Bicyclopyrone; PC Code 018986;
NOA449280
ENVIRONMENTAL CHEMISTRY METHOD REVIEW REPORT
Residue Method for the Determination of NOA449280 in air

Data Requirement: EPA Guideline: 850.6100
OECD Data Point: IIA 4.5

Reports: **Analytical Method:** MRID 47842020. Crook, S. and Oppilliart, S. 2012. NOA449280 Analytical Method GRM030.07A for the Determination of NOA449280 in Air. Report No. GRM030.07A. Task No. T013551-05. Unpublished study prepared by ADME Bioanalyses, 75 Chemin de Sommières 30310 Vergèze, France; and Syngenta Ltd., Jealott's Hill International Research Centre, Bracknell, Berkshire, RG42 6EY, UK. Submitted by Syngenta Crop Protection, LLC, Greensboro, NC.

Independent Laboratory Validation: MRID 47841959. Oppilliart, S. 2008. NOA449280 Validation of Residue Method AGR/MOA/NOA449280-2 for the determination of NOA449280 in Air. Report Number: Report Number: T013551-05-REG. Study Number: T013551-05. Task Number: T013551-05. Unpublished study prepared by Eurofins | ADME Bioanalyses, 75 Chemin de Sommiéres 30310 Vergéze, France. Submitted by Syngenta Crop Protection, LLC, Greensboro, NC.

Compliance Statements: These studies were reported to be conducted in compliance with GLP practices

Classification: This analytical method is classified as **Fully Reliable** (EPA classification: Acceptable) for the determination of NOAA449280 in air. The study is reported to be a method to sample NOA449280 in air. However, actual air recoveries were not attempted. The target compound was merely spiked to the filter units prior to air exposure. The independent laboratory validation portion of the data package did not include validation data from an independent laboratory, but reported the same data reported in the initial analytical method study. No other independent laboratory validation data were submitted.

PC Code: 018986

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Executive Summary

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This analytical method, GRM030.07A, is designed for the quantitative determination of NOA449280 air. The Residue Method actually determines the stability and extractability of NOA449280 that is spiked at a rate of 0.0027 ug for each isomer to a Tenax OVS air sampler tube with 2 layers of Tenax adsorbent. Air is then drawn across the tube by a calibrated air sampler motorized pump at a rate of 0.5L/min for a period of up to 6 hours.

The sampler is removed and the adsorbent layers separated for analysis. NOA449280 is then desorbed by ultrasonication in acetone. An aliquot of the acetone solution is then diluted with ulta pure water. Detemination is by LC-MS/MS. The LOQ is reported as 0.015 ug/m^3 or (0.000015 ug/L)

Table 1. Analytical Method Summary

Analyte(s) by Pesticide	MRID		EPA Review	Matrix	Method Date	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation						
NOA449280	47842020	47841959[1]	None	Tenax adsorptive media[2]	1/31/2012	Syngenta	LC/MS-MS	0.015 µg/m^3

[1]This study appears to be the same study as the original method study and does not appear to be an independent validation of the method. [2]Although this study is titled as a method for determining NOA449280 in air, it only tests the ability to extract the residue from the filter material. No in-situ testing of the filter cartridge as in field adsorbent was performed.

I. PRINCIPLE OF THE METHOD

After preparation of stock solutions, known amounts of NOA44920 in acetonitrile were applied to the upper glass fiber filter of the Tenax air sampling tube. Fortification should not be added at more than a 50-µL dose at a time. After fortification the Tenax tube should be allowed to dry for 15-20 minutes.

After calibrating an appropriate air sampling pump the treated filters as well as untreated controls were attached and air was drawn through the filters at a rate of 0.5 L/min for 6 hours.

The upper glass filter and teflon ring were removed and placed in a 15-ml polypropylene sampling tube. (The lower filter was retained in a separate tube for later analysis in case recoveries were low.) Ten mL of acetone was added and the tube shaken gently. The tube and contents were then ultrasonicated for 5 minutes. The contents of the tube were then allowed to settle.

A 0.2mL aliquot of the settled solution is transferred to a suitable test tube and brought to volume with 0.080mL of ultra pure water. The dilution is then shaken and mixed thoroughly. The sample is again diluted 5 fold with ultrapure water in an autosampler vial. (total dilution factor is 25)

Analysis was by LC-MS/MS.

II. RECOVERY FINDINGS

Table 2: Recovery Data for NOA449280 from Upper Sorbent Layer Obtained During Method Validation (Transition *m/z* 400.0 → 324.2).

NOA449280 (Transition *m/z* 400.0 → 324.2)					
Matrix	Fortification Level (µg/tube)	Recovery (%)	Mean (%)	RSD (%)	Range (%)
Air	0.0027	93, 96, 94, 92, 99	95	3	92 - 99
	0.027	83, 81, 80, 84, 84	82	2	80 - 84
	Overall		*89*	*8*	*80 - 99*

Table 3: Recovery Data for NOA449280 from Upper Sorbent Layer Obtained During Method Validation (Transition *m/z* 400.0 → 228.1).

NOA449280 (Transition *m/z* 400.0 → 228.1)					
Matrix	Fortification Level (µg/tube)	Recovery (%)	Mean (%)	RSD (%)	Range (%)
Air	0.0027	94, 91, 96, 96, 97	95	2	91 - 97
	0.027	81, 83, 80, 82, 83	82	1	80 - 83
	Overall		*88*	*8*	*80 - 97*

III. METHOD DEFICIENCIES AND REVIEWER'S COMMENTS

The major deficiency of this ECM-ILV package is that the study presented as an independent laboratory validation is, in fact, a repackaging of the same data reported in the method study. To be an acceptable set of studies, a separate study using the analytical method, should be conducted by an independent laboratory and the results of that study reported. Although the analytical method study reports good recoveries, the method will not be considered fully acceptable until an independent laboratory produces similar acceptable recoveries.

Additionally the study is reported to be a method to sample NOA449280 in air. However, actual air recoveries were not attempted. The target compound was spiked to the filter units prior to air exposure. As such, no actual capture of the test substance on the filters was attempted. So the capture efficiency of the filter cartridges is unknown. The method does appear to indicate that, once captured, the recovery methods and analysis are effective. However, these results are not validated by an acceptable ILV study.

Figure 1: NOA449280

Compound Code Number	:	**NOA449280**
CAS Number	:	**352010-65-5**
IUPAC Name	:	**4-Hydroxy-3-[2-(2-methoxy-ethoxymethyl)-6-trifluoromethyl-pyridine-3-carbonyl]-bicyclo[3.2.1]oct-3-en-2-one**
Molecular Formula	:	**$C_{19}H_{20}F_3NO_5$**
Molecular Weight	:	**399.39**