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November 4, 2015

Via FedEx and Regulations.gov

Division of Dockets Management (HFA-305)
Food and Drug Administration
5630 Fishers Lane, Room 1061
Rockville, Maryland 20852

Re: Comments on *Natural Resources Defense Council* et al.; *Filing of Food Additive Petition on Perchlorates*; Docket No. FDA-2015-F-0537

Dear Sir or Madam:

On behalf of our client, BASF Corporation, we are writing to provide you with comments regarding the Natural Resources Defense Council (NRDC)[1] October 15, 2014 petition regarding perchlorates. These Comments are intended to supplement Comments submitted on June 29, 2015.

As the Agency is aware, the NRDC Petition requests, among other items, that the U.S. Food and Drug Administration (FDA) revoke its 2005 approval of Threshold of Regulation (TOR) No. 2005-006.[2] The listing for Threshold of Regulation No. 2005-006 currently permits the use of sodium perchlorate monohydrate (CAS Reg. No. 7791-07-3) as a conductivity enhancer in the manufacture of antistatic agents for use in polymeric food packaging, at a level not to exceed 1.2 percent by weight of the finished polymer. The finished polymer may be used in contact with Food Type VIII ("Dry solids with the surface containing no free fat or oil").[3]

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[1] The Petition was co-sponsored by the Center for Food Safety (CFS), Breast Cancer Fund, Center for Environmental Health, Environmental Working Group, Improving Kids' Environment, Clean Water Action, Center for Science in the Public Interest and Children's Environmental Health Network.

[2] *See* http://www.accessdata.fda.gov/scripts/fdcc/?set=TOR&id=2005-006.

[3] As the Agency is aware, the TOR request was originally submitted on behalf of Ciba Specialty Chemicals Corporation on June 17, 2005. BASF later acquired Ciba Specialty Chemicals Company, and the existing TOR, in 2008. The TOR was revised in FDA's online TOR exemptions database on August 17, 2015, to correct certain typographical errors with respect to the intended use limitations. FDA's Food

(continued ...)

BASF originally submitted Comments to this Docket on June 29, 2015 indicating its' belief that the facts supporting the TOR exemption remain valid, and representative of food-contact materials used in dry food applications. To confirm the validity of these facts, BASF also agreed to conduct analytical work to demonstrate that sodium perchlorate monohydrate does not migrate to dry food.

In a May 18, 2015 Pre-Notification Consultation (PNC) with the Agency at its' College Park, Maryland office,[4] BASF agreed to demonstrate that the migration of sodium perchlorate would not be detectable at an analytical sensitivity of 10 parts per billion (ppb). The purpose of this analytical work, therefore, was to demonstrate that the potential dietary exposure to sodium perchlorate is safe and below the threshold level where regulation is necessary.

BASF has completed this analysis, and submits a September 4, 2015 Migration Report, *Migration of Sodium Perchlorate from Low Density Polyethylene containing 20% Irgastab P18 into Dry Food Simulant* ("Report") in support of the same. The analytical report demonstrates that sodium perchlorate would not, in fact, be detected at an analytical sensitivity of 10 ppb, when used as intended.

If you have any questions regarding these comments, or if we can be of further assistance in any other way, please do not hesitate to contact me.

Cordially yours,

Devon Wm. Hill

(...continued)
Types and Conditions of Use are *available at:* http://www.fda.gov/Food/IngredientsPackagingLabeling/PackagingFCS/FoodTypesConditionsofUse/default.htm.

[4] PNC 1642, "Meeting with the Society of the Plastics Industry (SPI) and BASF Corporation to discuss the allowed use of perchlorates in food contact applications."

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Migration Report

Migration of Sodium Perchlorate from Low Density Polyethylene containing 20% Irgastat P18 into Dry Food Simulant

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Migration Report

Migration of Sodium Perchlorate from Low Density Polyethylene containing 20% Irgastat P18 into Dry Food Simulant

I. Summary

The migration behavior of sodium perchlorate from low density polyethylene (LDPE) loaded with 20% Irgastab P18 was investigated with Tenax® as the dry food simulant. The polymer was exposed to Tenax® at 40°C with intervals removed and analyzed at 2h, 1 day, 4 days and 10 days. Perchlorate (as $Cl^{16}O_4$) was found to be less than 1.5 ng/in^2 in the simulant, corresponding to 150 ppt in food.

Table 1 Summary of migration results

Samples	Amount in Simulant (ng/in^2)	Amount in Food (ppt)
2 hrs	< 1.5	< 150
1 day	< 1.5	< 150
4 days	< 1.5	< 150
10 days	< 1.5	< 150

Note:

- The results were calculated based on the surface area of both sides of the migration plaques (4 in^2).
- The calculation was based upon the assumption that 1 square inch of surface is in contact with 10 grams of food.
- The limit of quantitation (LOQ) of perchlorate was 1.5 ng/in^2
- Data are reported as concentration of perchlorate ion $Cl^{16}O_4$

II. Objectives

Sodium perchlorate is used as conductivity enhancer in antistatic agents that are used in polymers. The purpose of current study is to investigate the migration behavior of sodium perchlorate from the polymer when in contact with dry foods. In this study, low density polyethylene was used as the model polymer. Antistatic agent Irgastab P18 containing sodium perchlorate was incorporated into LDPE at an approximate concentration of 20% (w/w). In this study, LC/MS/MS was used for sensitive analysis of perchlorate ions in the Tenax® at low ppb levels.

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III. Polymer Plaques

Polymer plaques were used as received. They are made of LDPE loaded with approximately 20% Irgastab P18. Irgastab P18 contains 1.8 % sodium perchlorate.

IV. Experimental

A. Apparatus and Reagents

- Wheaton™ glass staining dish with screw cap (1.02×1.02×2.7 in) Cat No. 08-813D
- Volumetric flasks, class A
- Fisher Scientific Isotemp Oven, Model 6841
- Balance: Mettler AT261
- Acetonitrile, HPLC Grade
- Ammonium acetate, 98%
- Barnstead Nanopure Diamond ultrapure water system
- BÜCHI Heating Bath B-490 and Rotavapor R-205 system
- Agilent 1200 High Performance Liquid Chromatograph
- Applied Biosystem API 4000 LC/MS/MS
- Sodium perchlorate monohydrate, ≥ 98%
- ^{18}O-labeled sodium perchlorate solution, atom purity 95%, Icon Isotopes
- Tenax®, 60-80 mesh, Sigma

B. Migration Experiments

Tenax® was used as a simulant for dry food. The Tenax® was washed upon receiving by overnight soxhlet extraction with acetonitrile (ACN). After thoroughly drying in a vacuum oven, it was used for the migration study.

Wheaton™ glass staining dishes with screw caps were used as migration vessels. One piece of LDPE plaque with surface area of 4 in^2 (double sides) was set upright in the middle of each vessel and 12 g of Tenax® was added into each vessel such that the plaques were fully covered. The simulant mass to surface area ratio was 3. The vessels were capped and put in the pre-heated oven for exposure. Each sampling time point was performed in triplicate. Blank polymer was treated in a similar manner.

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The migration study was done at four time points; 2 hours, 1 day, 4 days and 10 days. The vessels were placed into a preheated oven at 40°C. The temperature was maintained throughout the 10 days test period. After the specified time intervals, the plaques were removed and all the Tenax® in each vessel was transferred into a 250 mL beaker for further processing.

Conditions of Exposure	Food Simulant
40°C for 2 hrs, 1 day, 4,days and 10 days	Tenax®

C. Analysis of Simulants

After the Tenax® was transferred into the 250 mL beaker, 0.1 mL of internal standard ^{18}O-labeled perchlorate solution at 1.5 ppm was added, followed by adding 150 mL of ACN. The solution was stirred for one minute, then allowed to stand for 5 minutes. A 50 mL aliquot of the supernatant was transferred into a rotary evaporator tube and concentrated to 10 mL. The solution was then filtered through 0.2 µm PTFE syringe filter for LC/MS/MS analysis. See appendix A for details.

V. Results

At all four time points, perchlorate was detected, but at levels lower than method LOQ of 1.5 ng/in^2. No increase in perchlorate concentration was observed with prolonged exposure time. Additionally, large RSD values for triplicate analysis, between 33 to 60% (Appendix-D) were obtained for all time point samples, suggesting that the perchlorate found in the simulant was most likely caused by surface abrasion.

The results shown below were calculated based on the surface area of both sides of the migration plaques (4 in^2) based on the assumption of 1 in^2 of plaque being in contact with 10 grams of food.

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Migration of Sodium Perchlorate from Low Density Polyethylene containing 20% Irgastat P18 into Dry Food Simulant

Table 2 Results for specified conditions

Samples	Amount in Simulant* (ng/in^2)	Estimated amount in Food * (ppt)
2 hrs	< 1.5	< 150
1 day	< 1.5	< 150
4 days	< 1.5	< 150
10 days	< 1.5	< 150

*, data are reported as concentration of perchlorate ion $Cl^{16}O_4$

VI. Validation

Blank polymer plaques with 10 days exposure to Tenax® was used for the validation. Tenax® was transferred into a beaker followed by spiking with 6 ng of perchlorate ($Cl^{16}O_4$), corresponding to the LOQ level of 1.5 ng/in^2 (150 ppt in food). Then 0.1 mL of internal standard ^{18}O-labeled perchlorate ($Cl^{18}O_4$) at 1.5 ppm and 150 mL of ACN was added. The extracts were further processed as stated in section 0 for LC/MS/MS analysis.

Table 3 Results of Validation

Validation replicate	Perchlorate spiked* (ng/in^2)	Amount Spike* (ng)	Perchlorate found* (ng/in^2)	Amount Found* (ng)	Recovery (%)
1	1.53	6.12	1.57	6.26	102.3
2	1.53	6.12	1.51	6.04	98.6
3	1.53	6.12	1.49	5.97	97.5
AVG					99.4
STD					2.5
RSD (%)					2.5

*, data are reported as perchlorate ion $Cl^{16}O_4$

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Migration of Sodium Perchlorate from Low Density Polyethylene containing 20% Irgastat P18 into Dry Food Simulant

VII. Appendices

A. Analytical method (including calculations)

B. Detailed sample analysis data

C. Chromatograms

D. Spiking validation at low perchlorate levels

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Migration of Sodium Perchlorate from Low Density Polyethylene containing 20% Irgastat P18 into Food Simulant.

Appendix-A Analysis Method

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Appendix-A Analysis Method

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Appendix-A Analysis Method

I. Principle

This method is suitable for quantitative analysis of perchlorate in migration study from low density polyethylene (LDPE) in to Tenax® as dry food simulant. Perchlorate ion ($Cl^{16}O_4$) was detected by LC/MS/MS with multiple reaction monitoring (MRM) mode. ^{18}O-labeled perchlorate ($Cl^{18}O_4$) was used as internal standard for quantitation. Ion transition of m/z 99→83 for $Cl^{16}O_4$ to $Cl^{16}O_3$ and m/z 107→89 for $Cl^{18}O_4$ to $Cl^{18}O_3$ were monitored for quantitation.

II. Apparatus and Reagent

- Volumetric flasks, class A
- Fisher Scientific Isotemp Oven, Model 6841
- Balance: Mettler AT261
- Acetonitrile, HPLC Grade
- Ammonium acetate, 98%
- Barnstead Nanopure Diamond ultrapure water system
- BÜCHI Heating Bath B-490 and Rotavapor R-205 system
- Agilent 1200 High Performance Liquid Chromatograph
- Applied Biosystem API 4000 LC/MS/MS
- Sodium perchlorate monohydrate, ≥ 98%
- ^{18}O-labeled sodium perchlorate solution, atom purity 95%, Icon Isotopes
- Tenax®, 60-80 mesh, Sigma

III. LC/MS/MS Method

LC Mode	Ion Exchange
Solvents	ACN 50%, Water 50%, Ammonium acetate 100 mM
Column	Waters IC-Pak Anion HR 4.6 mm i.d.×75 mm
Flow Rate	0.35 mL/min
Gradient	Isocratic
Column Temperature	35 °C
Detection	MS/MS, ESI(-), MRM mode, monitor ion transition of m/z 99→83 for $Cl^{16}O_4$ to $Cl^{16}O_3$ and monitor ion transition of m/z 107→89 for $Cl^{18}O_4$ to $Cl^{18}O_3$

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Appendix-A Analysis Method

IV. Standard preparation and calibration curve

Prepare two standard stock solutions A and B at a concentration of 1000 ppm in water using two independent weighings of 35 mg of sodium perchlorate standard into two 25 mL volumetric flasks (stock solutions A and B). Working standards were prepare at the concentration range of 0.2 to 25 ppb with the dilution scheme below. To each standard, internal standard ^{18}O-labeled perchlorate at 7.4 ppb was added.

Solution A/Solution B: ^{18}O-labeled sodium perchlorate as internal standard was received as 1 mg of ^{18}O-labeled sodium perchlorate dissolved in 0.55 g $H_2{}^{18}O$, which equals 1497 ppm of $Cl^{18}O_4$ ion in solution (IS stock solution). A 0.1mL aliquot of IS stock solution was transferred into 10 mL volumetric flask and diluted to the mark with water to obtain 14.97 ppm (IS Solution A). 0.5 mL of IS Solution A was transferred into 100 mL volumetric flask. Dilute to mark with water to obtain 75 ppb IS solution B.

Solution C: 5 ppm of perchlorate in ACN. Transfer 0.5 mL of stock solution A to a 100 mL volumetric flask. Dilute to mark with ACN.

Solution D: 5 ppm of perchlorate in ACN. Transfer 0.5 mL of stock solution B to a 100 mL volumetric flask. Dilute to mark with ACN.

Solution E: 25 ppb perchlorate in ACN with IS ^{18}O-labeled perchlorate at 7.5 ppb. Transfer 0.5 mL of solution C and 10 mL of IS stock solution B to a 100 mL volumetric flask. Dilute to mark with ACN.

Solution F: 50 ppb perchlorate in ACN with IS ^{18}O-labeled perchlorate at 7.5 ppb. Transfer 1 mL of solution D and 10 mL of IS stock solution B to a 100 mL volumetric flask. Dilute to mark with ACN.

Solution G: 5 ppb perchlorate in ACN with IS ^{18}O-labeled perchlorate at 7.5 ppb. Transfer 2 mL of solution E and 1 mL of IS stock solution B to a 10 mL volumetric flask. Dilute to mark with ACN.

Solution H: 10 ppb perchlorate in ACN with IS ^{18}O-labeled perchlorate at 7.5 ppb. Transfer 2 mL of solution F and 1 mL of IS stock solution B to a 10 mL volumetric flask. Dilute to mark with ACN.

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Solution I: 1 ppb perchlorate in ACN with IS ^{18}O-labeled perchlorate at 7.5 ppb. Transfer 2 mL of solution G and 1 mL of IS stock solution B to a 10 mL volumetric flask. Dilute to mark with ACN.

Solution J: 2 ppb perchlorate in ACN with IS ^{18}O-labeled perchlorate at 7.5 ppb. Transfer 2 mL of solution H and 1 mL of IS stock solution B to a 10 mL volumetric flask. Dilute to mark with ACN.

Solution K: 0.2 ppb perchlorate in ACN with IS ^{18}O-labeled perchlorate at 7.5 ppb. Transfer 2 mL of solution I and 1 mL of IS stock solution B to a 10 mL volumetric flask. Dilute to mark with ACN.

Solution L: 0.4 ppb perchlorate in ACN with IS ^{18}O-labeled perchlorate at 7.5 ppb. Transfer 2 mL of solution J and 1 mL of IS stock solution B to a 10 mL volumetric flask. Dilute to mark with ACN.

By using LC/MS/MS method at MRM mode, ion transition of m/z 99→83 for ClO_4^{16} to ClO_3^{16} and m/z 107→89 for ClO_4^{18} to ClO_3^{18} was recorded. A calibration curve was established by plotting the ratio of peak area for ClO_4^{16} to peak area for ClO_4^{18} against the ratio of ClO_4^{16} concentration to ClO_4^{18} concentration. A typical calibration curve is shown below.

Concentration of ClO_4^{16} (ppb)	Concentration of ClO_4^{18} (ppb)	Ratio of ClO_4^{16} concentration to ClO_4^{18} concentration	Ratio of peak area for ClO_4^{16} to peak area for ClO_4^{18}
0.19	7.5	0.03	0.03
0.38	7.5	0.05	0.05
0.96	7.5	0.13	0.13
1.94	7.5	0.26	0.24
4.81	7.5	0.64	0.63
9.70	7.5	1.30	1.21
24.12	7.5	3.22	3.05

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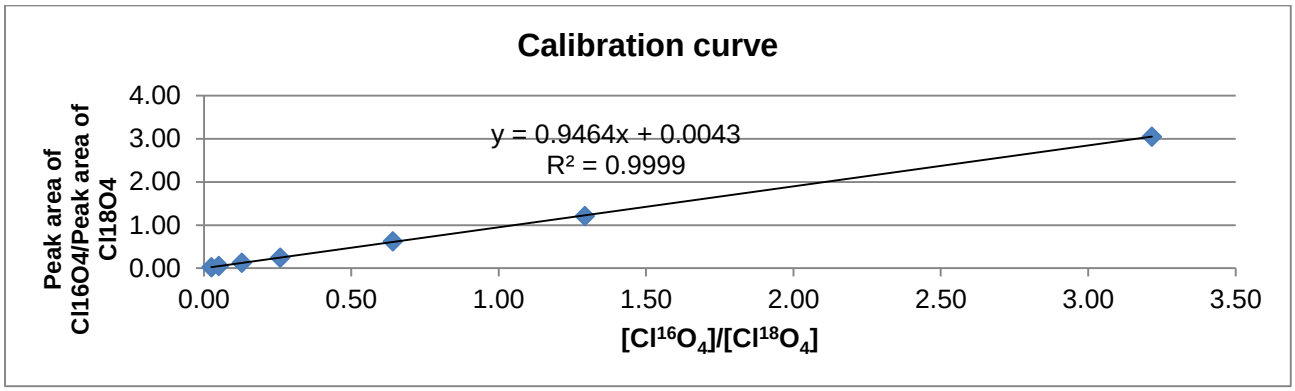
Appendix-A Analysis Method



V. Sample processing

After each migration exposure interval, the food simulant Tenax® was transferred into a 250 mL beaker and 0.1 mL of internal standard ^{18}O-labeled perchlorate solution at 1.5 ppm was added, followed by the addition of 150 mL of ACN. The solution was stirred for one minute and allowed to stand for 5 minutes. A 50 mL aliquot of the supernatant was then transferred into rotary evaporator tube for concentration to 10 mL. The solution was then filtered through 0.2 µm PTFE syringe filter for LC/MS/MS analysis.

VI. Calculations

By using the LC/MS/MS method for sample analysis, the ratio of peak area for ClO_4^{16} to peak area for internal standard ClO_4^{18} was recorded and the concentration of perchlorate in the food simulant was calculated as following.

- $y = mx + b$

where x = ratio of ClO_4^{16} concentration to ClO_4^{18} concentration, m = slope of the line, y = ratio of peak area for ClO_4^{16} to peak area for ClO_4^{18}, and b = y-intercept

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Appendix-A Analysis Method

- ppb $Cl^{16}O_4$ in solution = ratio of peak area for $Cl^{16}O_4$ to peak area for $Cl^{18}O_4$ X ppb IS $Cl^{18}O_4$
- ng analyte/in^2 =ppb $Cl^{16}O_4$ x 150 mL / surface area (4 in^2)
- ppt analyte in food* = ng analyte/in^2 x 100

The calculation is based upon the assumption that 1 square inch of surface is in contact with 10 grams of food.

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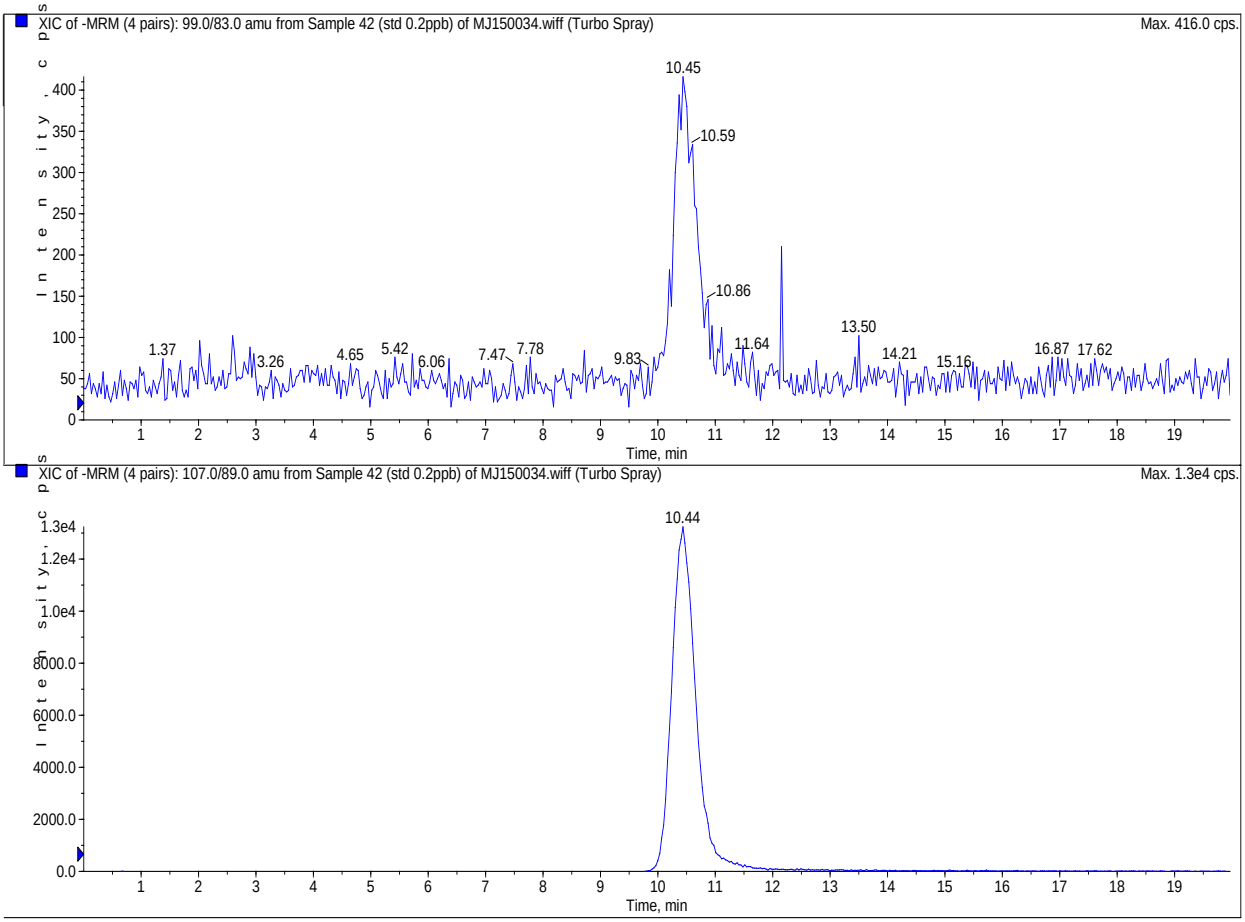
Appendix-A Analysis Method

VII. Chromatograms

Figure 1. Standard of $Cl^{16}O_4$ at 0.2 ppb (upper) with internal standard $Cl^{18}O_4$ at 7.5 ppb (lower)



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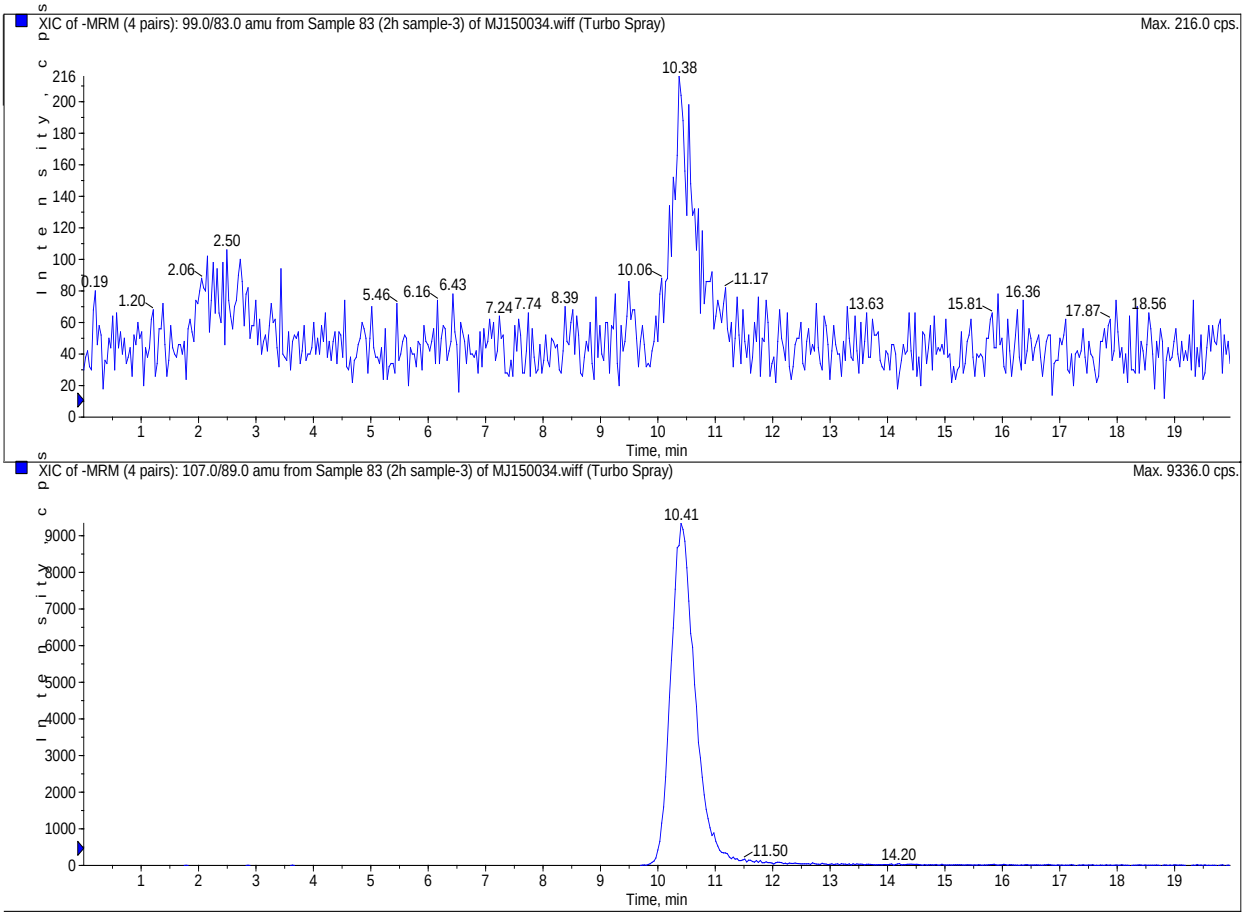
Appendix-A Analysis Method

Figure 2. Sample analysis (2 h exposure) with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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Appendix-A Analysis Method

VIII. Validation

The method was validated by its specificity, linear range, repeatability and detection limits.

A. Specificity

LC/MS/MS at MRM mode monitors ion transition of m/z 99→83 for $Cl^{16}O_4$ to $Cl^{16}O_3$ and monitor ion transition of m/z 107→89 for $Cl^{18}O_4$ to $Cl^{18}O_3$ for quantitation. Ion transition of m/z 101→85 for $^{37}Cl^{16}O_4$ to $^{37}Cl^{16}O_3$ is used for confimation of perchlorate identity.

B. Range

0.2 to 25 ppb for perchlorate in solution for LC/MS/MS analysis.

C. Repeatability

Six individual injections of perchlorate at 0.2 ppb was done for assessing repeatability. RSD obtained was 4.5%.

D. Linearity

Linear regression coefficient R^2 is better than 0.999.

E. Detection limits

LC/MS/MS method limit of detection is 0.1 ppb and limit of quantitation is 0.2 ppb for perchlorate in solution. For analysis of perchlorate migration into Tenax® as food simulant, limit of detection is 0.4 ng/in^2 and limit of quantitation is 1.5 ng/in^2 (Appendix-D).

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Appendix-B Detailed Sample Analysis Data

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Appendix-B Detailed Sample Analysis Data

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Appendix-B Detailed Sample Analysis Data

I. Detailed sample analysis data

Table 1 Detailed data analysis - Perchlorate levels in food simulant

Sample replicate	2 hrs		1 day		4 days		10 days		Validation with blank polymer at LOQ	
	Ratio*	ng/in^{2**}	Ratio*	ng/in^{2**}	Ratio*	ng/in^{2**}	Ratio*	ng/in^{2**}	Ratio*	ng/in^{2**}
1	0.017	0.51	0.027	0.96	0.024	0.82	0.013	0.31	0.040	1.6
2	0.011	0.30	0.016	0.54	0.020	0.66	0.021	0.63	0.039	1.5
3	0.019	0.59	0.015	0.53	0.007	0.18	0.013	0.31	0.038	1.5
AVG		0.47		0.68		0.56		0.42		1.52
STD		0.15		0.25		0.33		0.18		0.04
RSD (%)		33		36		60		44		3

*, ratio of peak area for $Cl^{16}O_4$ to peak area for $Cl^{18}O_4$

**, the numbers were estimated by extrapolating the calibration curve to lower than LOQ level. Numbers are reported as perchlorate ion $Cl^{16}O_4$

With the method stated in Appendix-A, Analysis method, the ratio of peak area for $Cl^{16}O_4$ to peak area for $Cl^{18}O_4$ was calculated from LC/MS/MS analysis chromatograms and with the calibration curve listed in Appendix-A, Section IV Standard Preparation and calibration curve, the ratio of $Cl^{16}O_4$ concentration to $Cl^{18}O_4$ concentration can be calculated. Knowing the concentration of internal standard $Cl^{18}O_4$, the perchlorate concentration in the solution for LC/MS/MS injection can thus be obtained. As can be seen from Table 1 **Detailed data analysis** the ratio of peak area for $Cl^{16}O_4$ to peak area for $Cl^{18}O_4$ for all time point are smaller than method LOQ. Additionally, although the RSD values are high for all four time points between 33 to 60%, the levels of perchlorate found in the simulant are less than 1 ng/in^2. This suggests that instead of migrating from the plaque into Tenax®, perchlorate found in Tenax® is more likely due to physical contact and scraping during sample handling.

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Appendix-C Chromatograms

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Appendix-C Chromatograms

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Appendix-C Chromatograms

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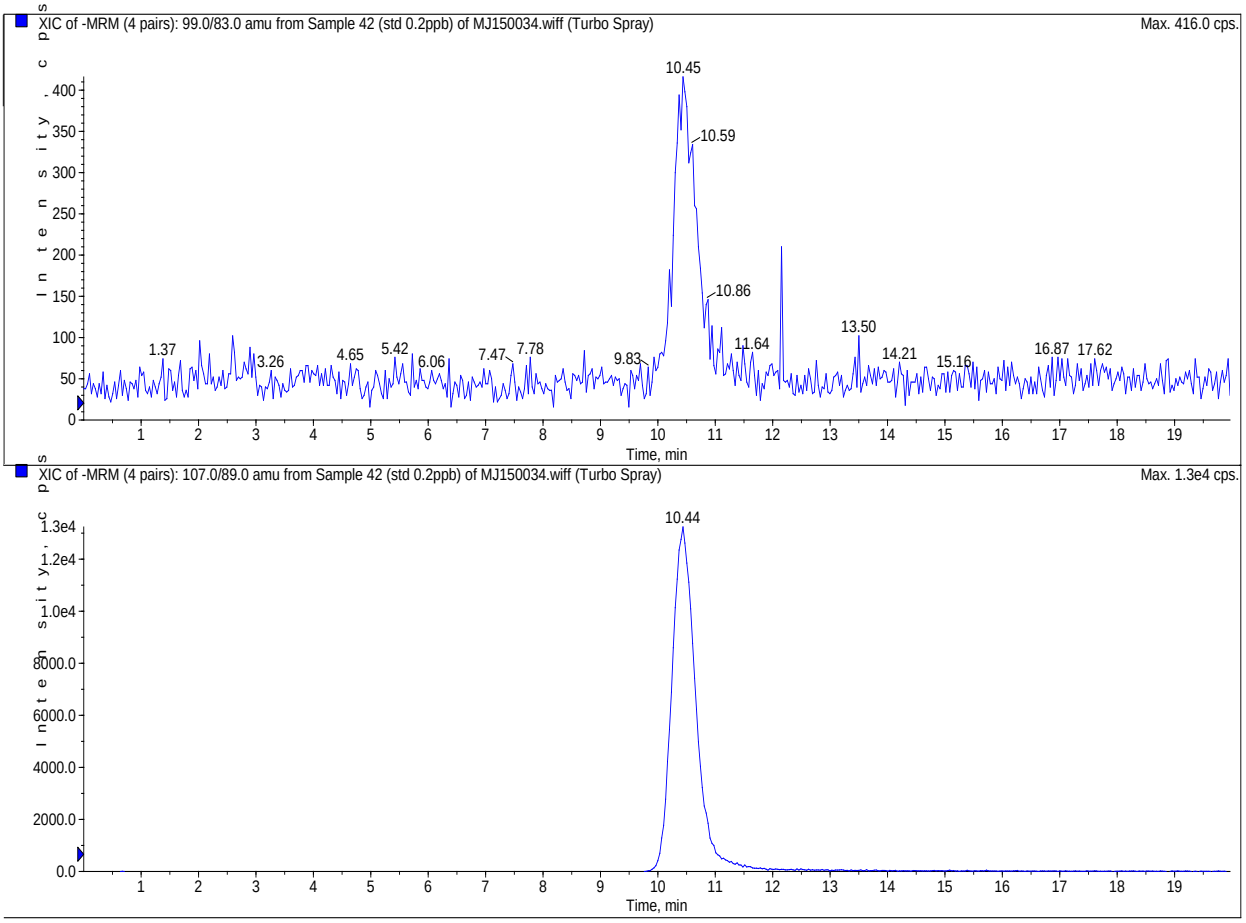
Migration of Sodium Perchlorate from Low Density Polyethylene containing 20% Irgastat P18 into Food Simulant.

Appendix-C Chromatograms

I. Figure 1 Standard of $Cl^{16}O_4$ at 0.2 ppb (upper) with internal standard $Cl^{18}O_4$ at 7.5 ppb (lower)



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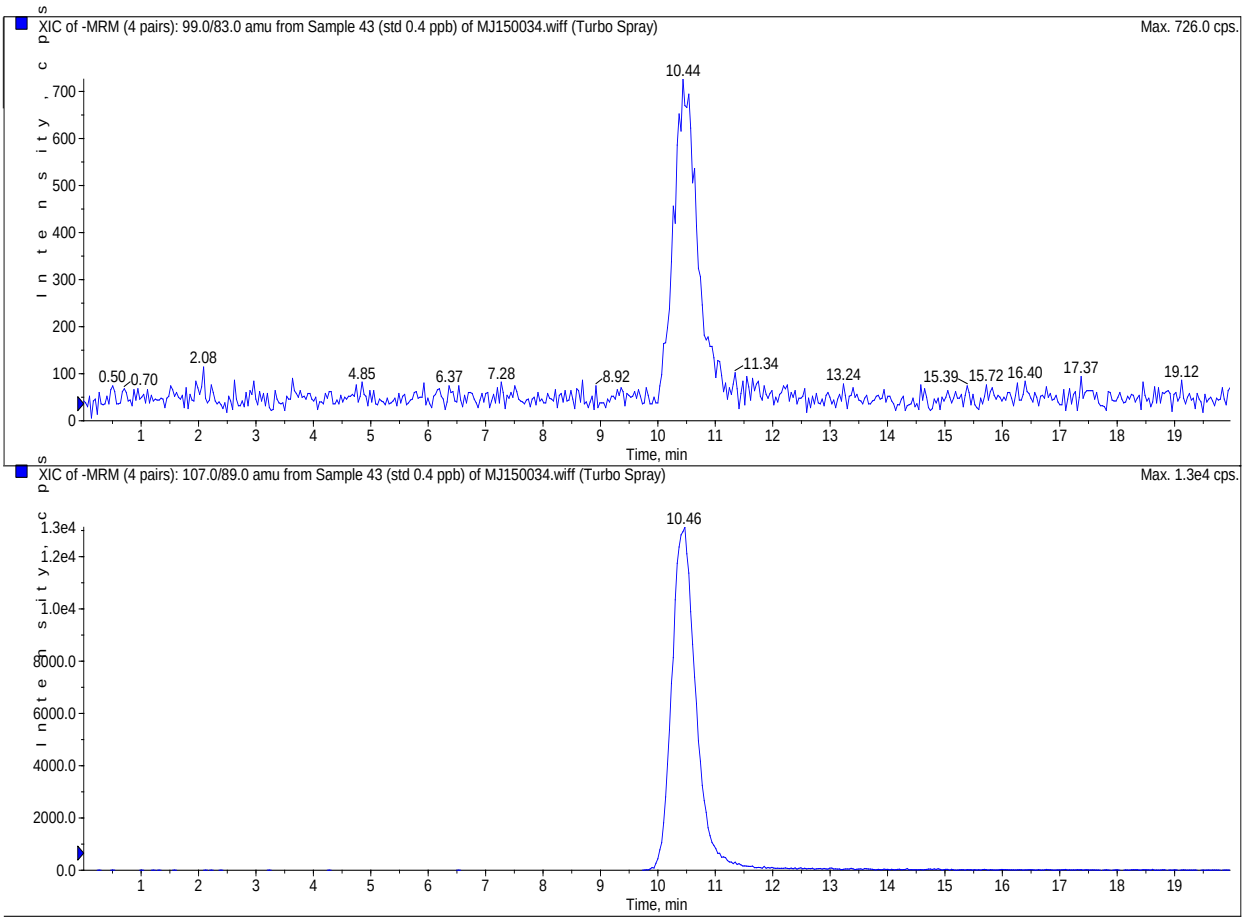
Migration Report

Migration of Sodium Perchlorate from Low Density Polyethylene containing 20% Irgastat P18 into Food Simulant.

Appendix-C Chromatograms

II. Figure 2 Standard of $Cl^{16}O_4$ at 0.4 ppb (upper) with internal standard $Cl^{18}O_4$ at 7.5 ppb (lower)



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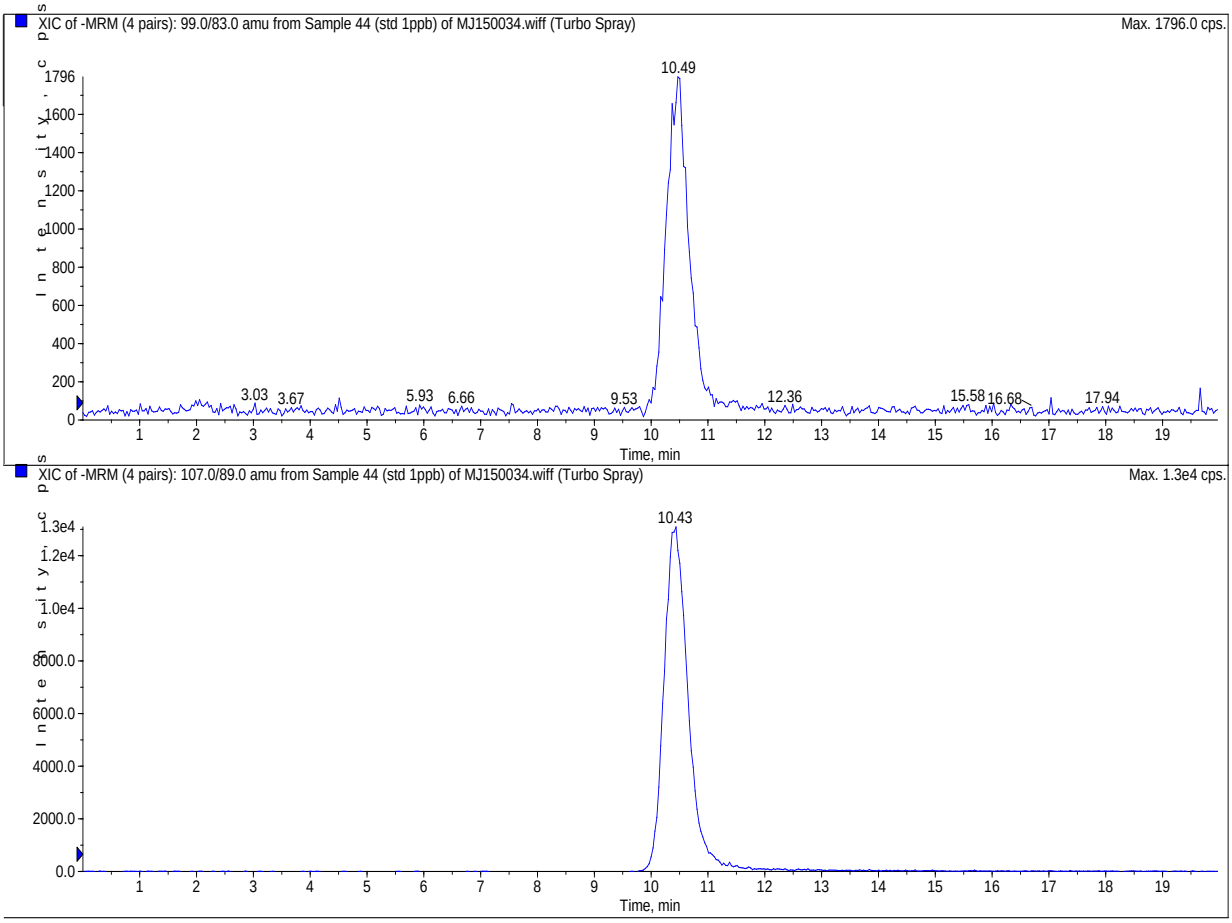
Appendix-C Chromatograms

III. Figure 3 Standard of $Cl^{16}O_4$ at 1 ppb (upper) with internal standard $Cl^{18}O_4$ at 7.5 ppb (lower)



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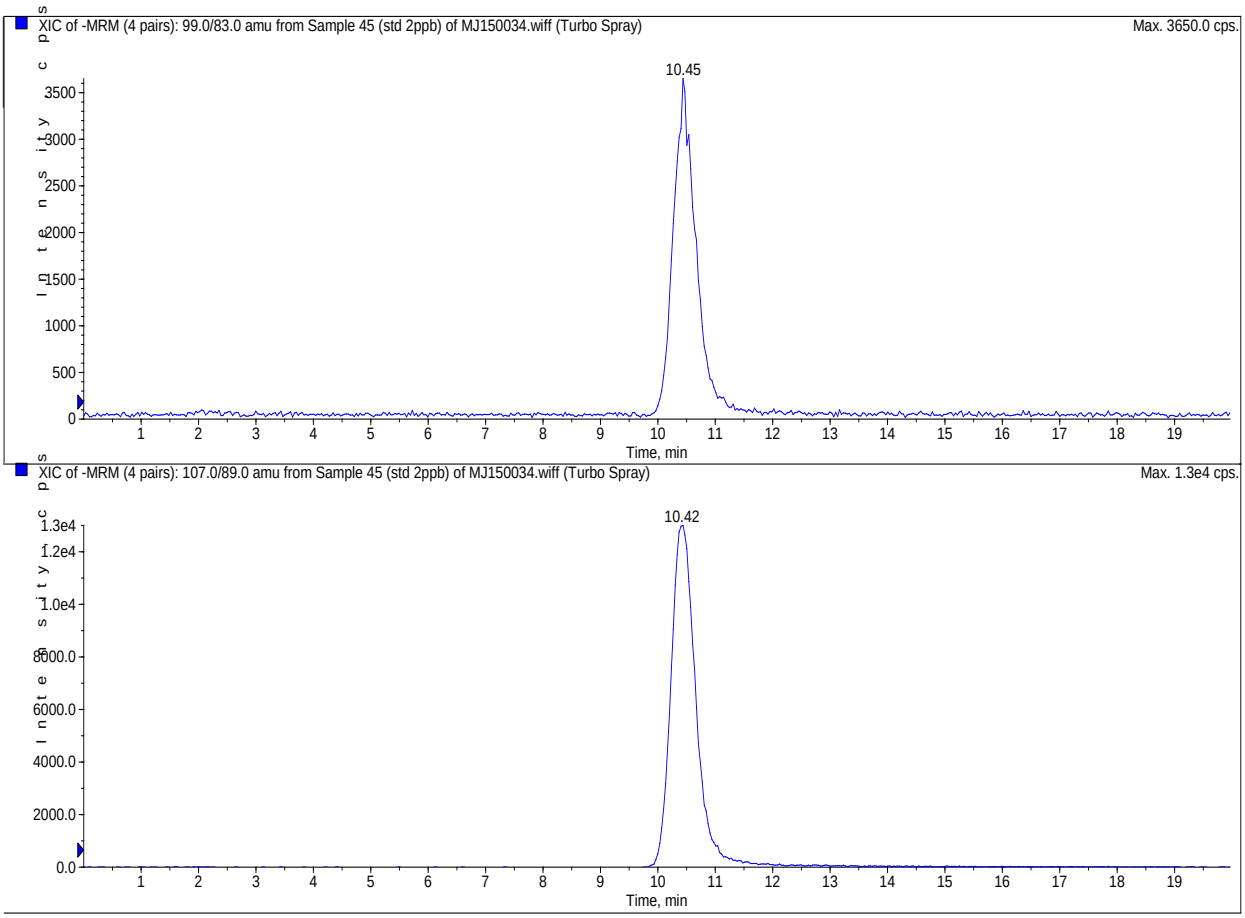
Appendix-C Chromatograms

IV. Figure 4 Standard of $Cl^{16}O_4$ at 2 ppb (upper) with internal standard $Cl^{18}O_4$ at 7.5 ppb (lower)



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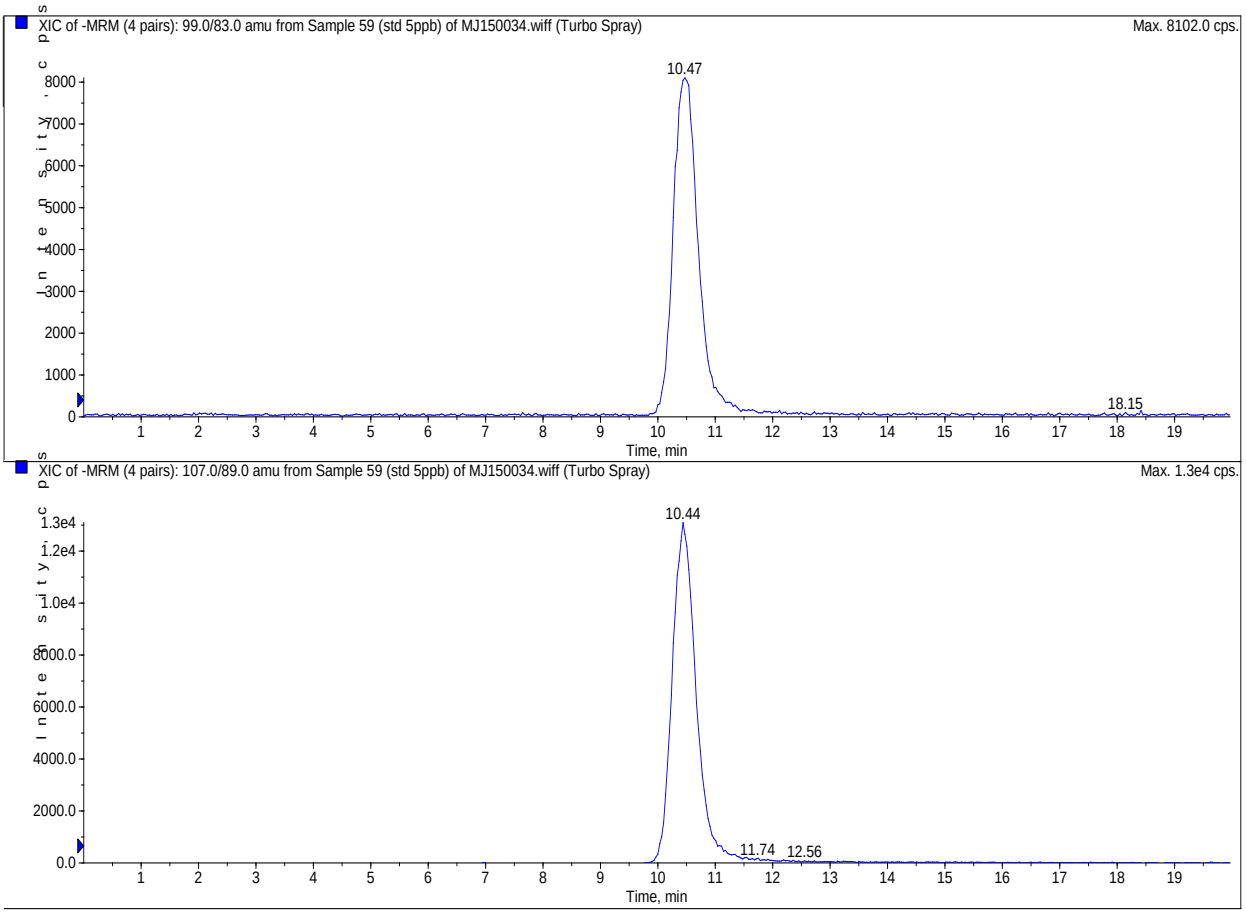
Appendix-C Chromatograms

V. Figure 5 Standard of $Cl^{16}O_4$ at 5 ppb (upper) with internal standard $Cl^{18}O_4$ at 7.5 ppb (lower)



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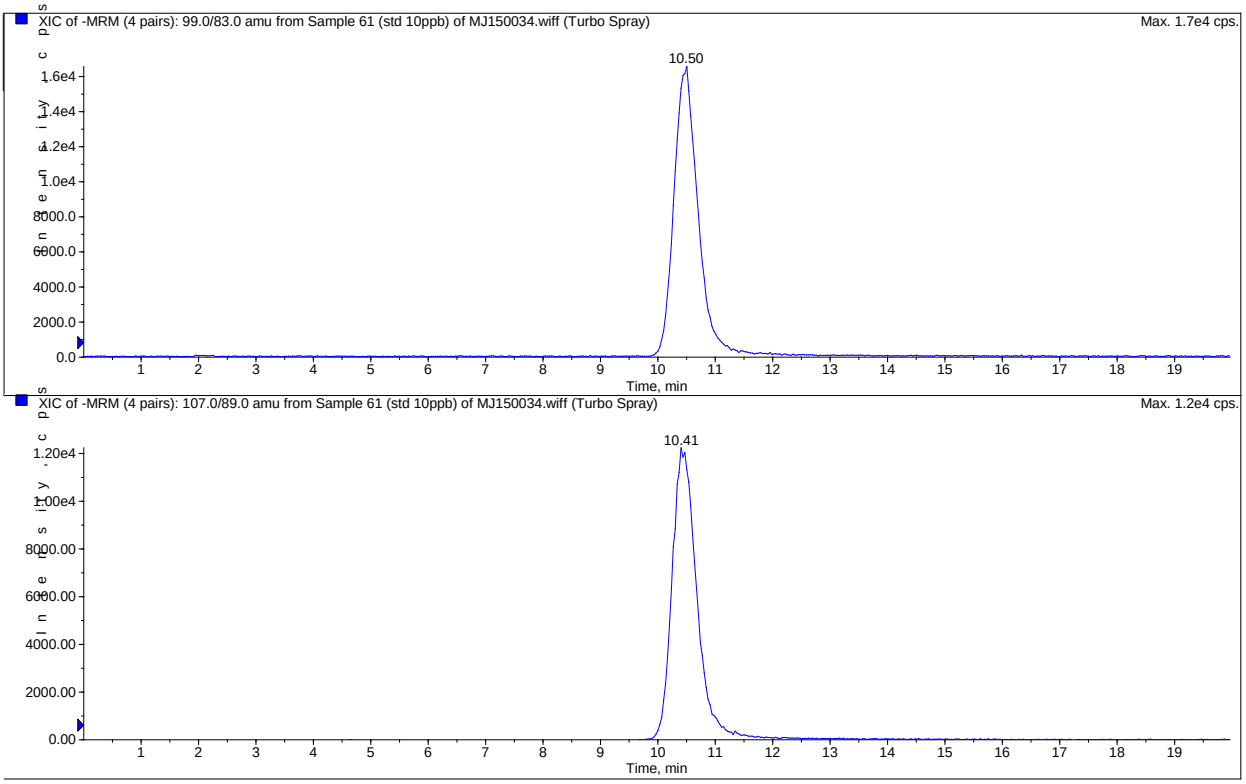
Appendix-C Chromatograms

VI. Figure 6 Standard of $Cl^{16}O_4$ at 10 ppb (upper) with internal standard $Cl^{18}O_4$ at 7.5 ppb (lower)



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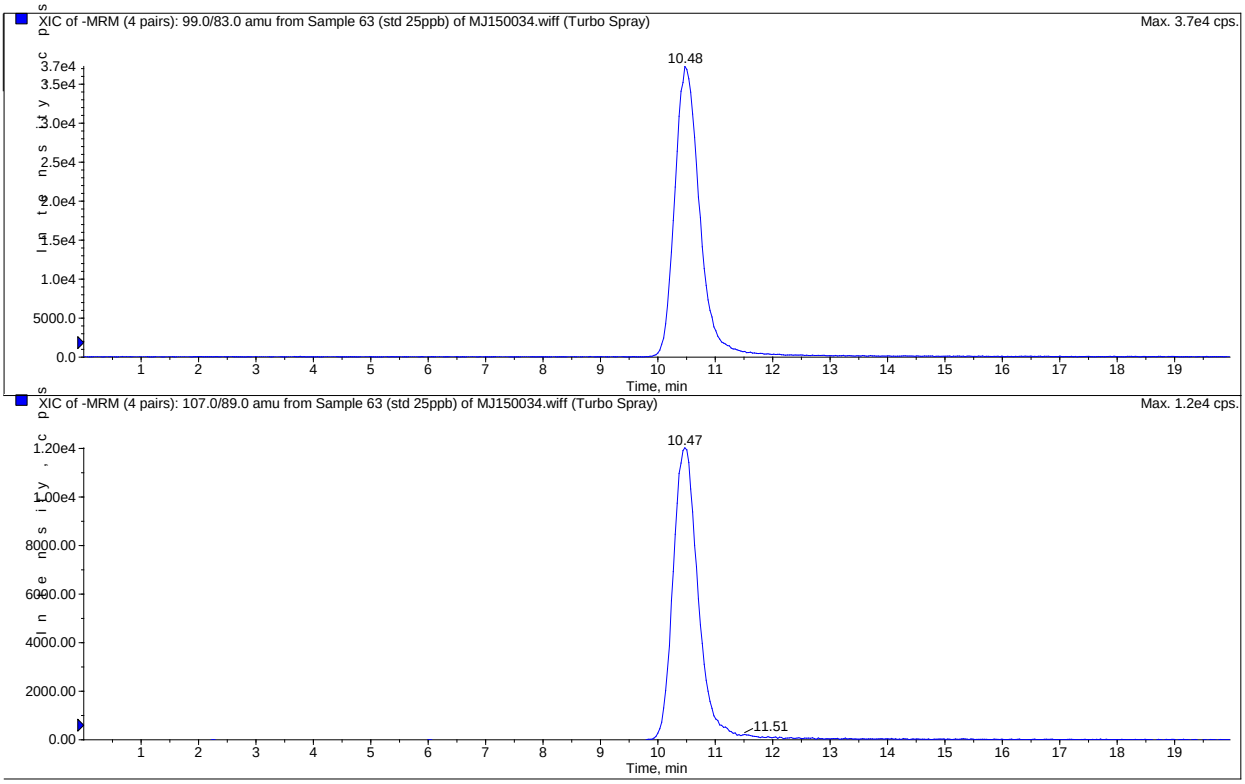
Appendix-C Chromatograms

VII. Figure 7 Standard of $Cl^{16}O_4$ at 25 ppb (upper) with internal standard $Cl^{18}O_4$ at 7.5 ppb (lower)



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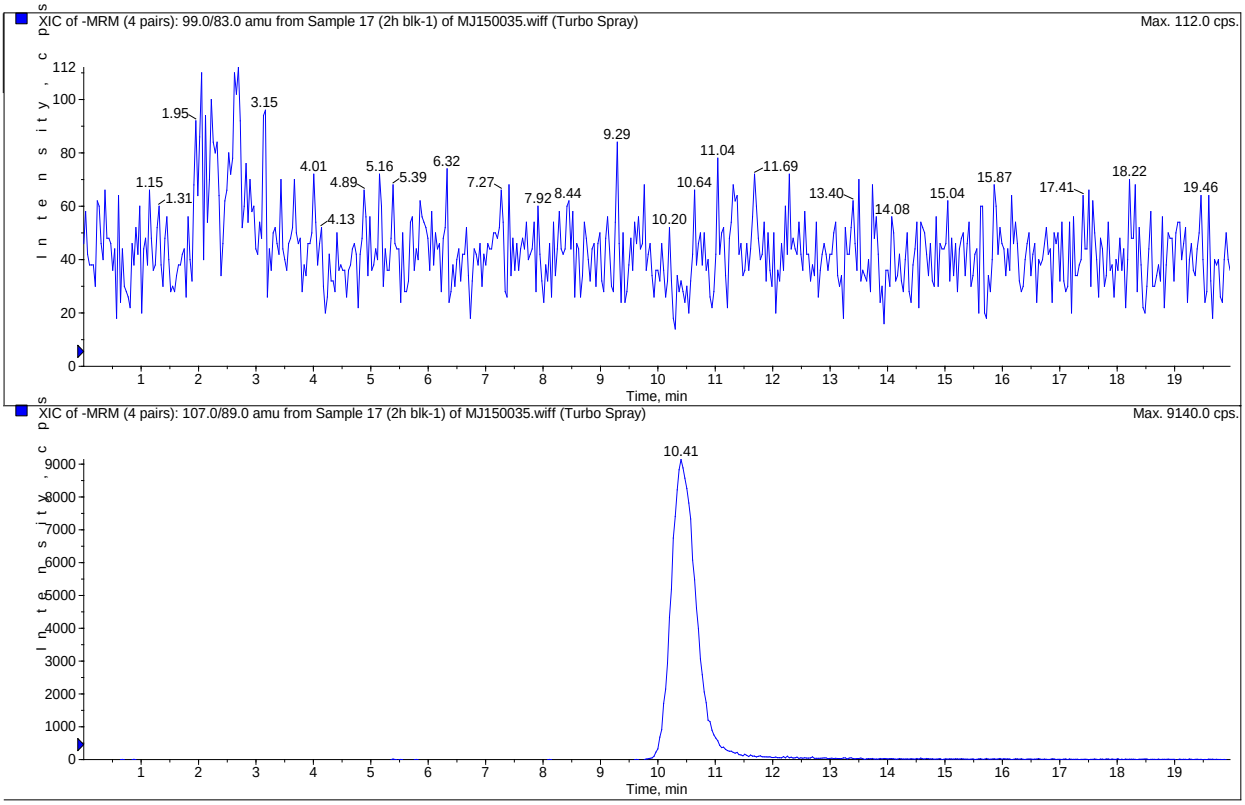
VIII. Figure 8. 2 Hrs exposure blank-1 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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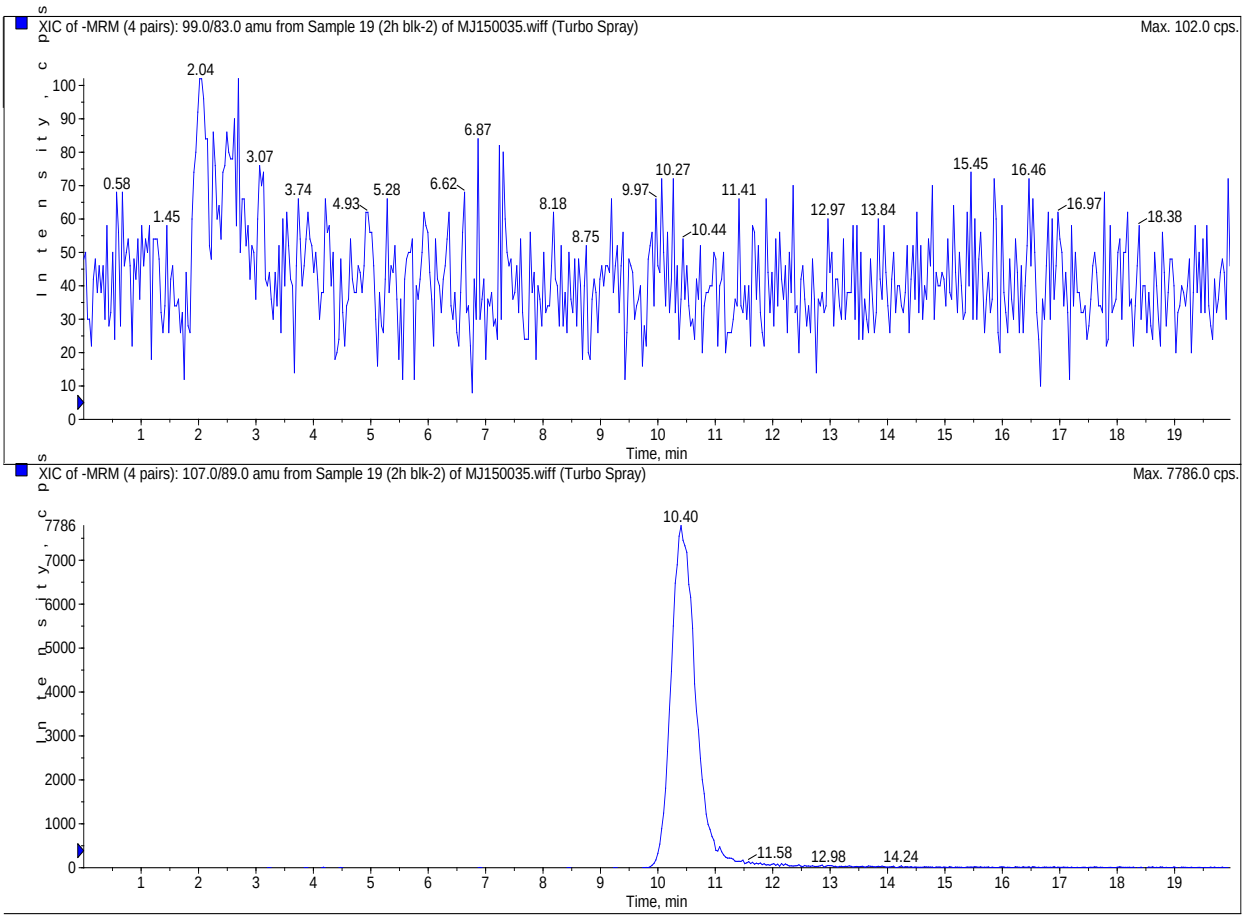
Appendix-C Chromatograms

IX. Figure 9. 2 Hrs exposure blank-2 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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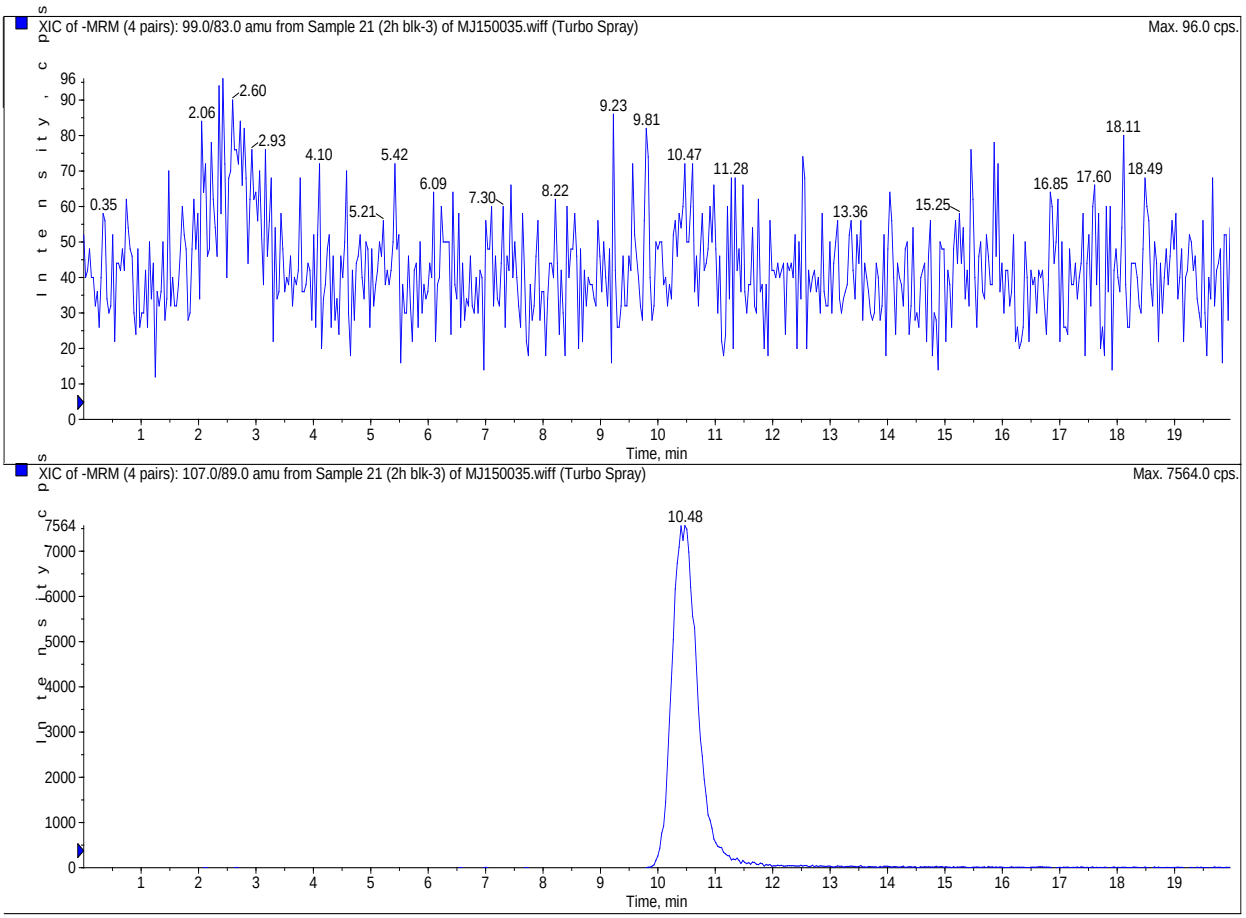
Appendix-C Chromatograms

X. Figure 10. 2 Hrs exposure blank-3 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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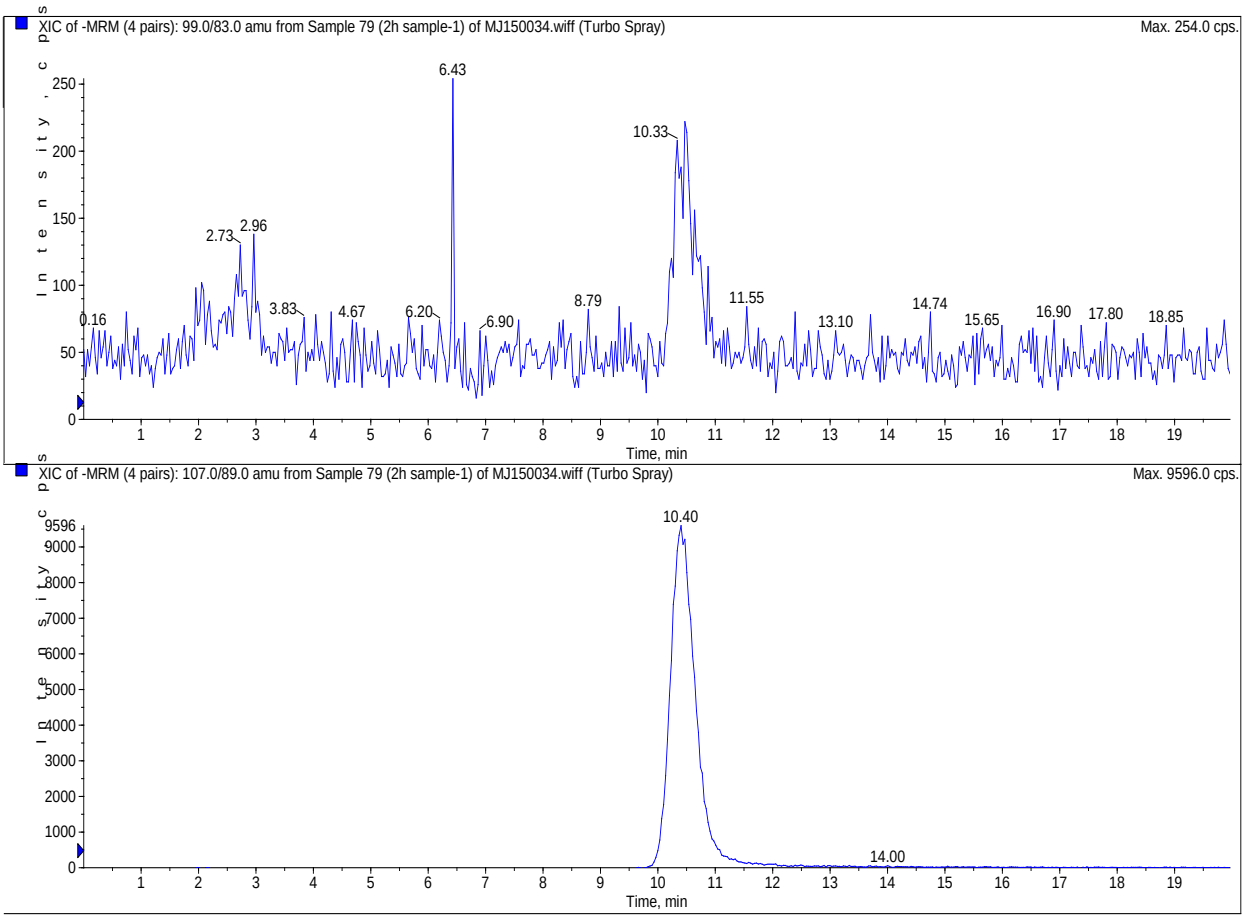
Appendix-C Chromatograms

XI. Figure 11. 2 Hrs exposure sample-1 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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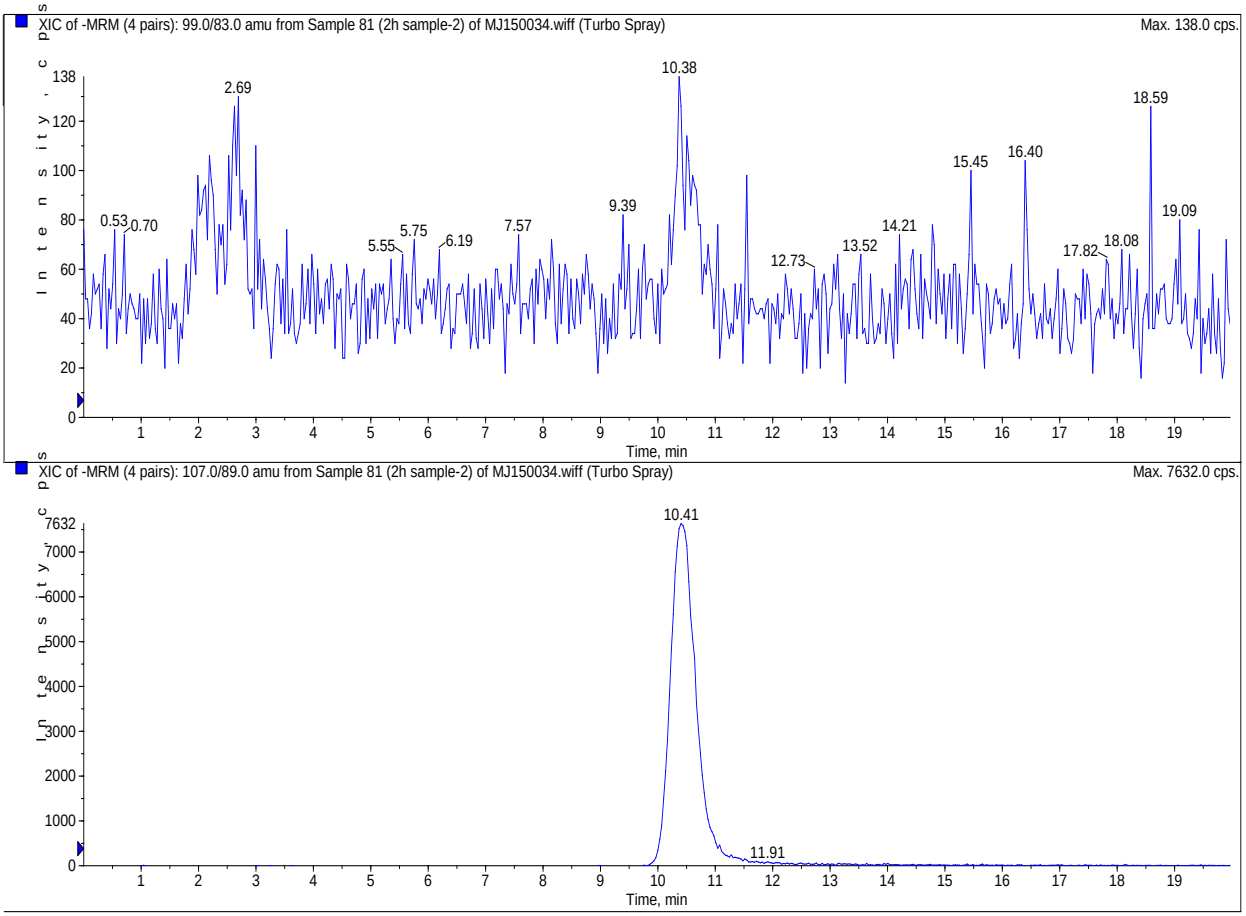
XII. Figure 12. 2 Hrs exposure sample-2 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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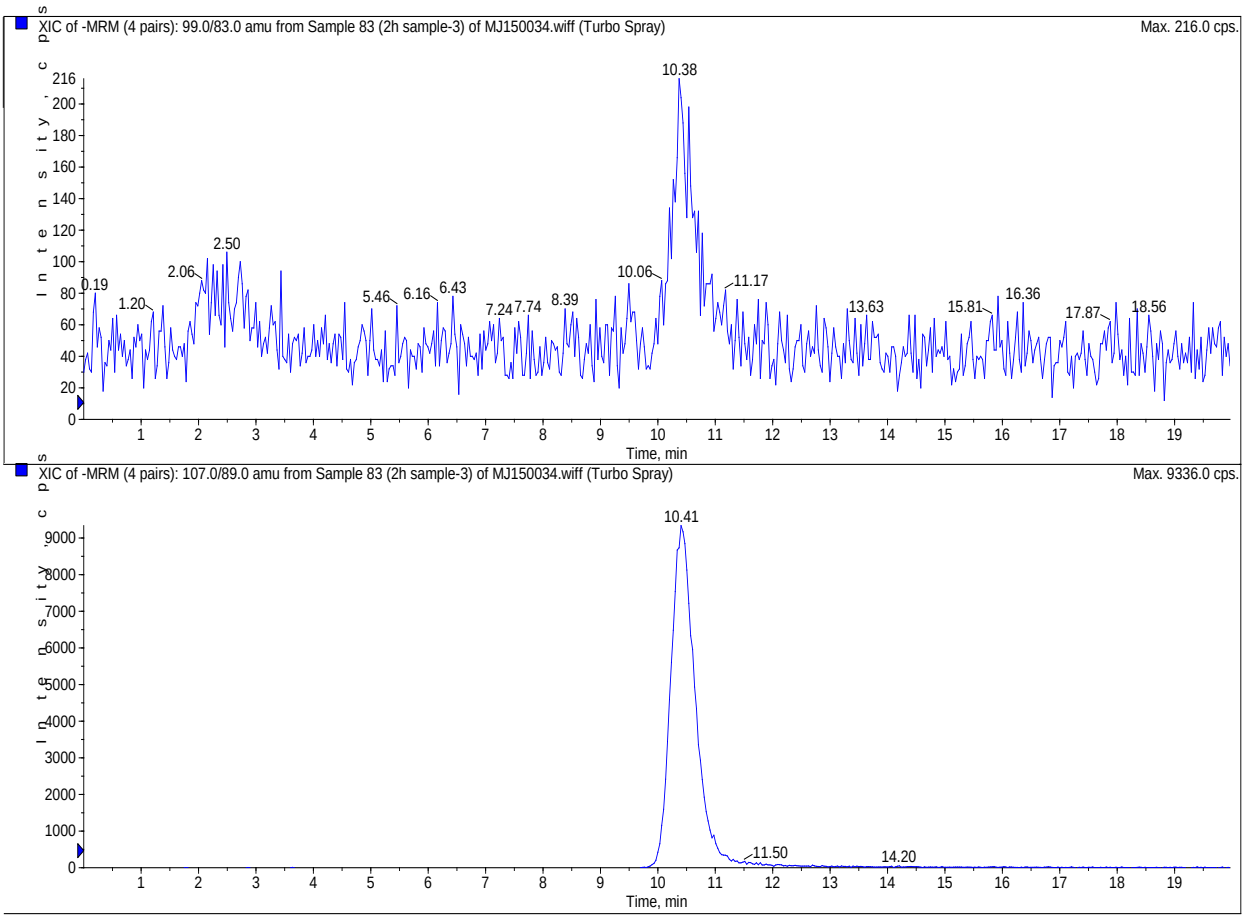
XIII. Figure 13. 2 Hrs exposure sample-3 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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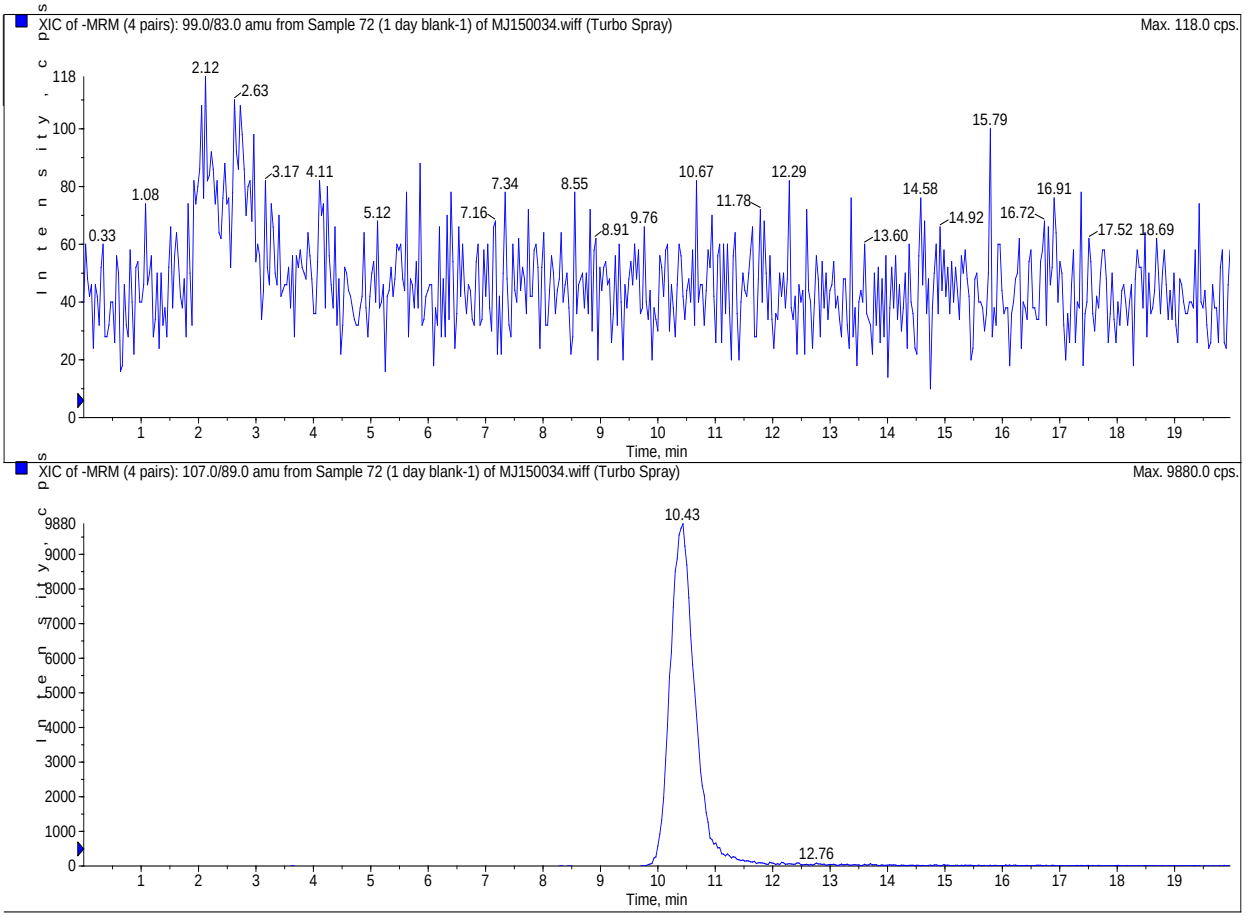
XIV. Figure 14. 1 Day exposure blank-1 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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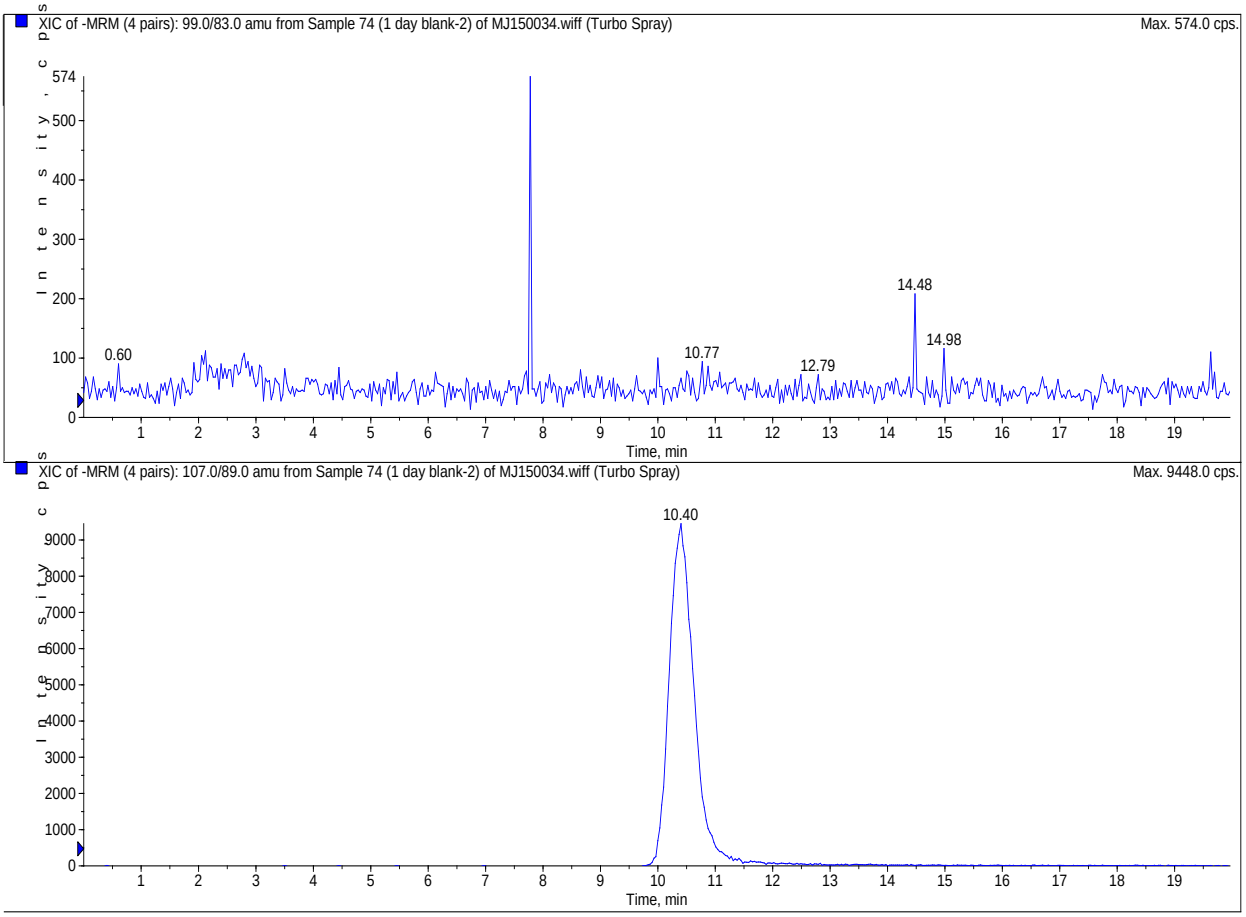
XV. Figure 15. 1 Day exposure blank-2 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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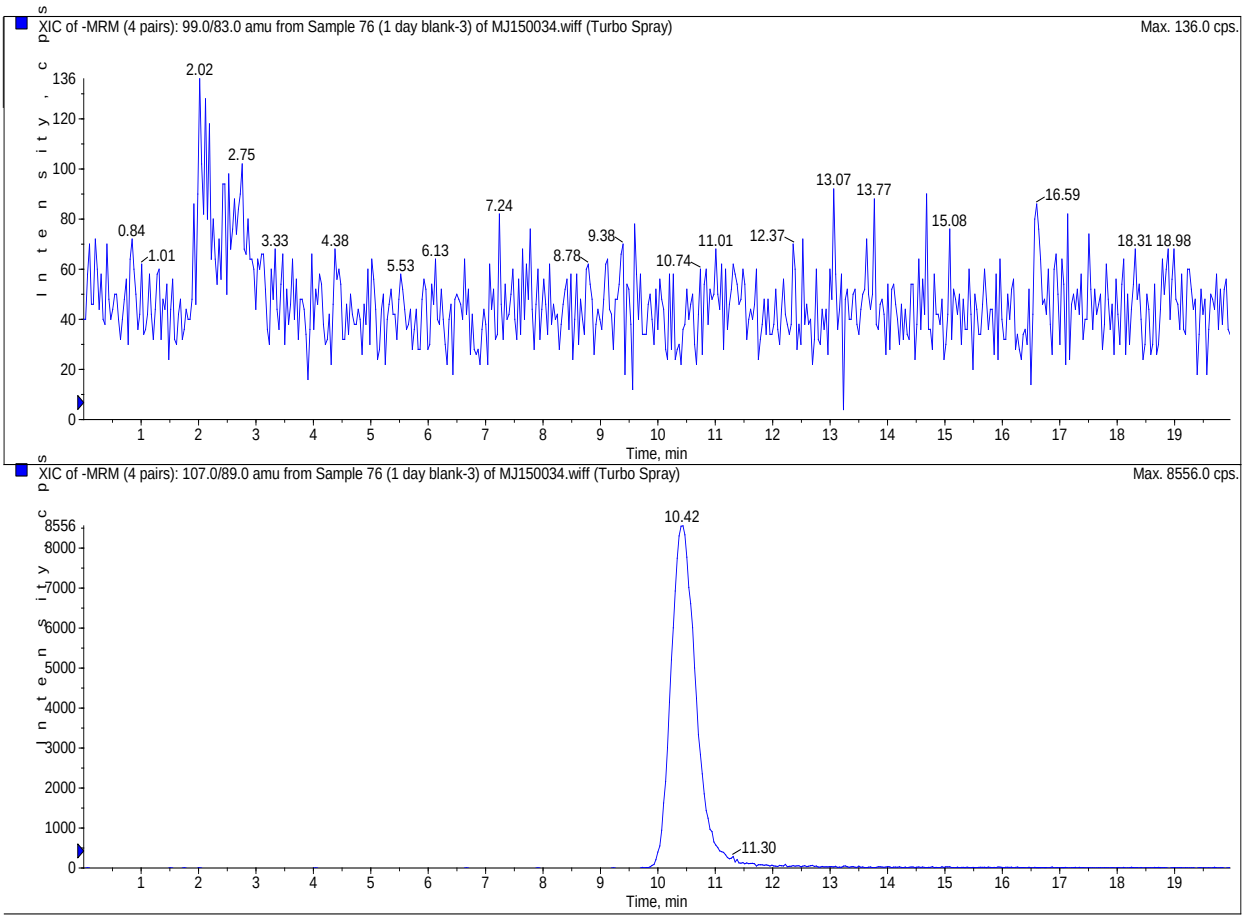
XVI. Figure 16. 1 Day exposure blank-3 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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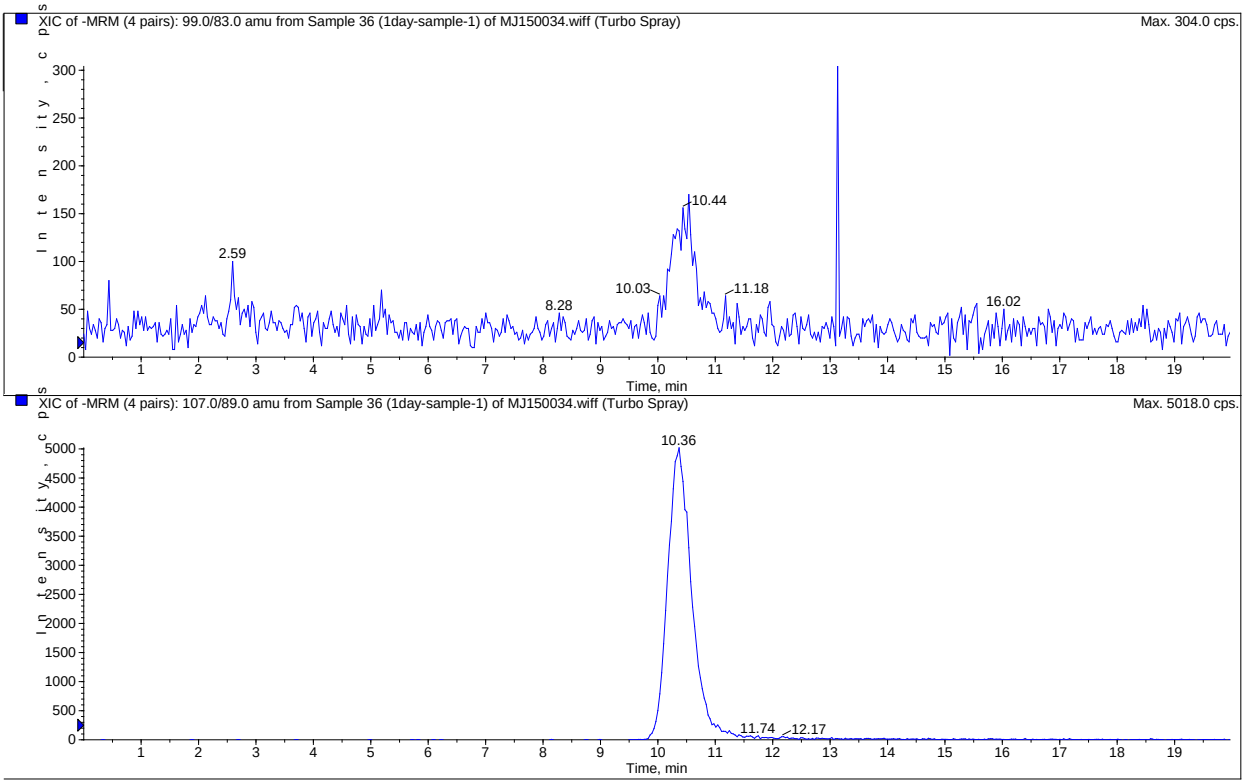
XVII. Figure 17. 1 Day exposure sample-1 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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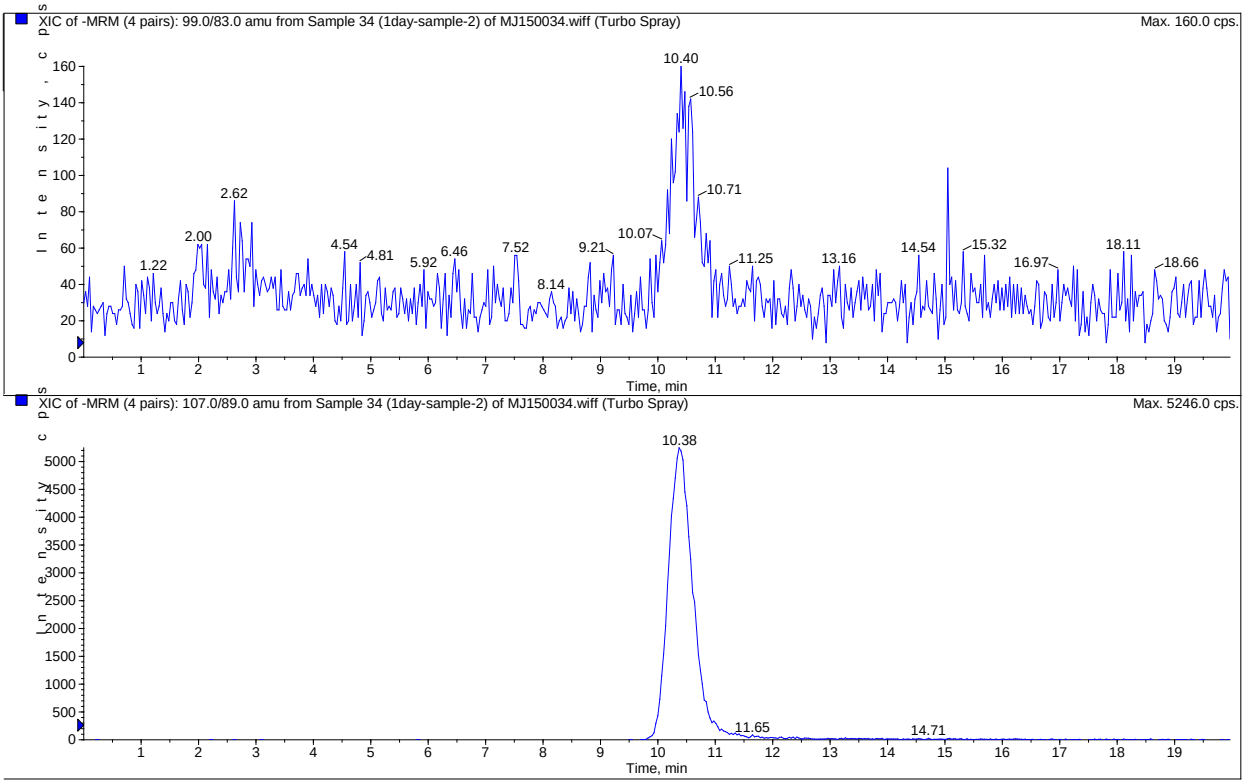
XVIII. Figure 18. 1 Day exposure sample-2 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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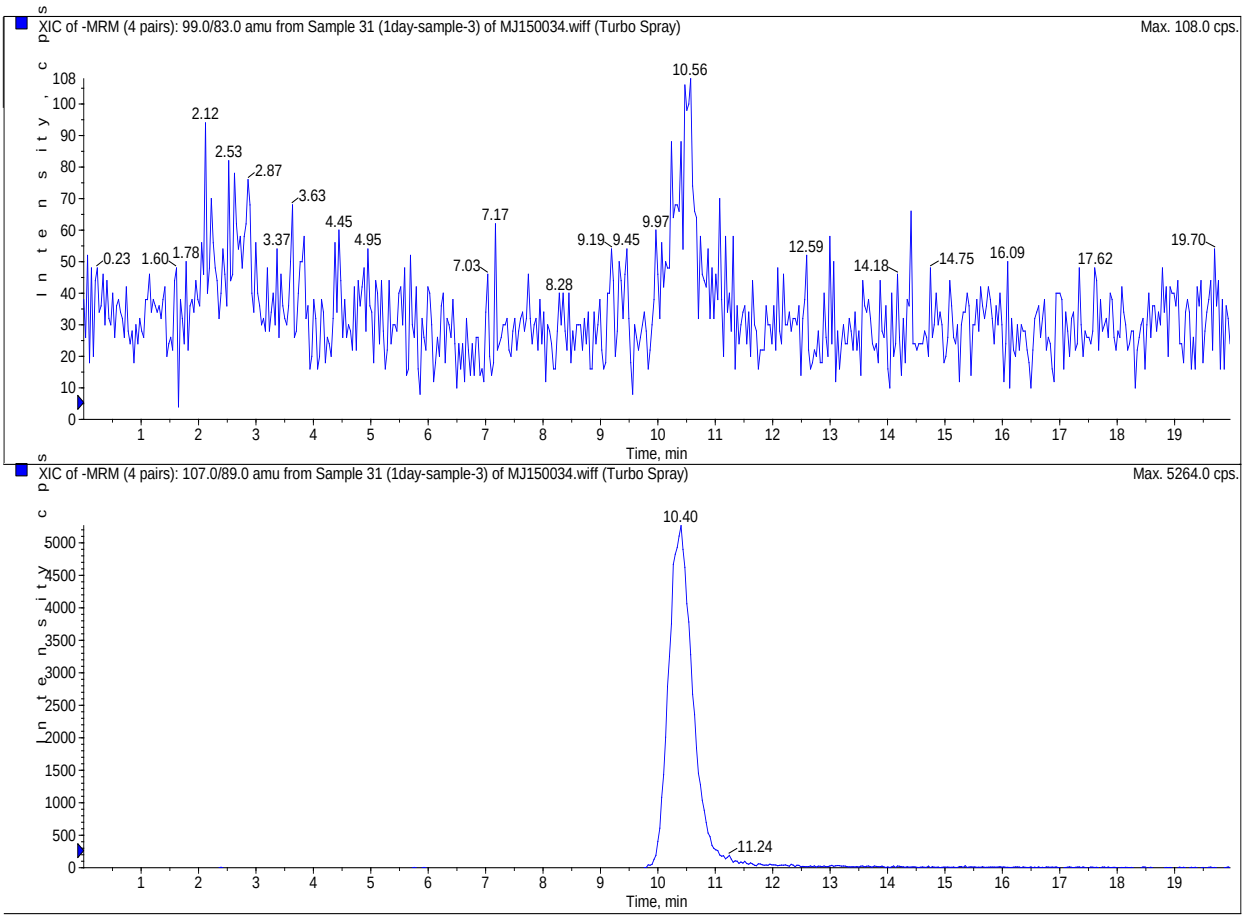
Appendix-C Chromatograms

XIX. Figure 19. 1 Day exposure sample-3 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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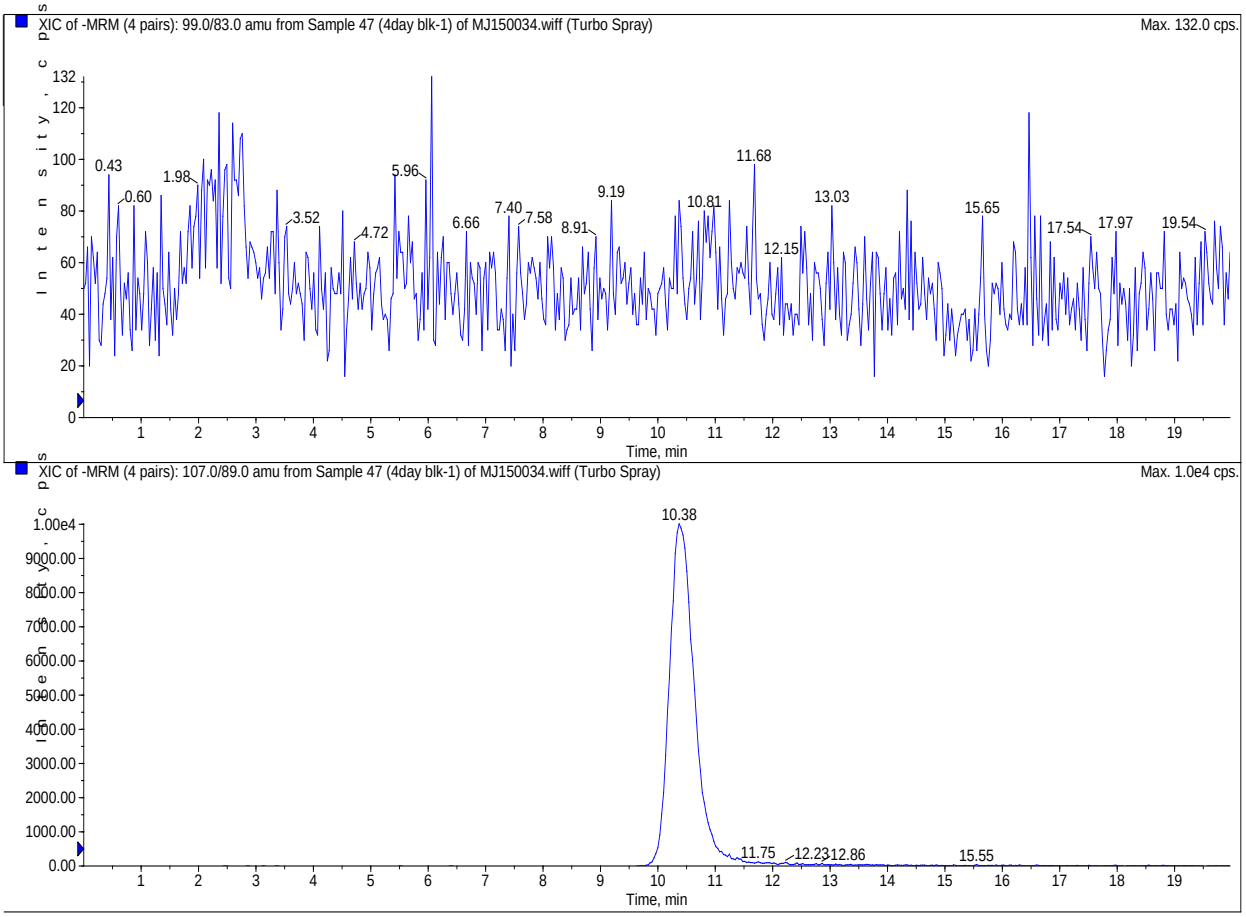
XX. Figure 20. 4 Days exposure blank-1 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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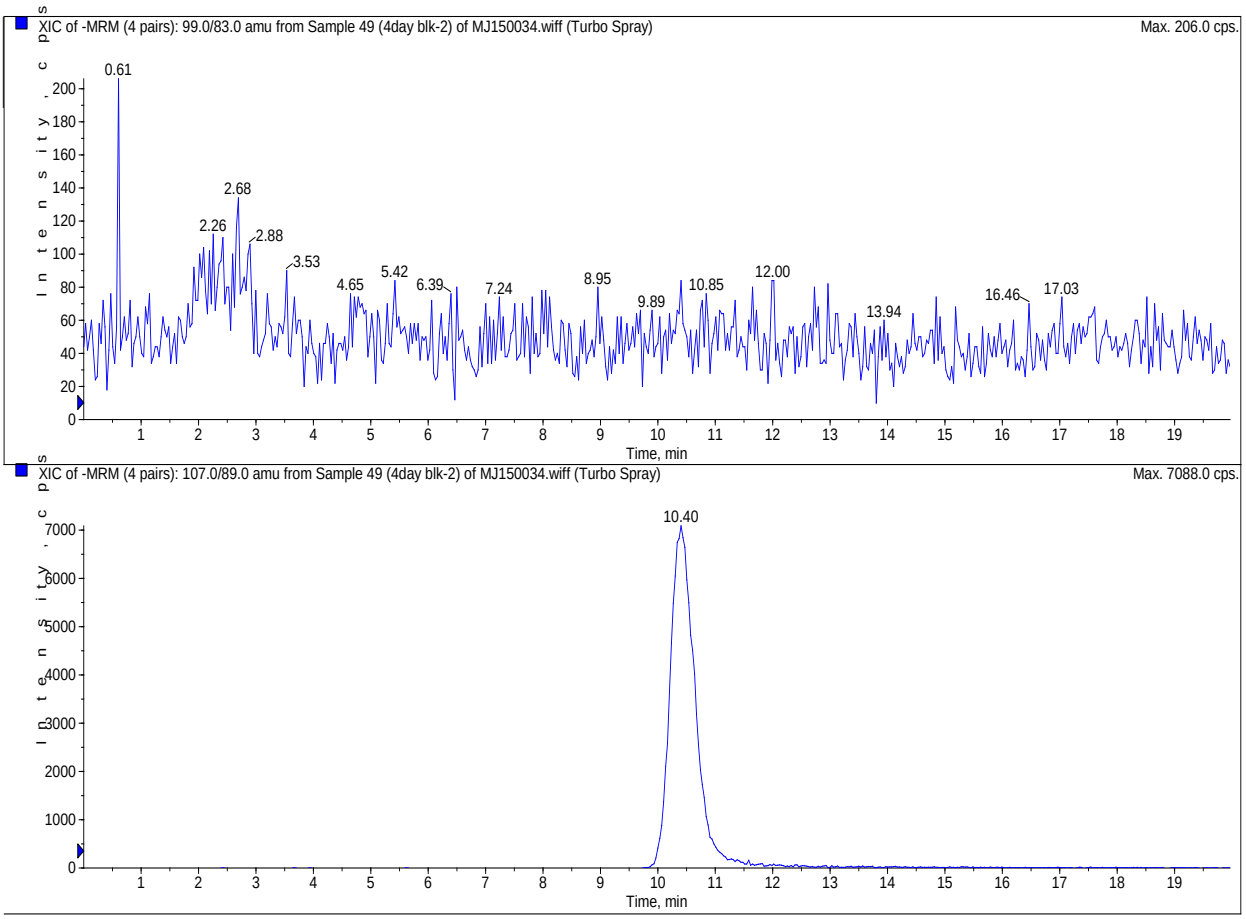
Appendix-C Chromatograms

XXI. Figure 21. 4 Days exposure blank-2 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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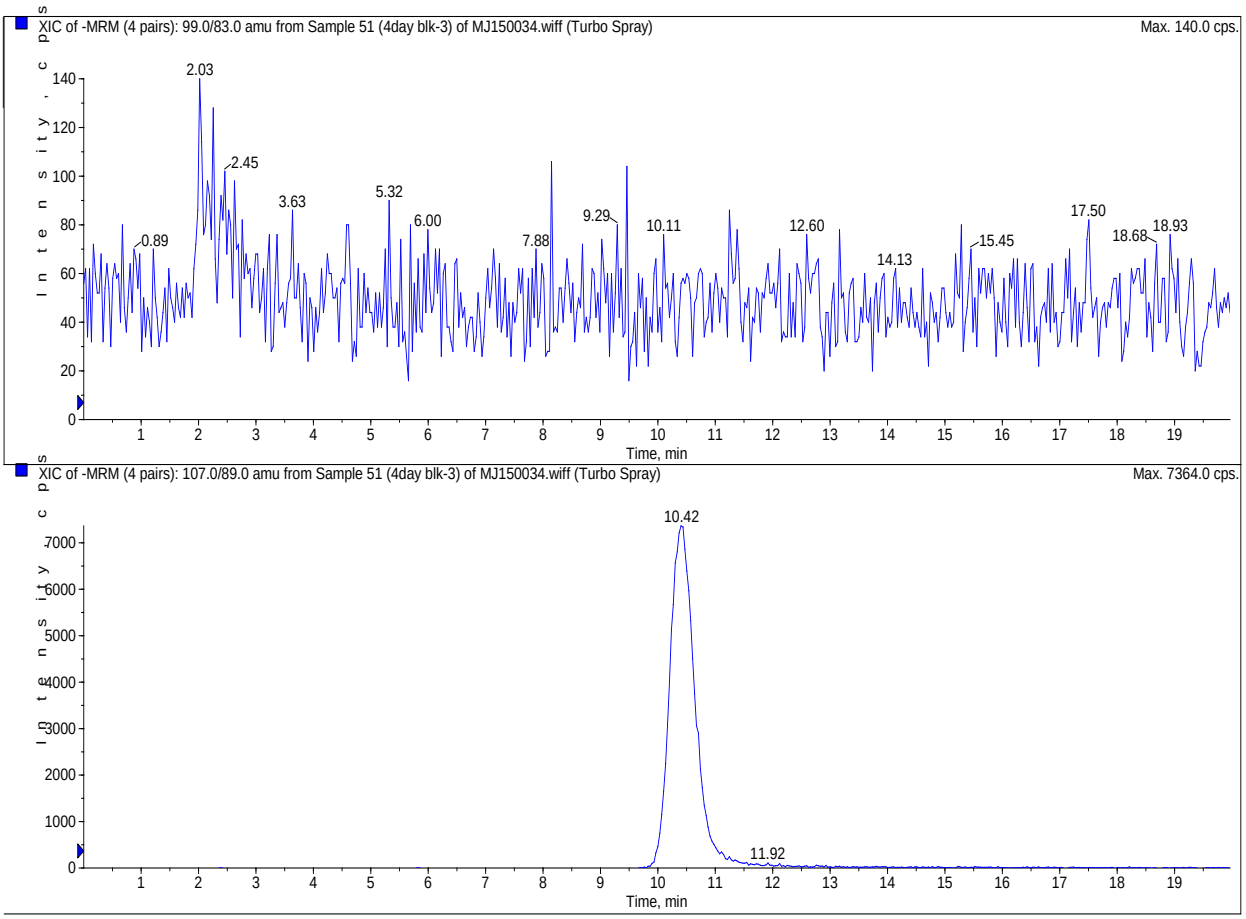
XXII. Figure 22. 4 Days exposure blank-3 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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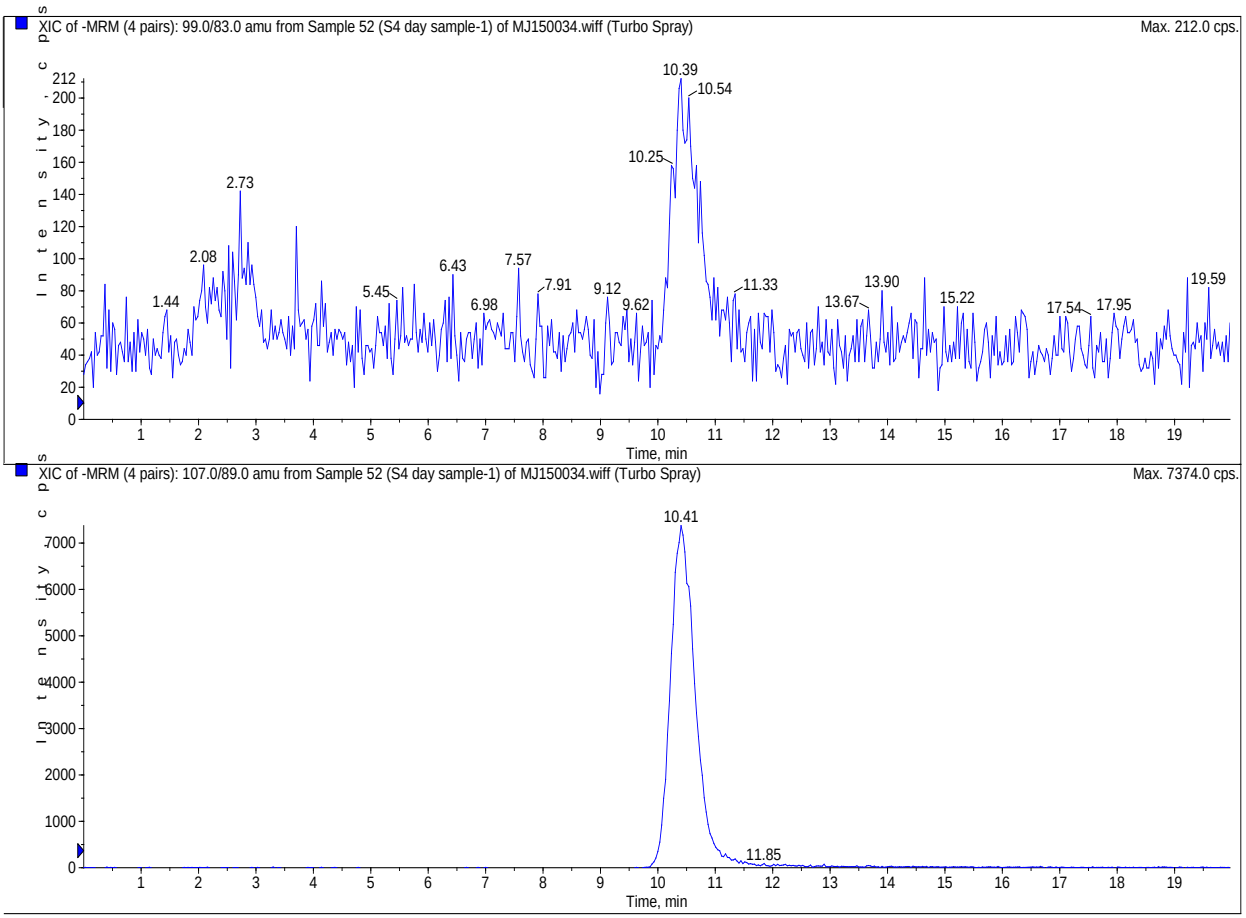
XXIII. Figure 23. 4 Days exposure sample-1 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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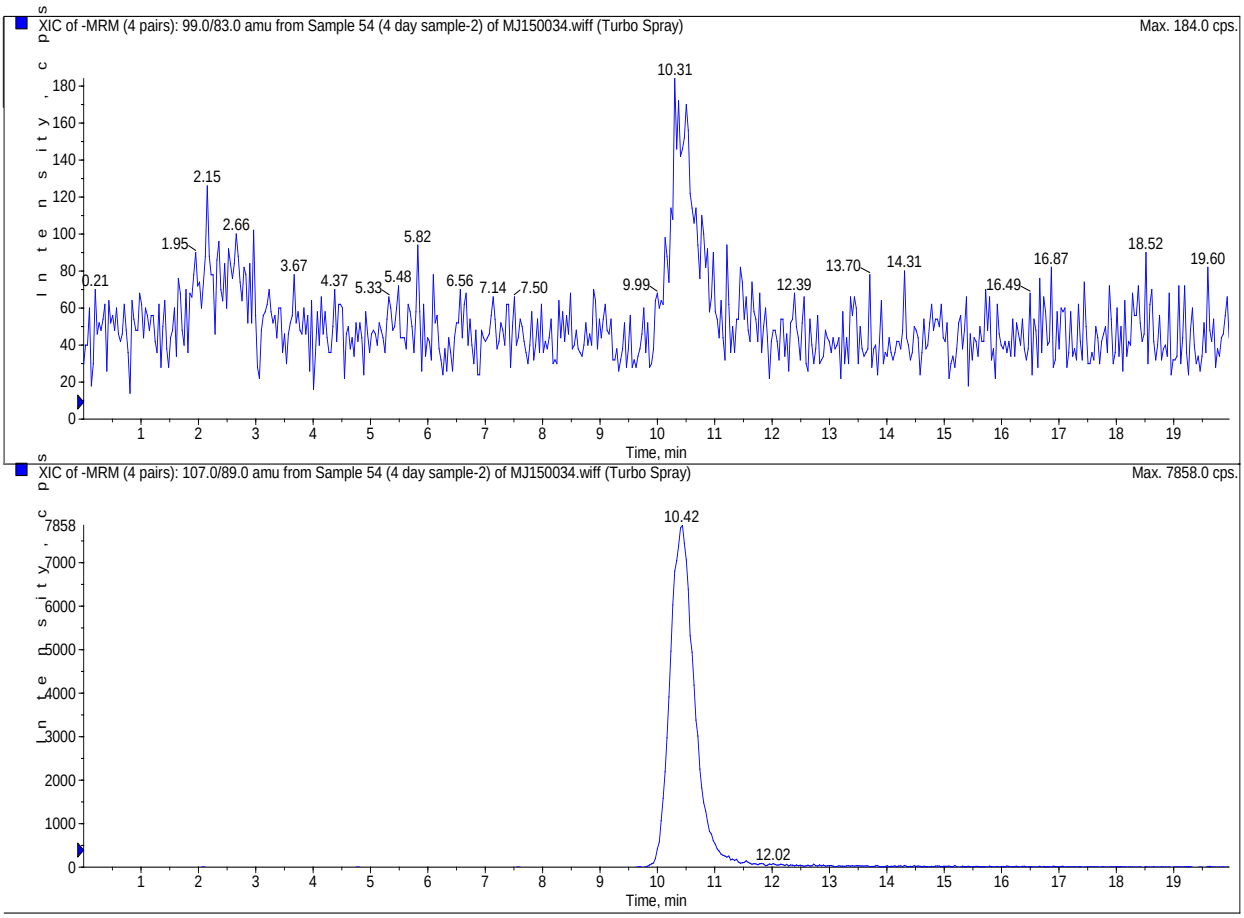
XXIV. Figure 24. 4 Days exposure sample-2 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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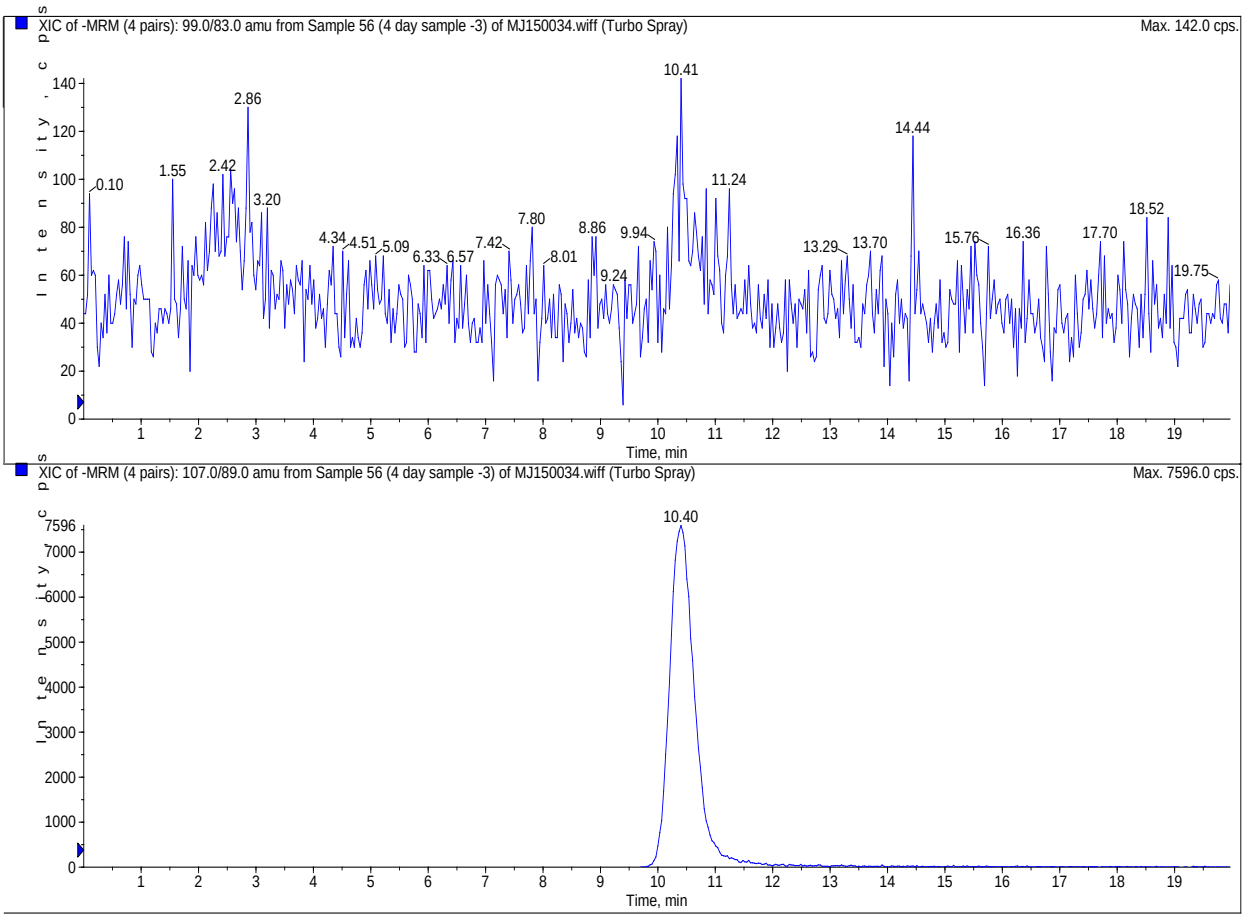
XXV. Figure 25. 4 Days exposure sample-3 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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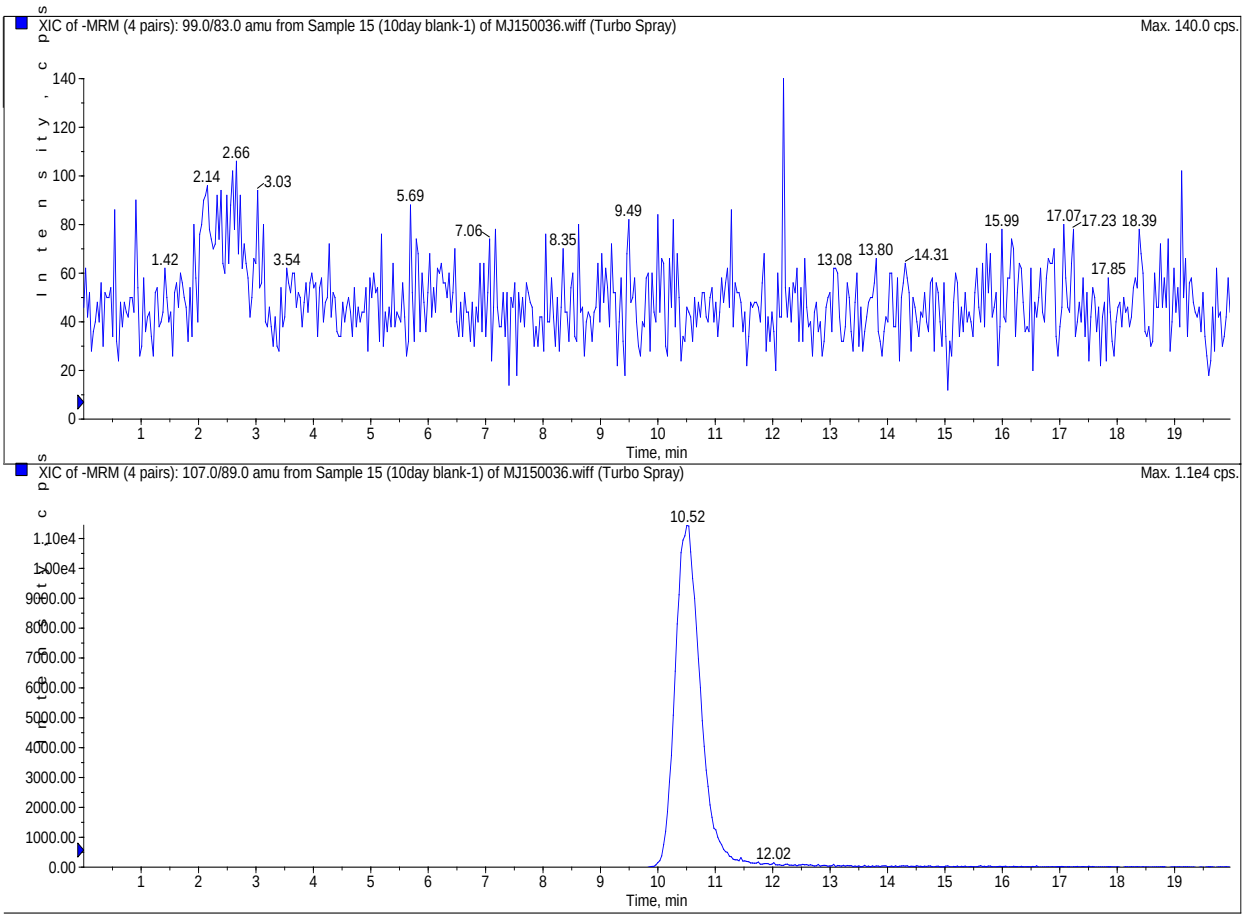
XXVI. Figure 26. 10 Days exposure blank-1 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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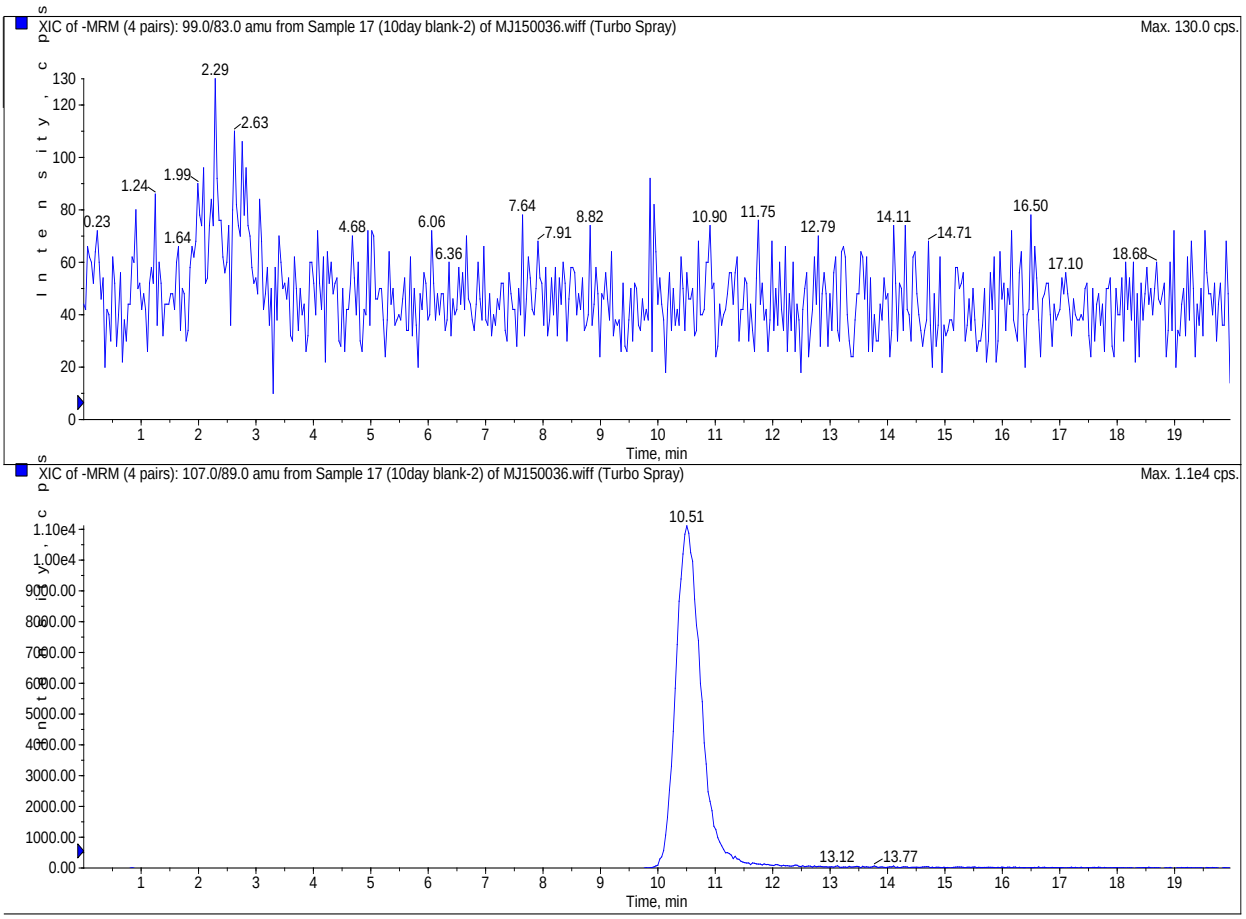
XXVII. Figure 27. 10 Days exposure blank-2 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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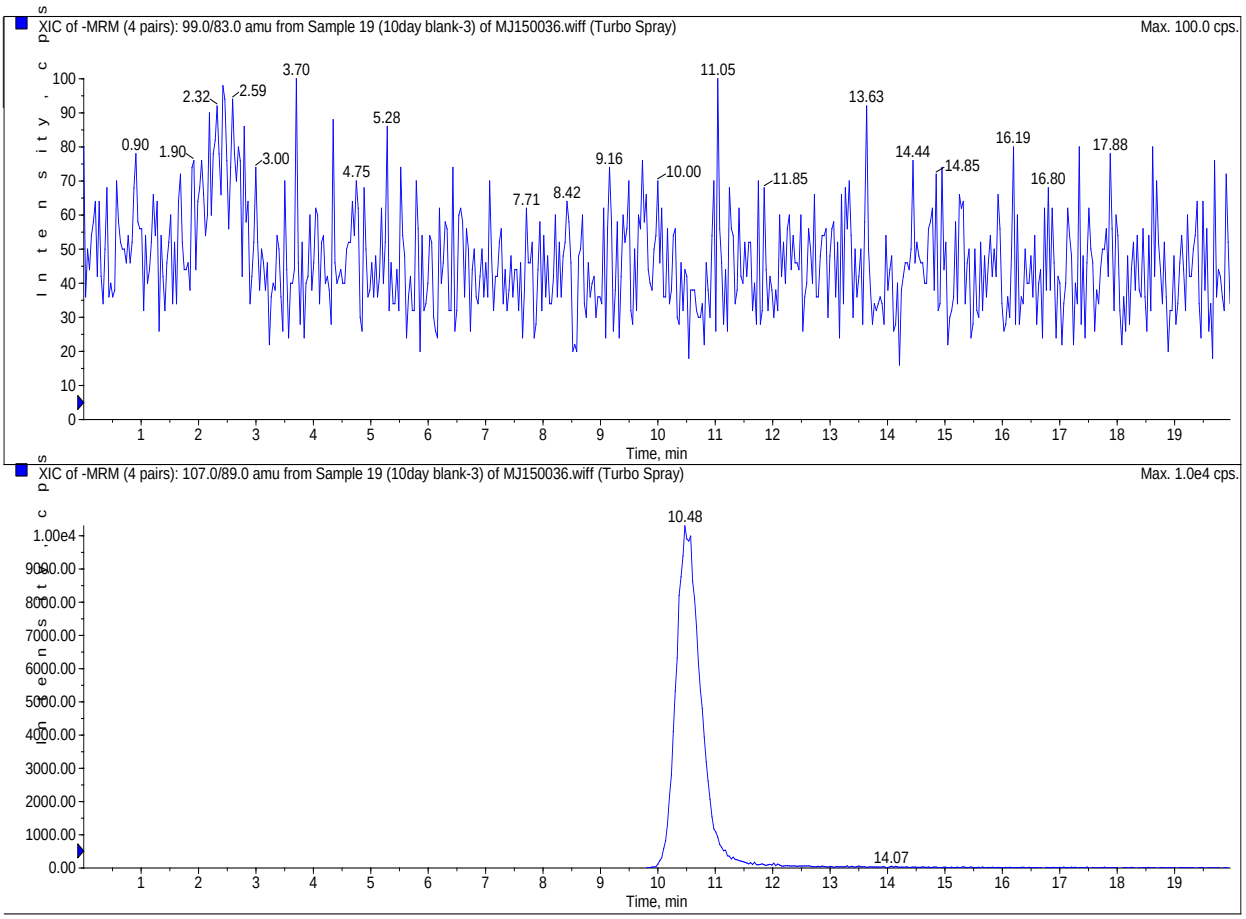
XXVIII. Figure 28. 10 Days exposure blank-3 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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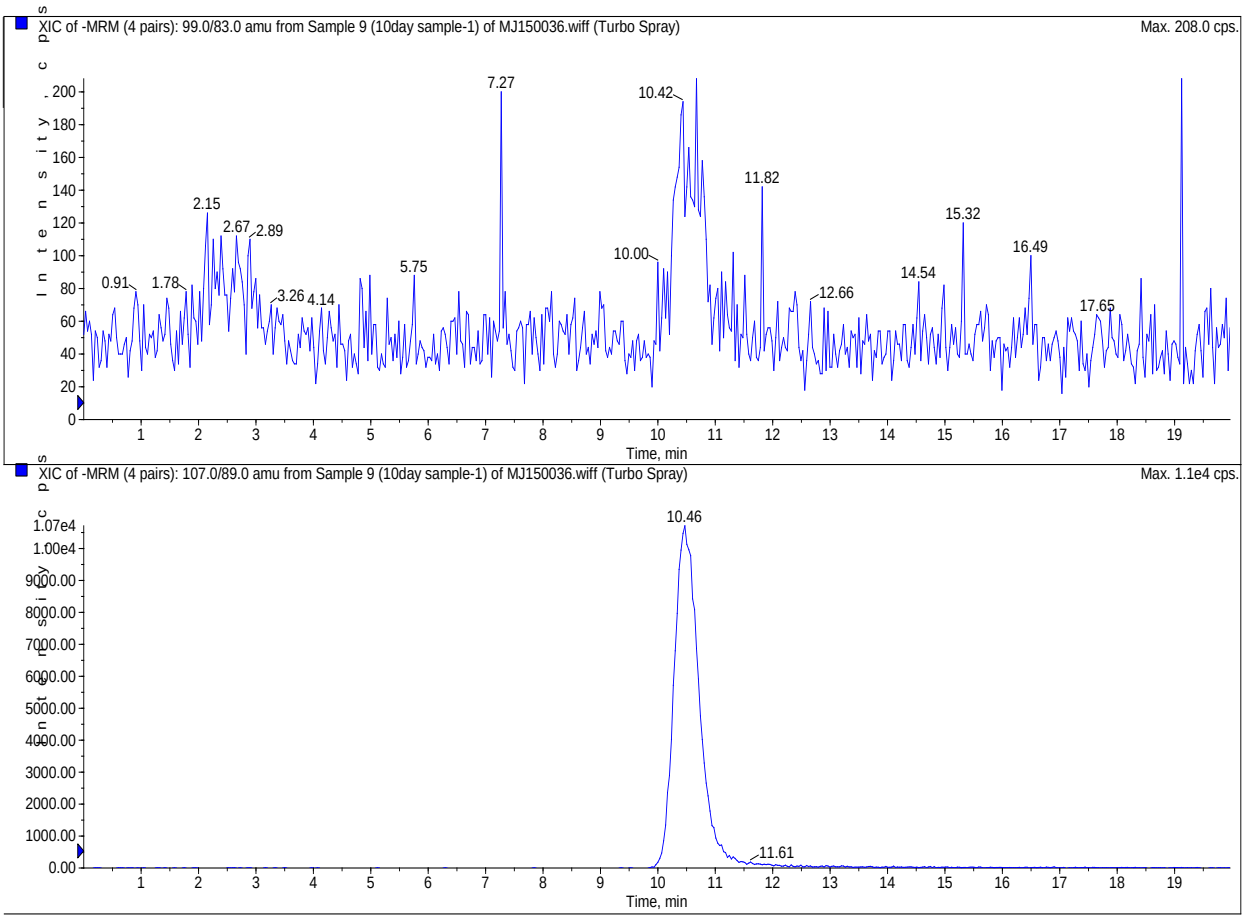
XXIX. Figure 29. 10 Days exposure sample-1 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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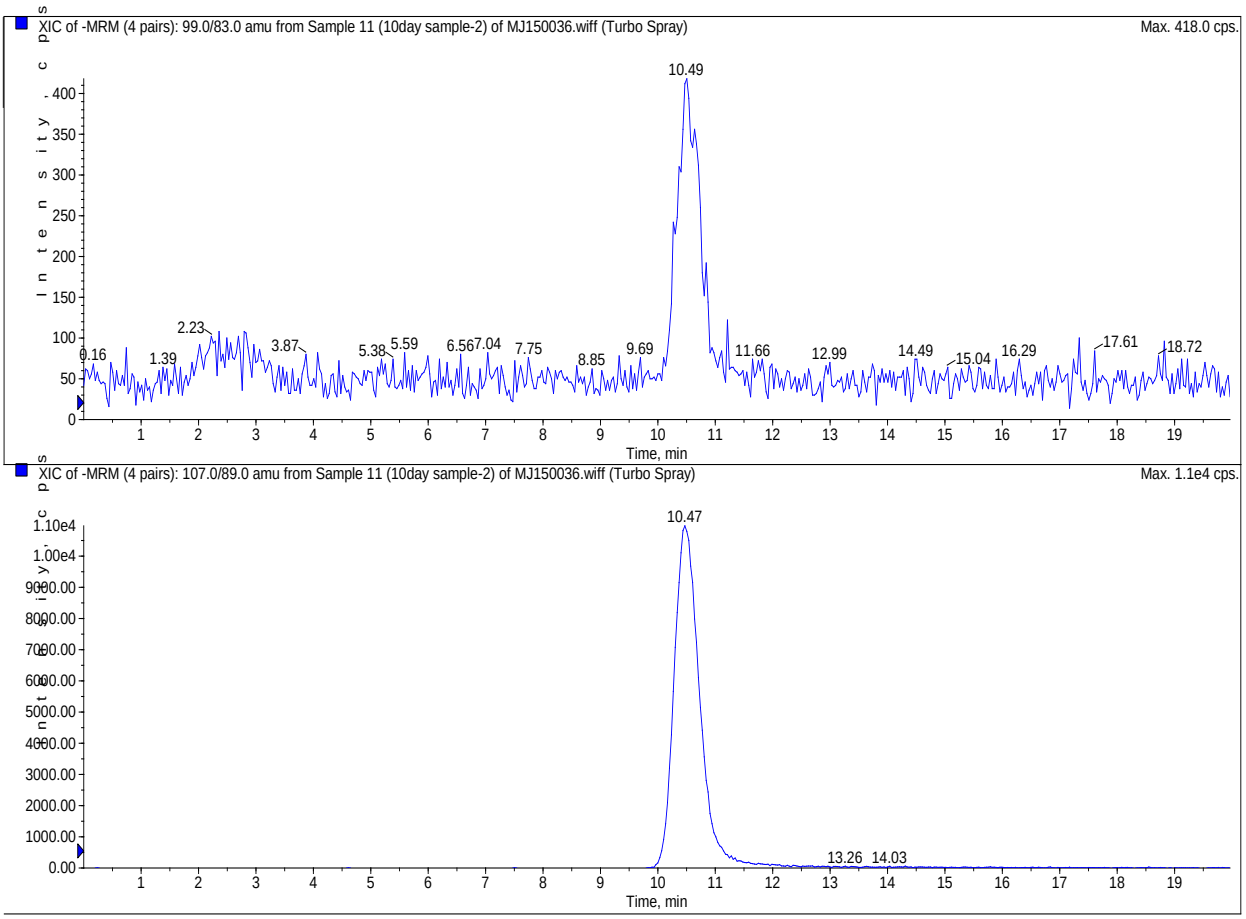
XXX. Figure 30. 10 Days exposure sample-2 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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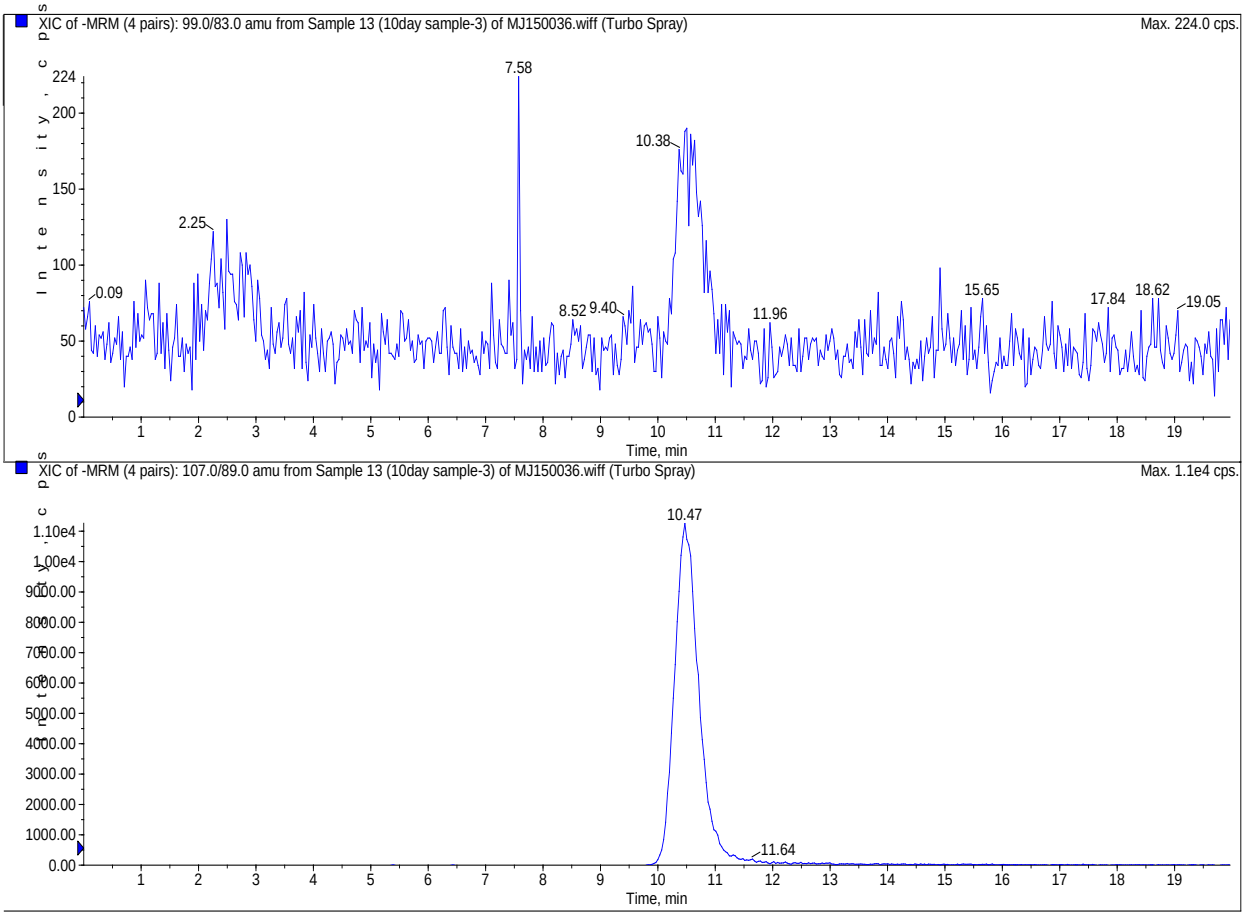
XXXI. Figure 31. 10 Days exposure sample-3 with $Cl^{16}O_4$ (upper) and internal standard $Cl^{18}O_4$ (lower)



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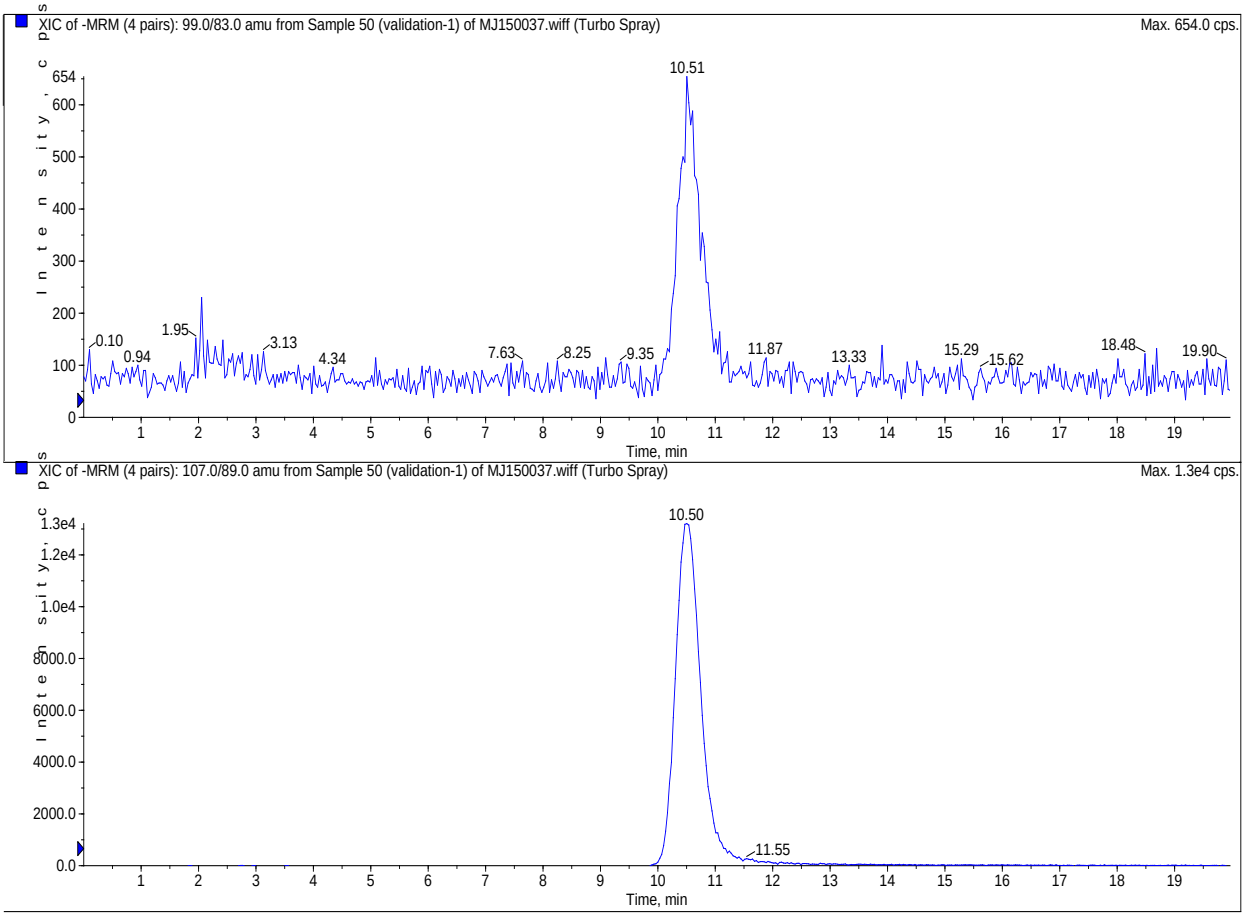
XXXII. Figure 32. Validation with 10 days blank-1. $Cl^{16}O_4$ (upper) was spiked at the level of 150 ppt in food and internal standard $Cl^{18}O_4$ (lower)



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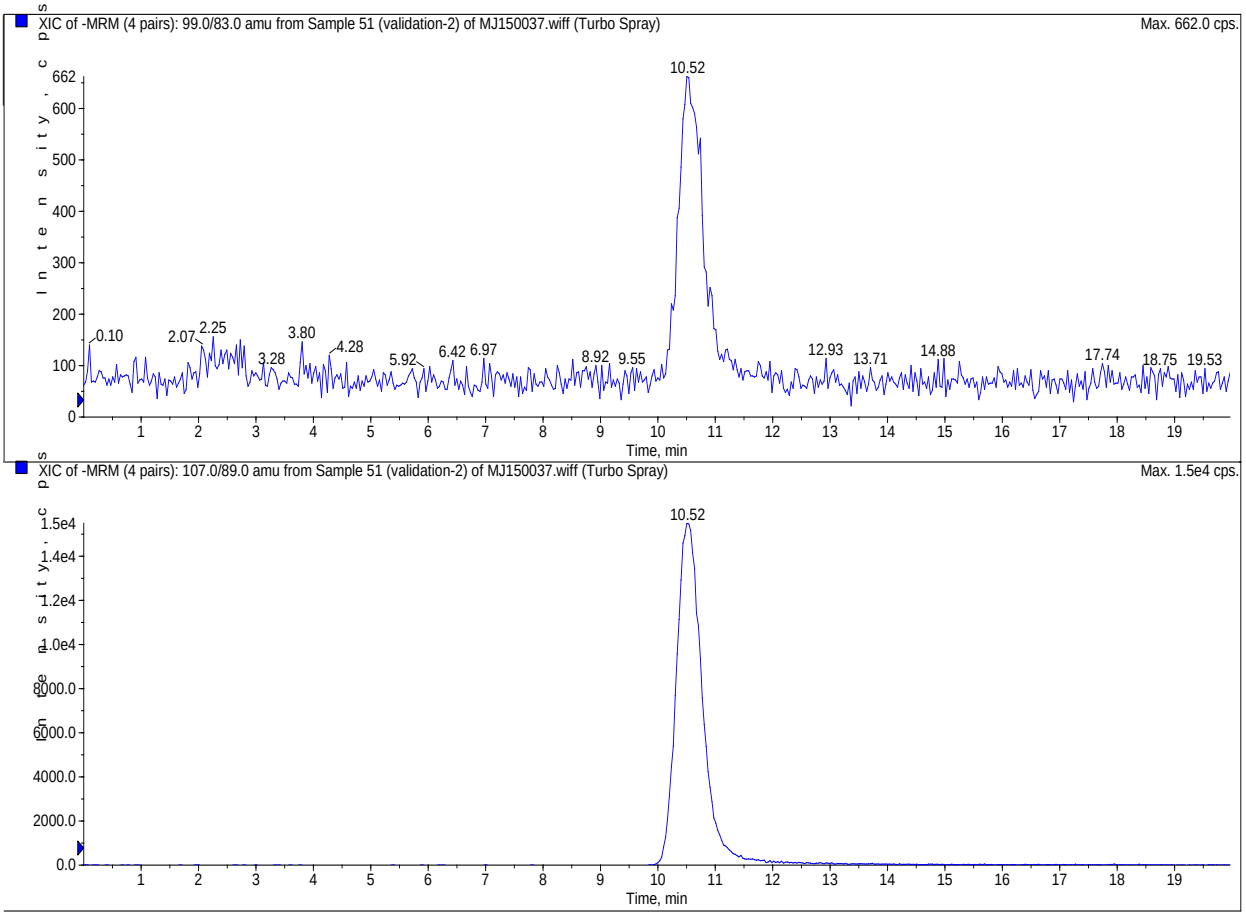
XXXIII. Figure 33. Validation with 10 days blank-2. $Cl^{16}O_4$ (upper) was spiked at the level of 150 ppt in food and internal standard $Cl^{18}O_4$ (lower)



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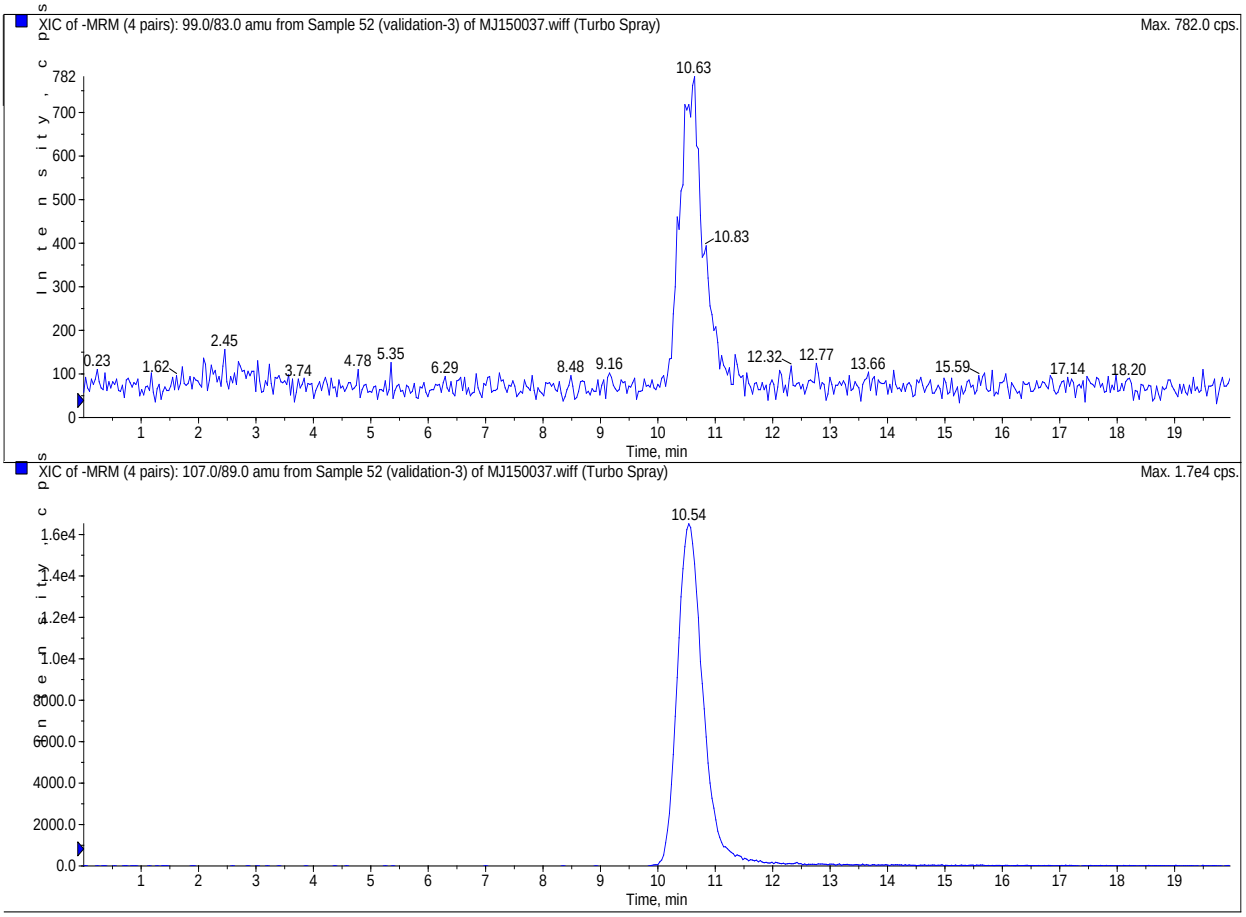
XXXIV. Figure 35. Validation with 10 days blank-3. $Cl^{16}O_4$ (upper) was spiked at the level of 150 ppt in food and internal standard $Cl^{18}O_4$ (lower)



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Appendix-D Spiking Validation at Low Perchlorate Concentrations

Issued:

Author: _____ Date: _____

Approved: _____ Date: _____

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Appendix-D Spiking Validation at Low Perchlorate Concentrations

I. Spiking validation with 10 days blank migration samples

Blank polymer plaque with 10 days exposure was used for validation. Tenax® was transferred into a beaker followed by spiking with 6 ng of perchlorate $Cl^{16}O_4$, corresponding to the LOQ level of 1.5 ng/in^2 (150 ppt in food). Then 0.1 mL of internal standard ^{18}O-labeled perchlorate $Cl^{18}O_4$ at 1.5 ppm and 150 mL of ACN was added. The solution was stirred for one minute and sit still for 5 min. 50 mL of the supernatant was then transferred into rotary evaporator tube for drying down to approximately 10 mL. The solution was then filtered through 0.2 µm PTFE syringe filter for LC/MS/MS analysis.

Table 1 Validation with 10 days blank migration samples

Validation replicate	Perchlorate spiked* (ng/in^2)	Amount Spike* (ng)	Perchlorate found* (ng/in^2)	Amount Found* (ng)	Recovery (%)
1	1.53	6.12	1.57	6.26	102.3
2	1.53	6.12	1.51	6.04	98.6
3	1.53	6.12	1.49	5.97	97.5
AVG					99.4
STD					2.5
RSD (%)					2.5

*, data are reported as perchlorate ion $Cl^{16}O_4$

II. Spiking validation with Tenax® at low perchlorate concentrations

Spiking experiments were also carried out with perchlorate spiked directly into Tenax® at one fourth and half of the method LOQ level at 0.4 and 0.8 ng/in^2. Except for the exposure procedure, all sample preparation procedures were the same as those for analysis of migration samples. Results are shown in Table 2 Spiking at 0.4 ng/in^{2}and Table 3 spiking at 0.8 ng/in^2

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Migration Report

Migration of Sodium Perchlorate from Low Density Polyethylene containing 20% Irgastat P18 into Food Simulant.

Appendix-D Spiking Validation at Low Perchlorate Concentrations

Table 2 Spiking at 0.4 ng/in^2

Validation replicate	Perchlorate spiked* (ng/in^2)	Amount Spike* (ng)	Perchlorate found* (ng/in^2)	Amount Found* (ng)	Recovery (%)
1	0.4	1.5	0.5	1.9	125
2	0.4	1.5	0.3	1.4	91
3	0.4	1.5	0.4	1.8	116
AVG					111
STD					18
RSD (%)					16

*, data are reported as perchlorate ion $Cl^{16}O_4$

Table 3 spiking at 0.8 ng/in^2

Validation replicate	Perchlorate spiked* (ng/in^2)	Amount Spike* (ng)	Perchlorate found* (ng/in^2)	Amount Found* (ng)	Recovery (%)
1	0.8	3.1	0.8	3.3	113
2	0.8	3.1	1.0	4.1	142
3	0.8	3.1	0.9	3.7	126
AVG					127
STD					15
RSD (%)					12

*, data are reported as perchlorate ion $Cl^{16}O_4$

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