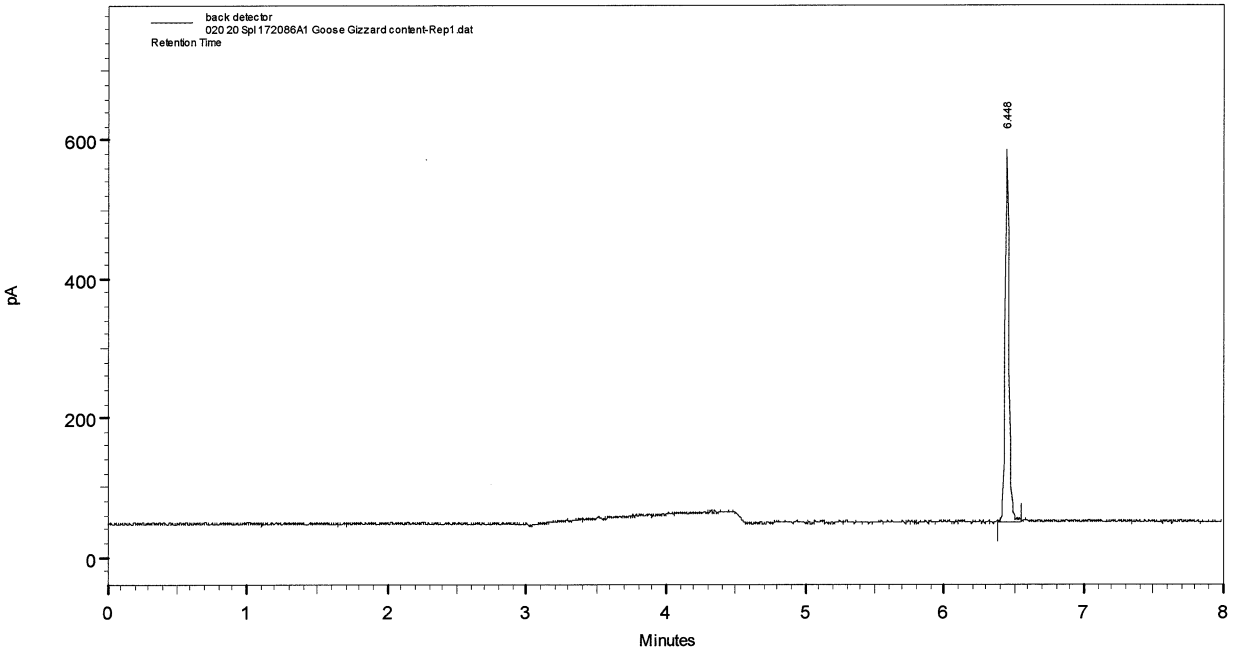


Typical Chromatography

Goose Gizzard Tissue

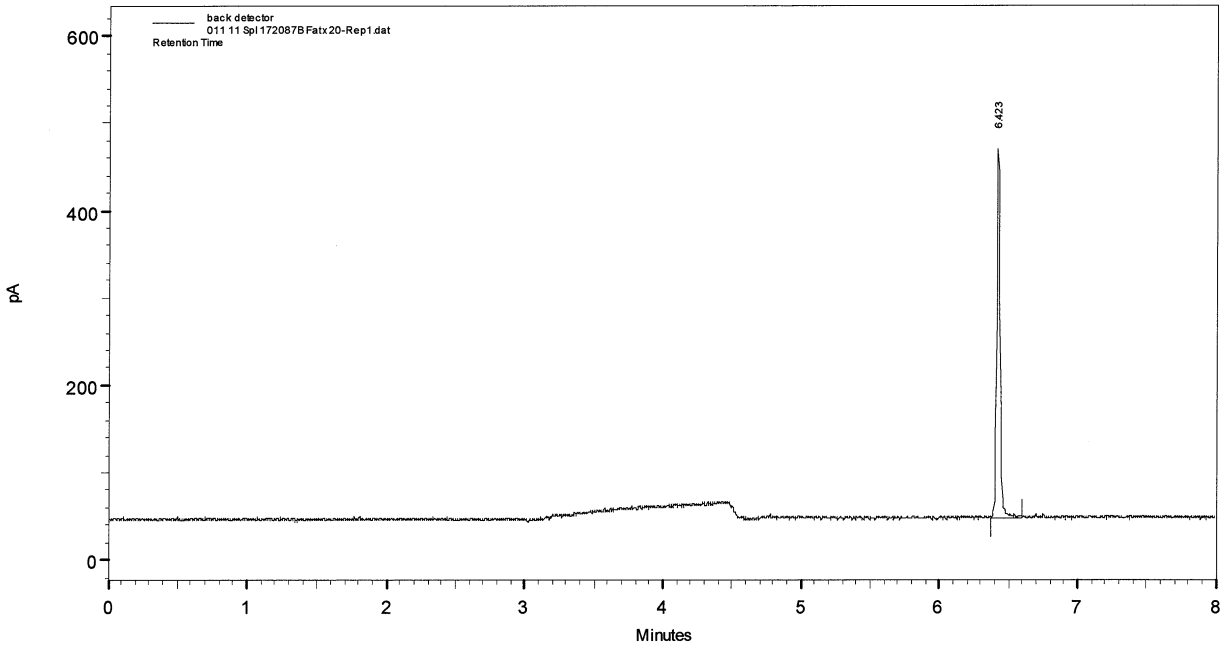


Goose Gizzard Contents (x 5000 dilution)

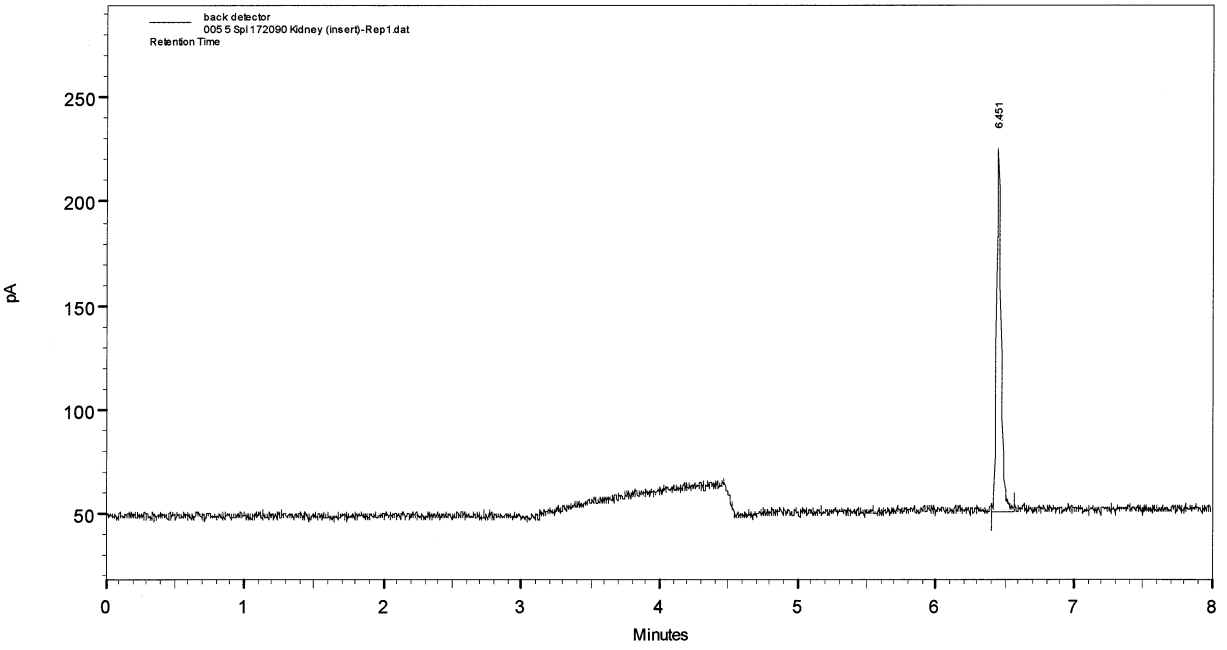


Typical Chromatography

Duck Fat (x 20 dilution)

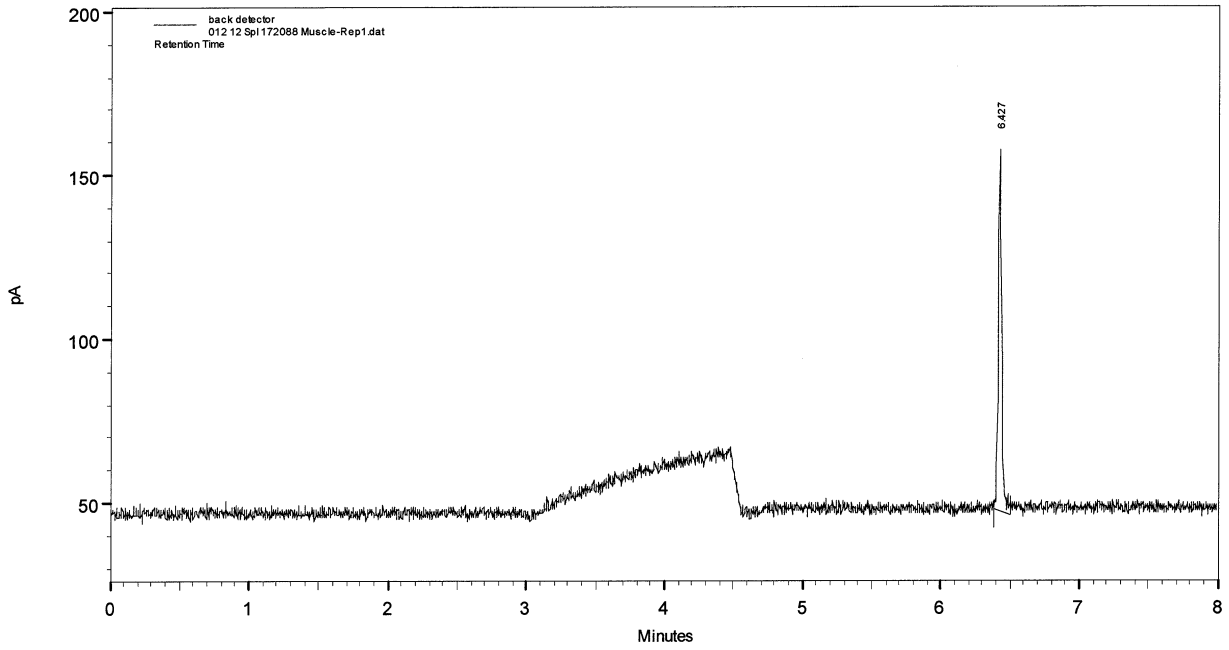


Duck Kidney

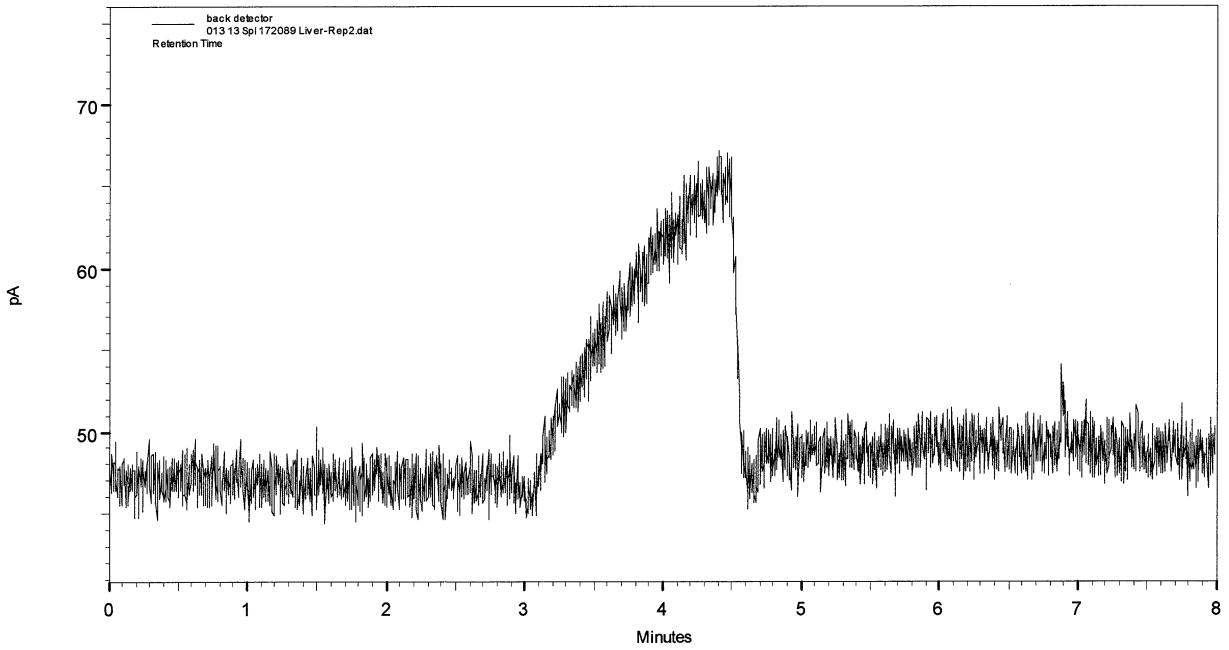


Typical Chromatography

Duck Muscle

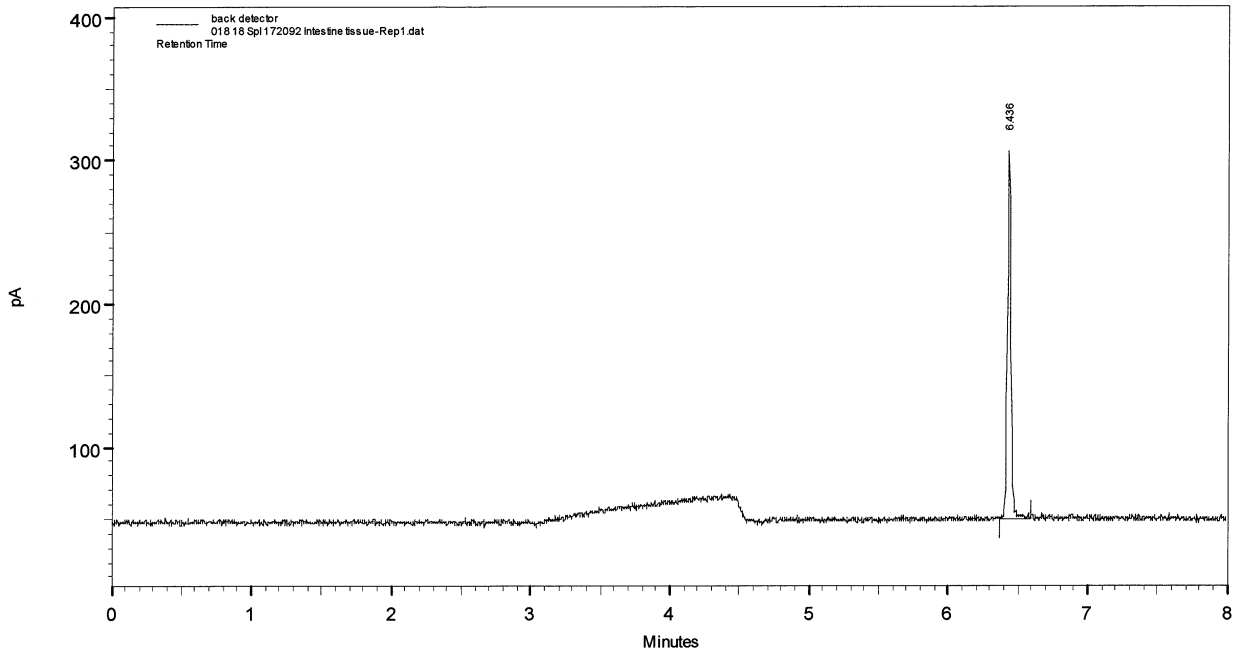


Duck Liver

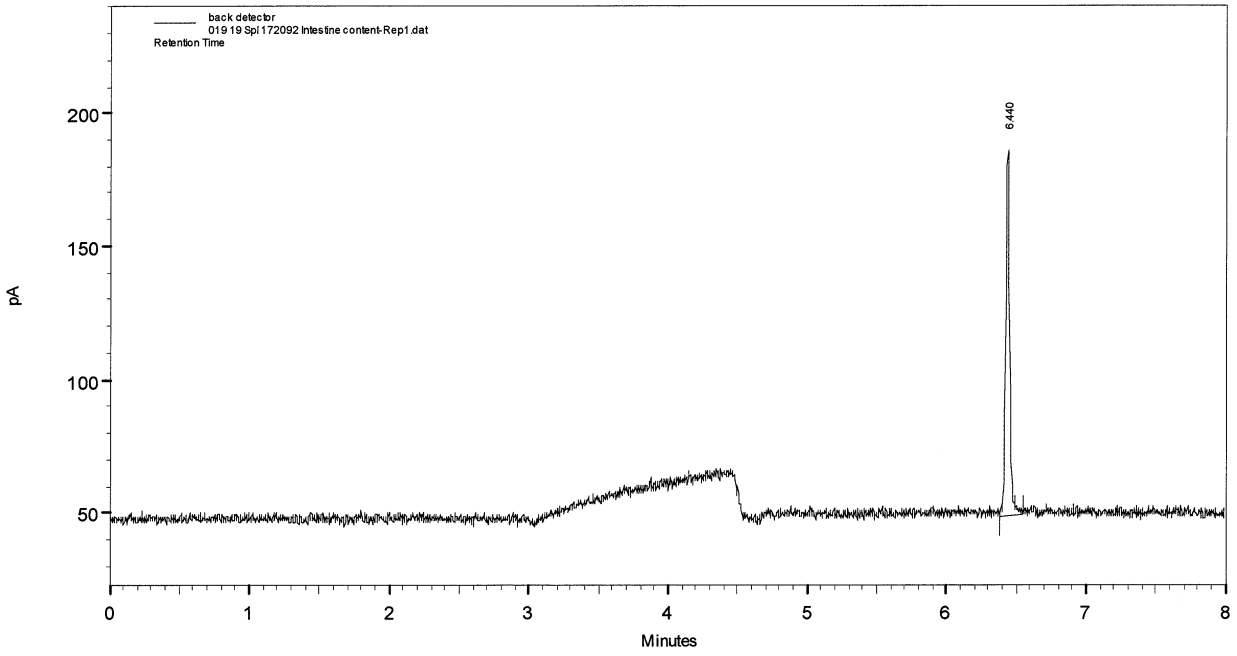


Typical Chromatography

Duck Intestine Tissue

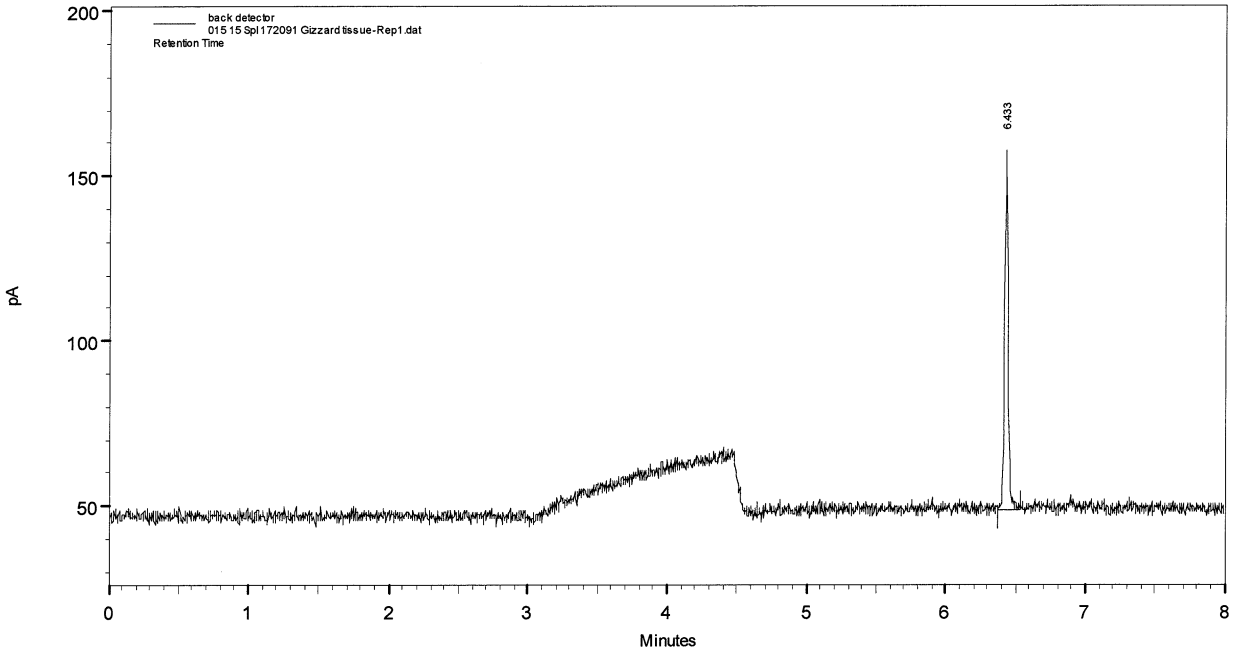


Duck Intestine Contents

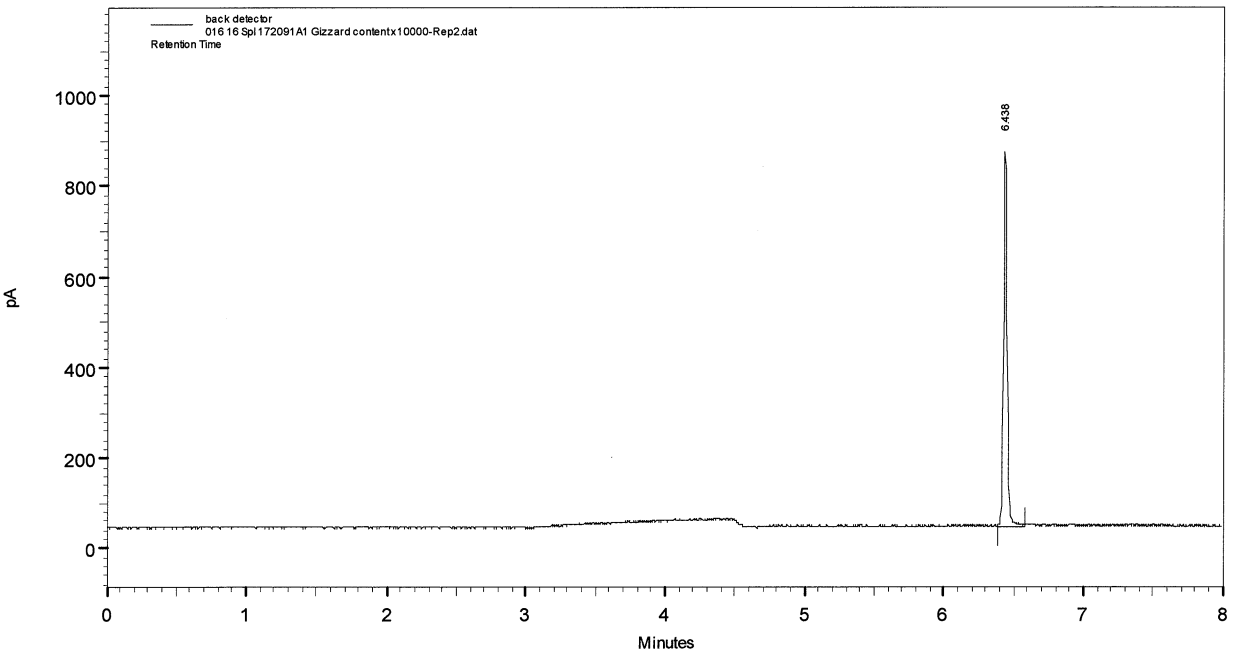


Typical Chromatography

Duck Gizzard Tissue



Duck Gizzard Contents (x 100 dilution)



3.3 Data Handling

The mean peak area and concentration of each standard were plotted on a calibration curve and the sample concentration (mg/L) interpolated from the curve. The concentration was then corrected for the purity of the standard and the dilution factor (if relevant).

The white phosphorus content (mg) present in gizzard contents and intestine contents was calculated using Equation 3.1.

Equation 3.1

$$M_{wp} = C_{wp} \times \frac{V_{iso}}{1000} \times D \times \frac{P}{100}$$

Where:

M_{wp}	=	mass of white phosphorus present in contents (mg)
C_{wp}	=	concentration of white phosphorus determined in the sample solution (mg/L)
V_{iso}	=	volume of iso-octane used for extraction (mL)
D	=	dilution factor
P	=	purity of white phosphorus standard (99.83%)

The white phosphorus residue (mg/kg) in tissue samples was calculated using Equation 3.2.

Equation 3.2

$$R_{wp} = C_{wp} \times V_{iso} \times \frac{1}{M_{tis}} \times D \times \frac{P}{100}$$

Where:

R_{wp}	=	residue of white phosphorus (mg/kg)
C_{wp}	=	concentration of white phosphorus determined in the sample solution (mg/L)
V_{iso}	=	volume of iso-octane used for extraction (mL)
M_{tis}	=	mass of tissue taken for extraction (g)
D	=	dilution factor
P	=	purity of white phosphorus standard (99.83%)

The sample of duck kidney tissue was reanalyzed using bracketing standards instead of a calibration curve as the detector had demonstrated linear response in the previous analyses.

The response factors of the standard peak areas (unit peak area per mg/L) were calculated using Equation 3.3.

Equation 3.3

$$RF = \frac{R_{STD}}{C_{STD}}$$

Where:

RF = response factor for the standard solution
R_{STD} = peak area for the standard solution
C_{STD} = concentration for the standard solution (mg/L)

The white phosphorus residue (mg/kg) in the tissue sample was calculated using Equation 3.4.

Equation 3.4

$$R_{wp} = \frac{R_{SPL}}{RF_{STD}} \times V_{iso} \times \frac{1}{M_{tis}} \times D \times \frac{P}{100}$$

Where:

R_{wp} = residue of white phosphorus (mg/kg)
R_{SPL} = mean peak area for the sample solution
RF_{STD} = mean response factor for the standard solutions (unit peak area per mg/L)
V_{iso} = volume of iso-octane used for extraction (mL)
M_{tis} = mass of tissue taken for extraction (g)
D = dilution factor
P = purity of white phosphorus standard (99.83%)

3.4 Results

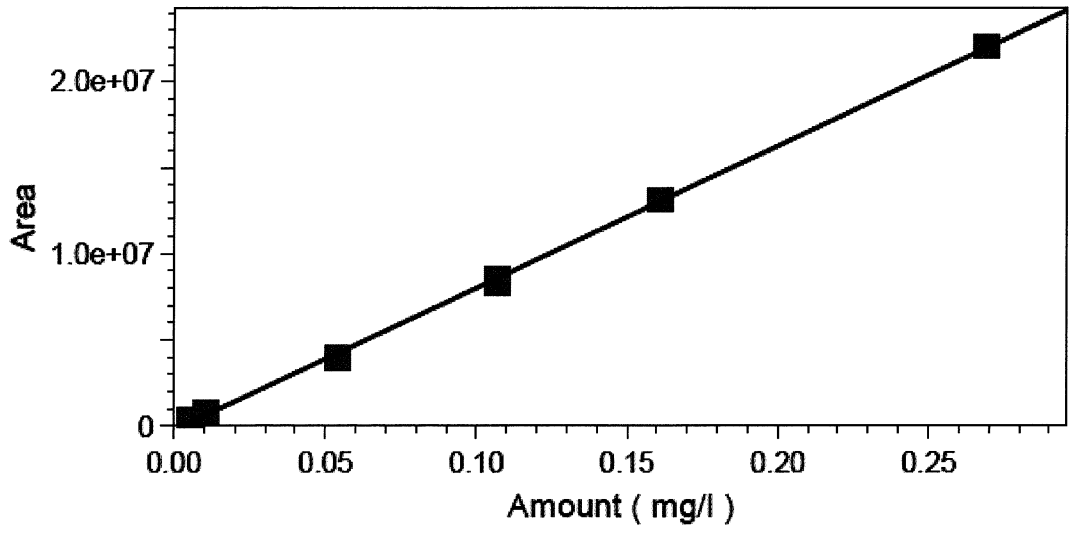
The mean peak areas relating to the standards and the swan sample solutions (except gizzard tissue and contents) are shown in the following table:

Table 3.2

Solution	Mean Peak Area
Standard Blank	None detected
Standard 5.37 x 10^{-3} mg/L	3.975 x 10^{5}
Standard 1.07 x10^{-2} mg/L	8.097 x 10^{5}
Standard 5.37 x 10^{-2} mg/L	3.991 x 10^{6}
Standard 0.107 mg/L	8.331 x 10^{6}
Standard 0.161 mg/L	1.314 x 10^{7}
Standard 0.269 mg/L	2.205 x 10^{7}
Standard 0.107 mg/L	8.564 x 10^{6}
Sample Blank	None detected
Swan Fat	None detected
Swan Kidney	None detected
Swan Muscle	None detected
Swan Liver	None detected
Swan Intestine Tissue	None detected
Swan Intestine Content	3.044 x 10^{6}

The calibration curve for the analysis from Table 3.2 is shown in Figure 3.1:

Figure 3.1



y = 8.24427e+007x - 223278.
Goodness of fit (r^2): 0.999460

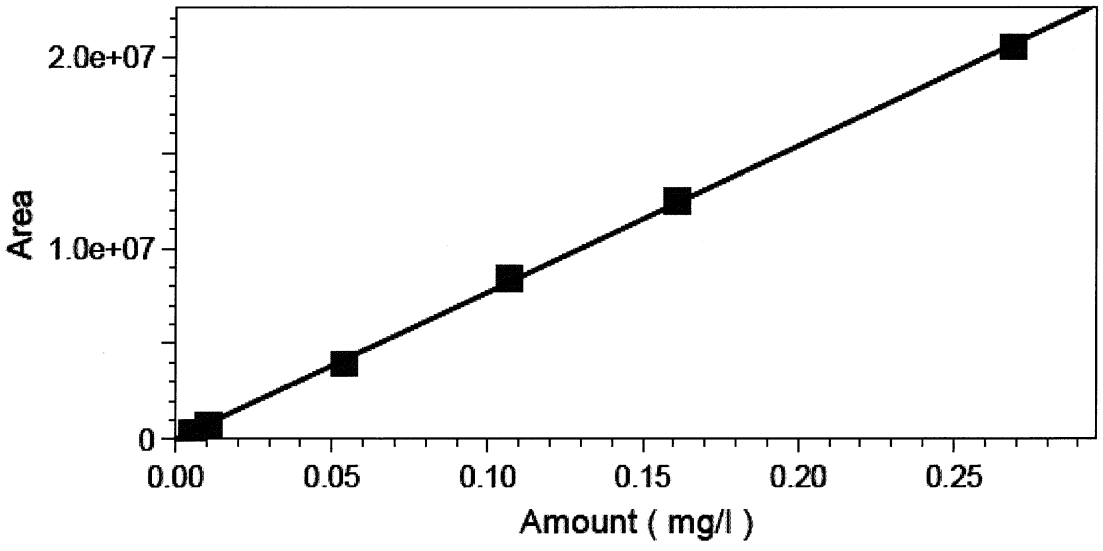
The mean peak areas relating to the standards and goose fat, liver, kidney and intestine content sample solutions are shown in the following table:

Table 3.3

Solution	Mean Peak Area
Standard Blank	None detected
Standard 5.37 x 10^{-3} mg/L	3.878 x 10^{5}
Standard 1.07 x10^{-2} mg/L	7.510 x 10^{5}
Standard 5.37 x 10^{-2} mg/L	3.942 x 10^{6}
Standard 0.107 mg/L	8.368 x 10^{6}
Standard 0.161 mg/L	1.248 x 10^{7}
Standard 0.269 mg/L	2.051 x 10^{7}
Standard 0.107 mg/L	8.440 x 10^{6}
Sample Blank	None detected
Goose Fat	3.057 x 10^{6}
Goose Kidney	None detected
Goose Muscle	1.209 x 10^{7}
Goose Intestine Content	1.417 x 10^{7}

The calibration curve for the analysis from Table 3.3 is shown in Figure 3.2:

Figure 3.2



y = 7.68416e+007x + 4235.24
Goodness of fit (r^2): 0.999524

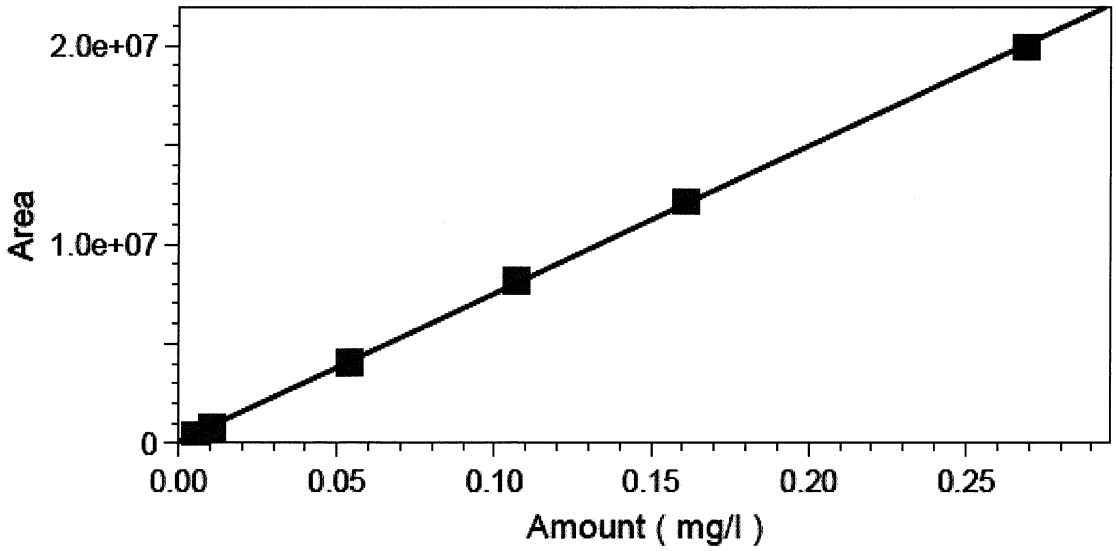
The mean peak areas relating to the standards, swan gizzard tissue and contents, goose liver, intestine and gizzard tissues, are shown in the following table:

Table 3.4

Solution	Mean Peak Area
Standard Blank	None detected
Standard 5.37 x 10^{-3} mg/L	4.389 x 10^5
Standard 1.07 x10^{-2} mg/L	8.026 x 10^5
Standard 5.37 x 10^{-2} mg/L	4.066 x 10^6
Standard 0.107 mg/L	8.233 x 10^6
Standard 0.161 mg/L	1.216 x 10^7
Standard 0.269 mg/L	1.993 x 10^7
Standard 0.107 mg/L	8.142 x 10^6
Swan Gizzard Tissue	9.826 x 10^5
Swan Gizzard Content	1.259 x 10^6
Goose Liver	None detected
Goose Intestine Tissue	5.940 x 10^6
Goose Gizzard Tissue	7.255 x 10^5

The calibration curve for the analysis from Table 3.4 is shown in Figure 3.3:

Figure 3.3



y = 7.42218e+007x + 112955.
Goodness of fit (r^2): 0.999679

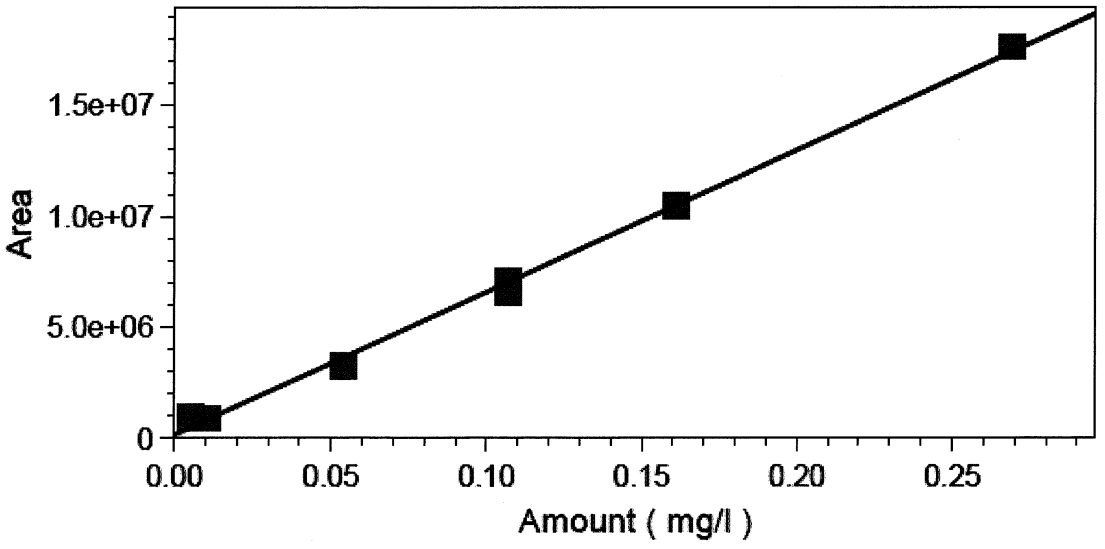
The mean peak areas relating to the standards, the goose gizzard contents and the duck sample solutions (except kidney) are shown in the following table:

Table 3.5

Solution	Mean Peak Area
Standard Blank	None detected
Standard 5.37 x 10^{-3} mg/L	9.664 x 10^5
Standard 1.07 x10^{-2} mg/L	9.119 x 10^5
Standard 5.37 x 10^{-2} mg/L	3.294 x 10^6
Standard 0.107 mg/L	6.596 x 10^6
Standard 0.107 mg/L	7.079 x 10^6
Standard 0.161 mg/L	1.048 x 10^7
Standard 0.269 mg/L	1.765 x 10^7
Sample Blank	None detected
Goose Gizzard Content	8.095 x 10^5
Duck Fat	5.753 x 10^6
Duck Muscle	1.510 x 10^6
Duck Liver	None detected
Duck Intestine Tissue	3.600 x 10^6
Duck Intestine Content	2.100 x 10^6
Duck Gizzard Tissue	1.489 x 10^6
Duck Gizzard Content	1.139 x 10^7

The calibration curve for the analysis from Table 3.5 is shown in Figure 3.4:

Figure 3.4



y = 6.41684e+007x + 167000.
Goodness of fit (r^2): 0.997391

The mean peak areas relating to the standards and the duck kidney sample are shown in the following table:

Table 3.6

Solution	Mean Peak Area
Standard Blank	None detected
Standard 1.07 $x10^{-2}$ mg/L	1.210×10^{6}
Standard 5.37 x 10^{-2} mg/L	3.870×10^{6}
Sample Blank	$5.507 \times 10^{5*}$
Duck Kidney	3.197×10^{6}

* The positive response for the sample blank in this analysis was attributed to contamination and therefore the duck kidney sample peak area has not been corrected.

There is no calibration curve for this analysis, as bracketing standards were used (see Section 3.3 for calculation).

The white phosphorus concentration determined in the analyzed tissue extracts is expressed as white phosphorus residue (mg/kg) in the original tissue in the following table:

Table 3.7

Tissue Sample	White Phosphorus Residue (mg/kg of tissue)
Swan fat	<LOD
Swan kidney	<LOD
Swan muscle	<LOD
Swan liver	<LOD
Swan intestine	<LOD
Swan gizzard	2.28×10^{-2}
Goose fat	7.85×10^{-2}
Goose kidney	<LOD
Goose muscle	0.284
Goose liver	<LOD
Goose intestine	0.155
Goose gizzard	1.61×10^{-2}
Duck fat	3.38
Duck kidney	6.91×10^{-2}
Duck muscle	3.89×10^{-2}
Duck liver	<LOD
Duck intestine	0.160
Duck gizzard	4.01×10^{-2}

The white phosphorus amount determined in the analyzed extracts of the gizzard contents and intestine contents is expressed as total white phosphorus content (mg) in the following table:

Table 3.8

Contents Sample	Total White Phosphorus Present (mg)
Swan Intestine contents	7.91×10^{-3}
Swan Gizzard contents	0.308
Goose Intestine contents	3.68×10^{-3}
Goose Gizzard contents	14.3
Duck Intestine contents	6.02×10^{-4}
Duck Gizzard contents	0.349

3.5 Limit of Detection

The limit of detection (LOD) for each analysis was based on three times baseline noise and calculated using the lowest calibration standard. Changes in the sensitivity of the instrumentation resulted in the LOD of each analysis to differ. For each tissue sample, the LOD was then calculated in terms of mg of white phosphorus per kg of sample weighed. For the gizzard and intestine contents, the LOD was calculated in terms of total mg of white phosphorus present in the sample. The LOD for each sample is presented in the following tables:

Table 3.9

Tissue Sample	Limit of Detection (LOD) (mg/kg of tissue)
Swan fat	4.53×10^{-3}
Swan kidney	4.73×10^{-3}
Swan muscle	4.18×10^{-3}
Swan liver	4.18×10^{-3}
Swan intestine	4.78×10^{-3}
Swan gizzard	1.38×10^{-2}
Goose fat	1.60×10^{-2}
Goose kidney	1.51×10^{-2}
Goose muscle	1.46×10^{-2}
Goose liver	1.98×10^{-2}
Goose intestine	1.40×10^{-2}
Goose gizzard	1.39×10^{-2}
Duck fat	2.30×10^{-3}
Duck kidney	4.93×10^{-3}
Duck muscle	2.20×10^{-3}
Duck liver	3.40×10^{-3}
Duck intestine	3.54×10^{-3}
Duck gizzard	2.31×10^{-3}

Table 3.10

Sample	Limit of Detection (LOD) (mg)
Swan Intestine contents	4.87×10^{-5}
Swan Gizzard contents	1.42×10^{-4}
Goose Intestine contents	1.61×10^{-4}
Goose Gizzard contents	2.37×10^{-5}
Duck Intestine contents	2.37×10^{-5}
Duck Gizzard contents	2.37×10^{-5}

3.6 Validation

The linearity of the detector response with respect to concentration was assessed over the nominal concentration range of 0.005 to 0.25 mg/L. This was satisfactory with a goodness of fit greater than 0.99 being obtained for all the analyses.

3.7 Discussion

In the typical chromatography, it can be seen that the retention times differ in some analyses. This is due to the analytical column being changed part way through the study due to deterioration the chromatography.

A small peak present in the swan liver sample was not considered to be due to phosphorus since there was a significant difference compared to the standard in that analysis.

3.8 Conclusion

The amount of white phosphorus (mg/kg) present in the tissue samples and the corresponding limits of detection (mg/kg) are shown in the following table:

Table 3.11

Tissue Sample	White Phosphorus Residue (mg/kg of tissue)	Limit of Detection (LOD) (mg/kg of tissue)
Swan fat	<LOD	4.53×10^{-3}
Swan kidney	<LOD	4.73×10^{-3}
Swan muscle	<LOD	4.18×10^{-3}
Swan liver	<LOD	4.18×10^{-3}
Swan intestine	<LOD	4.78×10^{-3}
Swan gizzard	2.28×10^{-2}	1.38×10^{-2}
Goose fat	7.85×10^{-2}	1.60×10^{-2}
Goose kidney	<LOD	1.51×10^{-2}
Goose muscle	0.284	1.46×10^{-2}
Goose liver	<LOD	1.98×10^{-2}
Goose intestine	0.155	1.40×10^{-2}
Goose gizzard	1.61×10^{-2}	1.39×10^{-2}
Duck fat	3.38	2.30×10^{-3}
Duck kidney	6.91×10^{-2}	4.93×10^{-3}
Duck muscle	3.89×10^{-2}	2.20×10^{-3}
Duck liver	<LOD	3.40×10^{-3}
Duck intestine	0.160	3.54×10^{-3}
Duck gizzard	4.01×10^{-2}	2.31×10^{-3}

The amount of white phosphorus (mg) present in the gizzard and intestine contents and the corressponding limits of detection (mg) are shown in the following table:

Table 3.12

Contents Sample	Total White Phosphorus Present (mg)	Limit of Detection (LOD) (mg)
Swan Intestine contents	7.91×10^{-3}	4.87×10^{-5}
Swan Gizzard contents	0.308	1.42×10^{-4}
Goose Intestine contents	3.68×10^{-3}	1.61×10^{-4}
Goose Gizzard contents	14.3	2.37×10^{-5}
Duck Intestine contents	6.02×10^{-4}	2.37×10^{-5}
Duck Gizzard contents	0.349	2.37×10^{-5}