

Laboratory 7a: Synthesis and Characterization of Spinel Compounds

Solid-state inorganic chemistry is a popular area of current research that is largely driven by the potential for interesting technological advances. Spinel is a class of materials whose particular solid-state structure imparts to them a variety of useful properties. Magnetite, Fe_3O_4 , is a naturally occurring magnetic ore that has many applications including magnetic recording. Other spinels are studied for their magnetoresistance properties in which their resistance to current changes in the presence of an applied magnetic field. As a final example, LiMn_2O_4 finds use as a cathode in lithium ion batteries. Spinel is also known for their bright colors in gemstones and pigments. The spinel cobalt blue, for example, is an inorganic pigment used to color ceramic, glass, and paint. In this experiment, you and two partners will synthesize three spinels: ZnCo_2O_4 , CoAl_2O_4 , and CoCr_2O_4 .

Pre-Lab

Write out balanced chemical equations for the synthesis of each spinel starting with the corresponding oxide reagents: ZnO , CoO , Al_2O_3 , and/or Cr_2O_3 . Complete this for each of the three spinels: ZnCo_2O_4 , CoAl_2O_4 and CoCr_2O_4 . You may need to add oxygen gas (O_2) to your equation to arrive at a balanced reaction.

Procedure

You will work with two partner to synthesize the three compounds, sharing results with each other. One person will synthesize ZnCo_2O_4 , a second will synthesize CoAl_2O_4 , and a third will synthesize CoCr_2O_4 . Use the amounts of ZnO , CoO , Al_2O_3 , and/or Cr_2O_3 specified below, which are sufficient to prepare 1.00 gram of each product.

ZnCo_2O_4 : 0.329 g ZnO , 0.606 g CoO

CoAl_2O_4 : 0.423 g CoO , 0.576 g Al_2O_3

CoCr_2O_4 : 0.330 g CoO , 0.669 g Cr_2O_3

Record the colors of your starting materials in your laboratory notebook. Weigh out the appropriate amount of each reactant and record the exact amount used in your lab notebook. Grind the reagents together in a mortar and pestle for **at least 15 minutes**. After grinding, empty the powder onto a piece of weighing paper and scrape out any remaining solid using a spatula. Record the appearance of your ground sample in your lab notebook. Weigh an empty ceramic crucible, transfer your sample into it, and then record the weight of the crucible and sample together. Prepare a label with your name and the name of the spinel you are synthesizing.

The reaction itself takes eight hours at a temperature of 1000°C . Bring your crucible and label to the hood with the muffle furnace and place your sample and label in one of the squares on the grid next to the furnace. The grid will help ensure that your sample is returned to you next.

Details on the sharing of results and on your report for this experiment will be included with the handout for Experiment 7b.

References

1. Rohrer, G. S. *Structure and bonding in crystalline materials*; Cambridge University Press, 2001, pg 162.
2. Huheey, J.E.; Keiter, E.A.; Keiter, R.L. *Inorganic Chemistry: Principles of Structure and Reactivity*, 4th ed.; HarperCollins: New York, 1993, pg 411.

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