

## Additional Information Regarding the Conduct of Thermogravimetric Analysis Data January 5, 2023

The Department of Pesticide Regulation (DPR) intends this summary to clarify the thermogravimetry study protocol entitled "[Estimation of Volatile Emission Potential of Pesticides by Thermogravimetry](#)" and to provide insight into DPR's evaluation of thermogravimetric analysis (TGA) data.

### Generating the Data

1. Nitrogen (N<sub>2</sub>) purge flow rate needs to be between 60 and 70 mL per minute.
2. Temperature of 35 °C is reached before loading the sample into the sample pan.
3. Initial sample mass needs to be between 9 and 11 mg.
4. After loading the sample and recording initial sample mass, the temperature is increased at a rate of 5 °C per minute.
5. For tests with a holding temperature of 115 °C:
  - a. The period of temperature-increase from 35 to 115 °C spans 16 minutes.
  - b. Once the holding temperature is reached, check for sample stabilization.
    - i. Sample stabilization is indicated by any 5-minute period after reaching 115 °C, during which total mass loss is less than or equal to 0.5% (equivalent to 0.1% per minute).
    - ii. If the sample does not stabilize within a period of 80 minutes after reaching 115 °C, the test is considered unacceptable, and the product must be retested at 55 °C.
  - c. The test ends 15 minutes after the end of the stabilization period.
6. For tests with a holding temperature of 55 °C:
  - a. The period of temperature-increase from 35 to 55 °C spans 4 minutes.
  - b. The holding temperature is maintained for 11 hours (660 minutes), at which point the test ends.

### Documenting the Data (Thermograms)

1. The x-axis is time in minutes. Zero minutes marks the start of the temperature increase period from 35 °C to the holding temperature.
  - a. For tests with a holding temperature of 115 °C: 16 minutes marks the time when the holding temperature is reached.
  - b. For tests with a holding temperature of 55 °C: 4 minutes marks the time when the holding temperature is reached.
2. The y-axis is sample mass as a percentage of the initial sample mass recorded on the submission sheet.
3. Axes' tick marks represent equidistant integer values (e.g., 0%, 5%, 10%, etc., for the y-axis).
4. Annotations need to be included for key moments.
  - a. For tests with a holding temperature of 115 °C: The start and end of the stabilization period, as well as the end of the test is annotated.
  - b. For tests with a holding temperature of 55 °C: The end of the test is annotated.
5. Annotations allow DPR staff to verify sample stability (115 °C only) and final mass loss. Each annotation includes a value for time and either mass, mass loss, or both mass and mass loss.

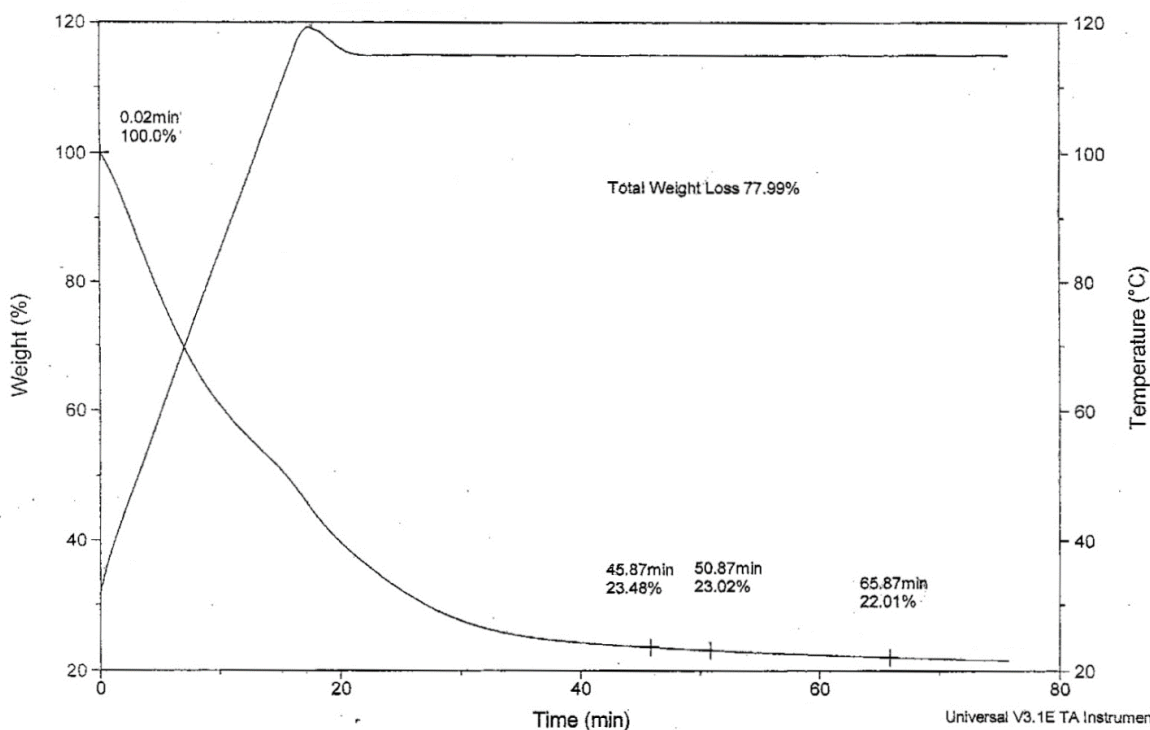
- a. Mass is acceptable in terms of mg and/or percent of initial mass.
  - b. Mass loss is acceptable in terms of mg.
  - c. Percent mass loss might be acceptable, depending on how it is calculated. The proper equation is  $\frac{mass_{initial} - mass}{mass_{initial}}$  or  $100\% - \frac{mass}{mass_{initial}}$ , where  $mass_{initial}$  is the initial sample mass that is recorded on the submission sheet.
  - d. Do not calculate percent mass loss by subtracting percent mass from percent mass at zero minutes, unless percent mass at zero minutes is 100%. DPR staff will recalculate any such percent mass loss values. Recalculation requires mass (in mg or percent), or mass loss in terms of mg. If these values are not also annotated and recalculation is not possible, the data are considered unacceptable.
6. Each data submission needs to include a minimum of three thermograms conducted at 115 °C. For tests conducted at 55 °C, all three thermograms from the 115 °C test are required to demonstrate failure to stabilize.

Examples of correctly annotated and developed thermograms (i.e., Figures 1 and 2) are provided at the end of this document for reference.

The results of the study are to be reported on the "[TGA Data Submission Form](#)." Results reported on the TGA Data Submission Form must match the conduct and results from the study. Percent water and other exempt ingredients are calculated from ingredients listed in the tested product's U.S. Environmental Protection Agency's Confidential Statements of Formula (CSFs) and from any additional CSFs for the product's ingredients.

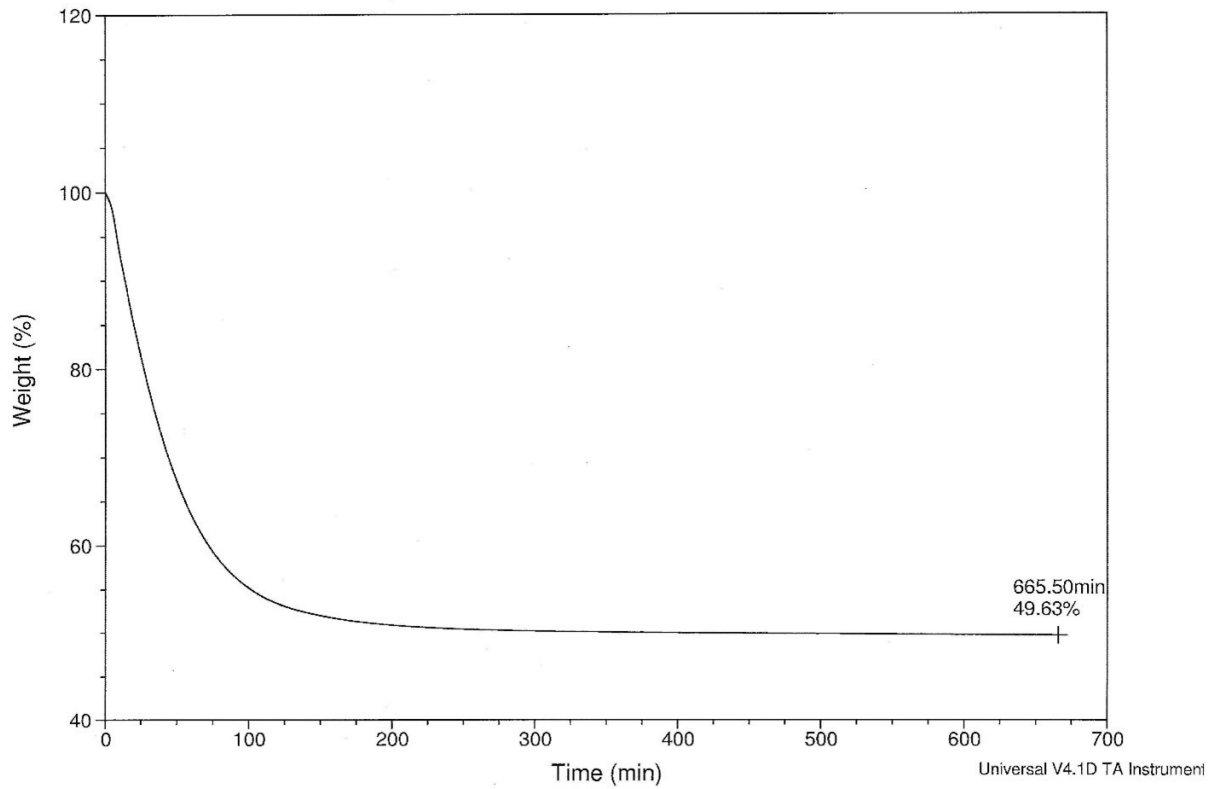
## Figures 1 and 2

Size: 10.0120 mg



**Figure 1.** This thermogram was run at 115 °C. The temperature curve begins at 35 °C and shows an increase of 5 °C per minute over 16 minutes. The mass curve achieves stability roughly between 46 and 51 minutes, with total mass loss less than 0.5% during this period. Final mass is annotated 15 minutes after the end of the stabilization period. Final mass loss equals 77.99%. Time and mass annotations are consistent with the axes. Initial sample mass is printed above the thermogram for easy comparison with the TGA submission sheet.

Size: 10.3680 mg



**Figure 2.** This is a thermogram of a sample tested at 55 °C. Final mass is annotated at 665.50 minutes. This complies with the minimum run time of 664 minutes. Final mass loss equals 50.37%. Initial sample mass is printed above the thermogram for easy comparison with the TGA submission sheet.