PURPOSE
To observe and identify metallic ions, using flame tests.

BACKGROUND
Have you ever wondered why a candle flame is yellow? The characteristic yellow of a candle flame comes from the glow of burning carbon fragments. The carbon fragments are produced by the incomplete combustion reaction of the wick and candle wax. When elements, such as carbon, are heated to high temperatures, some of their electrons are excited to higher energy levels. When these excited electrons fall back to lower energy levels, they release excess energy in packages of light called photons, or light quanta. The color of the emitted light depends on its energy. Blue light is more energetic than red light, for example. When heated, each element emits a characteristic pattern of light energies, which is useful for identifying the element. The characteristic colors of light produced when substances are heated in the flame of a gas burner are the basis of flame tests for several elements.

In this experiment, you will perform the flame tests used to identify several metallic elements.

MATERIALS (PER PAIR)
safety goggles potassium nitrate, KNO₃
8 small test tubes calcium nitrate, Ca(NO₃)₂
test-tube rack strontium nitrate, Sr(NO₃)₂
paper towel lithium nitrate, LiNO₃
scoopulas copper(II) nitrate, Cu(NO₃)₂
50-mL beaker sodium nitrate, NaNO₃
platinum wire or nichrome barium nitrate, Ba(NO₃)₂
wire loop 6M hydrochloric acid, HCl
gas burner cobalt-blue glass
unknown salt

SAFETY FIRST!
In this lab, the solutions you will be using contain harmful materials. Avoid skin contact with these chemicals. Observe all precautions, especially the ones listed below. If you see a safety icon beside a step in the Procedure, refer to the list below for its meaning.

Caution: Wear your safety goggles. (All steps.)
Caution: Hydrochloric acid is corrosive and can cause severe burns. (Step 2.)

Caution: Do not taste any of the substances or touch them with your hands. (Step 1.)

Caution: Do not at any time touch the end of the wire loop used in the flame tests. This wire gets extremely hot and can cause severe burns. (Steps 2–5.)

Note: Return or dispose of all materials according to the instructions of your teacher. (Step 6.)

PROCEDURE

As you perform the experiment, record your observations in Data Table 1.

1. Place a test-tube rack on a paper towel. Write the chemical name for each of the seven metal salts next to a position in the rack where a test tube will be placed. Use scoopulas supplied with each salt to place pea-sized samples of each metal salt into a test tube. Place the tubes in the test-tube rack.

2. Pour about 15 mL of 6M HCl into a clean, labeled 50-mL beaker. Dip the wire loop into the 6M HCl and then heat it in the hot flame of a gas burner, as shown in Figure 6.1a. Continue this procedure until no color comes from the wire when it is put into the flame.

3. Dip the clean wire loop into a sample of metal salt and heat the sample in the burner flame, as shown in Figure 6.1b. Record the color of the flame in Data Table 1. Test the remaining samples, cleaning the wire loop as described in Step 2, before each new sample is tested. Record your observations.

4. View the flame colors produced by NaNO₃ and KNO₃ through cobalt-blue glass. Record your observations.

5. Perform a flame test on your unknown salt. Record your observations.

6. Dispose of the unused portions of your samples as directed by your teacher.

The solutions can be stored and reused or use the following disposal methods for chemical waste.

Disposal 1: NaNO₃, KNO₃, Ca(NO₃)₂, Sr(NO₃)₂, LiNO₃, Cu(NO₃)₂.

Disposal 2: HCl(aq).

Disposal 3: Ba(NO₃)₂.

Figure 6.1a

Figure 6.1b
**OBSERVATIONS**

**DATA TABLE 1: FLAME TESTS**

<table>
<thead>
<tr>
<th>Ion</th>
<th>Flame Color</th>
</tr>
</thead>
<tbody>
<tr>
<td>sodium, Na⁺</td>
<td>yellow</td>
</tr>
<tr>
<td>potassium, K⁺</td>
<td>violet and yellow</td>
</tr>
<tr>
<td>calcium, Ca²⁺</td>
<td>brick red</td>
</tr>
<tr>
<td>barium, Ba²⁺</td>
<td>green</td>
</tr>
<tr>
<td>strontium, Sr²⁺</td>
<td>bright red</td>
</tr>
<tr>
<td>lithium, Li⁺</td>
<td>crimson</td>
</tr>
<tr>
<td>copper, Cu²⁺</td>
<td>blue-green</td>
</tr>
<tr>
<td>sodium, Na⁺ (cobalt glass)</td>
<td>none</td>
</tr>
<tr>
<td>potassium, K⁺ (cobalt glass)</td>
<td>violet</td>
</tr>
<tr>
<td>unknown</td>
<td></td>
</tr>
</tbody>
</table>

**ANALYSES AND CONCLUSIONS**

1. List the elements that produced the most easily identified colors.
   
   \[ \text{Ca}^{2+}, \text{Ba}^{2+}, \text{Sr}^{2+}, \text{Li}^{+}, \text{Cu}^{2+} \text{ are quite easily identified.} \]

2. Which elements are least easily identified? Explain.
   
   \[ \text{Na}^{+} \text{ and K}^{+} \text{ are difficult to distinguish.} \]

3. Which element produces the most intense color?
   
   \[ \text{The color given by Sr}^{2+} \text{ is a very bright red and is probably the most intense.} \]

4. Would flame tests be useful for detecting metal ions present in a mixture of metal ions? Explain.
   
   \[ \text{The detection of metal ions in mixtures would be difficult. For example, Li}^{+} \text{ and Sr}^{2+} \text{ both give red flames and cannot be distinguished in a mixture.} \]
5. The energy of colored light increases in the order red, yellow, green, blue, violet. List the metallic elements used in the flame tests in increasing order of the energy of the light emitted.

\[
\text{Sr}^{2+}, \text{Li}^+, \text{Ca}^{2+} \rightarrow \text{Na}^+, \text{Ba}^{2+}, \text{Cu}^{2+} \rightarrow \text{K}^+
\]

5. List the metallic elements used in the flame tests in increasing order of the energy of the light emitted.

6. What is the purpose of using the cobalt glass in the identification of sodium and potassium?

\[
\begin{align*}
\text{K}^+ & \text{ nearly always gives a yellow flame because of the presence of trace amounts of Na}^+. \\
\text{Cobalt glass filters out the yellow, allowing the violet of K}^+ \text{ to be seen. The glass helps to distinguish Na}^+. \text{ If only Na}^+ \text{ is present, no flame is seen through the cobalt glass. If K}^+ \text{ is present, violet light is seen through the glass.}
\end{align*}
\]

**GOING FURTHER**

**Do Research**

In this lab, you observed that each element emits a unique color of light when heated in a flame. If these light emissions were examined through a prism, you would observe that the emitted light is actually composed of different wavelengths of light that may lie in the violet region, the green region, or the red region of the visible spectrum. Each element has a unique emission spectrum. Look up the emission spectra for the elements tested in this lab. Do research on how scientists apply these emission spectra to investigate the chemical composition of stars. For example, what is the emission spectrum of the sun, and what does this spectrum reveal about the types of elements in the sun?

**Scientists compare the emission spectrum of the sun with the wavelengths of lines emitted by elements in the laboratory to determine the kinds of elements in the sun. At least 67 different elements have been detected in the sun’s emission spectrum. The sun is mainly composed of helium and hydrogen.**
PURPOSE
To construct a simple flame spectrograph and measure a wavelength of light produced by the electronic excitations of sodium ions.

BACKGROUND
You should recall from Experiment 6 that flame tests are useful for identifying metal ions that produce characteristic colors. Separating these characteristic colors into discrete wavelengths of light produces a pattern of individual lines that uniquely identifies the metal ion. This pattern of lines is called an emission spectrum. With a reference source of emission spectra, you would find it relatively easy to identify a particular metal ion.

You can separate the lines in the visible region of a flame emission spectrum by using an optical prism or a diffraction grating. A spectrograph is an instrument designed to produce electronic excitations, separate the emitted light into its component wavelengths, and then record the wavelengths of emitted light. In this experiment, you will construct a simple spectrograph and measure the wavelength of a strong excitation of sodium ions.

MATERIALS (PER PAIR)
safety goggles
2 meter sticks
diffraction grating
cardboard piece with narrow slit
50-mL beaker

SAFETY FIRST!
In this lab, observe all precautions, especially the ones listed below. If you see a safety icon beside a step in the Procedure, refer to the list below for its meaning.

Caution: Wear your safety goggles. (All steps.)

Caution: Hydrochloric acid is corrosive and can cause severe burns. (Step 2.)

Caution: Do not let your skin or clothing contact the burner flame or the hot wire used in the flame tests. (Steps 2–4.)
Caution: Exercise care when working with an open flame. Tie back hair and loose clothing. Do not use the burner near flammable materials. (Steps 2–4.)

Note: Return or dispose of all materials according to the instructions of your teacher. (Step 7.)

PROCEDURE
As you perform the experiment, record your data and calculation results in Data Table 1.

1. Set up the apparatus shown in Figure 7.1.

2. Pour approximately 15 mL of 6M hydrochloric acid, HCl, into a 50-mL beaker. Always cover the beaker with a watch glass when the beaker is not being used. Clean the wire loop by first dipping it into the HCl and then heating it in the hot flame of a gas burner. Continue to dip and heat the wire until no color comes from the wire as it is heated.

3. Dip the clean wire loop into the NaCl solution.

4. Place the wire loop in the burner flame. Observe the flame through the slit in the cardboard and the diffraction grating, as shown in Figure 7.1. You should see a series of lines to the left and right of the slit. Pick out the brightest line to the left side of the slit and have your partner record this position on the meter stick as position A. Repeat this procedure on the right side of the slit and record this as position B.

5. Measure the distance from the diffraction grating to the slit and record this as distance Y.

6. Dispose of the solutions as directed by your teacher.

Use the following disposal methods for chemical waste.

Disposal 2: NaCl(aq).
Disposal 3: HCl(aq).

Figure 7.1
OBSERVATIONS

DATA TABLE 1: WAVELENGTH FOR THE SODIUM EMISSION LINE

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>left image (A)</td>
<td>15.0 cm</td>
</tr>
<tr>
<td>right image (B)</td>
<td>15.8 cm</td>
</tr>
<tr>
<td>distance X (average of left and right images)</td>
<td>15.4 cm</td>
</tr>
<tr>
<td>distance Y</td>
<td>40.0 cm</td>
</tr>
<tr>
<td>distance Z</td>
<td>42.9 cm</td>
</tr>
<tr>
<td>diffracting grating constant (d)</td>
<td>$1.90 \times 10^{-4}$ cm/line</td>
</tr>
<tr>
<td>sin $\theta$</td>
<td>0.359</td>
</tr>
<tr>
<td>wavelength ($\lambda$)</td>
<td>682 nm</td>
</tr>
</tbody>
</table>

ANALYSES AND CONCLUSIONS

1. Find the average of distances A and B (in centimeters). Record this answer as distance X in your data table.

$$\frac{15.0 \text{ cm} + 15.8 \text{ cm}}{2} = 15.4 \text{ cm}$$

2. Calculate the distance Z, using the Pythagorean theorem. Refer to Figure 7.1 for relationship of distances.

$$Z = \sqrt{X^2 + Y^2}$$

Record the value of Z in the data table.

$$Z = \sqrt{(15.4 \text{ cm})^2 + (40.0 \text{ cm})^2}$$

$$= \sqrt{237.16 \text{ cm}^2 + 1600 \text{ cm}^2}$$

$$= 42.9 \text{ cm}$$

3. Calculate sin $\theta$, using the following relationship:

$$\sin \theta = \frac{X}{Z}$$

Record sin $\theta$ in the data table.

$$\sin \theta = \frac{15.4 \text{ cm}}{42.9 \text{ cm}}$$

$$= 0.359$$
4. The wavelength (\( \lambda \)) of the sodium flame emission line being investigated in this experiment is given, in nanometers, by the Bragg equation:

\[
\lambda = d \times \sin \theta \times \left( \frac{1 \times 10^7 \text{ nm}}{1 \text{ cm}} \right)
\]

\( d \) represents a diffraction grating constant: \( d = \frac{1}{n} \) where \( n \) is the number of lines, per centimeter, scribed on the diffraction grating. Calculate the value of \( d \) for your grating and enter it in Data Table 1.

The number of lines per inch is often 13 400; \( d \) may be calculated as follows:

\[
d = \frac{1 \text{ in.}}{13 400 \text{ lines}} \times \frac{2.54 \text{ cm}}{1 \text{ in.}} = 1.90 \times 10^{-4} \text{ cm/line (constant)}
\]

5. Compute the wavelength of the bright line you viewed on the meter stick, using the Bragg equation. Record this value in the data table.

\[
\lambda = (1.90 \times 10^{-4} \text{ cm}) \times (0.359) \times \left( \frac{1 \times 10^7 \text{ nm}}{1 \text{ cm}} \right) = 682 \text{ nm}
\]

6. The accepted value of \( \lambda \) for the observed transition is 589.0 nm. Calculate the percent error in your value.

\[
\text{percent error} = \frac{682 \text{ nm} - 589 \text{ nm}}{589 \text{ nm}} \times 100 = 15.8\%
\]

7. Identify the possible sources of error in your determination of \( \lambda \).

The major source of error is the measurement of the values for \( X \) and \( Y \).

8. How can a spectrographic experiment help identify a particular metal ion?

The ions of each element give characteristic patterns of lines in the spectrograph.

Even elements that produce flame-test results that appear to the eye to be the same color produce very different sets of flame emission lines.

**GOING FURTHER**

**Do Research**

Atomic absorption spectroscopy is one of the most sensitive methods available for the detection of various metals. Do research to find out how this method is used to quantify the amount of an element in a particular sample and compare the detection limits of this method with other spectroscopic methods of analysis.

The detection limits of many modern atomic spectroscopic methods lie well below 1 ppm for a number of metallic elements. The ultrasensitivity of this technique is a valuable asset to scientists studying materials that contain only trace amounts of a given metal.
PURPOSE
To determine the absorption spectrum of an aqueous solution of chromium(III) ions.

BACKGROUND
Many compounds absorb light from regions of the electromagnetic spectrum. A spectrophotometer is a device designed to determine the wavelengths of light that a compound absorbs. When an aqueous sample of a compound is placed in the light path of a spectrophotometer, the sample may absorb all the light, some of the light, or no light at all. The absorption of light depends upon the materials in the sample and the wavelength of the light. Light absorption occurs at wavelengths whose energy corresponds to the energy necessary to cause electronic excitations of atoms, ions, or molecules in the sample. From the spectrophotometer data, a graph can be made that plots the light intensity transmitted through the sample versus the wavelength of the light; such a graph is called an absorption spectrum. The range of wavelengths absorbed by the sample appear as bands of minimum intensity.

Absorption spectra are useful for two reasons. First, the absorption spectrum of a substance is a unique characteristic of that substance. This makes the spectrum useful for the identification of unknown substances. Second, the intensity of the absorption bands can be related to the concentration of the substance in the sample. Thus, the intensity of the absorption band can be used to determine the amount of a particular substance in a mixture.

In this experiment, you will determine the absorption spectrum of an aqueous solution of chromium(III) ions.

MATERIALS (PER PAIR)
safety goggles  
Spectronic 20 spectrophotometer  
2 small test tubes or 2 glass cuvettes  
10-mL graduated cylinder  
plastic wash bottle  
distilled water  
0.02M chromium(III) nitrate, Cr(NO_3)_3  
tissue paper

SAFETY FIRST!
In this lab, observe all precautions, especially the following ones. If you see a safety icon beside a step in the Procedure, refer to the following list for its meaning.
Caution: Wear your safety goggles. (All steps.)

Caution: Chromium(III) nitrate is toxic and can irritate your skin. (Steps 5–8.)

Note: Return or dispose of all materials according to the instructions of your teacher. (Step 8.)

PROCEDURE

As you perform the experiment, record your percent transmittance data in Data Table 1.

1. Turn on the spectrophotometer and allow it to warm up for about 20 minutes.

2. Set the wavelength control knob to 375 nanometers (375 nm). Adjust the amplifier control knob to produce 0 percent transmittance (0%T) at this wavelength.

3. Add 3 mL of distilled water to a clean, small test tube. Wipe the outside of the tube with a tissue to make certain that it is clean and dry. Avoid getting fingerprints on the tube. Dislodge any air bubbles present in the water by gently tapping the tube with a finger.

4. Place the tube in the sample holder and close the cover. Adjust the light control knob until the spectrophotometer reads 100%T.

5. Remove the first sample from the spectrophotometer. Add 3 mL of 0.02\text{M} chromium(III) nitrate, \text{Cr(NO}_3\text{)_3}, to another clean test tube. Use a tissue to clean and dry the tube. Insert the tube of chromium(III) nitrate into the sample holder. Close the cover of the holder. Read the percent transmittance and record the reading in Data Table 1. Remove the sample from the holder.

6. Turn the wavelength dial to 400 nm. Use the amplifier control knob to adjust the percent transmittance to 0%T. Place the water sample in the holder. With the light control knob, adjust the meter to 100%T. Replace the water sample with the chromium(III) nitrate sample. Measure and record the percent transmittance at 400 nm.

7. For the remainder of the wavelengths listed in Data Table 1, continue the procedure of setting 0%T, setting 100%T, and measuring the percent transmittance of the chromium(III) nitrate solution.

8. Unless directed otherwise by your teacher, return the aqueous chromium(III) nitrate to the dropper bottle.

Step 2.

To avoid damage, the spectrophotometer knobs must not be twisted past the point at which resistance is encountered. Twisting the amplifier control knob counterclockwise past the resistance point turns the spectrophotometer off.

Step 4.

Show students how to tilt the bottom of the test tube slightly to the right (facing the spectrophotometer) to fit into the holder.

The energy of the radiation striking the detector is not constant at all wavelengths, because the energy output of the source is not constant. The amplifier control knob opens or closes the slit to bring the energy at the detector to the constant value designated 100%T. Failure to adjust to 100%T at each wavelength will produce an “absorption spectrum” that includes the energy profile of the source as well as the spectrum of the absorbing species in the sample solution.

Use the following disposal method for chemical waste.

Disposal 6: \text{Cr(NO}_3\text{)_3}.
OBSERVATIONS

ANALYSES AND CONCLUSIONS

1. Graph percent transmittance versus wavelength. The curve you plot is the absorption spectrum of chromium(III) ions in the visible region of the electromagnetic spectrum.

2. At what wavelengths do chromium(III) ions absorb the maximum amounts of light? What colors of light correspond to these wavelengths?

   There are two absorption maxima (transmittance minima). One is at about 420 nm and the other is at about 580 nm. These wavelengths of light correspond to the wavelengths of violet (400–450 nm) and orange (600–650 nm) light.

DATA TABLE 1: PERCENT TRANSMITTANCE AND ABSORBANCE OF 0.02M Cr(NO₃)₃ SOLUTION AT VARIOUS WAVELENGTHS

<table>
<thead>
<tr>
<th>Wavelength (nm)</th>
<th>% Transmittance (%T)</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>375</td>
<td>64.0</td>
<td>0.194</td>
</tr>
<tr>
<td>400</td>
<td>48.5</td>
<td>0.314</td>
</tr>
<tr>
<td>405</td>
<td>47.0</td>
<td>0.328</td>
</tr>
<tr>
<td>415</td>
<td>46.5</td>
<td>0.333</td>
</tr>
<tr>
<td>425</td>
<td>53.2</td>
<td>0.274</td>
</tr>
<tr>
<td>440</td>
<td>60.0</td>
<td>0.222</td>
</tr>
<tr>
<td>455</td>
<td>71.5</td>
<td>0.146</td>
</tr>
<tr>
<td>470</td>
<td>80.0</td>
<td>0.097</td>
</tr>
<tr>
<td>490</td>
<td>82.5</td>
<td>0.084</td>
</tr>
<tr>
<td>500</td>
<td>83.0</td>
<td>0.081</td>
</tr>
<tr>
<td>520</td>
<td>72.0</td>
<td>0.143</td>
</tr>
<tr>
<td>530</td>
<td>67.5</td>
<td>0.171</td>
</tr>
<tr>
<td>540</td>
<td>63.0</td>
<td>0.201</td>
</tr>
<tr>
<td>550</td>
<td>58.5</td>
<td>0.233</td>
</tr>
<tr>
<td>570</td>
<td>54.0</td>
<td>0.268</td>
</tr>
<tr>
<td>575</td>
<td>53.0</td>
<td>0.276</td>
</tr>
<tr>
<td>580</td>
<td>53.0</td>
<td>0.276</td>
</tr>
<tr>
<td>600</td>
<td>56.0</td>
<td>0.252</td>
</tr>
<tr>
<td>625</td>
<td>66.3</td>
<td>0.178</td>
</tr>
</tbody>
</table>
Sample Data:
Percent Transmittance (%T) versus Wavelength (nm)

Sample Data:
Absorbance (A) versus Wavelength (nm)
3. Based on the answer to problem 2, would you expect a red solution to absorb or transmit red light? Explain.

The previous answer suggests that colored solutions absorb light of a complementary color. Blue Cr(NO₃)₃ solution absorbs orange light. (Orange is the complement of blue.) A red solution absorbs green light (the complement of red) and transmits red light. (A yellow solution would absorb its complement, yellow.)

4. The amount of light that is absorbed by a solution is commonly expressed either in terms of percent transmittance, as in this experiment, or in terms of absorbance (A). Absorbance is defined as:

\[ A = \log \frac{100}{%T} \]

or

\[ A = 2 - \log %T \]

Given the relationship shown in the preceding formula, convert the percent transmittance values in Data Table 1 to absorbance values. Plot a graph of absorbance versus wavelength. Compare and analyze the shapes of the two curves. Might it be more useful to use transmittance values sometimes and absorbance values at other times? Explain the advantages and disadvantages of using these different units.

An absorption maximum appears to be sharper than its corresponding transmittance minimum. The concentration of a substance is directly proportional to the area under the peaks in an absorbance spectrum, but not in a transmittance spectrum. For this reason, analytical work is most often expressed in absorbance.
GOING FURTHER

Develop a Hypothesis

Based on the results of this lab, propose a hypothesis about how the absorption of light by a solution of Cr(NO₃)₃ varies with the concentration of Cr³⁺ ions in solution.

The amount of light absorbed is directly proportional to the concentration of the absorbing species in solution.

Design an Experiment

Propose an experiment to test your hypothesis. In your protocol, include a method for determining the concentration of Cr³⁺ ions in an unknown solution of Cr(NO₃)₃. If resources are available and you have your teacher’s permission, perform the experiment.

Measure the absorption of a series of standard solutions of chromium(III) ions at the wavelength of an absorption maximum determined in this experiment. Construct a plot of absorption versus concentration. Draw a best-fit line through these points. The concentration of chromium(III) solutions in an unknown solution can be determined by measuring the absorption and reading its concentration from the graph.