Silicon from Sand

A School Science Report by Prof Bunsen Science

INDEMNITY

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1. SYNOPSIS

The element silicon is at the centre of the computer age as most microchips contain the semi-conductor, silicon. It is the 2nd most abundant element in the earth's crust and can easily be extracted from sand in the school science laboratory using two chemicals.

2. SCIENTIFIC INFORMATION

(from Wikipedia)

Silicon is the chemical element in the periodic table that has the symbol Si and atomic number 14. A tetravalent metalloid, silicon is less reactive than its chemical analog carbon. It is the second most abundant element in the Earth's crust, making up 25.7% of it by mass. It does not occur free in nature. It mainly occurs in minerals consisting of pure silicon dioxide (SiO_2) in different crystalline forms (quartz, chalcedony, opal) and as silicates. These minerals occur in clay, sand and various types of rock like granite and sandstone. Sand is basically tiny crystals of silica (silicon dioxide).

Silicon is the principal component of most semiconductor devices and, in the form of silica and silicates, in glass, cement, and ceramics. It is also a component of silicones (breast implants, hardware sealant, etc.) a name for various plastic substances often confused with silicon itself.

Silicon is widely used in semiconductors devices as it remains a semiconductor at higher temperatures. Because silicon is an important element in semiconductor and high-tech devices, the high-tech region of Silicon Valley, California, is named after this element.

Crystalline Silicon 99% pure

Characteristics

In its crystalline form, silicon has a dark gray color and a metallic luster (see photo above). Even though it is a relatively inert element, silicon still reacts with halogens and dilute alkalis, but most acids do not affect it.

Occurrence

Measured by weight, silicon makes up 25.7% of the Earth's crust and is the second most abundant element on Earth, after oxygen. Pure silicon crystals are rarely found in nature; natural silicon is usually found in the form of silicon dioxide (also known as silica) and silicate.

It is estimated to be the seventh most plentiful element in the universe.

Production
Silicon is commercially prepared by the reaction of high-purity silica with wood, charcoal and coal in an electric arc furnace using carbon electrodes. At temperatures over 1900 °C, the carbon reduces the silica to silicon according to the chemical equation
$SiO_2 + C \rightarrow Si + CO_2$
Liquid silicon collects in the bottom of the furnace, and is then drained and cooled. The silicon produced via this process is called *metallurgical grade silicon* and is at least 99% pure.

3. Silicon extraction in the school laboratory

Crystalline silicon can be prepared in a school laboratory using a version of the thermite reaction in which high temperatures are achieved in the reaction of aluminium and sulphur. The method was first described by Kuhn in 1902.

Required
Chemicals
Fine, dry aluminium powder
Powdered sulphur
Dry sand (beach sand or washed builder's sand works well)
Small clay flower pot
Fine magnesium powder
Magnesium ribbon (5cm)
Hydrochloric acid — *Dilute 200ml conc. HCl to 500ml*
Sodium hydroxide solution — *Dissolve 50g NaOH in 250ml water*

Aluminium, sulphur, sand and the clay pot

Other
Mortar & pestle
Clay flower pot
Glass beaker & plastic tub
Electronic balance 100g ± 0.1g
Test tube, bunsen burner
Wires with croc clips (2), ammeter, battery pack, lamp holder with 3.2V lamp
Spatula
Heat resistant pad (1m x 1m)

Transparent safety shield or fume cupboard
Heat-protective gloves
Spatula & metal tongs
Gas lighter with long stem
Hammer

Safety

- The near-impossibility of smothering and high temperatures generated make thermite reactions potentially hazardous. Appropriate precautions must be taken before thermite is ignited.
- The reaction should be performed in a fume cupboard or outdoors behind a safety shield as it produces intense heat and molten metal;
- A large quantity of smoke is produced as well as sparks flying up to 2m horizontally;
- Keep flammable material away from the area;
- A fire extinguisher should be readily available at all times. Water should not be used to extinguish the reaction as explosive hydrogen gas may form;
- Use heat-protective gloves and metal tongs to handle the fragments of the pot after the reaction has taken place;
- Wear protective clothing and safety glasses.

Disposal

Allow the solids produced to cool to room temperature. The clay flower pot invariably cracks and should not be re-used.

Method

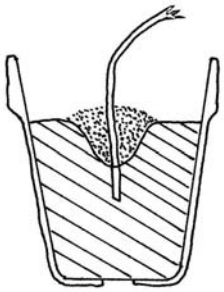
Dry a small quantity of sand in an oven for 3 hours.
Grind the sand to a powdered form in a mortar and pestle.

Mix the following to form an intimate mix (may be pre-weighed for students):

8g of dry aluminium powder
10g of powdered sulphur and
7g of dry powdered sand

The best mix is obtained by shaking the mixture for a minute in a sealed plastic container:

Transfer all of the mix to a small clay flower pot. Make a small indentation at the top and fill this with magnesium powder to facilitate ignition. Use a frayed magnesium ribbon as fuse.



Position the pot on a heat resistant pad in a fume cupboard or outdoors away from any combustible material. Use a safety shield. Wear protective eye wear, gloves and a lab coat.

Light the frayed end of the magnesium ribbon using a long stemmed gas lighter. It may take a few seconds before the ribbon starts burning. Once this happens – step back immediately. *The magnesium flame is a brilliant white (same as in fireworks).* Ignition of the mixture is not instantaneous and might take as long as 60 seconds. Do not approach the reaction vessel until you are sure no ignition is possible.

If the reaction mixture fails to ignite, replace the ribbon or add more magnesium powder. Try again.

The reaction releases large quantities of smoke and sparks fall over a wide area.

The chemical equations are

$$2Al + 3S \rightarrow Al_2S_3 + heat$$

and

$$3SiO_2 + 4Al \rightarrow 2Al_2O_3 + 3Si$$

The residue will have an orange-red glow and be hot for some time. Do not touch!!

Purification of the silicon

Leave the clay pot to cool for 15 minutes. Break the pot apart and separate the silicon / residue from the clay pieces.

Break the residue into smaller pieces using light taps from a hammer. The silicon is now separated from the aluminium sulphide and aluminium oxide by adding the residue to diluted hydrochloric acid in a glass beaker (acid - take care!). Copious amounts of hydrogen sulphide will be evolved – foul smelling gas - use a fume cupboard:

$$Al_2S_3 + 6HCl \rightarrow 2AlCl_3 + 3H_2S$$

Leave the residue in the acid until gas formation subsides. This process may take up to 20 minutes. The finer the residue, the faster this reaction.

Now, carefully do away with the acid under running water and wash the residue in the beaker for 30 seconds. The silicon will be found in the form of hard, black, crystalline globules as large as peas. It is insoluble in acid.

Yield calculations
Students now weigh the silicon residue and determine the yield:

Eg. Yield = 1.1g silicon / 7g sand x 100
= 15.7%

One or more globules can be boiled in a glass beaker using dilute hydrochloric acid for further purification.

Proving that the globules are silicon

Students can use any or both of the following methods to prove the existence of silicon:

A. Silicon's reaction with caustic soda

Silicon is insoluble in common acids but will react with alkalis liberating hydrogen gas. This provides a test that the substance is silicon. Place a cleaned small silicon globule in a test tube, add concentrated sodium hydroxide solution (caustic soda) and heat gently for 1 – 2 minutes. The characteristic match flame 'pop' at the mouth of the test tube will confirm the presence of hydrogen.

$Si + 2NaOH + H_2O \rightarrow Na_2SiO_3 + 2H_2$

$2H_2 + O_2 \rightarrow 2H_2O + E$

B. Demonstrating the reverse temperature effect

Clamp one cleaned globule of silicon using two crocodile clip wires. Check that the clips do not make direct contact.

Connect the following items in series to the two wires:
One battery pack: 2 x AA (3.0V)
One 3.2 V lamp
One ammeter (multimeter)

The lamp lights up. Record the ammeter reading.

Now heat the silicon with a bunsen burner. The lamp brightens and the ammeter reading increases.

Optional: Exchange the silicon with an iron nail and repeat the process. The opposite results should be detected.
Metals are conductors and silicon is a semi-conductor. Semi-conductors have a negative resistance-temperature coefficient since the number of free charge carriers increases with temperature.