KANSAS DEPARTMENT OF AGRICULTURE LABORATORY

Preparation of Tank Mix Samples For Gas Chromatography or High Pressure Liquid Chromatography

Prepared by: _____ Date: _____

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Annual Review:

Initialed _____ Date _____
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1. Purpose

To determine by way of Gas Chromatography or High Pressure Liquid Chromatography the percentage of an analyte in a tank mix.

2. Scope

This SOP concerns the typical preparation of tank mixes to be run by either the HPLC or GC.

3. References

3.1 National Enforcement Investigation Center
3.2 Official Methods of Analysis of AOAC International, 17th Edition

4. Safety and Hazardous Waste

4.1 Standard personal protective equipment for the laboratory shall be used. MSDS information may be accessed in the laboratory file. Any hazardous waste shall be disposed of according to the laboratory SOP KDAL-AM-WD-1. Usual good laboratory practices shall be adhered to during the procedures.

5. Reagents / Supplies / Equipment

Ring Stand
Separatory Funnel 250ml
Beakers
Graduated Cylinders
Round Bottom Flask 250ml
Funnel
Glass Wool
Rotary Evaporator
Rotary Evaporator Stand
Hot Plate / Water pan
Water Bottle
Sodium Sulfate
Methylene Chloride

6 Outline of Procedure

6.1 Pre-preparation
6.2 Standards Book and Worksheet
6.3 50/50 dilutions

6.4 Filtered to Vial
6.5 Phase Over
6.6 Calculations

7 Specific Procedure

7.1 Pre- Preparation
- 7.1.1 Obtain the sample according to SOP KDAL-PL-CC-3.
- 7.1.2 Sample is kept in locked cabinet in Formulations room.
- 7.1.3 Pesticide Formulations Worksheet is in file on top of counter by the east wall.
- 7.1.4 Determine appropriate solvent.
- 7.1.5 Obtain standard in standards room.

7.2 Standards Book and Worksheet

- 7.2.1 Record information regarding the preparation of the standard.
- 7.2.2 Record what was done with the sample on the Pesticide Formulations Worksheet.
- 7.2.3 Record any dilutions or any notes about sample or standard in the note section on worksheet.

7.3 50/50 Dilutions

- 7.3.1 Sample goes on GC.
- 7.3.2 Shake sample to make uniform.
- 7.3.3 Weigh 12.5 grams of sample into 25 ml volumetric flask.
- 7.3.4 Go to volume with solvent. Mix well and vial.

7.4 Filter to Vial

- 7.4.1 This sample would typically go on HPLC.
- 7.4.2 Supplies are in drawer under balance.
- 7.4.3 Take a 3cc syringe and put a 0.2 micron filter on the end. Place end of filter in vial, pull plunger from syringe and pour sample into syringe.
- 7.4.4 Replace plunger and inject sample through the filter into the vial and cap.

7.5 Phase Over

- 7.5.1 Set up stand for 250ml separatory funnel in hood.
- 7.5.2 Weigh up 12.5 grams of sample in beaker.

7.5.3 Pour sample into separatory funnel and add small amounts of sodium sulfate, shaking funnel in between each addition. Keep repeating until sample becomes opaque.
7.5.4 Pour 20 ml methylene chloride and shake.2 min. Let separate into layers.
7.5.5 Put a powder funnel packed with glass wool in a 250ml round bottom flask.
7.5.6 Pour sodium sulfate over glass wool until about an inch deep.
7.5.7 Let bottom layer from separatory funnel run through funnel into round bottom flask. Repeat two more times.
7.5.8 Set up hot plate and rotary evaporator in hood.
7.5.9 Rotary evaporate to dryness.
7.5.10 Decide on solvent and final volume and mix well.
7.5.11 Vial sample according to what instrument you're going to use.

8. Calculations

8.1 Pesticide Formulation Worksheet is in Excel.

External Standard Tank Mix Calculator

Fipronil

Sample Number	**06-0078**		Std Resp	1701015
				1695748
Standard Average Response	1696796			1687081
Sample Average Response	1821648			1703340
Standard Conc.	0.6	mg/mL		
Sample Weight	163.2	mg		
Standard Volume	1		Sam Resp	1701710
Sample Volume	25	mL		1941585
Concentration Percent	**9.867**	%		
	98.675	mg/g		

8.2 Enter the following information into the spreadsheet

8.2.1 Standard Concentration
8.2.2 Sample Weight
8.2.3 Sample Volume
8.2.4 Std Resp-Area of Standard
8.2.5 Sam Resp-Area of sample

9. History

Version 1 original copy on January 8, 2007 written by Janice Ramos