

STUDY TITLE**RESPONSE TO EPA REVIEW AND SUBMISSION OF SUPPLEMENTAL DATA
TO REPORT:****VALIDATION OF THE RESIDUE ANALYTICAL METHOD FOR IKI-220 AND
METABOLITES (TFNA, TFNG, TFNA-AM, TFNA-OH AND TFNG-AM) IN
SURFACE WATER AND DRINKING WATER****(MRID#45854816)**

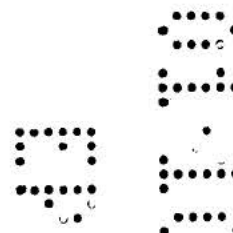
NOTE: FLONICAMID (CAS #158062-67-0) is *N*-(cyanomethyl)-
4-(trifluoromethyl)-3-pyridinecarboximide. Other identifiers are
IKI-220 and F-1785.

DOCUMENT NO.

RCC 834131

DATA REQUIREMENT

Pesticide Assessment Guidelines
160-General



FIRST AMENDMENT TO REPORT

Study Title:

VALIDATION OF THE RESIDUE ANALYTICAL METHOD FOR IKI-220 AND METABOLITES (TFNA, TFNG, TFNA-AM, TFNA-OH AND TFNG-AM) IN SURFACE WATER AND DRINKING WATER

Data Requirements / Test Guidelines:

Residue Analytical Method, Guideline 96/46/EC, July 16, 1996

European Commission, Guidance Document on Residue Analytical Methods, SANCO/825/00 rev. 6, June 20, 2000

European Commission, Residues: Guidance for Generation and Reporting Methods of Analysis in Support of Pre-Registration Data Requirements for Annex II (part A, Section 4) and Annex III (part A, Section 5) of Directive 91/414, SANCO/3029/99 rev. 4, July 11, 2000 - Working Document

Concerning:

2.4.3 Standard solutions (page: 20)

Present:

As described in the report

New:

Due to the matrix effect, matrix matched calibration standards were prepared.

Defined volumes of the mixed 10 µg/mL stock solution were diluted using **untreated control water extract [dissolved in water/acetonitrile (9+1), see section 2.6]** to obtain mixed standard solutions in the range of 1.0, 0.5 and 0.2 µg/mL containing IKI-220 and its metabolites.

Defined volumes of the mixed 1.0 µg/mL stock solution were diluted using **untreated control water extract [dissolved in water/acetonitrile (9+1), see section 2.6]** to obtain mixed standard solutions in the range of 0.1, 0.05, 0.02 and 0.01 µg/mL containing IKI-220 and its metabolites.

These solutions were stored in a refrigerator at about 4 °C.

Reason for alteration:

Matrix matched calibration standards were used.

Concerning:

2.6 Analytical Method (page: 21)

Present:

As described in the report

New:

The analytical method was developed and validated during the course of this study.

An ENV+ column was conditioned by passing 5 mL dichloromethane, 5 mL methanol, 5 mL water (distilled) and 5 mL hydrochloric acid (about 0.1 mol/L) one after another using a vacuum manifold.

The ENV+ column was filled with 5 mL hydrochloric acid (**about 0.1 mol/L**) and a 70 mL reservoir was installed.

1000 mL water sample were transferred into a 1000 mL laboratory flask. Samples were fortified at this stage and 2 mL of hydrochloric acid (**32%**) was added (**the pH-value should be <3**). After shaking, the acidified sample was passed through the ENV+ column at a flow rate of < 10 mL/minute. Then the column was taken to dryness with a vacuum for about 10 sec and the water discarded.

Thereafter, the ENV+ column was filled with 5 mL of methanol and let drip without vacuum for approx. 1 minute. Then the methanol was eluted with a few vacuum. The same process was repeated twice. The methanol solutions were combined in a 100 mL round-bottomed flask and evaporated to dryness under vacuum at about 50-60 °C using a rotary evaporator. The residue was dissolved in 10 mL of ethanol and evaporated again to dryness. The residue was dissolved in 2.0 mL of water/acetonitrile (9+1 v/v) using an ultrasonic bath.

The concentrations of IKI-220 and its metabolites were determined after LC separation using a MS/MS Detector.