REPORT L.NR. 7871-2023

Environmental Analysis of Imidacloprid in Norwegian Marine and Terrestrial Environments

Photo: Tom Dale

Norwegian Institute for Water Research

REPORT

Main Office
Økernveien 94
NO-0579 Oslo, Norway
Phone (47) 22 18 51 00

NIVA Region South
Jon Lilletuns vei 3
NO-4879 Grimstad, Norway
Phone (47) 22 18 51 00

NIVA Region East
Sandvikaveien 59
NO-2312 Ottestad, Norway
Phone (47) 22 18 51 00

NIVA Region West
Thormøhlensgate 53 D
NO-5006 Bergen Norway
Phone (47) 22 18 51 00

NIVA Denmark
Njalsgade 76, 4th floor
DK 2300 Copenhagen S, Denmark
Phone (45) 39 17 97 33

Internet: www.niva.no

Title	Serial number	Date
Environmental Analysis of Imidacloprid in Norwegian Marine and Terrestrial Environments	7871-2023	9 June 2023
Author(s) Kine Bæk Malcolm Reid	Topic group Environmental monitoring	Distribution Public
	Geographical area Norway	Pages 18

Client(s)	Client's contact person
Benchmark Animal Health AS	Meghan Rochford, Technical Environmental Specialist
	Printed NIVA Project number O-210324

Summary

Norwegian Institute for Water Research (NIVA) was contracted by Benchmark Animal Health Norway AS to perform environmental sampling and chemical analysis of Imidacloprid. This associated with medicinal delicing treatments in aquaculture in 2022. The sampling scheme for this investigation was designed to ensure coverage of all 13 Aquaculture Production Zones in Norway. Samples from terrestrial environments (freshwater and sediments) with no link to marine aquaculture were also included. Sample preparation and analysis of Imidacloprid in water and sediment was performed in accordance with a validated method.

Imidacloprid was below limits of quantification in all samples.

Four keywords

1. Imidacloprid
2. Environmental Monitoring
3. Chemical Analysis
4. Aquaculture

This report is quality assured in accordance with NIVA's quality system and approved by:

Kine Bæk	*Malcolm Reid*	*Malcolm Reid*
Project Manager/Main Author	Quality Assurance	Research Manager

ISBN 978-82-577-7607-7
NIVA-report ISSN 1894-7948

Environmental Analysis of Imidacloprid in Norwegian Marine and Terrestrial Environments

Table of contents

Summary

Norwegian Institute for Water Research (NIVA) was contracted by Benchmark Animal Health Norway AS to perform environmental sampling and chemical analysis of Imidacloprid. This associated with medicinal delicing treatments in aquaculture. Dialogue with Benchmark Animal Health Norway AS confirmed 50 distinct sites were treated with a combined 18,040.2 kg of Imidacloprid-based medication in the period congruent to environmental sampling. Benchmark Animal Health Norway AS reported a maximum combined release of 0.27kg of Imidacloprid to marine waters during this timeframe. This release is a conservative estimate based on a maximum allowable discharge concentration of 0.3 µg/L.

The sampling scheme for this investigation was designed to ensure coverage of all 13 Aquaculture Production Zones in Norway. Samples from terrestrial environments (freshwater and sediments) with no link to marine aquaculture were also included.

NIVA collected the following samples during the course 2022:

- 101 marine water samples from across the Norwegian coastal area
- 21 sediment samples across the Norwegian coastal area
- 18 freshwater samples from Norwegian rivers.

Marine waters were sampled via NIVAs FerryBox system. This platform allows for the collection, analysis and presentation of oceanographic and water quality data. The 10 rivers sampled within this monitoring programme discharge to Skagerrak, the North Sea, the Norwegian Sea and the Barents Sea. Water and sediment from Norway's largest lake, Mjøsa were also included. Sampling was carried out according to standard NS-ISO 5667-6:2014 "Water quality — Sampling — Part 6: Guidance on sampling of rivers and streams". Sediment samples were collected by means of a van Veen grab (0.15 m2) or by hand.

Sample preparation and analysis of Imidacloprid in water and sediment was performed in accordance with the validated method (NIVA Ref 0325/22).

Imidacloprid was below 1 ng/L in all water samples, and below 0.1 ng/g in sediment samples.

Acknowledgements

Assistance in the collection of marine samples was kindly provided by Tor Erik Eriksen, Pernilla Marianne Carlsson, Carl Ballantine, Rita Næss, Florian Wester and Cathrine Brecke Gundersen. Two marine samples were collected with support of the Urban Fjord programme which is administered by the Norwegian Environment Agency. Freshwater samples were collected with support of The Norwegian River Monitoring Programme and Milfersk (environmental contaminants in freshwater food webs) which are administered by the Norwegian Environment Agency.
Chemical analysis was carried out by Belinda Valdecanas. Analytical method development was by Thomas Runderget and Jose-Antonio Baz Lomba.

1 Introduction

Norwegian Institute for Water Research (NIVA) was contracted by Benchmark Animal Health Norway AS to perform environmental sampling and chemical analysis of Imidacloprid.

NIVA collected the following samples during the course 2022:

- 101 marine water samples from across the Norwegian coastal area
- 21 sediment samples across the Norwegian coastal area
- 18 freshwater samples from Norwegian rivers.

NIVA was to perform analysis upon completion of method validation. Method sensitivity for solids detection was required to 0.1 µg/kg and for water samples to 0.02 µg/L Imidacloprid.

2 Methods

2.1 Sampling

The sampling scheme was designed to ensure coverage of all 13 Aquaculture Production Zones in Norway (Figure 1). Samples from terrestrial environments (freshwater and sediments) with no link to marine aquaculture were also included.

2.1.1 Marine Waters

Marine waters were sampled via NIVAs FerryBox system (Table 1 and Table 2). This platform allows for the collection, analysis and presentation of oceanographic and water quality data. It combines information from sensors installed on board ships sailing along fixed routes with data from environmental satellites and discrete water samples.

Water is collected via an intake in the hull at a depth of 4 meters. The water is pumped with the aid of a peristaltic pump via a short pipe system past the sensors, where automatic measurements are taken. Water samples are collected automatically into sample bottles in a refrigerator where they are kept cold and dark. Samples are transferred to land after docking in Oslo. The samples are processed at NIVA's sample reception at NIVA and stored frozen at -20degC until analyses.

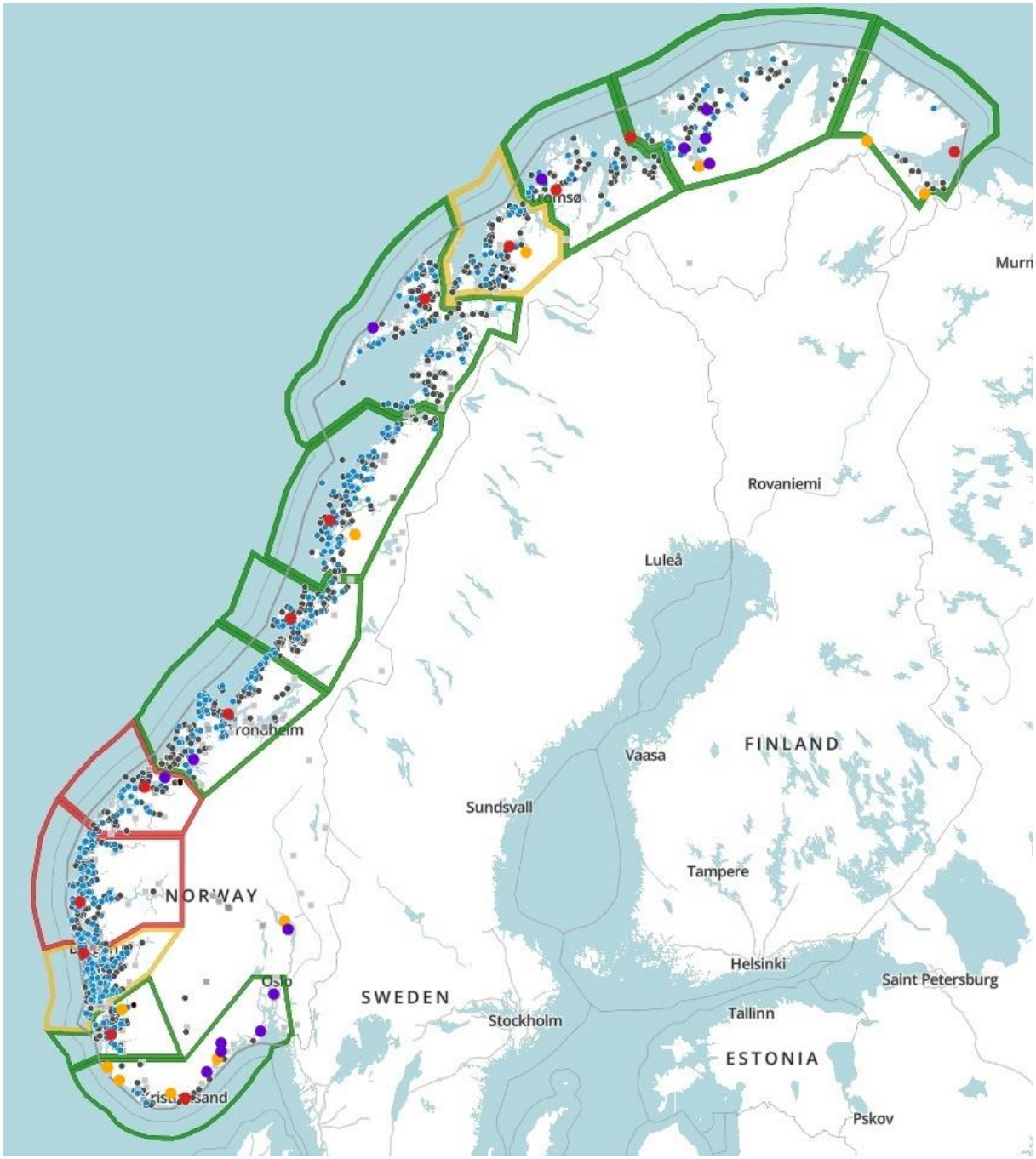

Figure 1. Geographic distribution of sampling locations in relation to the 13 Aquaculture Production Zones in Norway. (Source: Directorate of Fisheries, Norway). Production zones are marked with coloured borders. Aquaculture facilities are marked with blue dots where fish were present, and black dots where fish are not housed as of 21 March 2022. Marine water sampling sites are marked with red dots. Freshwater sampling sites are marked with yellow dots. Sediment sampling locations are marked with purple dots.

Table 1. Sampling locations for marine waters. Zones 1 and 2 were sampled at the fixed location provided. Zones 3-13 were sampled as linear transects within the rectangular area defined by latitude/longitude minima and maxima.

Aquaculture Zones		Longitude		Latitude	
#	Name	Min.	Maxi.	Min.	Max.
1	Swedish Border to Jæren	8.063		58.177	
2	Ryfylke	5.800		59.067	
3	Karmøy to Sotra	4.76	5.27	60.44	60.99
4	Nordhordaland to Stadt	4.69	5.60	61.22	62.32
5	Stadt to Hustadvika	6.85	8.90	62.68	63.47
6	Nordmøre to Sør-Trondelag	8.51	9.91	63.40	63.51
7	Nord-Trøndelag to Bindal	11.37	11.37	64.94	64.94
8	Helgeland to Bodø	12.88	12.88	66.15	66.15
9	Vestfjorden and Vesteråsen	15.43	15.43	68.70	68.70
10	Andøya to Senja	17.82	17.83	69.15	69.15
11	Kvaløya to Loppa	19.46	19.46	69.81	69.81
12	Vest-Finnmark	21.13	21.14	70.20	70.20
13	Øst-Finnmark	30.08	30.09	69.76	69.76

Table 2. Sampling dates for marine waters. Zones 1-13 represent each of the thirteen Aquaculture Production Zones in Norway

Zone	Sampling Dates (dd/mm 2022)								
1	22/03	28/04	1/05	8/06	1/07	16/09	8/10	30/11	
2	25/04	23/05	20/06	25/07	22/08	20/09	24/10	14/11	20/12
3	19/03	21/04	15/06	4/08	3/09	30/11	22/12		
4	19/03	21/04	24/05	15/06	29/07	3/09	30/11	22/12	
5	18/03	20/04	23/05	14/06	29/07	2/09	29/11	21/12	
6	18/03	20/04	23/05	14/06	28/07	1/09	29/11	21/12	
7	28/03	13/06	28/07	1/09	28/11	20/12			
8	17/03	19/04	22/05	13/06	27/07	1/09	28/11	20/12	
9	16/03	18/04	21/05	12/06	27/07	22/08	27/11	19/12	
10	16/03	18/04	21/05	12/06	26/07	22/08	27/11	19/12	
11	26/03	17/04	20/05	11/06	26/07	22/08	26/11	18/12	
12	15/03	17/04	20/05	11/06	25/07	22/08	26/11	18/12	
13	14/03	16/04	19/05	10/06	25/07	22/08	17/12		

2.1.2 Freshwater Samples

The 10 rivers sampled within this monitoring programme discharge to Skagerrak, the North Sea, the Norwegian Sea and the Barents Sea (Table 3, Figure 1). The rivers are selected based on geographical location and access to existing infrastructure for sampling. Three samples were also taken from Norway's largest lake, Mjøsa.
Sampling was carried out according to standard NS-ISO 5667-6:2014 "Water quality — Sampling — Part 6: Guidance on sampling of rivers and streams".
All samples were collected in the period 7 February – 3 October 2022. Samples were transported under refrigeration and stored frozen at -20degC until analyses.

Table 3. River and lake samples included in the programme.

Location	Sampling Dates (d/m/2022)	UTM (east)	UTM (north)	UTM zone	Catchment (km^2)	Drainage basin
Storelva	7/2 3/10	498897	6503307	32	408	Skagerrak
Otra	7/2 3/10	438737	6449755	32	3738	Skagerrak
Bjerkreimselva	7/2 3/10	325246	6487028	32	705	North Sea
Orreelva	7/2 3/10	299152	6515475	32	105	North Sea
Vikedalselva	7/2 3/10	325319	6599745	32	118	North Sea
Vefsna	2/5	418710	7292351	33	4122	Norwegian Sea
Målselva	2/5	406570	7660047	34	3239	Barents Sea
Altaelva	9/5	586586	7759686	34	7373	Barents Sea
Tana	9/5	543964	7791926	35	16389	Barents Sea
Pasvikelva	2/5	386937	7709634	36	18404	Barents Sea
Mjøsa	24/8	612056	6739772	32	16420	Vorma River
Mjøsa	24/8	612423	6740405	32	16420	Vorma River
Mjøsa	24/8	612920	6740904	32	16420	Vorma River

2.1.3 Sediment Samples

Sediments were collected by means of a van Veen grab (0.15 m2). Four grabs of the top layer (0-2 cm in grab samples with undisturbed surface) were prepared for one sample. See Table 4 for locations.
All samples were collected in the period 5 July to 17 October 2022. Samples were transported under refrigeration and stored frozen at -20degC until analyses.

Table 4. Sampling locations for marine and terrestrial sediments

Location	Latitude	Longitude	Water Depth (m)	Sediment Class
Grefstadvika Grimstad	58.35685	8.66125	<5	Marine
Ølbergholmen Larvik	59.00682	10.13149	<5	Marine
Bekkelaget Oslofjord	59.88230	10.74668	20	Marine
Alna Oslofjord	59.89477	10.74815	55	Marine
Nævestadfjorden Agder	58.67676	9.03974	<5	Marine
Songevannet Agder	58.67995	9.01146	<5	Marine
Lagfjorden Agder	58.68328	9.07223	<5	Marine
Ildjernsflua Oslofjord	59.85923	10.62620	60	Marine
Unstadstranden Lofoten	68.27017	13.58269	<5	Marine
Tingvollfjorden Gjemnes	62.90828	8.10438	<5	Marine
Vegglandet Hammerfest	70.54665	23.23308	185	Marine
Lerresfjord Alta	70.29295	23.46017	120	Marine
Fannefjorden Molde	62.79312	7.63085	<5	Marine
Langfjorden Alta	70.13801	22.88768	65	Marine
Grøtfjord Tromsø	69.74020	18.47905	<5	Marine
Lathari Alta	69.98533	23.44843	<5	Marine
Tollevika Alta	69.97888	23.25958	<5	Marine
Blåmannsvika Tromsø	69.74724	18.63271	<5	Marine
Mjøsa Hamar	60.77760	11.05769	5	Freshwater Lake
Mjøsa Hamar	60.78317	11.06478	<5	Freshwater Lake
Mjøsa Hamar	60.78751	11.07419	<5	Freshwater Lake

2.2 Analysis

Sample preparation and analysis of Imidacloprid in water and sediment was performed in accordance with the validated method. For details, see the Validation Report (NIVA Ref 0325/22).

In brief, water samples were defrosted and an aliquote of 50-mL was transferred (volumetrically) to 50mL Falcon tubes and spiked with 100 µL of a 10 ng/mL solution of imidacloprid-d4 in methanol. Solid Phase Extraction (SPE) was then performed on Oasis HLB 6cc 200mg extraction cartridges. The SPE eluates were dried under a stream of nitrogen and reconstituted in 0.5mL of water/acetonitrile 1:1 for analysis.

Sediments samples of 10g were spiked with 50 µL of a 10 ng/mL solution of imidacloprid-d4 in methanol. Extraction was performed by shaking with 5 mL milliQ followed by additional shaking upon addition of 10 mL acetonitrile. Aliquots (5 mL) of the acetonitrile phase were then transferred to a clean tube and evaporated down to 1 mL. Sample clean-up was via addition of dispersive-SPE sorbent (PSA, 40–60 µm), and the final volume of cleaned acetonitrile transferred to a new glass-vial (2 mL). The volume was reduced to dryness under a stream of nitrogen and then reconstituted in 1 mL 10% acetonitrile in water. A final addition of 0.1 mL hexane was added before the vial was again shaken vigorously for 1 minute. The hexane phase is removed, and the remaining volume retained for analysis.

Quantitative analysis was performed via Liquid chromatography Mass Spectrometry (LCMS) on a Waters Xevo TQ-S combined with a Waters Acquity UPLC system. Method performance was validated to better than 80-120% accuracy with 10% relative standard deviation in the range of 1-100 ng/L Imidacloprid in water, and 0.1 – 10 ng/g Imidacloprid in sediment.

Quality control of the analysis included review of spiked samples of marine water and sediments. Such control samples were spiked with the analytes at known concentrations, and analysis was subsequently performed in parallel with the true samples. Control samples of water were spiked to give 2 ng/L imidacloprid, while sediments were at both 1 ng/g and 10 ng/g imidacloprid.

3 Results

3.1 Water Samples

None of the target analytes were detected in any samples. Imidacloprid, 5-hydroxy-imidacloprid, desnitro-imidacloprid and imidacloprid-urea were all below 1.0 ng/L. See Appendix A for tabulated results.

Blank samples and spiked control samples at 2 ng/L imidacloprid were analysed at intervals. One blank sample and one spiked-control sample was analyzed for every batch of real samples, and with an interval of no greater than 15 samples. Blank samples showed no detectable compound, and results of the spiked samples were within validated method performance criteria of 80-120% accuracy. See Appendix B for details.

3.2 Sediment Samples

None of the target analytes were detected. Imidacloprid and 5-hydroxy-imidacloprid were below 0.1 ng/g in all samples. See Appendix A for tabulated results.

Blank samples and spiked control samples at 1 ng/g and 10 ng/g imidacloprid were analysed at intervals. One blank sample and one spiked-control sample was analyzed for every batch of real samples, and with an interval of no greater than 15 samples. Blank samples showed no detectable compound, and results of the spiked sediments were within validated method performance criteria of 80-120% accuracy for Imidacloprid. See Appendix B for details.
Accuracy was poor for 5-hydroxy-imidacloprid (58-62%) because of significant signal suppression from the complex sediment matrix in the samples. The interference observed in these samples was not evident in sediments used for method validation. It is recommended that deuterated internal standards for each of the metabolites are included in any future analyses to minimize the impact of matrix interference on accuracy, irrespective of the complexity of the sample.

4 Conclusion

Imidacloprid was below 1 ng/L in all water samples, and below 0.1 ng/g in sediment samples.

Subsequent dialogue with Benchmark Animal Health Norway AS confirmed 50 distinct sites were treated with a combined 18,040.2 kg of Imidacloprid-based medication in the period congruent to environmental sampling. Benchmark Animal Health Norway AS reported a maximum combined release of 0.27kg of Imidacloprid to marine waters within the Norwegian Aquaculture zones during this timeframe. This release is a conservative estimate based on a maximum allowable discharge concentration of 0.3 µg/L.

5 Appendices

A. Result Tables

i. Marine Water Samples

Table A1. Results of the analysis of Imidacloprid, 5-hydroxy-imidacloprid and Imidacloprid-urea in marine water samples

Zone	Sampling Date (dd-mmm-2022)	Measured concentration (ng/L)		
		Imidacloprid	5-hydroxy-imidacloprid	Imidacloprid-urea
1	22-Mar	<1.0	<1.0	<1.0
	28-Apr	<1.0	<1.0	<1.0
	1-May	<1.0	<1.0	<1.0
	8-Jun	<1.0	<1.0	<1.0
	1-Jul	<1.0	<1.0	<1.0
	16-Sep	<1.0	<1.0	<1.0
	8-Oct	<1.0	<1.0	<1.0
	30-Nov	<1.0	<1.0	<1.0
2	25-Apr	<1.0	<1.0	<1.0
	23-May	<1.0	<1.0	<1.0
	20-Jun	<1.0	<1.0	<1.0
	25-Jul	<1.0	<1.0	<1.0
	22-Aug	<1.0	<1.0	<1.0
	20-Sep	<1.0	<1.0	<1.0
	24-Oct	<1.0	<1.0	<1.0
	14-Nov	<1.0	<1.0	<1.0
	20-Dec	<1.0	<1.0	<1.0
3	19-Mar	<1.0	<1.0	<1.0
	21-Apr	<1.0	<1.0	<1.0
	15-Jun	<1.0	<1.0	<1.0
	4-Aug	<1.0	<1.0	<1.0
	3-Sep	<1.0	<1.0	<1.0
	30-Nov	<1.0	<1.0	<1.0
	22-Dec	<1.0	<0.2	<1.0
4	19-Mar	<1.0	<1.0	<1.0
	21-Apr	<1.0	<1.0	<1.0
	24-May	<1.0	<1.0	<1.0
	15-Jun	<1.0	<1.0	<1.0
	29-Jul	<1.0	<1.0	<1.0
	3-Sep	<1.0	<1.0	<1.0
	30-Nov	<1.0	<1.0	<1.0
	22-Dec	<1.0	<1.0	<1.0

5	18-Mar	<1.0	<1.0	<1.0
	20-Apr	<1.0	<1.0	<1.0
	23-May	<1.0	<1.0	<1.0
	14-Jun	<1.0	<1.0	<1.0
	29-Jul	<1.0	<1.0	<1.0
	2-Sep	<1.0	<1.0	<1.0
	29-Nov	<1.0	<1.0	<1.0
	21-Dec	<1.0	<1.0	<1.0
6	18-Mar	<1.0	<1.0	<1.0
	20-Apr	<1.0	<1.0	<1.0
	23-May	<1.0	<1.0	<1.0
	14-Jun	<1.0	<1.0	<1.0
	28-Jul	<1.0	<1.0	<1.0
	1-Sep	<1.0	<1.0	<1.0
	29-Nov	<1.0	<1.0	<1.0
	21-Dec	<1.0	<1.0	<1.0
7	28-Mar	<1.0	<1.0	<1.0
	13-Jun	<1.0	<1.0	<1.0
	28-Jul	<1.0	<1.0	<1.0
	1-Sep	<1.0	<1.0	<1.0
	28-Nov	<1.0	<1.0	<1.0
	20-Dec	<1.0	<1.0	<1.0
8	17-Mar	<1.0	<1.0	<1.0
	19-Apr	<1.0	<1.0	<1.0
	22-May	<1.0	<1.0	<1.0
	13-Jun	<1.0	<1.0	<1.0
	27-Jul	<1.0	<1.0	<1.0
	1-Sep	<1.0	<1.0	<1.0
	28-Nov	<1.0	<1.0	<1.0
	20-Dec	<1.0	<1.0	<1.0
9	16-Mar	<1.0	<1.0	<1.0
	18-Apr	<1.0	<1.0	<1.0
	21-May	<1.0	<1.0	<1.0
	12-Jun	<1.0	<1.0	<1.0
	27-Jul	<1.0	<1.0	<1.0
	22-Aug	<1.0	<1.0	<1.0
	27-Nov	<1.0	<1.0	<1.0
	19-Dec	<1.0	<1.0	<1.0
10	16-Mar	<1.0	<1.0	<1.0
	18-Apr	<1.0	<1.0	<1.0
	21-May	<1.0	<1.0	<1.0
	12-Jun	<1.0	<1.0	<1.0
	26-Jul	<1.0	<1.0	<1.0

	22-Aug	<1.0	<1.0	<1.0
	27-Nov	<1.0	<1.0	<1.0
	19-Dec	<1.0	<1.0	<1.0
11	26-Mar	<1.0	<1.0	<1.0
	17-Apr	<1.0	<1.0	<1.0
	20-May	<1.0	<1.0	<1.0
	11-Jun	<1.0	<1.0	<1.0
	26-Jul	<1.0	<1.0	<1.0
	22-Aug	<1.0	<1.0	<1.0
	26-Nov	<1.0	<1.0	<1.0
	18-Dec	<1.0	<1.0	<1.0
12	15-Mar	<1.0	<1.0	<1.0
	17-Apr	<1.0	<1.0	<1.0
	20-May	<1.0	<1.0	<1.0
	11-Jun	<1.0	<1.0	<1.0
	25-Jul	<1.0	<1.0	<1.0
	22-Aug	<1.0	<1.0	<1.0
	26-Nov	<1.0	<1.0	<1.0
	18-Dec	<1.0	<1.0	<1.0
13	14-Mar	<1.0	<1.0	<1.0
	16-Apr	<1.0	<1.0	<1.0
	19-May	<1.0	<1.0	<1.0
	10-Jun	<1.0	<1.0	<1.0
	25-Jul	<1.0	<1.0	<1.0
	22-Aug	<1.0	<1.0	<1.0
	17-Dec	<1.0	<1.0	<1.0

ii. Freshwater Samples

Table A2. Results of the analysis of Imidacloprid, 5-hydroxy-imidacloprid and Imidacloprid-urea in freshwater samples

Location	Sampling Date (dd-mmm-2022)	Measured concentration (ng/L)		
		Imidacloprid	5-hydroxy-imidacloprid	Imidacloprid-urea
Storelva	7-Feb	<1.0	<1.0	<1.0
	3-Oct	<1.0	<1.0	<1.0
Otra	7-Feb	<1.0	<1.0	<1.0
	3-Oct	<1.0	<1.0	<1.0
Bjerkreimselva	7-Feb	<1.0	<1.0	<1.0
	3-Oct	<1.0	<1.0	<1.0
Orreelva	7-Feb	<1.0	<1.0	<1.0
	3-Oct	<1.0	<1.0	<1.0
Vikedalselva	7-Feb	<1.0	<1.0	<1.0
	3-Oct	<1.0	<1.0	<1.0

Vefsna	2-May	<1.0	<1.0	<1.0
Målselva	2-May	<1.0	<1.0	<1.0
Altaelva	9-May	<1.0	<1.0	<1.0
Tana	9-May	<1.0	<1.0	<1.0
Pasvikelva	2-May	<1.0	<1.0	<1.0
Mjøsa	24-Aug	<1.0	<1.0	<1.0
	24-Aug	<1.0	<1.0	<1.0
	24-Aug	<1.0	<1.0	<1.0

iii. Sediment Samples

Table A3. Results of the analysis of Imidacloprid and 5-hydroxy-imidacloprid in sediment samples

Location	Sampling Date (dd-mmm-2022)	Measured concentration (ng/g)	
		Imidacloprid	5-hydroxy-imidacloprid
Grefstadvika Grimstad	5-Jul	<0.1	<0.1
Ølbergholmen Larvik	7-Jul	<0.1	<0.1
Bekkelaget Oslofjord	18-Aug	<0.1	<0.1
Alna Oslofjord	18-Aug	<0.1	<0.1
Nævestadfjorden Agder	19-Aug	<0.1	<0.1
Songevannet Agder	19-Aug	<0.1	<0.1
Lagfjorden Agder	19-Aug	<0.1	<0.1
Ildjernsflua Oslofjord	29-Aug	<0.1	<0.1
Unstadstranden Lofoten	4-Oct	<0.1	<0.1
Tingvollfjorden Gjemnes	10-Oct	<0.1	<0.1
Vegglandet Hammerfest	11-Oct	<0.1	<0.1
Lerresfjord Alta	12-Oct	<0.1	<0.1
Fannefjorden Molde	13-Oct	<0.1	<0.1
Langfjorden Alta	13-Oct	<0.1	<0.1
Grøtfjord Tromsø	16-Oct	<0.1	<0.1
Lathari Alta	17-Oct	<0.1	<0.1
Tollevika Alta	17-Oct	<0.1	<0.1
Blåmannsvika Tromsø	17-Oct	<0.1	<0.1
Mjøsa Hamar	24-Aug	<0.1	<0.1
	24-Aug	<0.1	<0.1
	24-Aug	<0.1	<0.1

B. Quality Control of Analysis

i. Water Samples

Table B1. Results of the analysis of Imidacloprid in Quality Assurance samples for water analysis

Control Name	Nominal Conc. Imidacloprid (ng/L)	Measured Conc. Imidacloprid (ng/L)	Accuracy (%)
Blank 1	-	<1.0	-
Blank 2	-	<1.0	-
Blank 3	-	<1.0	-
Blank 4	-	<1.0	-
Blank 5	-	<1.0	-
Blank 6	-	<1.0	-
Blank 7	-	<1.0	-
Blank 8	-	<1.0	-
Blank 9	-	<1.0	-
Blank 10	-	<1.0	-
Blank 11	-	<1.0	-
Spike 1	2.0	2.0	100
Spike 2	2.0	2.1	105
Spike 3	2.0	2.1	104
Spike 4	2.0	2.0	102
Spike 5	2.0	2.1	106
Spike 6	2.0	2.0	98
Spike 7	2.0	2.0	100
Spike 8	2.0	2.2	110
Spike 9	2.0	2.0	101
Spike 10	2.0	2.2	110

Table B2. Results of the analysis of 5-OH-Imidacloprid and Imidacloprid-Urea in Quality Assurance samples for water analysis

Control Name	Nominal Conc. (ng/L)	5-OH-Imidacloprid		Imidacloprid-Urea	
		Measured Conc. (ng/L)	Accuracy (%)	Measured Conc. (ng/L)	Accuracy (%)
Blank 1	-	< 1.0	-	< 1.0	-
Blank 2	-	< 1.0	-	< 1.0	-
Blank 3	-	< 1.0	-	< 1.0	-
Blank 4	-	< 1.0	-	< 1.0	-
Blank 5	-	< 1.0	-	< 1.0	-
Blank 6	-	< 1.0	-	< 1.0	-
Blank 7	-	< 1.0	-	< 1.0	-
Blank 8	-	< 1.0	-	< 1.0	-
Blank 9	-	< 1.0	-	< 1.0	-

Blank 10	-	< 1.0	-	< 1.0	-
Spike 1	2.0	2.3	114	2.1	103
Spike 2	2.0	2.3	117	2.0	100
Spike 3	2.0	2.3	115	2.2	108
Spike 4	2.0	2.2	111	2.0	101
Spike 5	2.0	2.5	126	2.0	101
Spike 6	2.0	2.0	99	2.0	99
Spike 7	2.0	2.0	101	2.0	102
Spike 8	2.0	1.9	97	2.0	101
Spike 9	2.0	2.1	105	2.1	103

ii. Sediment Samples

Table B3. Results of the analysis of Imidacloprid and 5-OH-Imidacloprid in Quality Assurance samples for sediment analysis

Control Name	Nominal Conc. (ng/g)	Imidacloprid		5-OH-Imidacloprid	
		Measured Conc. (ng/g)	Accuracy (%)	Measured Conc. (ng/g)	Accuracy (%)
Blank 1	-	< 0.1	-	< 0.1	-
Blank 2	-	< 0.1	-	< 0.1	-
Spike 1	1.0	0.9	90	0.579	58
Spike 2	10.0	8.3	83	5.921	59
Spike 3	10.0	9.1	91	6.177	62

NIVA: Norway´s leading centre of competence in aquatic environmentes

The Norwegian Institute for Water Research (NIVA) is Norway's leading institute for fundamental and applied research on marine and freshwaters. Our research comprises a wide array of environmental, climatic and resource-related fields. NIVA's world-class expertise is multidisciplinary with a broad scientific scope. We combine research, monitoring, evaluation, problem-solving and advisory services at international, national and local levels.

Norwegian Institute for Water Research

Økernveien 94 · NO-0579 Oslo, Norway
Telephone: +47 22 18 51 00
www.niva.no · post@niva.no