

Detection of Chemical Isomers with Gas Chromatography

There are many different types of chemical isomers, including *structural isomers* and *stereoisomers*. A structural isomer is one in which molecules with the same molecular formula have different bonding patterns or atomic arrangements. A stereoisomer differs in that the molecule bonds are in the same order, but the spatial arrangement is different. Separating isomers is an important use of instrumentation. Gas chromatography can be used to identify structural isomers. In this experiment, you will identify and isolate the structural isomers, *o*-xylene and *p*-xylene.

OBJECTIVES

- Explain the difference between various types of chemical isomers.
- Use gas chromatography to separate and identify chemical isomers of xylene.

MATERIALS

One of the following

- Chromebook, computer, or mobile device with Vernier Instrumental Analysis app¹
- LabQuest 2 (software is pre-installed; v2.9.0 or newer required)²
- LabQuest 3 (software is pre-installed; v3.0.7 or newer required)²

Go Direct Mini GC

1 μ L Hamilton syringe

goggles and gloves

paper towel or lint-free wipe

n-hexane

o-xylene

p-xylene

mixture of *p*-xylene and *o*-xylene

¹ Instrumental Analysis v1.3 or newer required; download the most recent version for free at www.vernier.com/ia

² Download the most recent version of LabQuest software for free at www.vernier.com/downloads

PRE-LAB ACTIVITY

Complete Table 1. This information is a common starting point for understanding the behavior of a set of substances that may be found in a mixture being tested by gas chromatography.

Table 1			
Compound	Boiling temperature (°C)	Molar mass (g/mol)	Chemical structure
<i>o</i> -xylene			
<i>p</i> -xylene			

PROCEDURE

1. Put on safety goggles and gloves.
2. Set up the Go Direct Mini GC by following the directions for your equipment:

Instrumental Analysis

- a. Launch Instrumental Analysis.
- b. Connect the Mini GC to your Chromebook, computer, or mobile device via USB or Bluetooth wireless technology. If using Bluetooth, click or tap Connect an Instrument, connect to your Mini GC, and click or tap Done.
- c. Click or tap Gas Chromatography.
- d. Set the temperature and pressure profile to the parameters listed in Table 2. Then, click or tap Apply to initiate the profile.

Table 2	
Start temperature (°C)	30
Hold time (min)	0
Ramp rate (°C/min)	5
Final temperature (°C)	95
Final hold time (min)	2
Pressure (kPa)	11

LabQuest

- a. Connect the Go Direct Mini GC to your device via USB or Bluetooth wireless technology. If using Bluetooth, tap the Sensors menu and choose Wireless Device Setup ► Go Direct. Select your instrument and tap OK.
 - b. Start data collection.
 - c. Set the GC Profile settings to the values in Table 2.
3. While the Mini GC is warming up, follow the procedure in this step to clean and flush the syringe with n-hexane. **Important:** The glass syringe is fragile. Be careful not to bend the needle or bend the plunger. Never pull the plunger back more than 50% of its total volume.
- a. Depress the plunger fully.
 - b. Submerge the tip of the syringe needle into the vial of n-hexane. **DANGER:** *n-Hexane, C₆H₁₄: Keep away from heat, sparks, open flames, and hot surfaces—highly flammable liquid and vapor. Do not eat or drink when using this product. Avoid breathing mist, vapors, or spray. May be fatal if swallowed and enters airways. May cause damage to organs. Causes skin and eye irritation. May cause drowsiness or dizziness. Suspected of damaging fertility or the unborn child. Do not handle until all safety precautions have been understood.*
 - c. Pull back the plunger to fill the barrel with 0.4 µL of n-hexane.
 - d. Expel the liquid onto a lint-free wipe or a paper towel.
 - e. Repeat Steps a–d at least two times, or until you are comfortable pulling up a liquid into the syringe and measuring the volume in the syringe barrel.
4. Follow the process in Step 3 to clean and flush the syringe with o-xylene, the first sample to be injected into the Mini GC. **WARNING:** *Flammable liquids. Flammable liquid and vapor. Keep away from heat, sparks, open flames, and hot surfaces. No smoking. May cause drowsiness or dizziness. Avoid breathing mist, vapors, or spray.*
5. Once the Mini GC has reached the correct start temperature and pressure, the LED will turn green. Collect a 0.1 µL volume of the pure sample for injection. Insert the needle into the injection port of the Mini GC. Depress the syringe plunger; immediately following, press the button on the Mini GC to initiate data collection. Be careful not to bend the plunger as you press it down. Pull the needle out of the injection port immediately. Data collection will automatically stop after 15 minutes, or you can choose to end it early if you are satisfied that your species has eluted completely.
6. Name your sample appropriately.
- Instrumental Analysis: Click or tap the y-axis label. Click or tap the pencil icon next to the data set you wish to rename. Name your sample set and then click or tap Rename.
 - LabQuest: From the Table screen, tap the Run 1 label. Name your sample set and tap Done.
7. Test the p-xylene and the xylene mixture. **WARNING:** *Flammable liquids. Flammable liquid and vapor. Keep away from heat, sparks, open flames, and hot surfaces. No smoking. May cause drowsiness or dizziness. Avoid breathing mist, vapors, or spray.*
- a. Click or tap Initiate Data Collection (Instrumental Analysis) or Collect (LabQuest). Then, click or tap Apply; you will use the same parameters as before.


Experiment 4

- b. While waiting for the instrument to warm up, follow the process in Step 3 to clean and flush the syringe with the *p*-xylene.
 - c. Repeat Steps 5–6.
 - d. Repeat this process to test the xylene mixture.
8. Save the file as directed by your instructor. Keep your test results open; you will need to refer to the various chromatograms to answer the Data Analysis questions.
 9. When you are done with data collection, make sure to turn the Mini GC power switch to the off position and disconnect the Mini GC from AC power.

DATA TABLE

Table 3		
Compound	Retention time (min)	Peak area
<i>o</i> -xylene		
<i>p</i> -xylene		
<i>o</i> -xylene in mixture		
<i>p</i> -xylene in mixture		

DATA ANALYSIS

1. Identify each isomer in your mixture.
2. Determine the retention time and peak area for each compound. To analyze the chromatogram, follow these steps:
 - Instrumental Analysis: Drag across the peak to select it, then click or tap Add Peak Integral, . Record the retention times in Table 3.
 - LabQuest: From the Analyze menu, choose Advanced, then Peak Integration. Select the signal you wish to analyze. Once the Peak Integration screen appears, highlight the peak you wish to analyze and tap Add. Record the retention times in Table 3.

EXTENSION

BTEX refers to a group of chemicals that includes benzene; toluene; ethylbenzene; and *o*-, *m*-, and *p*-xylenes. BTEX compounds are highly volatile contaminants, usually found in gasoline, with high solubility and toxicity. Therefore, they represent a high risk for public health and are classified as priority pollutants regulated by many environmental organizations around the world. Gas chromatography is a common method for their isolation and presence determination. Given what you have learned in this experiment, determine a temperature/pressure profile for a sample of BTEX. Obtain a sample from your instructor and verify your profile. **WARNING: Flammable liquids. Flammable liquid and vapor. Keep away from heat, sparks, open flames, and hot surfaces. No smoking. May cause drowsiness or dizziness. Avoid breathing mist, vapors, or spray.**