Thermogravimetric Analysis

After this module you will be able to…

- Identify the components of a TGA instrument
- Describe what can be measured by a TGA experiment
- Interpret a TGA plot

Table of Contents

Lesson 1: Introduction to Thermogravimetric Analysis (TGA)

What is TGA?

Thermogravimetric analysis (TGA) measures the mass of a sample as the temperature is changed over a period of time. TGA has many applications in the fields of environmental, food, pharmaceutical, and petrochemicals. In this research TGA is used to measure the boiling points of the various components of the bio-oil product. The boiling points of usable bio-oil products should fall within certain ranges as shown below in table 1. Any compounds with boiling points above 700ºC could not be used as a fuel source.

Type of Fuel	Boiling Point (ºC)
Bottle gas and chemicals	25 - 110
Gasoline	110 - 200
Jet fuel, stove fuel, and diesel oil	200 - 300
Lubricating oil for engines, ship fuel, and machine oil	300 - 400
Lubricants and candles, ship fuel	400 - 550
Ship fuel, factory fuel, and central heating	550 - 700
Asphalt and roofing	700 - 800

Table 1. Boiling point range of fuel.[1]

Lesson #2: Components of a TGA Instrument

A basic TGA instrument has four key parts: balance, sample platform, furnace, and purge gas system. The balance in a TGA is a highly sensitive instrument that accurately measures the mass of the sample at specific time intervals. The maximum sample weight of the Q500 TGA is 1 g with a sensitivity of 0.1 µg. The sample platform is where the sample is loaded and unloaded. The furnace heats the sample to the desired temperature. The purge gas system flows inert gas over the sample to remove any vaporized sample or decomposition products. The components of a TGA are shown in Figure 1 below.

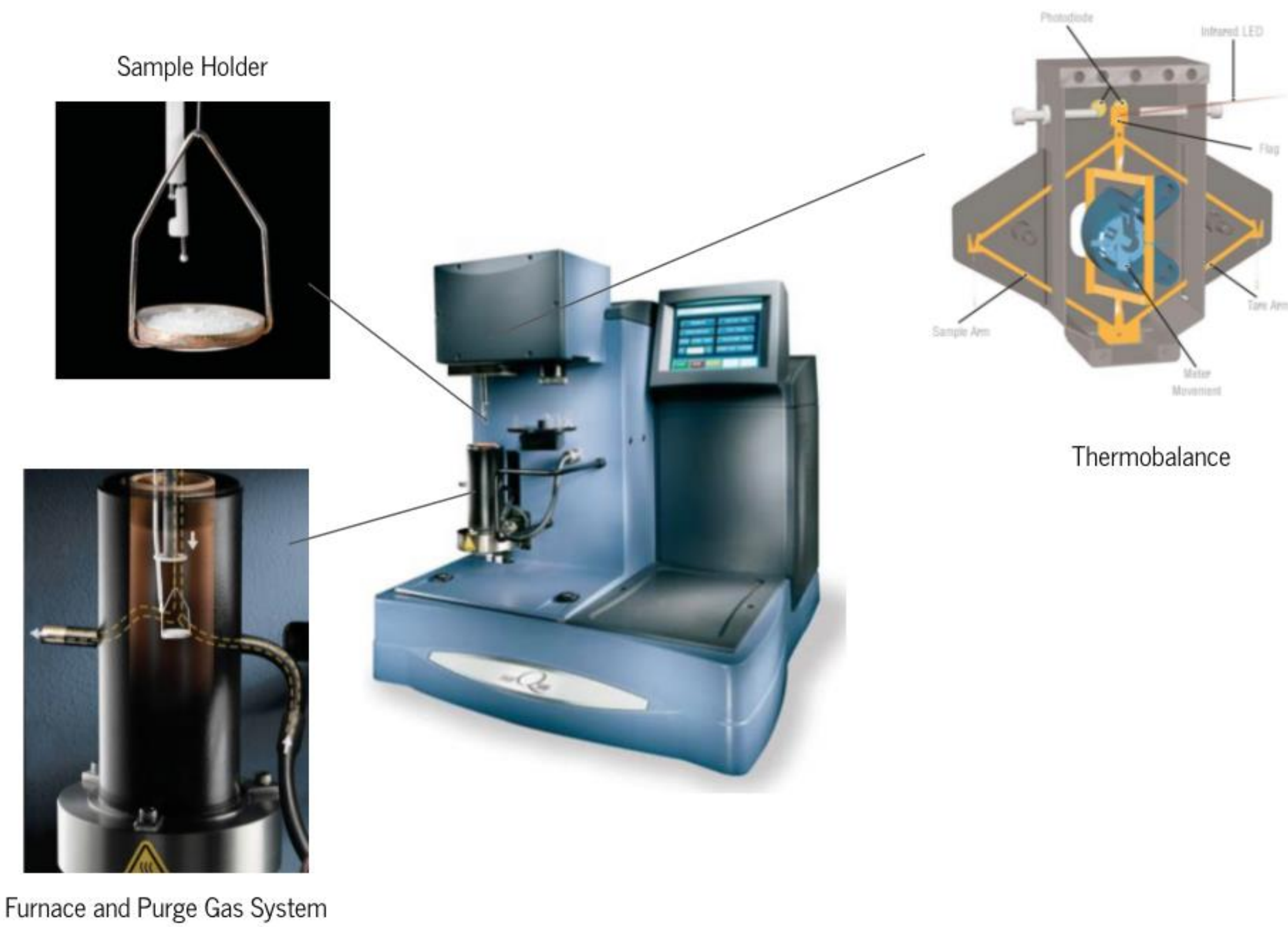

Figure 1. The components of a TGA instrument.

Lesson #3: TGA Experiments & Plots

Types of Experiments

There are three types of experiments that can be performed with a TGA instrument:

1. In isothermal thermogravimetry, temperature is kept constant and the sample weight is recorded as a function of time. This provides information about the stability of a substance at a given temperature.
2. In quasistatic thermogravimetry, the temperature is raised then is kept constant for a set amount of time. During the isothermal intervals, the sample is able to reach stability before the next increase in temperature. This is a good experiment to test the decomposition of substances at different temperatures.
3. In dynamic thermogravimetry, the sample weight is recorded as the temperature changes in a linear manner. This allows you to study how much gaseous products are removed as a function of temperature. Most of the TGA experiments run in this research lab are dynamic thermogravimetry.

TGA Plots

For dynamic thermogravimetry, the data plotted is sample mass percent as a function of temperature. Figure 2 shows the TGA curve for hydrated calcium oxalate. Each dip in the curve represents the loss of a new gaseous substance. Calcium oxalate is going to decompose at higher temperatures to produce carbon dioxide. The components of the bio-oil products in this lab will vaporize to their gaseous state. Instead of measuring the decomposition products, the TGA instrument is measuring the boiling points of the components. These boiling points play a role in determining what the oil can be used for. See table 1 for more information.

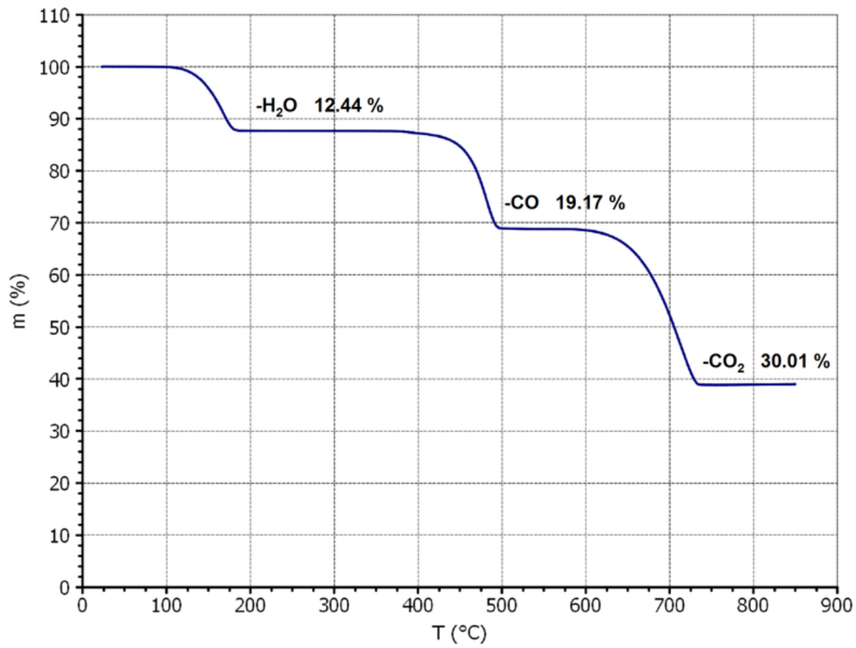

Figure 2. The TGA plot for the decomposition of calcium oxalate.

To help decipher the graph further, the first derivative of mass percentage vs. temperature can also be plotted. The first derivative is often overlaid onto the original plot to make it easier to identify the temperature at which a mass change occurs. The first derivative also provides an idea of the relative amount of mass that decomposed or evaporated at that temperature. That plot is shown below in Figure 3. From the first derivative plot you are able to see at what temperature the phase change occurred and also the relative amount of gas that was lost at each temperature.

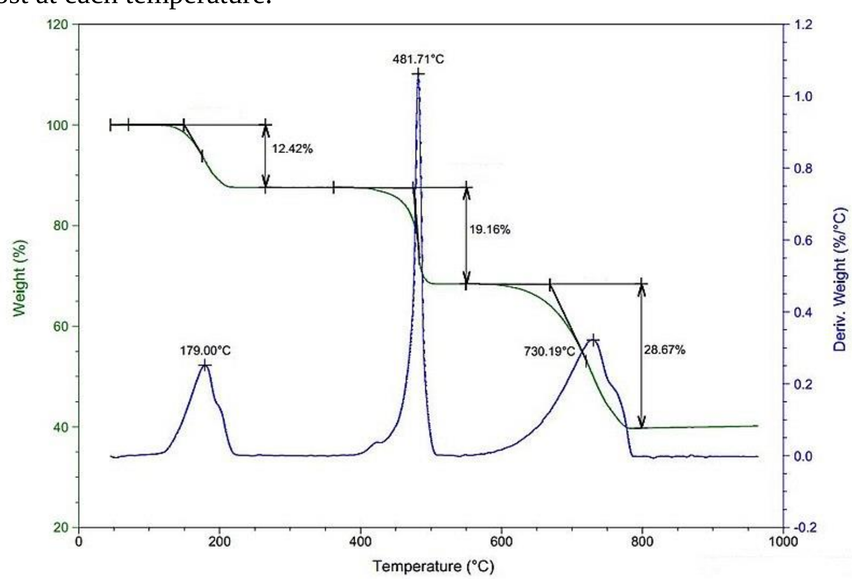

Figure 3. The TGA plot layered with the first derivative.

Image Credits

Figure 1: “TA Instruments Thermal Analysis” accessed via https://www.tainstruments.com/pdf/TGA%20Brochure.pdf

Figure 2: "Decomposition of calcium oxalate monohydrate - thermogravimetric analysis (TGA)" by Steffen 962 is licensed under CC0 1.0 Universal Public Domain Dedication

Figure 3: "Thermogravimetric Analysis of Calcium Oxalate" by DocMatSte is licensed under CC Attribution - Share Alike 4.0 International

Check Quiz

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1. What useful information does a TGA experiment provide?
   a. Molecular formula of major product
   b. Boiling points distribution of a mixture
   c. Elemental composition of a mixture
   d. Physiological effects of pharmaceuticals

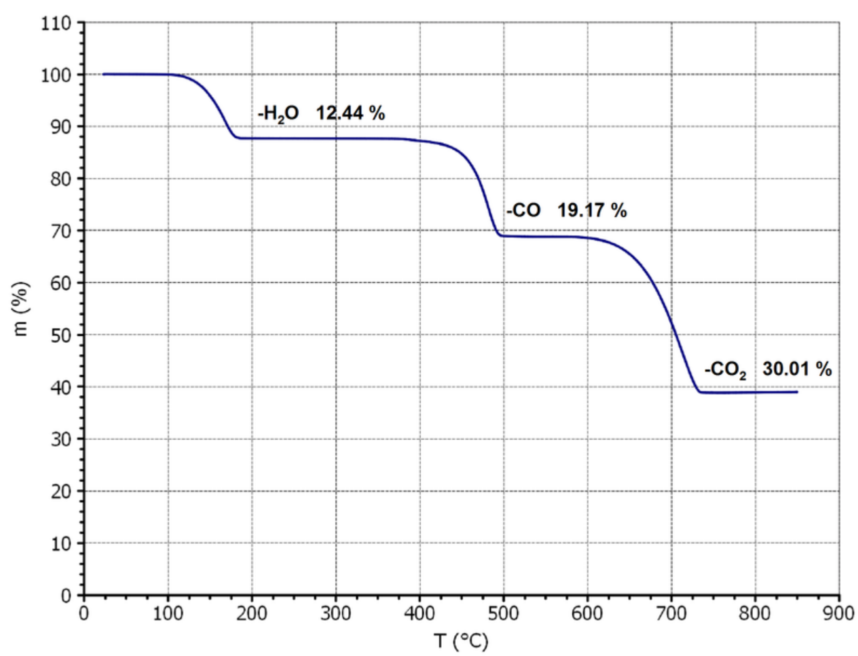
2. Which of the following parts of a TGA is responsible for removing vaporized or decomposition products?
   a. Balance
   b. Sample platform
   c. Furnace
   d. Purge gas system

3. Name each type of TGA experiment.

   Isothermal (A) Quasistatic (B) Dynamic (C)

   Temperature is increased, remains constant for a set period of time, and is raised again. _______
   Temperature is kept constant. _______
   Temperature is raised continuously. _______

4. Use the TGA plot below to determine what percentage of products decomposed or boiled between 100-300ºC.



Answer Key

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1. **Answer B**; TGA heats a sample and records the loss of mass as a

function of temperature. The mass loss is caused by a compound boiling or decomposing into a gas.

2. **Answer D**; The purge gas system runs a constant flow of inert gas over the sample. It carries away any gaseous products that were formed by heating the sample.

3. **Answer B**; Temperature is increased, remains constant for a set period of time, and is raised again. This experiment typically measures the thermal stability of a compound at various temperatures.

   Answer A; Temperature is kept constant. This experiment typically measures the thermal stability of a compound at one temperature.

   Answer C; Temperature is raised continuously. This experiment determines the boiling point distribution of a sample.

4. **12.44%** of products entered the gaseous phase between 100-300ºC. The mass % decreases from 100% at 100ºC to about 88% at 300ºC.