

Analytical method for ipflufenquin (NF-180) in sediment

Reports: ECM: EPA MRID No.: 50920983. Dix, M.E. 2018. NF-180 - Validation of the Analytical Method for the Determination of a Test Substance in Sediment. Report prepared by Smithers Viscient, Wareham, Massachusetts, sponsored by Nippon Soda Co., Ltd., Tokyo, Japan, and submitted by Nippon Soda Co., Ltd. (c/o Nisso America, Inc.), New York, New York (pp. 1-3); 79 pages. Smithers Viscient Study No.: 12681.6134. Final report issued March 27, 2018.

ILV: EPA MRID No.: 50920985. Garrigue, P. 2019. Independent Laboratory Validation of Residue Analytical Method for the Determination of NF-180, QP-1-1, and QP-1-7 in Soil and NF-180 in Sediment. Report prepared by SGS France – Laboratoire de Rouen, Saint Etienne du Rouvray, France, sponsored by Nippon Soda Co., Ltd., Tokyo, Japan, and Nippon Soda Co., Ltd. (c/o Nisso America, Inc.), New York, New York, and submitted by Nippon Soda Co., Ltd. (c/o Nisso America, Inc.), New York, New York (pp. 1, 3); 75 pages. GLP Study No.: BPL19-0009. Final report issued July 11, 2019.

Document No.: MRIDs 50920983 & 50920985

Guideline: 850.6100


Statements: ECM: The study was conducted in compliance with USEPA FIFRA Good Laboratory Practice (GLP) standards (40 CFR Part 160), with the exception that routine soil contaminant screening analyses were conducted at facilities other than Smithers Viscient (p. 3; Appendix 3, pp. 72-79 of MRID 50920983). Signed and dated Data Confidentiality, GLP and Quality Assurance statements were provided (pp. 2-4). The statement of authenticity was included with the QA statement.

ILV: The study was conducted in compliance with French GLP standards (Appendix II Article D 523-8; 2007), except for the characterization of the test system (p. 3; Appendix G, p. 75 of MRID 50920985). Signed and dated Data Confidentiality, GLP and Quality Assurance statements were provided (pp. 2-4; Appendix G, p. 75). The statement of authenticity was included with the QA and GLP statements (pp. 3-4).


Classification: This analytical method is classified as **Supplemental**. Only one sediment matrix was included in the ILV; it could not be determined if the ILV was provided with the most difficult sediment matrix with which to validate the method and if the ILV sediment matrix covered the range of soils/sediments used in the two sediment/water test systems included in the aerobic and anaerobic aquatic metabolism studies and the five sites included in the terrestrial field dissipation studies.

PC Code: 129120

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This Data Evaluation Record may have been altered by the Environmental Fate and Effects Division subsequent to signing by CDM/CSS-Dynamac Joint Venture personnel. The CDM/CSS-Dynamac JV role does not include establishing Agency policies.

Executive Summary

The analytical method, Smithers Viscient Study No. 12681.6134, is designed for the quantitative determination of ipflufenquin in sediment at the stated LOQ of 0.05 µg/g using HPLC/MS/MS. The LOQ is less than the lowest toxicological level of concern in sediment. The ECM used non-USDA characterized artificial sediment and marine sediment matrices; the ILV used a characterized loamy sand sediment matrix. It could not be determined if the ILV was provided with the most difficult sediment matrix with which to validate the method and if the ILV sediment matrix covered the range of soils/sediments used in the two sediment/water test systems included in the aerobic and anaerobic aquatic metabolism studies and the five sites included in the terrestrial field dissipation studies. The ILV validated the method for ipflufenquin in the first trial with insignificant analytical instrument and equipment modifications. No updated ECM is required. All ILV and ECM data regarding repeatability, accuracy, precision, linearity, and specificity were satisfactory for ipflufenquin in tested sediment matrices.

Table 1. Analytical Method Summary

Analyte(s) by Pesticide ¹	MRID		EPA Review	Matrix	Method Date (dd/mm/yyyy)	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation						
Ipflufenquin (NF-180)	50920983	50920985		Sediment ^{2,3}	27/03/2018	Nippon Soda Co., Ltd. (c/o Nisso America Inc.)	LC/MS/MS	0.05 µg/g

¹ Ipflufenquin = 2-[2-(7,8-Difluoro-2-methyl-3-quinolyloxy)-6-fluorophenyl]propan-2-ol.

² In the ECM, artificial sediment, prepared by the ECM according to OECD Guideline No. 218 (OECD, 2004) by mixing the following components (based on dry weight): 2.8 kg sphagnum peat, 11.2 kg kaolin clay, and 42 kg fine sand (i.e., 5.0, 20, and 75%, respectively), and marine sediment (82% sand, 4% silt, 14% clay; pH 7.2; 2.0% organic carbon) were used in the study (USDA Soil Texture Classification not reported; pp. 12-13; Appendix 3, pp. 71-79 of MRID 50920983). The particle distribution and % organic carbon content of the marine sediment was determined by Agvise Laboratories, Northwood, North Dakota, but particle distribution sizes were not reported.

³ In the ILV, loamy sand sediment (Nesthauser See; 83.1% sand, 9.3% silt, 7.6% clay; pH 7.54 in CaCl₂; 1.48% organic carbon; 2.54% organic matter) was obtained from 5-10 cm depth of Lake Nesthauser See, near Paderborn, Germany, characterized by a facility outside of the ILV, and used in the study (USDA Soil Texture Classification; p. 18; Appendix F, pp. 73-74 of MRID 50920985).

I. Principle of the Method

Sediment (5.00 g) in sample flasks was fortified with 0.0250 or 0.250 mL of 10.0 mg/L fortification solutions or 0.500 mL of 1000 mg/L fortification solutions, if necessary (pp. 15-19 of MRID 50920983). The sediment samples were extracted twice using 20 mL of acetonitrile:water:formic acid (90:10:0.1, v:v:v) with shaking (150 rpm) for 30 minutes. The sample was centrifuged for 10 minutes at 3000 rpm. The supernatant was transferred to a 50-mL volumetric flask, and the combined extracts were diluted to 50 mL with acetonitrile:water:formic acid (90:10:0.1, v:v:v). Aliquots of the diluted subsamples were transferred to an autosampler vial prior to LC-MS/MS analysis. Further dilutions with acetonitrile:water:formic acid (90:10:0.1, v:v:v) were performed, if necessary.

Samples are analyzed using a MDS Sciex API 5000 mass spectrometer coupled with a Shimadzu LC-20AD HPLC (pp. 12, 20-21 of MRID 50920983). The following LC conditions were used: Supleco Ascentis Express C18 column (2.1 mm x 100 mm, 2.7 μ m particle size; column temperature 40°C), gradient mobile phase of A) 0.1% formic acid in reagent grade water and B) 0.1% formic acid in acetonitrile [time, percent A:B; 0.010 min. 95.0:5.00, 0.50 min. 90.0:10.0, 6.00-7.00 min. 0.00:100, 7.10-8.50 min. 95.0:5.00], injection volume of 50 μ L, MS/MS with Electrospray Ionization (ESI) source in positive polarity (source temperature 500°C). Two ion pair transitions were monitored for ipflufenquin (quantitation and confirmation, respectively): m/z 348.1 \rightarrow 330.0 and m/z 348.1 \rightarrow 180.2. Retention time was *ca.* 5.2 minutes.

The ILV performed the ECM method for ipflufenquin in sediment as written, except for insignificant analytical instrument and equipment modifications (pp. 12-13, 19, 26-30 of MRID 50920985). The LC/MS/MS instrument was an API 5500 Q-trap mass spectrometer coupled with a Shimadzu LC-30AD HPLC System. The LC conditions were the same as those of the ECM, except that injection volume was reduced to 10 μ L. The ILV noted that the injection temperature was initially 5°C, but this minor modification had no impact on the study. Two ion pair transitions were monitored for ipflufenquin (quantitation and confirmation, respectively): m/z 348.0 \rightarrow 330.1 and m/z 348.0 \rightarrow 180.0. Retention time was *ca.* 4.3 minutes.

In the ECM and ILV, the Limit of Quantification (LOQ) was 0.05 μ g/g for ipflufenquin in sediment matrices (pp. 22-23, 28-30 of MRID 50920983; p. 35 of MRID 50920985). The Limit of Detection (LOD) values were 0.0006-0.004 μ g/g in the ECM and 0.0125 μ g/g in the ILV for ipflufenquin in sediment matrices.

II. Recovery Findings

ECM (MRID 50920983): Mean recoveries and relative standard deviations (RSDs) were within guidelines (mean 70-120%; RSD \leq 20%) for analysis of ipflufenquin at fortification levels of 0.05 $\mu\text{g/g}$ (LOQ), 0.5 $\mu\text{g/g}$ (10 \times LOQ), and 100 $\mu\text{g/g}$ (2000 \times LOQ) in two sediment matrices using matrix-matched and solvent-based calibration standards (p. 24; Tables 1-8, pp. 33-40). Two ion pair transitions were monitored, one quantitation and one confirmation; quantitation and confirmation recovery results were comparable. Artificial sediment and marine sediment were used. Artificial sediment was prepared by the ECM according to OECD Guideline No. 218 (OECD, 2004) by mixing the following components (based on dry weight): 2.8 kg sphagnum peat, 11.2 kg kaolin clay, and 42 kg fine sand (i.e., 5.0, 20, and 75%, respectively; pp. 12-13; Appendix 3, pp. 71-79 of MRID 50920983). The marine sediment was classified as 82% sand, 4% silt, 14% clay; pH 7.2; 2.0% organic carbon (USDA Soil Texture Classification not reported). The particle distribution and % organic carbon content of the marine sediment was determined by Agvise Laboratories, Northwood, North Dakota, but particle distribution sizes were not reported.

ILV (MRID 50920985): Mean recoveries and RSDs were within guidelines for analysis of ipflufenquin at fortification levels of 0.05 $\mu\text{g/g}$ (LOQ) and 0.5 $\mu\text{g/g}$ (10 \times LOQ) in one sediment matrix using solvent-based calibration standards (p. 34; Table 15, p. 35; DER Attachment 2). Two ion pair transitions were monitored, one quantitation and one confirmation; quantitation and confirmation recovery results were comparable. The loamy sand sediment (Nesthauser See; 83.1% sand, 9.3% silt, 7.6% clay; pH 7.54 in CaCl_2 ; 1.48% organic carbon; 2.54% organic matter) was obtained from 5-10 cm depth of Lake Nesthauser See, near Paderborn, Germany, characterized by a facility outside of the ILV, and used in the study (USDA Soil Texture Classification; p. 18; Appendix F, pp. 73-74). The method was validated for ipflufenquin in the first trial with insignificant analytical instrument and equipment modifications (pp. 12-13, 19, 26-30, 35). No updated ECM is required.

Table 2. Initial Validation Method Recoveries for Ipflufenquin in Sediment^{1,2}

Analyte	Fortification Level (µg/g)	Number of Tests ⁴	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%) ⁵	Relative Standard Deviation (%)
Matrix-matched Calibration Standards						
Artificial Sediment³						
Quantitation Ion Transition						
Ipflufenquin (NF-180)	0.05 (LOQ)	5	89.6-101	96.4	4.70	4.88
	0.50	5	94.7-100	96.8	1.95	2.02
	100	5	95.3-102	98.5	2.75	2.79
Confirmation Ion Transition						
Ipflufenquin (NF-180)	0.05 (LOQ)	5	93.7-109	101	5.90	5.87
	0.50	5	96.4-101	98.4	1.84	1.86
	100	5	98.4-106	101	2.72	2.69
Marine Sediment³						
Quantitation Ion Transition						
Ipflufenquin (NF-180)	0.05 (LOQ)	5	93.2-105	100	5.03	5.01
	0.50	5	99.3-104	102	2.05	2.01
	100	5	95.8-103	99.4	2.77	2.79
Confirmation Ion Transition						
Ipflufenquin (NF-180)	0.05 (LOQ)	5	94.1-108	100	6.00	5.98
	0.50	5	100-110	106	3.70	3.51
	100	5	95.2-106	99.5	4.56	4.58
Solvent-based Calibration Standards						
Artificial Sediment³						
Quantitation Ion Transition						
Ipflufenquin (NF-180)	0.05 (LOQ)	5	92.9-102	98.2	3.35	3.41
	0.50	5	93.5-105	100	4.28	4.27
	100	5	94.6-101	98.3	2.51	2.56
Confirmation Ion Transition						
Ipflufenquin (NF-180)	0.05 (LOQ)	5	93.2-98.1	95.2	1.80	1.89
	0.50	5	94.7-101	98.7	2.38	2.41
	100	5	99.1-101	100	0.837	0.834
Marine Sediment³						
Quantitation Ion Transition						
Ipflufenquin (NF-180)	0.05 (LOQ)	5	94.3-106	101	4.97	4.90
	0.50	5	98.8-103	102	2.03	2.00
	100	5	95.3-102	99.0	2.74	2.77
Confirmation Ion Transition						
Ipflufenquin (NF-180)	0.05 (LOQ)	5	93.4-106	99.3	5.76	5.80
	0.50	5	97.2-107	102	3.55	3.47
	100	5	92.3-103	96.4	4.38	4.54

Data (uncorrected recovery results; pp. 22-23) were obtained from p. 24; Tables 1-8, pp. 33-40 of MRID 50920983.

1 Ipflufenquin = 2-[2-(7,8-Difluoro-2-methyl-3-quinolyloxy)-6-fluorophenyl]propan-2-ol.

2 Two ion pair transitions were monitored for ipflufenquin (quantitation and confirmation, respectively): m/z 348.0→330.1 and m/z 348.0→180.0.

3 Artificial sediment, prepared by the ECM according to OECD Guideline No. 218 (OECD, 2004) by mixing the following components (based on dry weight): 2.8 kg sphagnum peat, 11.2 kg kaolin clay, and 42 kg fine sand (i.e., 5.0, 20, and 75%, respectively), and marine sediment (82% sand, 4% silt, 14% clay; pH 7.2; 2.0% organic carbon) were used in the study (USDA Soil Texture Classification not reported; pp. 12-13; Appendix 3, pp. 71-79 of MRID 50920983). The particle distribution and % organic carbon content of the marine sediment was determined by Agvise Laboratories, Northwood, North Dakota, but particle distribution sizes were not reported. The marine sediment texture was determined as loamy sand by the reviewer using USDA-NRCS technical support tools, assuming USDA particle size distributions.

Table 3. Independent Validation Method Recoveries for Ipflufenquin in Sediment^{1,2,3}

Analyte	Fortification Level (µg/g)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%) ⁴	Relative Standard Deviation (%)
Solvent-based Calibration Standards						
Sediment						
Quantitation ion transition						
Ipflufenquin (NF-180)	0.05 (LOQ)	5	95-103	98	3	3.5
	0.05	5	100-120	107	8	7.9
Confirmation ion transition						
Ipflufenquin (NF-180)	0.05 (LOQ)	5	89-105	97	6	5.8
	0.05	5	96-119	108	9	8.6

Data (uncorrected recovery results; p. 31) were obtained from Table 15, p. 35 of MRID 50920985 and DER Attachment 2.

1 Ipflufenquin = 2-[2-(7,8-Difluoro-2-methyl-3-quinolyloxy)-6-fluorophenyl]propan-2-ol.

2 Two ion pair transitions were monitored for ipflufenquin (quantitation and confirmation, respectively): m/z 348.0→330.1 and m/z 348.0→180.0.

3 The loamy sand sediment (Nesthauser See; 83.1% sand, 9.3% silt, 7.6% clay; pH 7.54 in CaCl₂; 1.48% organic carbon; 2.54% organic matter) was obtained from 5-10 cm depth of Lake Nesthauser See, near Paderborn, Germany, characterized by a facility outside of the ILV, and used in the study (USDA Soil Texture Classification; p. 18; Appendix F, pp. 73-74 of MRID 50920985). The sediment texture was verified by the reviewer using USDA-NRCS technical support tools.

4 Standard deviations were reviewer-calculated using the data in the study report since the study author did not report these values (see DER Attachment 2). Rules of significant figures were followed.

III. Method Characteristics

In the ECM and ILV, the LOQ was 0.05 µg/g for all analytes in sediment matrices (pp. 22-23, 28-30 of MRID 50920983; p. 35 of MRID 50920985). In the ECM and ILV, the LOQ was determined as the lowest fortification level. In the ECM, the LOQ was also defined as the fortification level at which the blank values did not exceed 30% of the Method Detection Limit (MDL). The reported LOD values were 0.0006-0.004 µg/g in the ECM and 0.0125 µg/g in the ILV for ipflufenquin in sediment matrices. In the ILV, the LOD was set to at least 30% of the LOQ. In the ECM, the LOD values for ipflufenquin in artificial and marine sediment were calculated based on the following equation:

$$\text{LOD} = (3x(N_{\text{ctl}})/(\text{Res}_{\text{PLS}}) \times \text{Con}_{\text{CLS}} \times \text{DF}_{\text{CTRL}}$$

Where, LOD is the limit of detection of the analysis, N_{ctl} is the mean signal to noise in height of the control samples (or Blanks), Res_{PLS} is the mean response in height of the two low calibration standards, Con_{CLS} is the concentration of the low calibration standard, and DF_{CTRL} is the dilution factor of the control samples (smallest dilution factor used, i.e. 2500). No calculations or comparisons to background levels were reported to justify the LOQ for the method in the ECM or ILV; no calculations or comparisons to background levels were reported to justify the LOD for the method in the ILV.

Table 4. Method Characteristics Ipflufenquin in Sediment

Test Material ¹		Ipflufenquin	
		Matrix-matched calibration standards	Solvent-based calibration standards
Limit of Quantitation (LOQ)	ECM	0.05 µg/g	
	ILV		
Limit of Detection (LOD)	ECM	0.0006 µg/g (Q, AS; calculated) 0.001 µg/g (C, AS; calculated) 0.004 µg/g (Q, MS; calculated) 0.003 µg/g (C, MS; calculated)	
	ILV	0.0125 µg/g (30% of the LOQ)	
Linearity (calibration curve r and concentration range)	ECM	r = 0.9985 (Q, AS) r = 0.9990 (C, AS) r = 0.9990 (Q, MS) r = 0.9960 (C, MS)	r = 0.9960 (Q & C, AS) ² r = 0.9995 (Q, MS) r = 0.9985 (C, MS)
		0.0100-0.100 ng/mL	
	ILV	Not performed ³	r = 0.9994 (Q & C) 0.005015-0.1003 ng/mL
Repeatable	ECM ⁴	Yes at LOQ and 10×LOQ in two non-USDA characterized sediment matrices.	
	ILV ^{5,6}	Yes at LOQ and 10×LOQ in one characterized sediment matrix.	
Reproducible		Yes at LOQ and 10×LOQ.	
Specific	ECM	Yes, matrix interferences were <2% of the LOQ (based on peak area). Minor peak tailing was observed.	
	ILV	Yes, matrix interferences were <3% (Q) and <13% (C) of the LOQ (based on peak area). Some minor baseline noise interference and non-interfering contaminants were noted. Minor peak tailing was observed.	

Data were obtained from pp. 22-23, 28-30 (LOQ/LOD); p. 24; Tables 1-8, pp. 33-40 (recovery results); pp. 25, 28-30; Figures 13-20, pp. 53-60 (calibration coefficients); Figures 1-12, pp. 41-52 (chromatograms) of MRID 50920983; p. 35 (LOQ/LOD); Table 15, p. 35 (recovery results); Appendix D, Figures 40-41, pp. 61-62 (calibration curves); Appendix D, Figures 42-51, pp. 63-67 (chromatograms) of MRID 50920985. Q = quantitation ion transition; C = confirmation ion transition. AS = artificial sediment; MS = marine sediment.

1 Ipflufenquin = 2-[2-(7,8-Difluoro-2-methyl-3-quinolyloxy)-6-fluorophenyl]propan-2-ol.

2 The ECM study author reported that the calibration curve for the solvent-based calibration standards failed to meet acceptance criteria since 25% of data from the calibration standards (3 of 12 values) was omitted (pp. 24-25, 28-30; Figures 13-20, pp. 53-60 of MRID 50920983). The ECM study author recommended matrix-matched calibration standards to be used for future validation.

3 Matrix effects of the test sediment matrix were studied in the ILV and found to be insignificant (<20%; p. 34 of MRID 50920985).

4 In the ECM, artificial sediment, prepared by the ECM according to OECD Guideline No. 218 (OECD, 2004) by mixing the following components (based on dry weight): 2.8 kg sphagnum peat, 11.2 kg kaolin clay, and 42 kg fine sand (i.e., 5.0, 20, and 75%, respectively), and marine sediment (82% sand, 4% silt, 14% clay; pH 7.2; 2.0% organic carbon) were used in the study (USDA Soil Texture Classification not reported; pp. 12-13; Appendix 3, pp. 71-79 of MRID 50920983). The particle distribution and % organic carbon content of the marine sediment was determined by Agvise Laboratories, Northwood, North Dakota, but particle distribution sizes were not reported. The marine sediment texture was determined as loamy sand by the reviewer using USDA-NRCS technical support tools, assuming USDA particle size distributions.

5 In the ILV, loamy sand sediment (Nesthauser See; 83.1% sand, 9.3% silt, 7.6% clay; pH 7.54 in CaCl₂; 1.48% organic carbon; 2.54% organic matter) was obtained from 5-10 cm depth of Lake Nesthauser See, near Paderborn, Germany, characterized by a facility outside of the ILV, and used in the study (USDA Soil Texture Classification; p. 18; Appendix F, pp. 73-74 of MRID 50920985). The sediment texture was verified by the reviewer using USDA-NRCS technical support tools.

6 The ILV validated the method for ipflufenquin in the first trial with insignificant analytical instrument and equipment modifications (pp. 12-13, 19, 26-30, 35. of MRID 50920985).

IV. Method Deficiencies and Reviewer's Comments

1. It could not be determined if the ILV was provided with the most difficult sediment matrix with which to validate the method and if the ILV sediment matrix covered the range of soils/sediments used in the aerobic and anaerobic aquatic metabolism and terrestrial field dissipation (TFD) studies since only one sediment matrix was included in the ILV: loamy sand sediment (Nesthauser See; 83.1% sand, 9.3% silt, 7.6% clay; pH 7.54 in CaCl₂; 1.48% organic carbon; 2.54% organic matter); USDA Soil Texture Classification; p. 18; Appendix F, pp. 73-74 of MRID 50920985). OCSPP guidelines state that "For a given sample matrix, the registrant should select the most difficult analytical sample condition from the study (*e.g.*, high organic content versus low organic content in a soil matrix) to analyze from the study to demonstrate how well the method performs". The organic content of the ILV sediment matrix was medium, and more than one sediment should be included to encompass the range of soils/sediments used in the aquatic metabolism and TFD studies. The submitted ipflufenquin aerobic and anaerobic aquatic metabolism studies (MRIDs 50920946 & 50920947) contained two US sediment/water systems: North Carolina (loamy sand) and Pennsylvania (loam; Table III, p. 43 of MRID 50920946; Table III, p. 41 MRID 50920947). The submitted ipflufenquin TFD study (MRID 50920948) contained five test sites: NAFTA Regions 10 (California; sandy loam, loamy sand, sand), 2 (Georgia; sand, loamy sand, sandy loam, sandy clay loam), 5 (Iowa; silt loam, clay loam, silty clay loam), 11 (Idaho; loam, sandy loam), and 1 (New York; sandy loam, loam, silt loam); (Ecoregions 11.1, 8.3, 9.2, 10.1, and & 8.1, respectively; p. 11; Tables 4a-4e, pp. 64-68 of MRID 50920948).
2. The determinations of LOD and LOQ in the ECM and ILV were not based on scientifically acceptable procedures as defined in 40 CFR Part 136 (pp. 22-23, 28-30 of MRID 50920983; p. 35 of MRID 50920985). In the ECM and ILV, the LOQ was determined as the lowest fortification level. In the ECM, the LOQ was also defined as the fortification level at which the blank values did not exceed 30% of the Method Detection Limit (MDL). In the ILV, the LOD was set to at least 30% of the LOQ. In the ECM, the LOD values for ipflufenquin in artificial and marine sediment were calculated based on the following equation: $LOD = (3 \times (N_{ctl}) / (Res_{PLS}) \times Con_{CLS} \times DF_{CTRL})$, where, LOD is the limit of detection of the analysis, N_{ctl} is the mean signal to noise in height of the control samples (or Blanks), Res_{PLS} is the mean response in height of the two low calibration standards, Con_{CLS} is the concentration of the low calibration standard, and DF_{CTRL} is the dilution factor of the control samples (smallest dilution factor used, *i.e.* 2500). No calculations or comparisons to background levels were reported to justify the LOQ for the method in the ECM or ILV; no calculations or comparisons to background levels were reported to justify the LOD for the method in the ILV. Detection limits should not be based on arbitrary values.

MDLs were also calculated in the ECM (pp. 23-24 of MRID 50920983).
3. In the ILV, the total time required to perform the method (extraction and analysis) with one sample set (30 samples, 1 control and 2 fortifications) was *ca.* 24 hours (p. 30 of MRID 50920985).

V. References

- U.S. Environmental Protection Agency. 2012. Ecological Effects Test Guidelines, OCSPP 850.6100, Environmental Chemistry Methods and Associated Independent Laboratory Validation. Office of Chemical Safety and Pollution Prevention, Washington, DC. EPA 712-C-001.
- 40 CFR Part 136. Appendix B. Definition and Procedure for the Determination of the Method Detection Limit-Revision 1.11, pp. 317-319.

Attachment 1: Chemical Names and Structures**Ipflufenoquin (NF-180)**

IUPAC Name: 2-[2-(7,8-Difluoro-2-methyl-3-quinolyloxy)-6-fluorophenyl]propan-2-ol
CAS Name: 2-[(7,8-Difluoro-2-methyl-3-quinolinyloxy)-6-fluoro- α,α -dimethylbenzenemethanol
CAS Number: 1314008-27-9
SMILES String: FC1=C(F)C=CC2=C1N=C(C)C(OC3=C(C(C)(C)O)C(F)=CC=C3)=C2

