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How to Test for Iron and Nickel – Canadian Conservation Institute (CCI) Notes 17/4

by Lyndsie Selwyn

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Introduction

Iron and nickel are industrial metals. Both are white metals, and both are magnetic, so they cannot be distinguished with a simple magnet test. Iron is much more susceptible to corrosion, so it is important to identify it. Nickel is often found in alloys, or as a plating layer. For example, nickel silver (also called German silver) is a white metal alloy that contains no silver; it usually contains copper, nickel, and zinc, and can therefore be identified with a spot test for nickel. Examples of objects containing these metals are shown in Objects containing iron and Objects containing nickel.

Spot tests for some metals (such as copper and lead) involve simply touching a wetted piece of test paper to the metal. Spot tests for iron and nickel, however, are more complicated because water alone does not dissolve enough iron or nickel ions into solution for the ions to be detected. The tests described in this procedure use electrolysis with a battery to force metal ions into solution, where they can be detected by the colour change in the test paper. No sample is removed from the object and no strong acids are used.

Spot tests are one of the simplest analytical techniques for identifying metals. This CCI Note describes the procedure and the required materials to detect iron or nickel in metals using commercial spot test papers. The first step in the procedure involves testing known samples of iron and nickel to gain experience using electrolysis and the test papers, and to confirm that the test papers are working properly. Then unknown metals can be tested. For more information on electrolysis and spot tests in conservation, consult The science behind electrolysis and test papers for metals.

Procedure: using spot test paper with electrolysis

Equipment and materials required for spot tests for iron and nickel

- Commercial spot test papers from Macherey-Nagel (consult Suppliers for information on obtaining these test papers)
    - Iron (Dipyridyl test paper 90725)
    - Nickel (Nickel 90730)
- Iron object or iron coupon
- Nickel object or nickel coupon
- 9-volt battery
- Homemade electrolysis cables that contain stainless steel tweezers and an alligator clip; for construction details, see Homemade electrolysis cables
- Stock solution of saturated sodium chloride; for preparation instructions, see Stock solution preparation: saturated sodium chloride (NaCl)
- Water (deionized or distilled)
- Plastic tweezers
- Scissors
- Pipettes (Pasteur or plastic) or eyedroppers
- Small containers (e.g. disposable 10 mL beakers)
- Marker to label containers
- Aluminum foil (optional)

General advice

1. Wear disposable gloves to avoid touching the test papers and metals with bare hands.
2. Choose an inconspicuous spot to test in case the test leaves a mark on the object.
3. Cut small triangular pieces of test paper from a larger sheet. Cut the triangles so that they have a sharp point. Using a sharp point also helps minimize any mark left by the test.
4. Use a pipette or eyedropper to transfer enough water to wet all of the test paper. Avoid adding too much water because this may wash away the reagent on the test paper.
5. Use plastic (not metal) tweezers to handle the test papers except during the electrolysis step.
6. Clean tweezers with water after each use to prevent contamination.
7. It may be necessary to degrease the metal surface before the test because a dirty surface can interfere with the test. (Use soapy water or acetone.) The test will not work if a coating such as wax or lacquer is present.
8. Optional: use a microscope to watch for a colour change on the test paper. This allows an even smaller spot to be tested.

9. When testing objects of unknown metal, start by testing known samples of iron and nickel to see what the reaction should look like and to ensure the test paper still works. Keep known metal samples with the test papers for this purpose.
10. Optional: use aluminum foil (folded several times if needed) to protect the object from being scratched by the teeth of the alligator clip on the electrolysis cable. Filing down the teeth in the clip is another way to reduce the risk of damaging the object.

Diagram of wiring for electrolysis

Shown in Figure 1 is the wiring diagram for carrying out the spot test with the aid of a battery. Allow only the test paper to touch the metal surface. **Do not** let the tip of the tweezers touch the metal surface or the alligator clip. Also, make sure the test paper between the tweezers and the tip is completely wet. If part of the paper remains dry, then the circuit will not be complete, no current will flow, and the metal will not corrode.

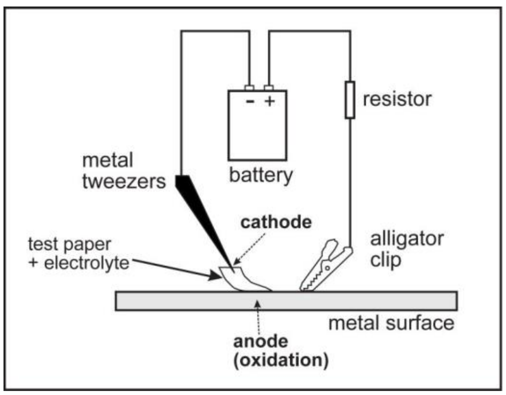

Figure 1: Wiring diagram for using a battery to force a metal to corrode and form metal ions in solution, which will then be present to react with the test paper.

Procedure

1. Transfer a few millilitres of sodium chloride stock solution into a small container.
2. Label the container.
3. Set up the battery and electrolysis cables.
4. Touch the alligator clip directly to the iron or nickel, and hold it steady to avoid scratching the object. Otherwise, sandwich the iron or nickel between aluminum foil and then clamp the alligator clip over the foil. Use enough aluminum foil to prevent the teeth of the clip from perforating the foil.
5. Hold the piece of iron or nickel test paper (point facing out) in the stainless steel tweezers.
6. Wet the test paper with a few drops of saturated sodium chloride using a pipette or eyedropper.

7. Touch the wet test paper to the metal surface.
8. Hold for 5 seconds to allow electrolysis to occur.
9. If iron ions are present, iron test paper will turn pink for Fe^{2+} ions and yellow for Fe^{3+} ions; if nickel ions are present, nickel test paper will turn from a pink to red colour, depending on the concentration of nickel ions.
10. The test paper may leave a pink or red mark on the object. After the test, use clean water to rinse the test area on the metal to remove any residual salt solution, and dry the surface immediately.
11. Use clean water to rinse the tweezers after each use.

Results of this procedure

The commercial test paper for iron is shown in Figure 2 and for nickel in Figure 3.

Figure 2: Macherey-Nagel commercial spot test papers for iron (dipyridyl paper 90725). Pictured are the case and a single sheet of test paper.

Figure 3: Macherey-Nagel commercial spot test papers for nickel (nickel test paper 90730). Pictured are the case and a single sheet of test paper.

Figure 4 shows a triangular piece of iron test paper placed against a piece of iron before the colour has changed. Note that aluminum foil has been used to protect the iron from the teeth of the alligator clip. Figure 5 shows iron test paper after it has reacted with a piece of iron and turned pink.

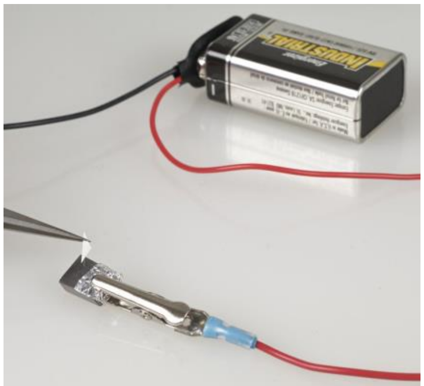

Figure 4: The electrolysis system for the iron spot test using dipyridyl paper, before the reaction has occurred.

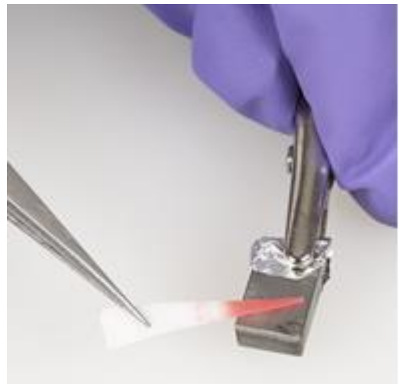

Figure 5: Detail of iron test paper, dipyridyl, reacting and turning pink.

Figure 6 shows nickel test paper after it has reacted with a piece of nickel and turned pink. In this case, the clip is pressed against the metal rather than clamped to it, and no aluminum foil is used.

Figure 6: Detail of nickel test paper, after a reaction has occurred and the paper has turned pink.

Additional information

Objects containing iron

Iron is the most widely used industrial metal today and is sure to be present in museum collections: heavy equipment in industrial collections; armaments and weaponry in military collections; and trucks and cars. It is a huge component in archaeological collections, often presenting conservation problems: cannons and cannonballs, nails, and chains. It is used in outdoor sculpture and in heritage buildings in various forms, such as wrought iron, cast iron, weathering steel, and stainless steel. Iron objects are often found plated with a variety of metals: aluminum, cadmium, chromium, copper, nickel, silver, lead-tin alloys, tin, and zinc. Plated objects may not appear to be iron, but once the coating fails, the iron underneath can rust.

It should be easy to find iron objects to test. Try testing some common objects that contain iron. Examples that tested positive for iron are shown in Figures 7 and 8.

Figure 7: Common nails.

Figure 8: Regular staples for a staple gun.

Objects containing nickel

Nickel is more expensive than iron, and is less common in collections. Nickel is used in corrosion-resistant alloys, such as Monel (a copper-nickel alloy with about 66% nickel). It is found in some coins and in most meteorites. It is used in jewellery, even though some people develop allergies to it. Nickel silver alloys are white copper-rich alloys that contain some nickel but no silver. Nickel is plated on iron-based or nickel-based alloys. It is difficult to plate nickel directly onto iron, so the iron is usually plated with copper, and nickel is plated onto the copper.

Examples that tested positive for nickel are shown in Figures 9 to 12.

Figure 9: Key chain (white metal on rectangle tested positive).

Figure 10: Outdoor light bulb (white metal at base tested positive).

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Figure 11: Fork made with a nickel silver alloy and electroplated with silver. The areas where the plating has worn off tested positive for nickel. (The paper turned pink, but there was also a yellow precipitate probably from interference by copper.)

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Figure 12: Canadian 5-cent coin made from an alloy containing 75% copper and 25% nickel. The spot test paper turned both pink and yellow (probably interference from copper).

Sensitivity and interferences

The iron test paper can detect 2 mg/L (2 parts per million) or more Fe^{2+} ions in solution. The nickel test paper can detect 10 mg/L (10 parts per million) or more nickel ions in solution.

In an ideal spot test, only one metal element produces the colour change. In general, however, other metal elements may also cause a similar colour change. This is called interference, and if it occurs, the test paper can give a positive result even though the metal being tested for is not present. For example, the instructions for the nickel test paper warn that iron can cause interference if ammonia is present, and copper and cobalt can cause interference if present at high concentrations. The instructions for the iron test paper do not list any elements that interfere with the test, but point out that Fe^{3+} ions produce a yellow colour that could mask the red from Fe^{2+}.

Storage

These test papers need to be stored below 30°C in a dry place away from sunlight. If stored properly, they last several years. Nevertheless, it is important to check that the test papers still work by testing known samples of iron and nickel.

Spot tests in conservation

In a spot test, a small drop of water is allowed to react with the metal, producing a coloured reaction product. The test may leave a small mark on an object, but because only a small amount of metal dissolves, the test can usually be done on an inconspicuous area of the object.

A large number of spot tests are available for metals, as described in the book by Feigl and Anger (1972). For detailed information on the use of metal spot tests in conservation (and on detection limits and interferences), see publications by Laver (1978), Townsend (1988), and Odegaard et al. (2005).

Additional procedures

Stock solution preparation: saturated sodium chloride (NaCl)

The following procedure can be used to prepare 100 mL of a saturated sodium chloride solution. The solubility of sodium chloride in water at 25°C is 35.9 g in 100 mL of water or 6.14 moles per litre.

Equipment and materials

- Sodium chloride (NaCl)
- Spatula
- Balance
- Water (distilled or deionized if available)
- Beaker (100 mL)
- Container to store solution

Procedure

1. Use spatula and balance to weigh out roughly 37 g of sodium chloride.
2. Place about 100 mL of water in a 100 mL beaker.
3. Add sodium chloride to the water and swirl to dissolve.
4. If all the sodium chloride dissolves, add a little more sodium chloride.
5. When solid sodium chloride crystals remain undissolved on the bottom of the beaker, the solution above it will be saturated. It can take a few days to achieve saturation.
6. Transfer the solution to a container for long-term storage.
7. Label the container.

Homemade electrolysis cables

Described below is one example of how to make electrolysis cables (Figure 13) that attach to a 9-volt battery.

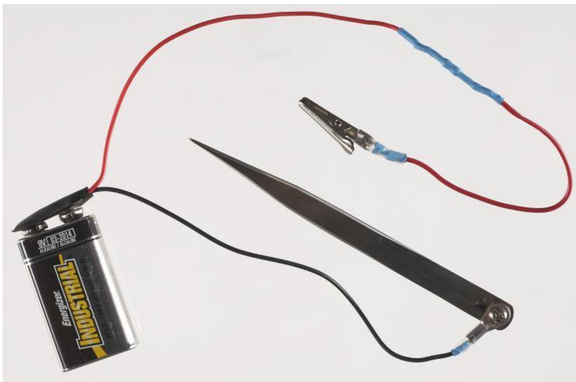

Figure 13: Homemade electrolysis cables connected to a 9-volt battery. The cables are made using a 9-volt battery snap, stainless steel tweezers, a resistor (300 to 360 ohms), an alligator clip, and heat shrink tubing.

Equipment and materials

- Battery, 9-volt
- Battery snap for 9-volt battery (Figure 14)
- Heat shrink tubing (e.g. 3/32" and 3/16")
- Heat gun or hair dryer
- Tweezers, stainless steel (the salt in the electrolyte attacks other types of metal tweezers)
- Hardware for attaching wire to tweezers (e.g. appropriate drill bit, 3 mm screw, 3 mm washer, 3 mm lock washer, ring terminal with hole to fit 3 mm screw); an example of a ring terminal is shown in Figure 15.
- Wire, red insulation (e.g. 22-gauge stranded wire)
- Resistor (300 to 360 ohms, 1/4 watt, Figure 16)
- Alligator clip (subminiature works well for small objects)

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Figure 14: Battery snap for a 9-volt battery.

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Figure 15: A ring terminal (non-insulated); also called an eyelet connector.

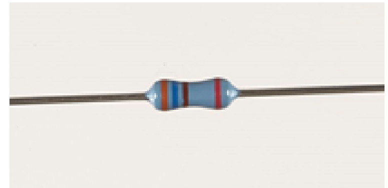

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Figure 16: 360-ohm resistor. The value or resistance is specified by the three rings on the left (orange, blue, brown); the red ring on the right specifies the tolerance (2%). Other acceptable values are 330 ohms (orange, orange, brown) or 300 ohms (orange, black, brown).

Procedure for black wire on battery snap

1. Cut a piece of heat shrink tubing long enough to cover a joint between the ring terminal and wire (see next steps).
    a. Slip heat shrink tubing over the black wire of the 9-volt battery snap; push it all the way to the edge of the snap.
    b. Attach the ring terminal to the end of the black wire (e.g. solder or crimp it).
    c. Slide the heat shrink tubing over the joint between the ring terminal and the wire.
    d. Heat the tubing to shrink it into place.
2. Drill and thread a hole in the stainless steel tweezers for a 3 mm screw (or omit the thread and use a nut instead).
3. Attach the ring terminal to the tweezers using the 3 mm hardware (Figure 17): place a washer over the screw, push the screw through the hole in the ring terminal, add a lock washer, and then thread the screw onto the tweezers.

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Figure 17: Hardware for attaching ring terminal to threaded hole in tweezers in correct order.

Procedure for red wire on battery snap

1. Cut a piece of heat shrink tubing long enough to cover the resistor and the solder joints at both ends of the resistor (see next steps).
    a. Slip the heat shrink tubing over the red wire of the 9-volt battery snap; push it all the way to the edge of the snap.
    b. Solder one end of the resistor (300 to 360 ohms) to the red wire of the battery snap.
    c. Cut a piece of insulated wire (e.g. red 22 gauge); use a length that provides easy manipulation of the clip and the tweezers during a test.
    d. Solder one end of the cut wire to the other end of the resistor.
    e. Slide the heat shrink tubing over the resistor and the two solder joints.
    f. Heat the tubing to shrink it into place.
2. Cut a piece of heat shrink tubing long enough to cover a solder joint between the alligator clip and the wire (see next steps).
    a. Slip heat shrink tubing over the red wire and push it further along the wire if the alligator clip is going to be attached by soldering.
    b. Attach the alligator clip to the red wire (by soldering or crimping).
    c. Slide the heat shrink tubing over the joint.
    d. Heat the tubing to shrink it into place.

Once the cables are complete, attach the 9-volt battery snap to a 9-volt battery.

Check components

To check the components in the homemade electrolysis cables, use a multimeter.

1. Voltage: check that the 9-volt battery is close to 9 volts.
2. Resistance: check the resistance of the two wires from the battery snap.
    a. Resistance of red wire from bigger connector on snap to alligator clip: it should be the same resistance as the resistor (e.g. 300 to 360 ohms)
    b. Resistance of black wire from smaller connector on snap to tweezers: it should be almost zero (unless the wire or a connection is broken)
3. Current: attach the battery snap to the battery and check the current flowing through the circuit. It should be about 30 mA (if 300 ohms), 27 mA (if 330 ohms), and 25 mA (if 360 ohms). If the current is roughly the predicted value, then the battery is still good and can be used for the spot test.

Resistors

The resistor is used to limit the current that can flow from the battery. The resistance (in ohms) and the tolerance are colour-coded with stripes. The resistance can be checked with a multimeter. Good values for resistance in the electrolysis cable are 300 ohms to 360 ohms.

Resistors are sold with different power ratings, but the rating may not be specified on the resistor itself. For the 9-volt battery cables, the resistors used were rated for 1/4 watt (0.25 watts). When the rating of a resistor is exceeded, the resistor can become warm and possibly burn out. The power through a resistor can be calculated from the voltage across it or the current through it, using the following equations:

$$P = I^2 R = I V = V^2/R$$

where P is the power (watts), I is the current (amps), R is the resistance (ohms), and V is the voltage (volts), and V = I R. If the entire voltage from the 9-volt battery were applied across the resistor, the power would be 0.27 watts in a 300 ohm resistor, 0.25 watts in a 330 ohm resistor, and 0.23 watts in a 360 ohm resistor. However, this calculation overestimates the power during electrolysis because only a fraction of the 9 volts from the battery is applied across the resistor. The rest is between the tweezers and the metal being tested. Also, the circuit is used only for a few seconds. Therefore, the 1/4 watt power rating of the resistors used here is acceptable.

The science behind electrolysis and test papers for metals

How test papers work

A chemical indicator has been incorporated into these commercial test papers. The indicator changes colour when exposed to a specific metal ion in solution. For these tests to work, that specific metal ion must be present in solution. Commercial test papers for lead and copper work well because these two metals corrode rapidly when exposed to water. Once their metal ions have dissolved in solution, they quickly react with the chemical indicator in the paper. Iron and nickel, unfortunately, do not react so quickly. According to the instructions for the test papers used in this procedure, an acid should be used to force the metals to corrode. But electrolysis can also be used, to eliminate the need to handle acids.

Electrolysis

Electrolysis occurs when an electric current forces a chemical reaction to occur. Electrolysis takes place in an electrolytic cell, which is composed of two metal electrodes immersed in an electrolyte (a salt dissolved in a solvent, usually water). In this procedure, the electrolyte is the salt solution used to wet the test paper. The two electrodes are the metal object being tested and the metal tweezers. Both electrodes must contact the electrolyte to complete the circuit; that is why it is essential that the test paper between the metal object and the tweezers be completely wet.

When the circuit is completed, the battery connected to the electrodes causes an electric current to flow. Electric current in the external circuit is carried by electrons, and in the electrolyte (in the wet paper), it is carried by ions from the salt in the saturated salt solution. A reaction must occur at the interface between each electrode and the electrolyte to supply or consume the electrons.

When the positive electrode is an iron object, electrolysis causes the iron (Fe) to corrode to form Fe^{2+} ions, releasing electrons:

$$Fe \rightarrow Fe^{2+} + 2e^{-}$$

At the negative electrode (the tweezers), electrons are consumed in some other reaction, such as

$$O_2 + 2\ H_2O + 4\ e^{-} \rightarrow 4\ OH^{-}$$

The reagent in the test paper then reacts with the iron ions in solution to produce a colour change.

Colour-change reactions

The manufacturer does not specify the reagent used in the nickel test paper. The iron test paper is based on dipyridyl, also known as 2,2'-bipyridine, $C_6H_8N_2$. Each bipyridine molecule contains two nitrogen atoms. The nitrogen atoms in three molecules bind to an iron ion to form a molecular ion $Fe(C_6H_8N_2)_3^{2+}$ (Figure 18), which produces an intense red colour.

Figure 18: Iron bipyridine ion, $Fe(C_6H_8N_2)_3^{2+}$, the red-coloured ion formed in the iron spot test.

Acknowledgements

Special thanks to Avital Lang, Ute Werner and Lucy ‘t Hart, former CCI interns, for their help in developing this CCI Note.

Suppliers

Note: The following information is provided only to assist the reader. Inclusion of a company in this list does not in any way imply endorsement by the Canadian Conservation Institute.

Macherey-Nagel test papers

The commercial test papers for iron (90725, Dipyridyl) and nickel (90730, Nickel) are made in Germany by Macherey-Nagel. (Note: Macherey-Nagel also makes another iron test paper called “Iron Test Paper” which is not suitable for this electrolysis test.) Each box of test papers contains 200 sheets with dimensions 20 x 70 mm. Distributors for these test papers include Aldert Chemicals in Canada and CTL Scientific Supply in the USA.

- Macherey-Nagel
- Aldert Chemicals
- CTL Scientific Supply

Iron and nickel

Thin sheets of pure iron and nickel to use as reference materials are available from chemical suppliers, such as Sigma-Aldrich.

Hardware for homemade electrolysis cables

The 3 mm hardware, 9-volt battery snap, alligator clips, wire, and resistors can be purchased from electronic stores.

Stainless steel tweezers

These can be purchased from many sources. The ones used here were purchased from Canemco – Marivac.

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