

Quick assays in mineral identification

A guide to experiments for mineral collectors and geoscientists in field work

by

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I General part

Introduction

A quick assay is a qualitative chemical analysis; it is a test to prove the presence or absence of a chemical element in the assayed matter. Normally, the test substance is heated by a small hot flame to temperatures in some cases as high as 1200° C. Sometimes the finely ground material is mixed with certain chemicals in order to obtain a melt. This procedure aims to produce a colour of the melt, or a sublimate, or a metallic regulus which is specific to a chemical element. Sometimes the produced melt is dissolved and certain chemicals are added to evoke a coloration which is specific for the element in question. Such assays are done on a semimicro scale, only 0.1 to 20 mg of the tested matter are normally used.

Most of these methods were developed by Swedish scientists (Cronstedt, Bergman, Gahn, Berzelius) in the 18th century when chemistry was developing into a modern natural science. Many reactions reproduce on a very small scale processes used in metallurgical work and ore processing since ancient times. Considerable improvements were achieved by Harkort, von Kobell, Brush, Bunsen, and Plattner in the 19th century, especially by the addition of wet chemical reactions. The entire combination of different methods was called blowpipe analysis, since a blowpipe was used to produce the hot flame in almost all the tests.

Approximately one hundred years ago it took well-trained geologists or chemists from several minutes to 2 hours to complete a qualitative analysis of a mineral or a metallurgical slack using this method. Even a quantitative determination of gold and silver was possible with a precision comparable to modern spectrographic methods. Significant improvements in the quantitative analysis of gold and silver had already been introduced by Sir Isaac Newton at the beginning of the 18th century, during his appointment as Master of the Mint in Great Britain. Supplementary spot tests were triggered by the rapid development of organic chemistry early in the 20th century.

Most of these methods were the required subject of university courses in chemistry, mineralogy and geology till approximately 1960. Since then in research institutes blowpipe analysis has have been progressively replaced by physical methods such as wavelength dispersive X-ray fluorescence spectrometry and by the combination of scanning electron microscopy (SEM) with energy dispersive X-ray fluorescence spectrometry (EDAX). Somewhat later the inductive coupled plasma torch (ICP) was applied to emission spectroscopy. All these analytical methods yield quantitative analytical results simultaneously for many elements. No wonder that the teaching of the old-fashioned blowpipe methods vanished almost completely from university curricula parallel to these developments.

Moreover, minerals have been identified exclusively by X-ray diffraction for the past fifty years. The file of d-values together with relative intensities achieved by this method permits the unambiguous identification of a mineral. Already the d-values contain information concerning the geometrical data of the unit cell; data concerning the symmetry of the crystal are provided by the systematic absence of some d-values. Thus it is not necessary to record the relative intensities exactly. The Debye-Scherrer method requires very tiny amounts of the mineral powder; for the X-ray powder diffractometer generally used today, only skilful users manage with amounts of one cubic millimeter and less, if necessary.

That is why the blowpipe method is indeed an odd, obsolete technique in a modern and fully equipped research institute. Unfortunately not everyone who is interested in qualitative inorganic analysis and mineral identification has access to the facilities of such research institutes. There is a simple reason for this: the price for each item of such modern equipment ranges from at least €60,000 to many times that amount. In addition, approximately 3 to 5% p. a. have to be invested for maintenance, expendable material and electric power. Reliable results are only achieved by well trained staff. Commercial institutes charge approximately € 30 or more per analysis or X-ray diffraction analysis. Moreover, such spectrometers are stationary instruments, which cannot be moved into the field. This situation is just starting to change. Russian scientists had constructed portable energy dispersive X-ray spectrometers for unmanned exploratory missions to the moon and Mars. They were equipped with a radioactive element as the source of radiation and a Peltier cooling of the detector crystal. In the 1990s such devices were further developed for civil use in the U.S.A., especially with respect to radiation safety regulations. At the present time such hand-held, battery operated multi-element spectrometers are on sale, but they are also expensive, with prices starting at € 35,000. For physical reasons they can be used only for elements with an atomic number of 18 or higher, due to the high absorption of long wave X-rays by air.

So who might still be interested in using an old-fashioned, but quick and cheap technique in qualitative chemical analysis? Perhaps four groups of people:

- Geoscientists at an early stage of exploration of minerals in the field, especially in remote environments.
- Mineral collectors who are interested in the history of mineralogy.
- People who are interested in chemistry and its history as a hobby, as well as teachers.
- Possibly, geoscientists working in scantily equipped institutes, services, and companies.

All these people are likely to have at least a basic knowledge of inorganic chemistry and mineralogy.

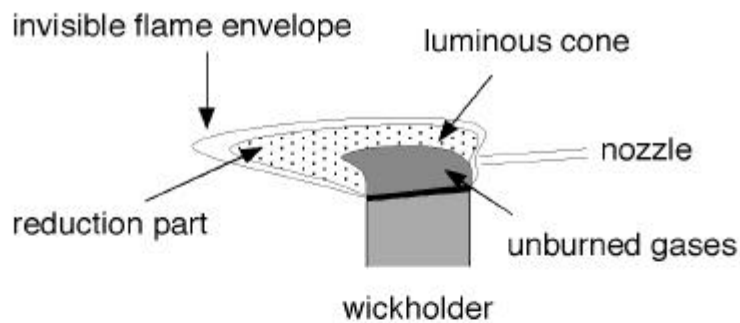
In most cases the aim of the analysis is to gain further information about a mineral which has not yet been unambiguously identified by the usual methods. First its properties should be recorded: hardness, streak, colour, lustre, cleavage, magnetic properties, and morphology. About 4200 mineral species are acknowledged by the International Mineralogical Association (IMA), but only 60 to 200 species can be determined by ocular inspection and the above-mentioned methods, depending on the experience of the geologist or collector. A qualitative analysis gives information about the main constituents of the mineral. Let us take an example. There are more than 400 phosphates registered as minerals. Imagine you have found a mineral, black to brown in colour, with a hardness of ≈ 5 , and the analysis shows the presence of P, Li, Fe, and Mn as the main constituents. By simply looking at a good textbook on mineralogy you will find out that this specimen is most probably sicklerite $\text{Li}(\text{Mn,Fe})\text{PO}_4$ or a member of the series triphylite LiFePO_4 - lithiophylite LiMnPO_4 .

Information about the qualitative composition of a mineral is also of great value when interpreting an X-ray powder diffraction pattern of this mineral. This holds true even in the days of modern electronic data processing with advanced and sophisticated programs; many

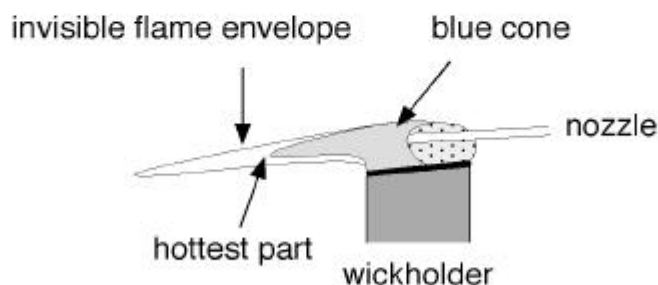
minerals are mixed crystals and that means the recorded d-values of a mineral may vary considerably depending on the composition of the crystal.

The heat source

Today the heat source exclusively used is a butane torch, but the traditional heat source was a small hot flame achieved by blowing a jet of air from a mouth-operated blowpipe through the luminous flame of a lamp with a rectangular wick, filled with oil or paraffin. A reducing flame was produced by placing the nozzle of the blowpipe just outside the flame, while an oxidizing flame was obtained by placing the nozzle approximately one-third within the flame. Wick and wickholder were cut at an angle of 15° to the horizontal direction, so, depending on the direction of the blowpipe, a flame directed at this angle downward or upward could be produced by blowing parallel to the wick's surface, figure 1. - The first written mention of such a device dates back to 1670, but it can be taken for granted that the basic technique is much older. The use of bellows in ore melting was already mentioned in the Bible (Jeremiah 6, 29 and Ezekiel 22, 20-21).



The reducing flame



The oxidizing flame

Figure 1: blow pipe flame: position of nozzle and paraffine flame

The preferred type of blowpipe for more than 100 years was invented by Gahn and first described by Berzelius in 1812. The mouthpiece was shaped like that of a trumpet and made

from wood or horn. It was connected to a conical brass tube approx. 180 mm long which was fixed to a cylinder of approx. 30 mm length and 12 mm diameter in order to collect the condensed water. A small tube about 60 mm long, ending in a 0.4-0.6 mm diameter nozzle, was plugged into a radial borehole of this cylinder, figure 2. Good nozzles were made from platinum; later on, nickel alloys or stainless steel were used. The air was not pressed by the operator's lungs, but by his cheek muscles. In the 19th century already many people had difficulty doing this properly, as can be guessed from early suggestions for bellow-operated blowpipes.



Fig. 2: blow pipe according to Gahn

Blowpipes of the type described by Gahn and Berzelius are no longer on sale. However, anybody interested in this technique can build a device producing a small jet of air by connecting a throwaway hypodermic needle (0.90 x 70 mm, DIN 13097) with a battery operated air pump by means of plastic tubing with 4 mm outer diameter and 1 mm wall thickness. Such membrane pumps are sold to aerate aquariums. In order to fix the needle at the desired height and inclination it is clamped by a flat magnet to a 30 x 400 mm iron sheet bent into a rectangle in the middle. The horizontal part of the metal sheet can be held in position by a paperweight or a stone, figure 3.

To produce the luminous flame you need a lidded tin box, preferably a spherical one, 20 to 35 mm in diameter. A 10 mm x 4 mm wickholder can be cut and folded from the thin metal of a beer or cola can. It should be fixed by soldering or clamping by a collet next to the inner side of the cylinder at the upper end. Remember the inclination of the upper end of the wickholder described above. Rectangular wicks are sold as spare parts for petroleum lamps, but several thick round wicks in a parallel position can be used instead. The device is filled with paraffin wax from the remains of old candles. Before use, the surface of the paraffin should be melted by means of a lighter. You can also use the luminous flame of a Bunsen burner instead of the paraffin lamp. Butane-operated Bunsen burners are available for nonstationary use.

Fortunately today we can work without a blowpipe at all, since there are various small butane gas burners on the market since the 80's of the 20th century. Some of them have an air-regulating screw as well as a valve to regulate the gas flow, figure 4. Items with a stable stand and piezo ignition should be preferred. Such butane torches are designed for use in soldering and welding; all constructions produce a hot oxidizing flame up to 1300° C, but only some devices can also produce a reducing flame. The oxidizing flame should have a minimum length of 12 mm and a maximum length of 40 mm; if the flame is too long the sample tends to be blown away. - Some cheap butane torches are designed for short use only. The device must be extinguished after 3 to 4 minutes use and any further use is only possible after the nozzle has cooled to room temperature. The nozzle is made of stainless steel and ceramic, but most other parts are plastic and may be deformed due to heating by radiation. This short time is not really a disadvantage because most tests are completed in less than three minutes.

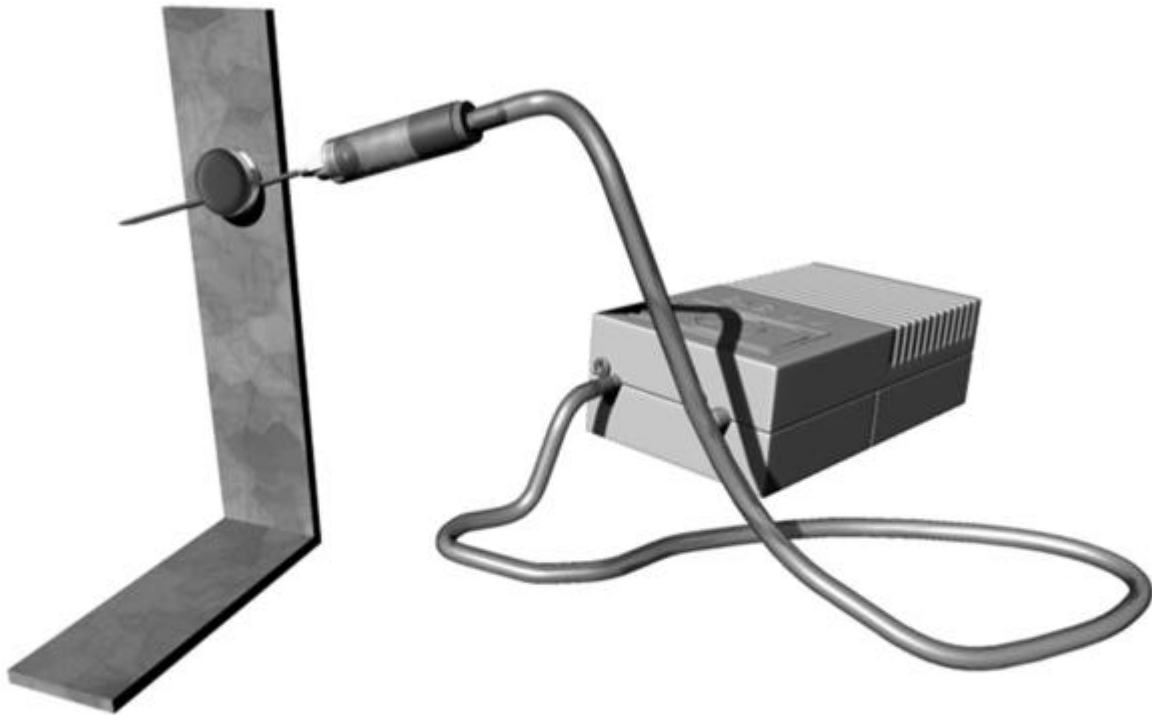


Fig. 3: substitute for a blowpipe: arrangement of a hypodermic needle connected to an air pump via plastic tubing

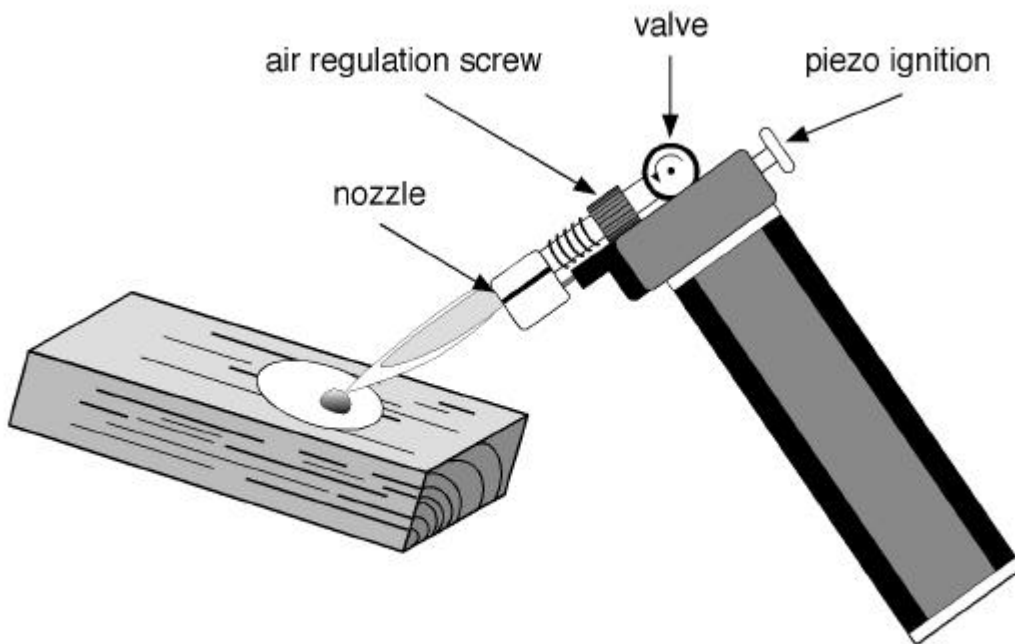


Figure 4: Heating on charcoal with a butane torch

International air traffic regulations strictly prohibit the transport of inflammable compressed gas or fluids on all aircraft, only pocket lighters are an exception. This should be taken into account when field use is planned. Nevertheless it is possible to transport empty burners; butane refill containers are available in most cities of the world.

The base and the vessels

The small flame has a low heat capacity; this does not matter since the mass of the analysed matter and the chemicals is also small. But it implies that any vessel holding the melt in its place must also have a low heat capacity; thus the vessel must be very small. Another possibility is to place the melt on a base with a very low heat conductivity, such as charcoal which has been used since the very beginning of this technique. Traditionally, 110 x 35 x 12 mm charcoal blocks were used and are still on sale. But you may cut your material using a fine saw from any dense charcoal sold for barbecues. The kinds made from basswood, pine, or willow are recommended. Used pieces can be recycled by grinding off the contaminated surface with a flat file or with abrasive paper. To hold the melt in place, a small cavity should be made with a penknife or a small coin. - Charcoal is a porous substance, therefore some melts are soaked up by the coal. This is the case for most alkali salts, only alkali phosphates, borates and silicates produce a bead on the surface. Any fusion on charcoal implies at least a weak reducing environment.

For these reasons other vessels must be used for some melts. Originally platinum was preferred, as small spoons of 12 mm diameter, small crucibles and as wire. The free end of an approx. 0.3 mm thick platinum wire is coiled into a small U-shaped loop through which an ordinary match will barely pass. The other end is melted into a small glass tube. Due to surface tension the melt to be handled adheres to the wire as a bead. For further treatment it can be shaken off from the wire by a sudden jerk. To do this you have to heat the bead as highly as possible holding the wire tight in your fist, then bang your wrist on the table. With some skill it is possible to catch the falling bead in a dish, or - more easily - on a triple layer of aluminium foil. For some fusion reactions this technique is still important today. Remaining melt on the wire can be removed by melting some potassium hydrogen sulphate KHSO_4 to the wire loop. Any remains of this sulphate bead can be dissolved by boiling with dilute hydrochloric acid HCl , then rinsing with distilled water.

Unfortunately platinum is heavily corroded at high temperatures by all sulphides, mixtures of chlorides and nitrates, alkali hydroxides, and under reducing conditions by any matter containing arsenic, antimony, lead, copper, bismuth, cadmium, nickel, or silver. Under reducing conditions free quartz reacts at 1000°C to SiO , which decomposes to $\text{Si} + \text{SiO}_2$. The Si reacts with Pt to a eutectic melt with a melting point of 830°C ; this melt causes severe grain boundary corrosion. Any heating of phosphorous acid in platinum vessels should be avoided for similar reasons. - Moreover platinum is a precious metal and additionally the cost of forming wires and vessels is usually as high as that of the material itself. (A 10 cm long piece of 0.3 mm Pt wire costs about €32.)

Hence magnesia rods with 2 mm diameter and 120 mm length were introduced in the early 20th century as a substitute for platinum wire. Originally they were made from magnesium oxide. They are still on sale, but nowadays such magnesia rods consist of a ceramic matrix of magnesium aluminium spinel MgAl_2O_4 and indialithe $\text{Mg}_2\text{Al}_4\text{Si}_5\text{O}_{18}$, which is the high temperature polymorph of cordierite. Such rods are used like a platinum wire for bead melting; after use the contaminated end is broken off and discarded. The advantage is the

possibility of reducing the melt, but the melt cannot be shaken off since the rods are brittle. "Magnesia" furrows made from the same ceramic matrix are also on sale, they are used especially for oxidizing melts. Remember that any melt formed on such magnesia devices may be contaminated with Mg, Al and Si. Normally this does not matter, but in rare cases these ions may disturb an intended test in solutions of the dissolved melt.

Small porcelain dishes and spot plates are recommended in the literature for melts and spot tests. Commercial spot plates are made from glazed porcelain and contain 6 to 12 flat depressions of equal size that hold 0.5 to 1 ml of liquid. Some authors recommend the melting of the analyzed matter with fusion mixtures in the flat depression of a spot plate, but at temperatures higher than 300° C the plate often cracks due to heat induced strain. Porcelain is made from a wet plastic mixture of kaolinite with a fine powder of quartz and potash feldspar. After the mass has been shaped to the desired form it is dried at normal temperature and then fired to temperatures of 1100° to 1200° C. Porcelain raw material is difficult to obtain, but you can also use normal potter's clay which is sold in hobby shops. Such clays are a wet plastic mixture of montmorillonite, sometimes halloysite with quartz and feldspar. On firing to high temperatures it is converted to a ceramic matrix consisting of mullite, quartz, glass, and residues of feldspar.

Small dishes of fired potter's clay were already used in the 19th century for the roasting of sulphides and in tests for gold and silver. At that time they were formed in a wooden mould impregnated with oil. Today it is easier to make them in another way. Take a piece of potter's clay with a consistency like plasticine and form a sphere the size of a small pea. Now press it on the surface of a ping-pong ball. By repeated pressing and flattening with the thumb form a small dish till the wall thickness is approximately 1 mm. The somewhat irregular rim of the clay layer does not matter for later use. A perfect spherical rim can be obtained by pressing a tube with 16 to 20 mm inner diameter against the freshly formed clay layer. Since the clay shrinks on drying, it falls off the ping-pong ball's surface after some hours of drying at normal temperature. Drying at normal temperature should be continued for at least two days before firing the clay to 1000 to 1100° C. Heating in an electric furnace is preferable, but a charcoal fire can also be used. In the latter case any adhering ash or coal should be removed by brushing and finally by glowing with an oxidizing flame.

Clay furrows may be formed on the surface of a plastic pill tube; the outer diameter of the tube should be 18 to 25 mm. The rims may be cut by a knife. In a similar way it is possible to produce small streak plates if you press finely powdered quartz to the surface during formation on a flat surface. Streaks can be dissolved on such plates in situ by appropriate melts. The fired material does not show a pure white colour like porcelain, but is white with a yellow or gray tinge, because potter's clay normally contains traces of iron compounds.

For use with solutions it is appropriate to glaze the dishes. This may be done in the following way. Mix 1 ml of waterglass thoroughly with a pinch of white toothpaste. Spread this mixture in a very thin film over the surface of the already fired dish by means of a fine brush. The glaze is formed by firing the dish again at 1000°C. Unglazed dishes made from fired potter's clay are slightly porous, they should be discarded after use, since they may be contaminated with remaining melt or solution.

You may even use small thin shards of porcelain from saucers or tea cups for melt reactions. Please test such shards before use by heating to the desired temperature in order to avoid cracking by thermal strain during the experiment. All melts produced on a ceramic matrix may contain elements of that matrix such as Al, Si, Mg, K, Ca, and Na; some glazes

may additionally contain Ti, B, Sn or F! A blank test should be done if the assay is intended to test for one of these elements; i.e. the experiment is done without adding the material to be tested.

There are three ways to hold the ceramic dish in place during the heating process:

- The dish is held by stainless steel forceps.
- An iron wire is coiled to a loop of appropriate size and fixed to a wooden handle.
- The ceramic dish is placed on a flat base with very low thermal conductivity such as charcoal, pumice, glass wool or ceramic wool. The glass wool may be placed in a flat tin box with a lid to cover it after use.

Small test tubes are used for dry heating (7x70 mm), bigger ones for the handling of solutions. A wooden clothes-peg (spring type) may be used to hold the tube. A spirit lamp should be preferred for heating. For temperatures up to 100° C bumping is avoided by using a waterbath. Use a small tin two-thirds full of tap water and add some sand grains. This waterbath can be placed on a tripod made from thick iron wire (figure 5).

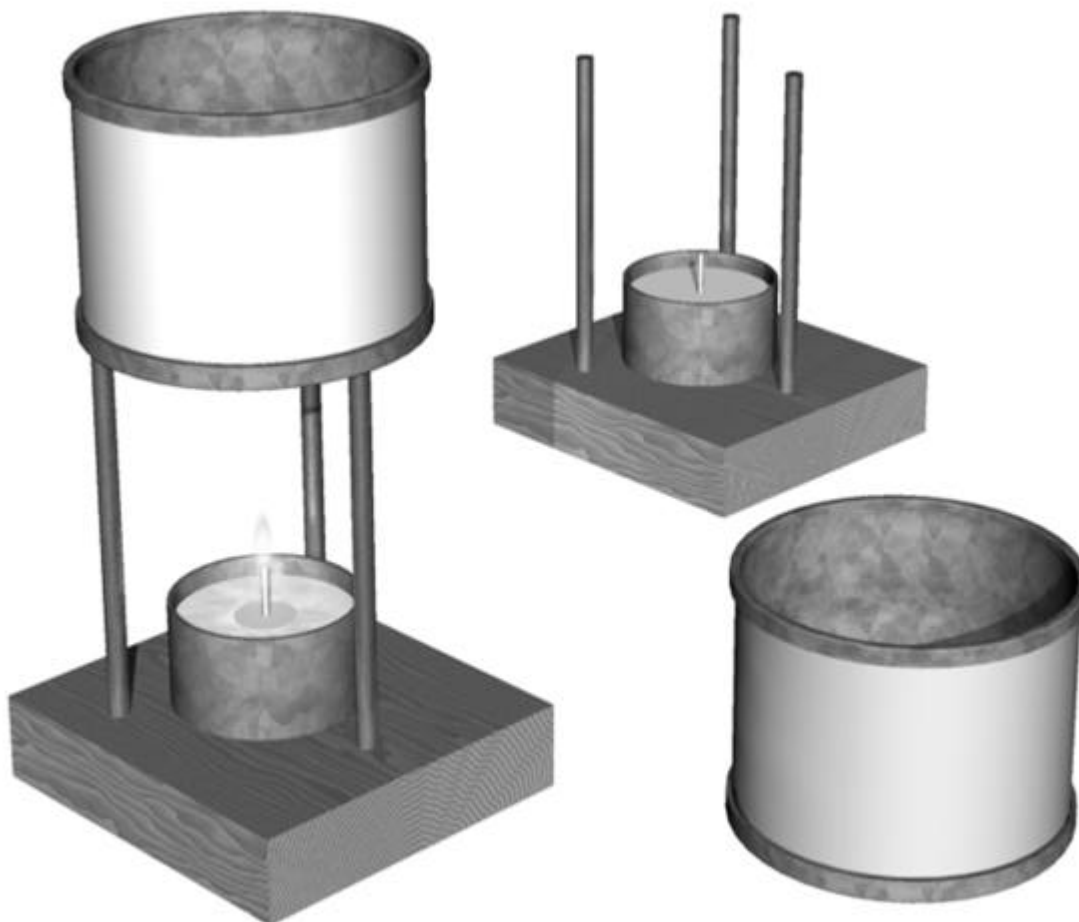


Fig. 5: dismantable water bath

A small funnel and two or three 50 or 25 ml beakers are sometimes needed for preliminary work for spot tests. If field use is intended, the funnel and one beaker should be made of

polyethylene. Glass for laboratory use such as the *Pyrex* or *Duran* brands are recommended for the test tubes and beakers. Some watch-glasses fitting the beakers are needed as well as microscope slides, small glass rods and cover glasses. Never try to heat watch-glasses by means of a flame; however they can be heated on a water bath.

Glass tubes of 140 mm length, with an internal diameter of 4 mm and 6 to 9 mm are used for some tests. Glass tubes are cut in the following way. A tiny notch is filed into one side of the glass tube, which is then half pulled, half broken in two. Put your thumbs on both sides on the opposite side of the notch, it is safer to wrap the glass tube in a towel before pulling!

Sample preparation

In sample preparation it is of the utmost importance to ensure the purity and homogeneous character of the mineral in question. Since, in most cases, only very little material is required for the tests, careful selection can generally secure enough in a pure condition. In rare cases you may even use a mineral intergrown with another (but well-known) mineral, like quartz or calcite.

With a few exceptions the mineral must be ground to a fine powder. As a rule the powder should feel like flour when rubbed between your fingertips. Most textbooks recommend the first grinding in a steel mortar, followed by a fine grinding in a small agate mortar of approx. 25 to 60 mm diameter. The agate mortar and pestle are used for grinding only, never for pounding hard bodies! Agate mortars should be cleaned by the repeated grinding of fine quartz powder. Agate is a slightly porous matter, avoid wetting it with any solutions, since this may cause contamination in subsequent use. - Mineral fragments may be powdered by wrapping in several folders of thick paper or plastic foil, and hammering on a flat piece of steel, in this way the use of a steel mortar can be avoided.

But very often you can obtain enough material by using a file. Diamond files are superior to all other devices, but they are expensive (approx. € 17 each). A so-called sapphire file, originally intended for nail care, and available from pharmacies or drugstores will do the same job. After use the file must be cleaned with soap and water, using an old tooth brush. In most cases the powder obtained by the file can be used for the test, but sometimes it is better to grind it to a finer state in the agate mortar. If no agate mortar is available, you can use a porcelain mortar instead for all minerals with a hardness lower than 6.5. Since the mortar has to be cleaned after use as well, a porcelain mortar is somewhat inconvenient, because its surface is very rough.

Another simple possibility is to obtain a fine mineral powder by rubbing the mineral piece on carborundum paper designed for wet grinding and polishing, the grinding paper should be discarded after use. - Minerals with a hardness of less than 5 on the Mohs scale may be rubbed on a clean streak plate; rubbing an area of 1 square centimetre usually yields enough matter in a dispersed state for a test. In the case of acid soluble minerals they can be dissolved on the streak plate in situ. Again the streak plate has to be cleaned with water, soap and a brush afterwards. Remaining residues can be dissolved by melting a mixture of ammonium chloride and ammonium nitrate on the streak plate which is placed on charcoal or held by forceps. For such purposes it is recommended to cut a streak plate in 10 to 20 mm wide strips by means of a diamond saw or a grinder.

The powdered sample is transferred by means of a fine watercolour brush to a glazed paper which has previously been sharply folded and flattened again. Rough and fine grains can be

separated by gently knocking against the paper from one side. The fine powder is now transferred to an aluminium foil. Any necessary mixing with dry chemicals can be done on the aluminium foil. The use of toothpicks is recommended for this purpose. Never use matches, since they are usually impregnated with chemicals. A watchglass or a microscopic slide should be used for wet mixing or mixing with potassium or ammonium hydrogen sulphate. – It is not necessary to buy glazed paper, it can be cut from advertising leaflets or the cover page of any magazine.

Beginners tend to use too much substance. Enough powder to cover a small knife-tip amounts to approximately 50 mm^3 , this applies to 20 to 60 mg, depending on the density of the mineral and the grain size of the powder. Such an amount is called a pinch in this paper. A small pinhead has a volume of approximately 1 mm^3 , such a volume of a powdered mineral weighs 0.4 to 1.2 mg.

The reagents

The good news first: for the most important tests you need only a few chemicals and normally only a very small amount of each. Generally minute amounts, a pinch or a knife-pointful, are used for a test. Thus a sufficient supply may be stored in approx. 5 ml volume plastic tubes with a tight-fitting hinged lid. All chemicals should be carefully labelled!

The bad news:

- If you want to do all possible spot tests, you have to keep more than 50 different chemicals in store.
- Although you will use very small amounts of any chemical, suppliers sell only large packages, much bigger than you need!

The simplest way to overcome these difficulties is to have a friend who works at a chemical laboratory, preferably at an university laboratory. Some of the chemicals are sold in small quantities by pharmacies. Another way is to place a joint order together with other interested collectors. Some hints about suppliers are given in part II.

Solid reagents.

Sodium carbonate, Na_2CO_3 (soda); $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ (natron) is also available.-

Dry sodium carbonate should be preferred. It melts at 854°C . The melt decomposes most chemical compounds, thus producing the sodium salts of the anions, the cations are transferred to carbonates, oxides or other compounds. NaHCO_3 , which is sold as baking soda at pharmacies, may be used as well.

Sodium tetraborate, $\text{Na}_2\text{B}_4\text{O}_7$; $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$ (borax) is also available.-

The dry reagent melts at 742°C . The melt cools to a clear glass. Sodium tetraborate melt dissolves various substances, especially metal oxides, in many cases yielding characteristic colours.

Ammonium sodium hydrogen phosphate, $\text{NH}_4\text{NaHPO}_4 \cdot 4\text{H}_2\text{O}$, is sometimes called phosphorous salt or *microcosmic salt*.-

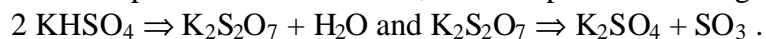
On heating, this salt changes to sodium metaphosphate NaPO_3 by losing water and NH_3 . If available NaPO_3 should be preferred. The melting point is 628°C , again a clear glass is obtained. Many oxides give a distinct colour to the glass.

The origin of the name *microcosmic salt*.

Beads of sodium metaphosphate saturated with some oxides cool to a clear glass. Small crystals grow in the glass on repeated heating and cooling. A microscope with a 80 to 100 x magnification is needed to inspect these crystals with various morphologies. Such beads resemble a microcosmos. Some authors tried to use the effect for analytical purposes in the 19th century, but these efforts failed. Borax beads sometimes show the same effect.

Potassium hydrogen sulphate KHSO_4 –

This compound melts at 210° C, it decomposes according to:



A variety of minerals are decomposed by fusion with the reagent. Such fusions may be made either in a platinum wire loop, on a magnesia rod, in porcelain, on glazed clay dishes, or in a test tube.

Ammonium hydrogen sulphate, NH_4HSO_4 –

Melting point 147° C. This compound or its concentrated solution can be used instead of concentrated sulphuric acid in some cases.

Potassium nitrate, KNO_3 (nitre) –

It is used only in a mixture with three parts of sodium carbonate in sensitive reactions for chromium and manganese.

Tin(II) chloride, $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ –

It is used in reductions.

Potassium oxalate, $\text{K}_2(\text{COO})_2$ –

Is used for reducing melts, mostly together with sodium carbonate.

Tin, Sn / zinc, Zn –

These metals are used for reductions. The granulated metals are preferred, thin foil is useful, too. Don't use soldering tin, since this is an alloy!

Magnesium, Mg –

Magnesium ribbon is useful for detecting phosphoric acid in minerals.

A mixture of one part ammonium chloride NH_4Cl and two and a half parts of ammonium nitrate NH_4NO_3 has a melting point of approx. 140° C. It may be used like aqua regia, which is a mixture of concentrated nitric and hydrochloric acid. The solid mixture should be kept dry, any contact to metals or reducing chemicals should be avoided! Melting should be done in porcelain or glass vessels or on a magnesia furrow.

Cobalt nitrate, $\text{Co}(\text{NO}_3)_2$ –

Just before use, the required amount of this salt is dissolved in 10 parts of water. The solution is used to identify some oxides.

Ammonium heptamolybdate, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ –

It is used for the detection of phosphates, arsenates, and silicates.

Liquid reagents

You will need distilled water, which is sold at pharmacies or filling stations. It is convenient to keep a supply of distilled water in a washbottle of the type used in chemical labs.

Hydrochloric acid, HCl –

Concentrated HCl is sold with either 32 or 36 weight%. A 1:1 dilution is generally used.

Nitric acid, HNO₃ –

Concentrated nitric acid has 65 weight%. It is extremely corrosive, keep it away from skin, fabric, metal and paper. Generally a dilution of 1 volume conc. HNO₃ with 2 volumes of water is sufficient.

Sulphuric acid, H₂SO₄ –

A diluted sulphuric acid 30 weight% is sold for lead batteries at filling stations in nearly all countries. This acid is sufficient for nearly all tests. – Some drops of the concentrated acid may be prepared by heating no more than 1 ml of the 30% acid in a test tube, shaking vigorously till white fumes are produced. Use and handle the remaining liquid only after cooling! - In most cases it is possible to use ammonium hydrogen sulphate instead of concentrated sulphuric acid.

The concentrated acids described above should be stored in small glass bottles with ground-glass-stoppers. The diluted acids can be stored in polyethylene bottles with screw tops. Before filling please leak-proof the bottle and the screw; fill with water, screw shut, and squeeze the bottle gently holding the neck of the bottle downward.

Ammonium hydroxide, NH₄OH –

The concentrated solution has 34 weight%, a 1:1 dilution is sufficient.

Methanol, CH₃OH –

It is used for a special boron test, or as a solvent for organic compounds, it is poisonous when swallowed! Denatured ethanol may be used instead.

Hydrogen peroxide, H₂O₂ –

A diluted solution is used in some tests for Ti, Cr, V, or Mn. – You may use a freshly prepared solution of sodium perborate instead. This is a constituent of tablets sold for cleaning dentures. Look for the name sodium perborate on the label showing the ingredients. A common brand is *Kukident*. A tiny part of a tablet dissolved in 1 – 2 ml water is sufficient for each test.

It is advisable to store only a very small amount of all wet chemicals, bottles holding 30 or 50 ml are sufficient! If you need only a few drops, use a dropper to take the amount from the bottle. - A glass tube with a narrow inner diameter (3, 4, or 5 mm) is heated in the middle over a flame, turning it slowly so that the glass will be uniformly heated. When the glass becomes quite soft, the tube is removed from the flame and quickly pulled in two parts. Break off the thus formed capillary in order to get a narrow opening. Soak the liquid by means of a rubber bulb, it is a good auxiliary construction to fix a plastic tubing to the end of the glass dropper and close the end of the plastic tubing by a glass rod (figure 6).

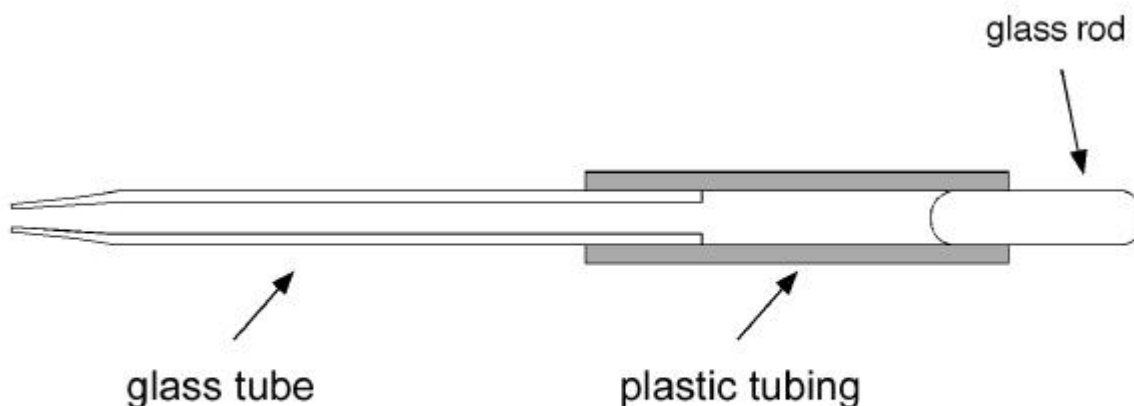


Fig. 6: dropper

For transport of any kind the leakproof plastic bottles should each be wrapped in a plastic bag secured by sellotape. Put this in a tin with a tight lid and fill the free volume at least partly by an absorbent material like silica gel. You can also use the super-absorbent material from a modern diaper like the brand *pampers*. Again the lid of the tin should be fastened with adhesive tape.

The transport of any corrosive liquids, acids and strong alkaline solutions on an aircraft is strictly prohibited by international air traffic regulations. You may overcome this difficulty by using 30% sulphuric acid for lead batteries which is available worldwide and can be bought at your destination.

- On dissolving 4.3g of NaCl in 10 ml of such acid and adding 10 ml of water you get a 15% HCl.
- A solution of 6.1g of ammonium nitrate in 10 ml of sulphuric acid 30% can be used instead of nitric acid.
- If you dissolve 2 g of NH_4Cl and 1.4g of NaOH in 10 ml of distilled water, you may use this solution like ammonium hydroxide.

Weigh these chemicals at home and seal or weld them separately in plastic for transport. Do not forget to label them correctly. Mark a volume of 10 ml by a waterproof pen on a test tube. The additional ions in these solutions must be considered only in the case of a few wet tests. If you only need an acid to detect calcium carbonate, you may use vinegar, or, better a 10% solution of citric acid in water.

Safety hints

All tests rely on chemical reactions, especially fusion processes can produce poisonous gases and fumes. If you are working in a laboratory please use the fume-cupboard. Otherwise you may work with such reactions in the open air or on a balcony. Ensure good ventilation and take care that no other persons are inconvenienced by the fumes. If you want to smell

anything you should take a deep breath beforehand, then stop breathing and take a tiny extra sniff by fanning the air towards your nose, followed by immediate breathing out. There is scarcely any health hazard if you always work with the recommended small amounts of sample and reagent.

Nitrates, nitrites, bromates, iodates, and sodium perborate are oxidizing chemicals, avoid any fusion with reducing chemicals like sulphur, powdered coal, oxalates, thiocyanates, organic compounds, or pure sulphides since this may lead to a rapid combustion or even an explosion.

The use of goggles is strictly recommended.

During the work do not smoke, eat or drink.

Wash your hands carefully after work.

If any drops of acid or other solutions are spilled on skin or fabric, rinse immediately with plenty of water. Traces of acids adhering to fabric can be neutralized with white toothpaste containing calcite.

If you are working with concentrated acids or if you heat strong acid or alkaline solutions the use of protective gloves is by no means a sign of weakness but a prudent decision. The thin rubber gloves designed for medical use are excellent for that purpose; but a thin, transparent plastic bag may be used as a substitute as well.

Great care has to be taken to avoid any delay in the formation of steam bubbles if it is necessary to heat a solution in a test tube! Such sudden steam evolution may sputter considerable amounts of the boiling liquid out of the test tube, this may cause serious burns especially if the solution contains strong acids or bases. The effect is known as "bumping", it is avoided by heating the test tube not at the bottom, but at the side and by gently shaking the test tube in a slanting position during heating. In any case the open end of the tube should never point towards the operator or any other person!

Hot things look the same as cold ones! Before touching any device that has been heated, please control its temperature by its radiation: bring the back of your hand *next* to it, but don't touch it! This part of human skin is very sensitive to infra-red radiation. - Finger protectors for thumb and forefinger may be cut from thick rubber tubing approx. 25 mm in length and slit once in an axial direction. Silicone rubber tubing should be preferred for such purposes, since it stands temperatures up to 200° C.

Never suck any liquids into glass tubes or plastic tubing by means of your mouth!

All solutions thrown into the sink must be well diluted with tap water first, and the sink rinsed with plenty of water.

Never store any chemicals in bottles or vessels designed and used for drinks or food! - Take care that no unauthorized persons, especially no children, have access to chemicals!

To protect your working table cover it with newspaper and a double layer of aluminium foil like that sold for kitchen use.

The classical tests

These are the tests developed and widely used in the 19th century. The mineral powder is used directly, no wet separation process is employed.

Heating in a closed tube

Glass tubes closed at one end, 8 to 10 cm long and 3 to 6 mm internal diameter should be preferred, but it is also possible to use small test tubes. The coarsely powdered assay of 1 to 50 mg of mass is transferred to the bottom of the tube, take care to avoid any fine material adhering to the upper parts of the tube wall. In some cases it is advantageous to use only a few pieces of approx. 1 mm diameter. The tube is heated at the bottom and held by a wooden clamp or clothes-peg at the top in an oblique, nearly horizontal position. If any water condenses in the upper part, put some cotton wool in the upper part to avoid water drops rolling back to the hot part and causing cracks. It is important to increase the temperature slowly.

You may observe the following phenomena:

- a. Change in the condition or appearance of the assay.
 - b. The formation of gases which collect in the tube.
 - c. The formation of sublimates or condensed liquids on the cold walls of the tube.
- a. Change in condition and appearance.

Only materials with a low melting point fuse in the closed tube. Such boiling melt points to minerals of the zeolite group, or to crystal hydrates. They can be distinguished by their hardness, zeolites are not scratched by steel.

Minerals containing liquid inclusions may snap and explode owing to the evolution of steam during heating. Sometimes they break up into very fine powder or dust, e.g. milky quartz. This phenomenon is called *decrepitation*.

Some minerals emit a bright, often coloured light when heated below redness. ($T \approx 300^\circ \text{C}$) The effect can be observed only in darkness. It is caused by the healing of lattice defects on heating. The lattice defects are always due to radioactive radiation which had hit the crystal since its formation. This effect is called *thermoluminescence*, it is often found on fluorite, quartz, calcite, apatite, zircon, and diamond in specimens from different outcrops.

Sometimes the colour of a mineral changes due to the healing of lattice defects on moderate heating, mostly from dark colours to lighter ones. Such bleaching is a common procedure used in treating raw gemstones, especially metamict zircons. – But minerals may also change colour after heating, owing to decomposition. For example the carbonates of copper, iron, and manganese become black on heating, due to the formation of black oxides. A dark red colour frequently occurs when hematite is formed.

b. The formation of gases

Carbon dioxide CO_2 is obtained from most carbonates, it may be identified by introducing a drop of a clear barium hydroxide or calcium hydroxide solution onto the inner wall of the tube next to the open side. The drop becomes white owing to the formation of the respective carbonates. The calcium hydroxide solution should be freshly prepared by agitating some $\text{Ca}(\text{OH})_2$ with water in a test tube, after allowing the surplus solid to settle for some minutes, take a drop of the clear solution by a glass rod for the test.

Oxygen O_2 may be formed by the decomposition of pyrolusite and other manganese oxides. To detect it, light a wooden toothpick with a torch flame, blow out the flame of the burning wood and insert the still glowing end of the toothpick into the tube. The presence of oxygen causes the glow to intensify or the flame to re-appear.

Sulphur dioxide SO_2 with a strong, pungent odour may be formed by the decomposition of sulphates or the part-oxidation of sulphides. It is detectable by the acid reaction it imparts to moistened pH-paper or by the decoloration of wet brown pyrolusite-paper. Such paper is prepared by soaking white filter paper with an aqueous solution of potassium permanganate and drying the paper. Soaking and drying is repeated once or twice till the paper is distinctly brown. The dry paper can be stored for months in tightly sealed glass or plastic vessels. For use cut a small strip, dip it in water, shake off any surplus water, and place it half inside the tube, bending the second half down. The decoloration is due to the reaction $\text{SO}_2 + \text{MnO}_2 \Rightarrow \text{MnSO}_4$, which only occurs in the presence of water.

Sometimes minerals containing fluorine as well as OH-groups cause hydrofluoric acid HF to form; it will etch the glass, gives an acid reaction to pH-paper and has a pungent odour. This reaction occurs only at higher temperatures. (e.g. topaz)

Many organic materials will develop a brown smoke, mostly accompanied by dark distillation products and an empyreumatic odour. Only amber or natural resins produce an aromatic odour.

A garlic-like odour points to the presence of arsenic, while selenium causes a peculiar odour resembling the smell of a rotten radish.

c. The formation of sublimates or condensed liquids on the cold walls of the tube.

Water is given off from zeolites and hydrates like gypsum at low temperatures. Minerals containing the hydroxyl group, like clay minerals, micas or amphiboles lose their water due to the reaction $2 \text{OH}^- \Rightarrow \text{H}_2\text{O} + \text{O}^{2-}$ only at higher temperatures. Any sublimate that occurs should be inspected through a lens and proved by heating the tube at the location of the sublimate:

Black like a black mirror, next to the assay dark gray crystals \Rightarrow **As**

Black similar to As, transferred to a streak plate and rubbed it turns red \Rightarrow **HgS**

Black fusible globules \Rightarrow **Se** or **Te**, small globules of Se transmit a reddish light.

Red to brown colours point to sulphides of **As** or **Sb**, but they look nearly black, the As-compounds are readily volatile.

Yellow, hot orange-red \Rightarrow **S**, may be produced in the closed tube by pyrite or marcasite.

White may be ammonium salts, or As_2O_3 , or Sb_2O_3 , or lead chloride, or Hg_2Cl_2 . Repeat the test adding five times the amount of dry sodium carbonate to the assay, ammonium salts give the NH_3 smell, mercury chloride decomposes to metallic mercury. To distinguish As- and Sb-oxides use the open tube test.

Gray metallic globules, which may be united by rubbing with a strip of paper \Rightarrow **Hg**

When sulphates decompose, small drops of concentrated sulphuric acid may sometimes occur, they look like oil. - Closed tubes should be discarded after use if any sublimate has formed.

Heating in an open tube

Glass tubes, open at both ends, are employed in heating and roasting substances in a current of air, thus bringing about oxidation. The tubes should be between 5 and 8 mm internal diameter and 15 to 17 cm long. They should be held in a slanting position of 20° to 30° in order to ensure a draft of air on heating. Straight tubes may be used, but the powder of the assay tends to fall out of the tube. To bend a tube, heat the tube 5 cm from one end, turning it constantly during heating. When this part becomes soft, bend it to an angle of 20° - 30° . The coarsely powdered sample is placed next to the bend. Bring the smaller part in a horizontal position and heat the tube first somewhat above the sample to produce a draught of air. The long part acts like a chimney, hold the tube at this upper end. Please increase the temperature very slowly to ensure complete oxidation. Heating in an open tube is one of the most important tests for minerals which are suspected to belong to the sulphide group; the test will give a reliable proof if S, As, Sb, Hg, Te, or Se are main constituents.

S: All sulphides are oxidized and sulphur dioxide is formed, which can be detected by its odour or by the colour change of wet pH-paper. Again brown moistened pyrolusite paper can be used to detect the formation of sulphur dioxide, which will bleach the brown paper. The minerals sphalerite ZnS and molybdenite MoS_2 are difficult to roast, they should be used as a fine powder for this test. –

As: All arsenides are oxidized to As_2O_3 , which is white and readily volatile. The sublimate forms as a ring, and where it deposits on the warm glass it is distinctly crystalline. With a good lens octahedrons can be observed. The typical garlic odour should not occur, since this is an indication of incompletely oxidized samples. –

Sb: This element is oxidized to Sb_2O_3 , which is white and slowly volatile. The sublimate forms as a dense, white smoke, which passes up the tube and partly settles on the upper side, partly it leaves the tube. It is volatile, but on further heating it changes to Sb_2O_4 , this compound is non-volatile, infusible, and its colour is pale straw-yellow when hot. Often a white "fur"-like deposit of this compound is formed on the underside.

Se: SeO_2 has a typical odour of rotten radish. Only large amounts produce a gray sublimate of Se next to the sample. It may turn to red at a distance, and, far from the sample there

appears a sublimate of SeO_2 made up of white, radiating, prismatic crystals, which are readily volatile on heating.

Te: Produces a white smoke, partly it passes through the tube, partly it settles as a thick, white sublimate on the lower side. Very similar to Sb_2O_4 , but on heating of the sublimate of TeO_2 small oil-like drops are formed!

Hg: Produces gray metallic globules; volatile. By rubbing the minute globules with a strip of paper, they may be made to unite.

Bi: If sulphides are present, a small white sublimate of bismuth sulphate is formed. When heated it melts to brown drops, the cooled drops are yellow and opaque. On increased heating they vanish due to the formation of Bi-silicates.

Pb: Sulphides with a considerable lead content may give a small amount of a sublimate of PbSO_4 . The colour is white when cold, on strong heating it vanishes due to the formation of colourless lead silicate.

The test for Pb and Bi is not very reliable and should be confirmed by heating on charcoal. Arsenates are already oxidized, they will not give reactions in the open tube. The same is true for most sulphates, only those of some transition metals are decomposed to the metal oxide and SO_3 , which itself decomposes at higher temperatures to SO_2 and oxygen. - Open tubes should be discarded after use if any sublimates were observed during the test.

Heating on charcoal

Most probably this is the oldest method to prove ores. Both reductions and oxidations are obtained on charcoal. If the mineral shows no decrepitation a small piece of 1 to 2 mm diameter is used, otherwise the sample is finely crushed and mixed with some water to a paste. The piece or the paste is laid on the coal into a flat cavity approx. 1 to 2 mm depth about 10 mm from one end of the coal. Now the surface of the charcoal is brought into a slanting position of approx. 20° with the assay at the lower end. The flame is directed towards the sample in such way that the area just ahead of the inner blue cone is close to the sample, while the remaining hot gases pass over the charcoal towards the upper end (figure 4). The combined effect of the flame and the burning charcoal gives considerable heat and intense reduction is achieved. Many elements are volatilized; passing into air they take on oxygen and deposit characteristic coatings of oxide on the coal. The sulphur of sulphides oxidizes to SO_2 with its typical odour. The garlic odour of As or the odour already described typical for the presence of Se occurs only under strong reducing conditions.

It is important to judge whether any sublimate is deposited next to the assay or far from it. Any colour should be recorded in the hot stage as well as after cooling. Some sublimates are volatile on direct heating. Please try gentle heating first and look for any coloration of the outer flame when touching the sublimate with the top of the flame.- Some charcoals produce a white-gray ash on burning. This may be confused with a sublimate. To test it, please do a blank test without assay.

In some cases a metallic bead is formed. - If no sublimate or metallic bead is achieved, the procedure should be repeated using a finely powdered mixture of one volume sample with three volumes of dry sodium carbonate. This mixture is slightly moistened before heating to avoid any blowing-off effects by the flame gases. The soda mixture should always be applied

if there is any likelihood that the mineral may belong to the sulphide group or is a gossan mineral!

If the substance is assumed to be a carbonate or sulphate it should be brought into contact with wet pH-paper after intense glowing of the pure sample on charcoal. The carbonates and sulfates of the alkaline earth group show an alkaline reaction, due to the formation of the appropriate hydroxides.

As: Produces a white, very volatile sublimate far from the assay. Sometimes the colour changes to gray. When touched with the flame the sublimate vanishes rapidly and gives a pale blue colour to the flame. Often the garlic odour is pronounced if the mixture with sodium carbonate is used.

Sb: The Sb_2O_3 sublimate is white, less volatile than that of As_2O_3 , and therefore nearer to the assay. Thin layers distant from the assay show a bluish colour. Touching with the flame causes the sublimate to move with a pale green shine.- A grayish-white, brittle bead is formed only if enough antimony is present. After extinction of the flame, the bead stays liquid for some time, producing white smoke. Eventually, the last smoke produces tiny white crystals on the bead's surface.

Se: The volatile sublimate is steel-gray, far from the assay it often changes to white, tinged with red. When touched with the flame it imparts an azure-blue colour to the flame. The typical odour similar to that of rotten radish is pronounced.

Te: A dense, white sublimate with a gray, sometimes brownish rim is typical. It is volatile; a green shine is produced only with a reducing flame.

Zn: The ZnO sublimate is canary-yellow only when hot, but white when cold. It is not volatile!

Cd: CdO deposits as a dark, almost black sublimate, which changes with decreasing thickness from reddish-brown to yellow, the outer coal surface shows an iridescence resembling peacock colours. The colour varies from blue-violet to black, followed by a change from copper-red to brass-yellow towards the cold side of the coal. This iridescence is very typical, it can be observed even in the presence of a large surplus of zinc. - The sublimate is volatile if touched by the flame, but it imparts no colour to it.

Bi: Bismuth produces a sublimate of Bi_2O_3 , it is dark orange-yellow when hot and changes on cooling to yellow with a yellowish-white rim. The coating is volatile if the coal is heated to visible glowing, but no colour is given to the flame. – Any formed metallic bead shows a gray to reddish-white colour and is brittle, although it may at first flatten to some extent when hammered.

Pb: Lead compounds produce a gray malleable metallic bead. The bead imparts a blue colour to the oxidizing flame; the PbO settles as a dark-yellow sublimate when hot, the cool oxide is sulphur-yellow with a bluish-white rim of lead carbonate. The sublimate is volatile on glowing, again imparting a blue colour to the flame.

A yolk-yellow sublimate of a mixed oxide of lead and antimony is formed next to the assay if both elements are present in the assay. The typical Sb_2O_3 sublimate is produced as well. - Many authors in the 19th century found it difficult to distinguish the yellow sublimates of Bi and Pb oxide. They resolved this problem by converting the oxides to iodides, which have quite different colours. The

coatings were moistened with a solution of iodine in alcohol. On repeated heating the bismuth oxide changed to a volatile brilliant red bismuth iodide, while the PbO changed to volatile yellow lead iodide.

Sn: Tin compounds are reduced to a bright white bead of metallic tin in the reducing flame; it assumes a white coating when cold. The bead is malleable, the white sublimate of SnO_2 is next to the assay and nonvolatile.

Cu: Copper compounds are reduced to red, malleable beads. They are fusible only at a rather high heat. Sometimes many small globules or spongy plates are formed. They are bright when in the reducing flame, but they oxidize and become black on exposure to the air. No coating is formed on the charcoal.

Ag: Minerals containing enough silver form a white and bright malleable bead. It is easily fusible. If less noble metals like lead, antimony, or zinc are present, an alloy bead is formed. Prolonged treatment with an oxidizing flame causes these less noble metals to oxidize and eventually the silver bead is formed. In such cases the white sublimate of oxides turns reddish by the deposition of traces of silver oxide Ag_2O . If copper is present its full oxidation is sometimes difficult since black copper oxide covers the bead. An easy way to detect any silver content of such beads is to transfer the bead to a ceramic dish. Add some borax and treat with an oxidizing flame. The oxidized copper dissolves in the borate melt with a blue colour and the silver bead remains.

Au: Gold is rather easily fusible, the yellow malleable bead is bright in the hot as well as the cold stage. On prolonged heating some gold is vaporized, producing a reddish to golden colour which can be observed only on a white base, but not on charcoal. If any alloy should be proved, melting with borax on a ceramic dish is recommended. If gold and silver is present the colour of the remaining bead depends on its silver content.

Mo: Minerals with a considerable molybdenum content produce a sublimate of MoO_3 which is pale yellow when hot, but white when cold. It is sometimes distinctly crystalline, and is volatile in the oxidation flame. This coating assumes a beautiful azure-blue colour if it is touched for an instant with a reducing flame. If the coal glows red during contact with the flame the coating is reduced to a lower oxide, which looks copper-red when cold.

Black magnetic globules or flakes, or a magnetic mass will be obtained on fusing iron containing minerals with sodium carbonate on charcoal. The same may happen if cobalt or nickel is present. For further wet analysis for Fe and Ni they should be dissolved in hot concentrated HCl. Cobalt can be detected by dissolving some of the powdered globule in molten borax; see borax and phosphorous salt beads.

Easily fusible globules with a metallic lustre, bright when in the reducing flame, but tarnishing on exposure to the air, are frequently obtained when combinations of certain metals like Pb, Ag, Cu, and Bi with S, As, or Sb are heated on charcoal. They can be distinguished from metallic beads by their brittleness. Complete oxidation of S, As, and Sb can be achieved by prolonged treatment with the oxidation flame.

White coatings may also result from the volatilization of some chlorides, namely the chlorides of lead, copper, bismuth, mercury, ammonium, and the alkalis. Touching with the flame renders such sublimates volatile, Pb and Bi forming a yellow new sublimate. PbCl_2 imparts a pale blue colour to the flame. If CuCl_2 is present it melts and produces an intensive

azure blue flame colour. These phenomena are only important if you are testing oxychlorides from an oxidation zone.

To prove whether a bead is brittle or malleable, put the bead on a flat piece of steel and give it a gentle blow with a hammer. Brittle beads will crash to pieces, while perfectly malleable metallic globules can be hammered to thin sheets. Less malleable globules can be hammered to sheets when they are heated after each blow. On proving alloys care should be taken especially with lead-gold alloys, some compositions in this system are extremely brittle!

Some scientists in the 19th century recommended heating on gypsum plates as well as on charcoal, since some coloured sublimates may be more easily detected on a white surface. In fact, this is true for the red-brown sublimate of silver oxide and the red one of gold. In all other cases the use of gypsum plates provides no advantage at all. - These gypsum plates were prepared by making a thin paste from calcined gypsum with water, pouring this upon a sheet of glass, and spreading it out evenly until it is 3 to 4 mm thick. Before the plaster sets, its surface is ruled off by means of a knife into rectangular blocks about 4 x 8 cm. They are removed after the plaster hardens.

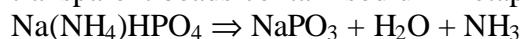
According to a suggestion by Goldschmidt the sublimates can be deposited on small cover glasses for microscopic use. The cover glasses have to be positioned on the charcoal at an appropriate distance from the assay. They are excellent for further microscopic observation. A small pocket microscope with a 30x magnification can be used. Such sublimates on a cover glass can be transferred to a test tube as well and used for further wet analysis.

The coloration of borax and phosphate beads

Borax is $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$; on heating it loses its water of crystallization and melts at 742°C to a colourless, transparent glass, which consists of a mixture of sodium metaborate and boric oxide. $\text{Na}_2\text{B}_4\text{O}_7 \Rightarrow 2\text{NaBO}_2 + \text{B}_2\text{O}_3$.

The hot melt reacts with most oxides of the transition metals, as well as the oxides of the main group elements and silicates. The reaction leads to metaborates, sometimes orthoborates are also formed. Any SiO_2 deriving from silicates or quartz is incorporated into the glass structure if a surplus of borax is employed. Many transition metals impart a characteristic colour to the glass. On reducing conditions some elements are transferred to a lower charge, e.g. $\text{Fe}^{3+} \Rightarrow \text{Fe}^{2+}$, and in such a way a characteristic colour change is evoked. Some elements can be reduced to the metallic state, like Cu or Ni.

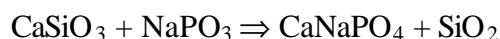
Very similar beads result on the melting of sodium ammonium hydrogen phosphate tetrahydrate, $\text{Na}(\text{NH}_4)\text{HPO}_4 \cdot 4 \text{H}_2\text{O}$ which is also called *microcosmic salt*. The colourless, transparent beads contain sodium metaphosphate in a glassy state:



Pure sodium metaphosphate NaPO_3 melts at 628°C , it is recommended to use this dry component instead of the *microcosmic salt*, if it is available.

Again the melt dissolves the oxides of transition metals by the formation of orthophosphates, e.g. $\text{CuO} + \text{NaPO}_3 \Rightarrow \text{NaCuPO}_4$

The colour of the phosphate beads is generally very similar to that of the borax beads, but there are some important exceptions. Silicates are also dissolved by the metaphosphate melt, according to the formula



Quite in contrast to the borate melt, in this case the formed silica is not incorporated into the glass structure! The silica "skeleton" of dissolved small silicate grains often can be detected in phosphate beads during and after fusion. However, it should be noted that many silicates dissolve completely in the bead, but the remaining SiO_2 is not visible to the naked eye or by using a lens. So the absence of any silica "skeleton" does not conclusively prove that a silicate is not present!

There are two further differences regarding both kind of beads:

- Borax beads are more viscous than the phosphate beads. Therefore it is easier to handle a borax bead if the molten bead is held in place by its surface tension.
- The colours of the phosphate beads are usually more pronounced.

The beads can be made in several ways. Use of the platinum wire loop is the oldest way, and it is the best method since you can look through the bead. Unfortunately great care must be taken when treating beads on a platinum loop with a reducing flame, because any formation of easy reducible elements will lead to corrosion of the platinum. Melting the bead on the tip of a magnesia rod avoids these pitfalls, but any judgement of the colour must be done against the background of the magnesia rod.

If necessary, beads can also be prepared in small cavities on charcoal. This technique achieves an intensive reduction; by adding a small amount of tin or tin chloride any content of Cu or Ni can be reduced to the metallic state. It can be extracted subsequently from the bead by melting with some lead.

To produce a bead, heat the platinum loop or the magnesia rod to a red glow, then dip it into the appropriate salt. Some material should adhere to the loop or rod. Now glow again till all material is molten. This procedure should be repeated till a clear colourless bead of sufficient mass is formed. Do not dip any hot loop or rod into a plastic storage vessel, it may melt! Put some borax or sodium metaphosphate on a piece of aluminium foil instead, and pick it up from there. - If the assay is a mineral of the sulphide group it has to be roasted before being used for bead coloration. Put a thin layer of the very finely powdered sulphide on a magnesia furrow and glow it till all material is converted to the oxides. Easily melting minerals can be roasted if they are mixed with some pure graphite powder. If any difficulties arise due to incomplete oxidation, mix the half-roasted powder with some ammonium carbonate and glow again.

The finely powdered assay is placed on aluminium foil and a minute amount is brought to the moistened cold colourless bead by dipping it into the powder. A trace of saliva may be used for moistening. Now melt to a bright glow for approximately a minute, then look at the hot bead as well as at the cold one using a lens. It is important to apply a minute amount of the assay as otherwise the bead may become too dark or opaque. If the colour of the bead is too faint, a repetition of the material take-up is possible. This stepwise take-up is a quicker way to success than the application of larger amounts at once!

To cause a distinctive colour it is necessary to dissolve 2 to 3 weight % of the appropriate oxide of the element in a given bead for the elements W, Mn, and Fe. Approximately 1 to 0.5 weight % of the oxide is sufficient for the elements Cu, Ni, V, U, Ti, Cr, and Ce. The test for cobalt is the most sensitive one, even 0.1% CoO causes a blue colour.

Generally the colour tint changes with the temperature because the absorption spectrum is shifted towards longer wavelength with increasing temperature. For this reason we see an increase of red, brown or yellow colours at high temperatures, but a decrease of the colour for blue and green on hot beads.

It is very important to know what kind of flame to use for melting beads, an oxidizing flame or a reducing one. You should test that in the following way, no matter what heat source is used, a butane torch, a bunsen burner or a blowpipe flame. Dissolve a sufficient amount of ammonium molybdate in a borax bead. In a really pure oxidizing flame the hot bead is clear and pale yellow, it becomes colourless on cooling. Look for the colour of the hot bead immediately after its removal from the flame, holding it against a white background. Even small amounts of reducing gas in the flame will cause a brown colour. Reciprocally the reducing power of a flame can be tested by a borax bead doped with a sufficient amount of manganese oxide. After melting with an oxidizing flame this bead shows a reddish violet when cold. A real reducing flame more or less quickly changes this bead to a colourless one, due to the reduction of the manganese to the divalent state. If your butane torch produces only an oxidizing flame you may place the hot bead into the luminous part of a pocket lighter flame for reduction.

Ambiguous results may occur if the assay contains several elements that render different colours to the beads. Nevertheless such coloration may give useful hints for further proofs by wet analysis. Especially mixtures of iron and copper, sometimes with nickel or cobalt are green in the hot stage; on cooling they change to various shades of yellow, green or blue, depending on the concentrations of the elements. Even a small amount of Co can be detected by an intensive reduction of such phosphate beads on charcoal with some tin chloride; the typical blue cobalt colour remains. - A blood red colour is caused on a phosphate bead in the reducing flame if iron-containing minerals of tungsten or titanium are tested.

Generally the oxides of the following elements give no colour to the borax bead: the alkalis, the alkaline earth elements, aluminium, tin, zinc, zirconium, tantalum, niobium, scandium, yttrium, lanthanum, thorium and all rare earths, with the exception of cerium and mixtures of neodymium and praseodymium. The elements lead, antimony, cadmium, and bismuth give only a pale yellow colour to the hot bead, but the cold bead is colourless. Only bismuth is reducible by the reducing flame causing a gray colour. - The same is true for the phosphate beads, with some exceptions: here the elements Ta, Nb, Cd, Pb, Sb, and Bi turn a hot bead a very pale yellow if they are present at high concentrations, but the cold bead is colourless. In the reducing flame beads containing Pb, Sb, and Bi become gray, while Nb will cause a faint brown colour. Mixtures of neodymium and praseodymium give a very pale rose colour to any bead. - This information is only for anyone who is interested in the method, none of the faint colours described above can be taken as a distinct evidence for the presence of an element in the assay!

Colour of the phosphate beads				
oxidizing flame		element	reducing flame	
hot	cold		hot	cold
yellowish green	colourless	Mo	green	green
pale yellow	colourless	W	green	blue
pale yellow	colourless	Ti	yellow	violet
green	green	Cr	green	green
yellowish green	yellow	V	green	green
yellowish green	greenish yellow	U	green	green
yellowishbrown	pale yellow	Fe	pale yellow	colourless
green	pale blue	Cu	colourless	red
blue	blue	Co	blue	blue
brownish red	yellow	Ni	brownish red	yellow to gray
violet	violet	Mn	colourless	colourless

Colour of the borax beads				
oxidizing flame		element	reducing flame	
hot	cold		hot	cold
pale yellow	colourless	Mo	brown	brown
pale yellow	colourless	W	yellow	yellow to brown
pale yellow	colourless	Ti	grayish	brownish-violet
orange	pale yellow	Ce	colourless	colourless
yellow	yellowish-green	Cr	green	green
yellow	yellowgreen	V	dirty green	green
yellow to orange red	yellow	U	pale green	pale green to colourless
yellow to orange red	yellow	Fe	green	pale green
green	blue	Cu	pale green	opaque red
blue	blue	Co	blue	blue
violet	reddish-brown	Ni	opaque gray	opaque gray
violet	reddish-violet	Mn	colourless	colourless

Flame coloration

The compounds of certain elements are volatilized in a non-luminous flame, some of the volatile species impart characteristic colours to the flame. The colours observed by the flame test are summarized in the following table. Some extremely rare elements like indium, rubidium and caesium are not included.

Flame tests	
Colour of the flame	Element
Persistent golden yellow	Sodium
Violet	Potassium
Carmine-red	Lithium
Brick-red to yellowish	Calcium
Crimson	Strontium
Yellowish green	Barium, molybdenum
Green	Boron, copper, thallium
Blue	Lead, arsenic, antimony, bismuth,
Blue-green	Phosphates

Tests may be made in different ways:

- A very small elongated fragment of the mineral can be held with clean stainless steel forceps. Take care that the flame touches only the mineral, not the forceps.

- A minute quantity of the very finely powdered material is taken on a magnesia rod or a platinum wire loop: the rod or wire is heated till it gives no coloration, then it is moistened with pure water or hydrochloric acid, and its end is touched to the powdered material to take up some of the mineral, which is then introduced into the flame. It is possible to use a soft iron wire with a loop instead of the platinum wire. This device corrodes quickly, but due to the cheap iron wire it can be replaced from time to time. Usually the wire is contaminated with sodium. Therefore before use the loop is dipped into HCl and heated until the wire glows without producing the yellow sodium colour.

- Oxides, carbonates and phosphates should be mixed with some HCl, the resulting slurry is again brought to the flame by a loop or the magnesia rod. Sulphates of the alkaline earth group give better results if they are previously glowed on charcoal. In this way they are at least partly reduced to alkaline earth sulphides which are soluble in HCl. Chlorides are the most volatile compounds, they should be used as far as possible.

Especially sodium compounds cause a persistent golden-yellow flame which masks all other flame colours. Unfortunately this test for sodium is extremely sensitive, already an amount of 7×10^{-8} mg Na causes a distinctive yellow flame. It is possible to overcome this difficulty by viewing the flame through two thicknesses of cobalt glass, which masks the yellow sodium colour. Flame tests with the cobalt glass are used to detect the alkali and alkaline earth elements; but the colour of the flame is changed by the cobalt glass; this change is indicated by the table at the end of this section. Sometimes it is convenient to fix the cobalt glass to a spectacles frame by means of adhesive tape. If no cobalt glass is available it is possible to look at the flame through a bottle filled with a diluted solution of potassium permanganate. Again the yellow sodium colour is masked in this way, but the colours are slightly changed. It is recommended to gain experience by trials with known composition.

Silicates should be tested for boron by fusion with a mixture of one part calcium fluoride with one and a half parts of ammonium hydrogen sulphate on a platinum or iron wire loop. Mixing of these chemicals should be done in the plastic lid of a tubule. In this way boron-containing minerals produce boron trifluoride, which is extremely volatile. Therefore a green colour is caused even if the loop is brought next to the flame without touching it.

Another way to test silicates for alkali elements is to glow the finely powdered mineral with the same amount of gypsum. By this procedure calcium silicate and the appropriate alkali sulphates are formed.

The flame colours of some elements mainly occurring as sulphides were already described in the section "heating on charcoal". Since sulphides do not contain alkalis or alkaline earth elements it is useless to test them for such flame colorations on a magnesia rod or a loop!

The colours are best seen in a dark room; since this is usually inconvenient a dark screen or charcoal as a background will be found advantageous. Flame tests should never be done in bright daylight.

Flame tests with cobalt glass				
Flame coloration		Flame colour through cobalt glass		Element
Golden-yellow		Nil		Sodium
Carmine-red		Purple-Red		Lithium
Violet		Crimson		Potassium
Brick-red		Light-green		Calcium
Crimson		Purple		Strontium
Yellowish green		Bluish-green		Barium

Fusibility

The ease with which minerals fuse is of considerable assistance in their identification. Small elongated splinters about 1.5 mm in diameter should be employed in testing fusibility. The splinter should be held by forceps so that its end projects beyond the metal, only this end is held to the flame. Older textbooks recommend the use of platinum forceps, but it is possible to use stainless steel ones instead. Any remaining fused material at the tips can be removed by means of fine abrasive paper. The fusibility of a mineral is determined by comparing its fusibility with that of a fragment of comparable size from a scale suggested by Von Kobell.

1. Stibnite Sb_2S_3 Fuses easily in the flame of a candle or a match. (525°C)
2. Chalcopyrite CuFeS_2 Fuses rather slowly in a candle flame. A small fragment in a closed glass tube fuses at a full red heat. ($\sim 800^\circ\text{C}$)
3. Almandine $\text{Fe}_3\text{Al}_2(\text{SiO}_4)_3$ Fuses readily to a globule at the tip of an oxidizing flame of a butane torch. ($\sim 1050^\circ\text{C}$)
4. Actinolite
 $\text{Ca}_2(\text{Mg,Fe})_5(\text{OH/Si}_4\text{O}_{11})_2$ Edges of a fragment are readily rounded by an oxidizing flame, only very fine splinters are fused to a globule. ($\sim 1200^\circ\text{C}$)
5. Orthoclase KAlSi_3O_8 The edges of a fragment are rounded with difficulty by an oxidizing flame. ($\sim 1300^\circ\text{C}$)
6. Bronzite $(\text{Mg,Fe})\text{SiO}_3$ Only the finest points and thinnest edges become rounded by an oxidizing flame. ($\sim 1400^\circ\text{C}$)

Sometimes no fragments are available and the powder does not fuse in a closed glass tube at full red heat. In such cases the mineral is ground with some drops of water in an agate mortar, the pulp is spread on charcoal and first slowly heated to dryness, then intensively glowed. The cake thus formed can be picked up cautiously with the forceps, holding the rim of the cake to the flame. This procedure was already suggested by Berzelius. Of course it cannot be used for sulphide minerals, since such minerals would be decomposed by glowing on charcoal!

Textbooks on mineralogy and mineral identification normally register the fusibility of minerals, unfortunately some recently published books have refrained from such hints. Anyway, in order to test the heat of the employed flame it is recommended to heat splinters of the standard minerals. Some iron-containing oxides are infusible in an oxidizing flame, but

are fusible with difficulty in a reducing flame, due to the reduction of iron to the divalent state; e.g. hematite or franklinite.

The oxidation melt

The finely powdered mineral is melted with a surplus of a mixture of soda and potassium nitrate (3 : 1). A magnesia furrow, a clay dish, a magnesia rod or a platinum wire loop may be used.

In the presence of manganese the melt shows a green colour with a bluish tinge. This is due to the formation of sodium manganate Na_2MnO_4 . The melt can be dissolved in water, resulting in a green solution. On acidification with acetic acid this solution changes its colour to red and a brown sediment settles. After some time the red colour fades and a colourless solution with a brown sediment remains. The red colour is caused by the formation of the permanganate ion, the brown sediment is a disordered pyrolusite.

The presence of chromium causes a yellow colour due to the formation of sodium chromate Na_2CrO_4 . The melt dissolves in diluted acetic acid; this yellow solution changes its colour to blue on the addition of hydrogen peroxide. The blue colour caused by chromium peroxide is very unstable, it quickly changes to green by the formation of trivalent chromium compounds.

The wet part of both tests can be carried out also on a micro scale. To do this, the melt is moistened with a drop of distilled water. The liquid is soaked after some seconds by the end of a strip cut from white filter paper. One or two drops of diluted acetic acid are placed at the rim of the soaked solution. In the test for chromium then add a drop of hydrogen peroxide or a solution of sodium perborate.

Both tests are rather sensitive, already a content of 0.2% of Cr or Mn of the tested matter is sufficient for a distinct coloration of the oxidizing melt.

The ammonium hypophosphite fusion

$\text{NH}_4\text{H}_2\text{PO}_2$, ammonium hypophosphite contains phosphorus in a positive monovalent state. On fusion it decomposes at a low temperature ($\sim 200^\circ\text{C}$) to a mixture of ortho phosphoric and meta phosphoric acid by the simultaneous formation of phosphine PH_3 , hydrogen and ammonia. Phosphine is a very strong reducing agent. Phosphoric acids decompose all silicates, carbonates and most oxides, except quartz. The melting with ammonium hypophosphite is a reducing fusion. For this reason it is useless to apply this assay for minerals of the sulphide group!

You must proceed with great caution when using this reaction. The evolved phosphine is extremely poisonous, it is self-inflammable and burns with flames of up to 5 cm length to a white smoke of phosphorous pentoxide, which again is seriously toxic on aspiration. Never do such fusion in a room, unless a well-ventilated fume cupboard is available! The reaction may be done in the open air if a secure ventilation by a low wind is guaranteed.

A pinch of the well-powdered mineral is mixed with half a teaspoon of the ammonium hypophosphite and placed in a clay dish or porcelain dish. Place the dish on a piece of charcoal or on glasswool. Heating is done by directing the flame of a butane torch towards the side of the vessel. The flame may be taken away till the burning of the melt stops. Finally the melt is heated again till a clear melt is formed. Sometimes a little precipitate is formed.

If the hot melt is blue, but changes its colour to a pale pink on cooling cobalt is present.

If the melt stays blue on cooling, put some drops of water on the cool melt:

- A violet colour indicates tungsten.
- A pale pink colour which turns to orange-red on the addition of hydrogen peroxide or sodium perborate solution indicates titanium.

Vanadium causes a reddish hot melt, it changes its colour on cooling to yellow and then to green. A small amount of water placed on the top of the cool melt becomes pale green, on addition of hydrogen peroxide the colour changes to a pale pink.

Green melts are caused by uranium or chromium: add some water, separate the green solution by transferring to a test tube and add ammonium carbonate and hydrogen peroxide. A yellow-orange colour indicates uranium, but the green colour is lasting if only chromium is present.

A reddish-brown colour of the melt indicates molybdenum, but this test does not work for molybdenite MoS_2 !

A colourless melt with a white precipitate may contain Nb, columbium. Add some concentrated hydrochloric acid and some pieces of tin foil and heat again. The formation of a blue colour is a proof for Nb.

The ammonium hypophosphite fusion test is not really a classical test, since it was introduced only in 1941 by American scientists. - Many pitfalls may arise in using it, since any easily reducible element may cause a dark colour, masking the above-mentioned colours. So arsenic, silver or tellurium cause a black colour, while black melts with a brown rim may occur in the presence of copper, bismuth or antimony. Yellow-brown melts are formed by minerals containing tin, lead or selenium.

Some tests for anion forming elements

The Hepar reaction for **sulphur**:

Mix the finely powdered mineral with sodium carbonate and melt the mixture on charcoal. Any sulphur content, even the sulphate ion, is changed by this procedure at least partly to sodium sulphide. Now remove the melt from the charcoal by means of a knife and place the material with a drop of water on a clean silver surface. A black stain of silver sulphide is readily formed. It should be noted that the rare elements Te and Se also produce black staining. - To remove any stains on the silver, the surface should be cleaned by scouring with toothpaste.

Test for **sulphides**:

Put a small amount of the mineral in a test tube, add a piece of pure zinc and add 3 ml concentrated hydrochloric acid. On warming the mixture, hydrogen sulphide evolves; a white filter paper strip soaked with lead acetate solution turns black if it is placed on the open end of the test tube. This test works also for sulphides which are insoluble in HCl. As, Se and Te may also cause a coloration. Ensure good ventilation, hydrogen sulphide is very poisonous! - It is possible to prepare the lead acetate paper and to store it after drying in any air-tight vessel till use. Before use it should be moistened again.

Test for **phosphorus** by reduction with metallic magnesium:

A small glass tube of 8 to 10 cm length is intensively heated at one end till the flame turns yellow, now the walls of the heated end are tweezed together by means of stainless steel forceps. Cool this closed end slowly by waving it to the flame and back, eventually by setting it down on charcoal. After complete cooling to room temperature place a piece of magnesium ribbon about 20 mm long into the tube next to the closed end, add a minute amount of the finely powdered mineral and tap the tube so as to bring the mineral powder as much as possible in contact with the magnesium metal. The mineral powder should be heated before to a low red glow on a magnesia furrow. Hold the tube with a clothes peg (spring-type) at the open end in a nearly horizontal position and heat the portion with the magnesium ribbon and mineral till the Mg ribbon is ignited. The use of goggles is recommended! Crack off the end of the hot tube by dipping it into water. The disagreeable odour of phosphine PH_3 , somewhat like the odour of garlic, is clear evidence for the presence of phosphorus in the mineral. The burning Mg reduces any phosphates and Mg_3P_2 is formed, this compound reacts with water to PH_3 . Ensure good ventilation!

Test for **fluorine**:

The finely powdered mineral is mixed with 5 parts of sodium metaphosphate, transferred to a new test tube and some drops of water are added. Cotton wool is used to close loosely the open end of the test tube. On melting and prolonged heating hydrofluoric acid will be given off; it etches the glass. Some silicon tetrafluoride thus formed is decomposed by water vapour and forms a white tarnish of silica in the upper part. Etching and tarnish are best seen after the tube has been cooled and rinsed with water. The test tube should be discarded after use. This test is applicable only if fluorine is a main constituent. No clear results are achieved for very low F contents sometimes found in amphiboles and some micas.

Test for **boron**:

The mineral powder is mixed with some drops of concentrated sulfuric acid or some ammonium hydrogen sulphate and approx. 4 ml methanol in a test tube. On gentle heating the volatile trimethylester of boric acid is formed, this compound burns with a green colour if the methanol vapor is ignited. The test can also be made in a potter's clay dish. If no methanol is available it is possible to use ethanol instead. - This test is applicable only to minerals which are decomposed by sulfuric acid. Any hard minerals may be silicates, they should first be melted with the same amount of sodium carbonate on charcoal. The resulting bead is ground and melted with potassium hydrogen sulphate. This melt is wetted with some drops of water; dry carefully before adding H_2SO_4 and CH_3OH , followed by heating and ignition.

Test for **silicon**:

Approximately 0.4 g of the mineral powder is mixed with a quarter of this amount of calcium fluoride and placed in a plastic tube like that used for tablets in pharmaceutical use. One millilitre of concentrated sulfuric acid is added and the tube is closed with the plastic lid, in which a hole has previously been made by means of a hot nail. It is possible to use a sheet of lead with an appropriate hole instead of the plastic lid. The hole is covered with a wet black cotton fabric kept wet by placing some wet cotton wool on the lid. Now heat the lower part of the plastic tube gently by dipping it into hot water for 10 minutes. Any SiF_4 , evolved in the reaction of $\text{CaF}_2 + \text{H}_2\text{SO}_4 + \text{silicate}$, is decomposed by the water according to the formula:

$$3 \text{SiF}_4 + 2 \text{H}_2\text{O} = \text{SiO}_2 + 2 \text{H}_2\text{SiF}_6$$
 After opening the lid the black fabric should have turned white in the presence of any silicate in the tested mineral.

Treatment with cobalt nitrate

This test was applied in the 19th century for the identification of infusible and light-coloured pure oxides. Cobalt nitrate is converted to a black oxide on heating. But characteristic colours of mixed oxides are formed by strong heating, if an extremely finely powdered white metal oxide is wetted with the appropriate amount of cobalt nitrate solution. The reaction between the cobalt oxide and the tested metal oxide is a solid state reaction. It occurs readily only at high temperatures, and with extremely finely powdered metal oxides! Such fine powders are for instance sublimates on charcoal or precipitates yielded by wet processes. Ambiguous results are achieved by the application of the test to mixtures of oxides! Actually the method was only used to identify ZnO, Al₂O₃ or MgO.

Two types of a non-volatile white sublimate may occur on heating of the assayed matter on charcoal. Such sublimate is moistened with a 10% cobalt nitrate solution at the rim of the sublimate and again intensively heated. ZnO exhibits a clear green colour, it is best seen when cold. This mixed oxide is called Rinmann's green. If the sublimate is SnO₂, only a bluish-green is achieved. Only careless workers apply this test to sublimates made up from Sb₂O₃, since this compound is volatile on further heating. Unfortunately Sb₂O₃ also forms a dirty green colour on glowing with cobalt solution.

Carbonates can be transformed to oxides by intensively heating on charcoal, afterwards they may be treated with cobalt nitrate followed by glowing. Only MgO develops a colour in the pink to flesh-colour range, but SrO and CaO become gray.

The deep blue colour of the oxide CoAl₂O₄ resulting on glowing aluminium oxide with cobalt nitrate solution has sometimes been used as an artificial pigment. It is called Thenard's blue. Its formation is a good test for Al, but it should be made only with the precipitate of aluminium hydroxide obtained by wet analysis. It was called Thenard's bluff, instead of Thenard's blue, by generations of students of chemistry who were frustrated by their careless work. Namely, amorphous silica, zinc silicate or finely powdered quartz melts with CoO to a blue glass, which may be mistaken for Thenard's blue.

Just to illustrate the many pitfalls of the method described, some colours achieved by other light oxides are added here: violet may be caused by zirconium oxide or by the phosphate, borate or arsenate of magnesia; titanium oxide produces a green with a yellow tinge, and barium oxide exhibits a reddish brown.

The charcoal-soda stick

Most of the methods described were used in the early 20th century by students of chemistry as preliminary tests in inorganic analysis. A Bunsen burner could be used as a heat source for nearly all tests, but there was one exception. For heating on charcoal a small hot flame is needed. Since no butane torch was available at that time, the use of a blowpipe was inevitable. Many students ran into difficulties when using that device, so they invented a surrogate. For the charcoal-soda stick a toothpick is dipped in molten natron. Don't use matches: they are impregnated with sodium phosphate. Natron has the formula Na₂CO₃ · 10 H₂O, it melts at 32°C by decomposing into a saturated solution of sodium carbonate and Na₂CO₃ · H₂O. Now the front end of the stick is held into the oxidizing Bunsen flame till the wood is converted to charcoal soaked with sodium carbonate. If any glowing is observed after taking the stick out of the flame it should be immediately dipped into molten natron. The powdered mineral is mixed with two parts of molten natron, brought to the tip of the prepared stick, and is now

heated in the reducing part of a Bunsen burner flame till complete melting occurs. The stick is quickly removed from the flame and the front end is ground with some water in a mortar. Any remaining charcoal is carefully washed away, and this elutriation isolates any metal beads or metallic flakes. The use of a good lens is recommended. Iron, nickel or cobalt form magnetic flakes, they can be isolated by a magnet covered with a thin plastic foil. After being transferred to a piece of filter paper they are dissolved in diluted hydrochloric acid. Fe causes a brown, Ni a green, but Co a pink solution. Easily reducible metals like Pb, Sn, Bi, Sb and Ag form small globules, but Cu shows red flakes or a red spongy mass. - However, heating on charcoal is by far superior to the charcoal-soda stick, because it allows the observation of sublimates, as well as the typical flame colours.

Heating with sodium thiosulphate

Most metal oxides are converted to metal sulphides by melting with $\text{Na}_2\text{S}_2\text{O}_3 \cdot 10 \text{H}_2\text{O}$, sodium thiosulphate. This method was used occasionally only in the 19th century, because many sulphides have a different colour from the appropriate oxides.

The 1:1 mixture is heated together with a small amount of oxalic acid in a closed tube or a test tube in a slanted, nearly horizontal position in order to avoid the rinsing back of water. Sometimes sublimates occur. - The following colours may be observed.

White: Zn / Red: Sb / Yellow: As or Cd / Green: Mn or Cr / Brown: Sn or Mo

Black sulphides are formed by the elements: Fe, Co, Ni, Cu, Pb, Bi, Hg, Ag, U, and Au.

Spot tests and the use of the wet separation process

It is possible to determine the main constituents of many minerals by the classical tests described, but there are also some exceptions. For instance there is no simple and conclusive test for aluminium, magnesium, zirconium, and beryllium, especially in silicates. Sometimes there is an interest in minor or trace amounts of Ag, Au, As, U, or other elements in minerals or in mineral aggregates, in this case some of the classical methods may fail due to their low sensitivity.

Many elements form typical colours with dye-stuffs and the sensitivity of such reactions is high enough to identify even trace amounts. But most of the used dyestuffs react with more than one element and the reacting ion must be present in an aqueous solution. Therefore the mineral must be dissolved in an acid; acid-insoluble minerals must be decomposed beforehand by an appropriate fusion process. In most cases, disturbing ions have to be removed by one or more subsequent precipitation processes, or the disturbing ion must be masked by a special reagent. Fortunately you need only very small volumes. Usually it is sufficient to add one drop of the dye solution to one drop of the solution to be tested for the element. Generally the typical colour change is best seen if the drops are combined on a white filter paper placed on glass or plastic. Such spot tests may be carried out also on spot plates or on a white glazed porcelain shard, but often the sensitivity is enhanced on filter paper due to capillary phenomena. The sensitivity may be enhanced again at a factor of two to ten by using dry filter paper impregnated with the dye or a special reactant.

Test sticks are now available for many cations and anions showing semi-quantitative results by the coloration of different zones; they are the result of the further development of this technique during the last fifty years. Such test sticks are used predominantly in water analysis

and environment control, but they are too expensive for occasional use in mineral determination.

Some tests require the use of strong acid or basic solutions which cause the decay or the swelling of any paper. Such reactions may be carried out in small test tubes, because looking along the axis of the tube the deepness of the coloration does not depend on the dilution of the solution. A high sensitivity is achieved sometimes by the fluorescence caused by ultraviolet radiation.

The following text presents selected fusion processes for the different mineral groups and selected tests for different elements.

The choice of fusion processes

As already stressed, the first step in identifying a mineral is an accurate ocular inspection of the matter in order to determine colour, lustre, streak colour, hardness, cleavage, magnetic properties, and morphology, like re-entrant angles and other hints to twinning. A rough hint to higher density is only possible for bigger lumps and for densities in the range of 4 or higher. It is strongly recommended to note all observations throughout the whole determination procedure. - The system of mineralogy arranges the minerals in groups, depending on their chemical composition and structure. The type of fusion has to be chosen with respect to the chemical composition of the mineral, so it is very important first to gain information about the kind of mineral group.

Some authors have published systematic tables for identifying mineral species by means of their physical properties and by simple chemical experiments. Only few people have used such tables more than once. There are several reasons for this. You have to follow strictly the path given by the scheme, but any erroneous estimation will lead to inconsistent results. Moreover any scheme will fail if two intergrown minerals are tested. A quicker result may be achieved by any somewhat experienced collector or geoscientist using a trial-and-error method. - Some hints are given here how to operate in this way.

Presumption of sulphide minerals

Minerals may belong to the *sulphide group* if they show a distinctive streak colour as well as a colour. (There are only two rare and unimportant exceptions among the more than 300 members of the sulphide group.) Such minerals should be tested using the classical tests in the open and closed tube and by heating on charcoal. If S, or As, or both are detected, the mineral is most probably a member of the sulphide group. (In rare cases arsenates, arsenites, sulphates, selenites or tellurites may be present.) The mineral can be dissolved by fusion in a test tube with a mixture of ammonium nitrate and ammonium chloride. Add some drops of dilute hydrochloric acid to arrive at a solution for further wet processing.

Dissolution in hydrochloric acid

Try to dissolve the mineral in hot hydrochloric acid, but act carefully with only tiny amounts if the mineral is black and produces a black streak colour. All oxides containing tetravalent manganese dissolve in hydrochloric acid, generating chlorine gas! The odour of this poisonous gas is pungent and a piece of wet iodide-starch paper becomes blue when held atop the open end of the tube. The formation of chlorine is avoided if oxalic acid is added to the hydrochloric acid. Also all iron oxides dissolve slowly in such a hot mixture of oxalic acid and hydrochloric acid, even magnetite. This method can be used to remove coatings of iron or manganese oxides from acid-insoluble minerals like quartz.

Any intensive effervescence of a mineral on dissolution points to carbonates, a test for carbon dioxide can be made by a drop of barium or calcium hydroxide solution on a glass rod.

A turbid solution, a flocculent precipitate or the formation of a jelly points to acid soluble silicates. Boil the solution for some time till a white or yellowish precipitate is formed. Sometimes it is necessary to heat carefully till complete dryness; add concentrated HCl after cooling and boil again. Dilute with distilled water before filtration. Now isolate the precipitate by filtration and washing with water. Any silica precipitate is soluble in a hot NaOH solution.

If a clear solution is obtained without effervescence and without the formation of hydrogen sulphide the mineral may belong to the following groups: phosphates or oxides/hydroxides or sulphates or chlorides/fluorides or borates or arsenites/arsenates or selenites/tellurites. Any white precipitate at least partly soluble on heating and crystallizing in tiny needles on cooling is presumably lead chloride; use a lens.

⇒ Tests should be made for P by flame coloration and by ignition with magnesium ribbon. With a few exceptions phosphates show a hardness lower than 6.

⇒ Sulphates are detected by the formation of a white precipitate of BaSO₄ on the addition of BaCl₂ solution. The Hepar test may give further corroboration, especially for acid insoluble minerals. Hardness lower than 4.

⇒ Heat the mineral in a test tube with potassium or ammonium hydrogen sulphate. Any chlorides give HCl, a white smoke of NH₄Cl is produced if a drop of ammonium hydroxide solution is held next to the open end of the tube by means of a glass rod or filter paper.

⇒ Test for fluorine by heating with sodium metaphosphate in a new tube, as already described.

⇒ Borates may be identified by the aforementioned tests for boron.

⇒ Test for As, Se or Te by the three classical methods: open and closed tubes, heating on charcoal.

The mineral may belong to the oxide/hydroxide group if none of the aforementioned tests gives a positive result. By a strong and prolonged heating on charcoal hydroxides are converted to oxides at temperatures above 900°C. Test for any change with respect to colour, hardness and appearance after the glow process of a tiny fragment. But you have to bear in mind that many silicates and some oxides may also change their properties on strong heating! Some oxides may be reduced or partly reduced by glowing on charcoal. If there is any likelihood of this, you may gain further information by melting a mixture of the ground mineral with soda on charcoal.

Presumption of silicates

Most silicates show a hardness between 5 and 8, but there are many exceptions for phyllosilicates, especially clay minerals. Silicates show mostly a vitreous lustre, the streak colour is normally white, even dark-coloured species produce only grey or brownish streaks. Many neo-, soro-, and phyllo-silicates and all zeolites are decomposed by hydrochloric acid. The cold solution sometimes forms a jelly, sometimes flocculent silica is precipitated. How to proceed in such cases was already described above. - Because many silicates are not soluble

in hydrochloric acid, they have to be decomposed by a fusion process. There are several possibilities:

- Melting of the mineral powder with the same amount of soda or a mixture of soda and potassium carbonate on charcoal. No complete fusion may be achieved for silicates containing zirconium, beryllium, or much aluminium or magnesium. Do not use too much soda, in such cases the sodium silicate is soaked up by the charcoal and the non-reducible metal oxides like magnesium or aluminium oxide form an infusible residue.

- One part of the mineral powder is mixed with one part of soda and one part of borax; this mixture is melted on charcoal.

Both fusion processes may lead to the reduction of reducible elements and therefore metallic beads, or flakes and sublimes may occur. - The soda or soda/borax beads have to be crushed and ground before dissolution in hydrochloric acid.

- Any reduction can be avoided by dissolving the silicate in molten NaOH (Fp = 322°C) or, better, in a mixture of NaOH and KOH (Fp = 170°C). Unfortunately these melts are soaked up by charcoal and all easily available vessel materials except silver are attacked. However, you can melt the mineral powder with two flakes of both alkali hydroxides on a teaspoon made from stainless steel. Such a melt is contaminated with iron, chromium, and nickel. Work cautiously since the melt is aggressive to almost everything, use goggles, and wait till the melt is solidified and completely cooled before adding any water! The addition of water to the hot melt can spill the corrosive liquid due to the sudden evaporation of the water! Place the cold spoon with the melt in a small beaker and add some distilled water. After solution some brown floccules of iron hydroxide remain, add hydrochloric acid till an acidic reaction is assured by pH-paper. In most cases gelatinization occurs or floccules of silica are formed. Boil the diluted solution in a test tube till the silica settles on cooling. The filtrate can be used for further tests, but not for the identification of iron, chromium and nickel, since they are a contamination caused by the use of the stainless steel spoon in flux melting.

Fusion with potassium hydrogen sulphate

Acid-insoluble oxide minerals may be melted after intensive grinding with potassium hydrogen sulphate (1 : 8) in a test tube for some minutes (Fp \cong 300°C). Turn the test tube in a slanted position during the cooling of the melt; in this way you reach a larger surface of the melt. Dissolve the cold melt in water. Check carefully whether the mineral has dissolved completely. If the oxide contains any Nb, Ta, W, Sn or Zr, the white oxides of these elements remain undissolved; only the tungsten oxide is yellow. Such insoluble residue should be filtered and washed with dilute HCl. If ZrO₂ is present, also some TiO₂ may remain, but some Ti and all other dissolved elements will be found in the solution. - Tests for the elements Nb, W, Sn, and Zr in the filtered residue are described in the chapter on special tests for selected elements.

A survey of fusion melts is given on the following page.

- Carbonate melts convert all bivalent metals into carbonates; while aluminium, titanium, silicon and phosphates are converted to water soluble compounds: alkali aluminate NaAlO₂, sodium titanate Na₄TiO₄, sodium silicate, alkali phosphate.

- Adding KNO₃ to carbonate melts cause oxidizing conditions; Cr and Mn are converted to water soluble chromate K₂CrO₄ and manganate K₂MnO₄, respectively.

- Adding potassium oxalate $K_2C_2O_4$ to a melt causes reducing conditions; the formation of chromate is prevented. This may be important, if a stainless steel teaspoon is used as the vessel.
- The same as for carbonate melts holds true for alkali hydroxide melts, of course no carbonates are formed, but hydroxides and oxides of bivalent metals. Al, P, Pb, Si, Ti, V, W, and Zn compounds are converted to water soluble compounds.
- Borate glass melts dissolve most minerals, with the exception of sulphides. The crushed glass can be dissolved in HCl.
- The same is true for phosphate melts, but the silica is not dissolved in most cases.
- Acid sulphate melts converts oxides and phosphates into sulphates and alkali phosphates. W, Sn, Nb, Ta, and Zr remain as oxides, acid insoluble silicates are not attacked.
- The fusion with a mixture of ammonium chloride and ammonium nitrate is a neat substitute for aqua regia, a mixture of concentrated HCl and HNO_3 . It decomposes all minerals of the sulphide group, as well as acid soluble silicates and some oxides. The solid mixture is easier to store and to transport than the aggressive liquids.

A survey of fusion melts

Composition	Melting point	Vessel	Fusion type
Na_2CO_3	854°C	charcoal, Pt	carbonate
K_2CO_3	901°C	charcoal, Pt	carbonate
Na_2CO_3/K_2CO_3	710°C	charcoal, Pt	carbonate
Na_2CO_3/KNO_3	400°C	magnesia furrow, Pt, porcelain	carbonate, oxidizing
$Na_2CO_3/K_2C_2O_8$	710°C	stainless steel, charcoal	carbonate, reducing
$Na_2B_4O_7$ borax	742°C	charcoal, Pt, magnesia rod	borate glass
borax/soda	»960°C	charcoal, Pt, magnesia	borate glass
$NaPO_3$	628°C	magnesia rod, Pt	phosphate glass
NaOH	322°C	stainless steel, Ag	alkali hydroxide
NaOH/KOH	170°C	stainless steel, Ag	alkali hydroxide
KOH	404°C	stainless steel, Ag	alkali hydroxide
$KHSO_4$	210°C	glass, Pt, porcelain	acid, sulphate
$K_2S_2O_7$	»300°C	glass, Pt, porcelain	acid, sulphate
NH_4HSO_4	147°C	glass, Pt, porcelain	acid, sulphate
NH_4Cl/NH_4NO_3	»142°C	glass, porcelain	acid, chloride/nitrate (oxidizing)

Some hints about filtration

Most wet analysis requires the separation of a solution and a precipitate or a remaining solid phase. In modern laboratories this is achieved by means of centrifugation, but good centrifuges are expensive. Moreover, such device can scarcely be used in the field. Therefore you have to rely on the old filtration method. Normally you have to fold the filter paper first to a half circle, and then again to a quarter circle. Now the cone is opened, one layer on one side, three layers on the other side. The cone is placed in the filter funnel and completely wetted with distilled water, any surplus of water in the cone is discarded. The use of filter paper with 55 mm diameter and a funnel with a top diameter of 35 mm is recommended. Use a moderately fine textured filter paper. For a quantitative analysis washing of the filter residue is necessary and the washing liquid dilutes the filtrate. Since all quick assays are qualitative analysis it is mostly possible to proceed in a more simple way.

1. If you need only the filtrate you may proceed in the usual way; after obtaining enough filtrate, discard the filter paper with the precipitate. Sometimes spot tests require only a filter paper soaked with the filtrate. In such cases you place atop the test tube a piece of filter paper larger than the open end and upon that the test paper. Now press a big rubber stopper against this double layer and turn the test tube upside down. Thus both papers are soaked. - In some cases it is possible to place a drop of the fusion slurry on filter paper, now turn the paper and place a drop of the reagent solution on the wetted spot.

2. If only the precipitate is needed you may proceed in the usual way, discarding the filtrate and the washing liquid. For further handling the precipitate can be scratched from the filter cone by means of a spatula, or the cone is punctured and the precipitate washed down into a test tube. - Sometimes it is necessary to gain the insoluble remains of a fusion melt, but the water soluble part is of no interest. No funnel is needed, place the crushed melt in the depression of a spot test plate, a clay dish or a watch glass. Treat with water, stir, and remove the washing water by soaking with filter paper, a paper towel, or kitchen paper. Repeat this operation at least four times. The soaking paper should be discarded.

3. If the filtrate as well as the precipitate is needed, you must proceed in the usual way, but the washing liquid should be discarded.

Remember quick assays work with small amounts, thus usually only small volumes of liquid are used; normally no more than 1 - 5 ml are to be filtrated.

II Special part

In the following text some information is given about identification tests for the different elements. For most elements some remarks are added on the abundance and the distribution of the element within the geological cycle. Such information will be useful to anyone interested in minerals, since 99.3% of the crustal rocks are composed of only 12 chemical elements, namely O, Si, Al, Fe, Ca, Na, K, Mg, Ti, H, P, and Mn. The abundance of all other elements ranges within the order of some hundreds of ppm down to 0.001 ppb for the rarest elements. Remember one ppm = 10^{-6} applies to a gram per ton and one ppb = 10^{-9} means a milligram per ton. - The elements are arranged in the alphabetical order of their element symbols.

Ag silver

Silver is a rare element, it makes up only 0.1 ppm of the earth's crust. It is concentrated in hydrothermal veins, where it forms some of its own minerals such as proustite Ag_3AsS_3 and pyrargyrite Ag_3SbS_3 . But the main amount of silver is incorporated in galenite PbS .

Any silver content of a mineral enters the metallic bead formed on melting the mineral with soda on charcoal. It is recommended to transfer this bead to a clay dish and to melt it with borax with the oxidizing flame, if there is any likelihood that silver is present. Only silver and other noble metals like gold, platinum, and palladium will remain bead-shaped under such conditions.

The following procedure may be used to detect trace amounts of silver in galenite, sphalerite or fahlore. 1 - 2 g of the finely powdered mineral is dissolved in 2 ml of concentrated nitric acid in a small porcelain crucible, a beaker or a test tube and the solution is boiled till a dry state is achieved. After complete cooling add 10 ml of water and some drops of nitric acid and boil again. Transfer this solution to a test tube. Put a piece of filter paper on top and upon that a dry piece of filter paper prepared with the saturated solution of 5-(4-dimethylaminobenzylidene)-rhodanine in acetone. Now press a rubber stopper larger than the open end of the tube against this arrangement and turn the test tube till both papers are wetted. A red colour of the test paper indicates the presence of Ag, Cu, Hg, Au, Pt, or Pd. If the colour is caused by Cu or Hg it will vanish on the addition of some drops of diluted HCl.

Al aluminium

Aluminium is one of the eight main elements of the earth's crust. Unfortunately there is no quick and simple assay for the detection of Al, especially in silicates. The following wet procedure may be used if necessary.

A pinch of the powdered mineral and a pinch of potassium oxalate are melted with two flakes of NaOH on a stainless steel teaspoon. Only after complete cooling the spoon with the solid melt is placed in a plastic beaker. Add 5 ml water and warm gently for 5 minutes. This alkaline solution is filtrated using a plastic funnel. The filtrate should be dropped into a test tube with 5 ml of diluted HCl. The acid solution is boiled till a dry state is reached. Add 5 ml of water and a drop of diluted HCl. Put a piece of filter paper on the top of the test tube and upon that a dry paper which was previously freshly impregnated with a solution containing 0.1% of the ammonium salt of aurintricarboxylic acid and 1% of ammonium carbonate. Now press a rubber stopper larger than the open end of the test tube against the paper and turn the test tube till both papers are wetted. Remove the upper paper and hold it for a time over an open bottle containing ammonia solution. The presence of Al is indicated by the formation of a red colour, which is best visible on the dried paper.

Another possibility is to place a drop of the above-mentioned filtrate of the alkaline solution on a spot test plate or a watch glass. Further, add a drop of a 0.1% solution of sodium alizarin sulfonate in water. Acetic acid is now added dropwise until the violet colour disappears, and then a further drop of acetic acid. A flocculent red precipitate appears in the presence of Al. A blank test should give no colour or only a faint pink. The blank test is necessary since NaOH may contain traces of Al.

As arsenic

The arsenic content of the earth's crust is only 2 ppm. As may be present in sulphide minerals and in some gossan minerals. Any considerable content in sulphide minerals is easily detected by melting with soda on charcoal and by heating in an open tube. In gossan minerals arsenic mostly exists in the pentavalent state, thus no reaction is found by the open tube reaction. But it is detected in a closed tube if the mineral powder is mixed with soda and charcoal powder before heating.

To detect even very small amounts of arsenic in minerals, the dissolved mineral is mixed with conc. HCl and tin chloride. On heating this mixture renders a black precipitate of As.

Since arsenic is a very poisonous element, tests are sometimes needed for even trace amounts in different matter. Such sensitive tests mostly use the formation of the volatile arsine AsH_3 by the action of nascent hydrogen. In acid solutions antimony interferes due to the formation of volatile SbH_3 . This is avoided by the reduction in alkaline solution. Arsenates do not react, they have to be reduced beforehand to the trivalent state by the addition of hydroxylamin sulphate or with tin chloride in a strong acid hydrochloric solution: Place 3ml of water in a normal test tube, add a flake of potassium hydroxide and some pure aluminium turnings or some crumpled pieces of aluminium foil. Loosely plug the tube with cotton wool and then place a piece of filter paper moistened with a 20% silver nitrate solution on the top of the tube. Warm the tube gently to produce a regular evolution of hydrogen. After 2 to 5 minutes remove the filter paper and examine the paper that covered the tube for any coloration. Trace amounts of As in the reagents used may cause a faint brownish colour. Only now remove the cotton wool plug, add 1 ml of the test solution and replace the cotton wool plug with a new piece of the silver nitrate paper. Again produce hydrogen by gentle warming for the same time, then remove the paper and compare the two spots. The second spot will appear black if much arsenic is present. The black colour is due to metallic silver which is formed according to the formula $\text{AsH}_3 + 6 \text{Ag}^+ = \text{As}^{3+} + 6 \text{Ag}$

If only small amounts of arsenic are present the spot sometimes shows a yellow colour which is caused by the complex $\text{Ag}_3\text{As} \cdot 3\text{AgNO}_3$. - Only trivalent arsenic reacts, arsenates should be reduced beforehand! - Ensure good ventilation since arsine AsH_3 is very poisonous!

Au gold

Gold is well known to everybody, though it is one of the rarest elements of the earth's crust with a content of only 5 ppb. It occurs as a trace amount in the lattice of some sulphide minerals, especially in pyrite and chalcopyrite. Native gold is also found in small quantities in crystalline schists and in granitic rocks, mostly associated with quartz. It collects in the sands and gravels which have resulted from the disintegration of rocks and mountain masses that have contained gold. Placer deposits formed in this way gave rise to the famous gold rushes in the 19th century. Owing to its weak chemical affinity gold does not form very stable compounds in nature, only some rare tellurides are known as gold bearing minerals. Placer gold sometimes contains some silver and traces of copper and iron.

Any sulphide ore may be dissolved by the fusion of a pinch of the powdered mineral with a surplus of the mixture of one part ammonium chloride with 2.5 parts of ammonium nitrate. The cooled melt is dissolved in some drops of hydrochloric acid. A drop of this solution is brought to a filter paper and a drop of a solution of SnCl_2 is added. A red colour indicates the presence of gold.

Another procedure may be used in testing for gold in panning residues. Transfer the wet heavies into a glass jar or beaker and decant the water. Now add two pinches of calcium hypochlorite and 3 ml of hydrochloric acid and cover the vessel with a watchglass. **Ensure good ventilation**, since chlorine evolves due to the reaction $\text{HOCl} + \text{HCl} = \text{Cl}_2 + \text{H}_2\text{O}$. The chlorine dissolves the native gold and soluble HAuCl_4 is formed. Wait for 10 minutes, add 10 ml of diluted HCl and shake cautiously. Now transfer some of the solution to a test tube and boil till no odour of chlorine remains. Some drops of a solution of SnCl_2 are now added to the cooled solution. The presence of gold is indicated by a purple colour due to a colloidal solution of gold, called "purple of Cassius". If there is only a tiny amount of gold the colour shows sometimes a bluish purple. Please refrain from weeping for joy in case of a positive reaction: this reaction is extremely sensitive. Already the 1/1000 part of a milligram of gold per millilitre causes a red colour.

If no calcium hypochlorite is available it is possible to use chloride of lime CaOCl_2 instead. The use of sodium hypochlorite solution is also possible. Such a solution is sold in some countries by pharmacies as "eau de javelle" for bleaching and disinfection. - Gold can be dissolved also in bromine water. - Add a surplus of solid sodium thiosulphate to any remaining solution in order to destroy any free chlorine or bromine!

B boron

The mean value for boron in the earth's crust is 3 ppm. In igneous and metamorphic rocks boron is mainly concentrated in the mineral tourmaline and in low amounts in micas. Silicates may be proved for boron by mixing the mineral powder thoroughly with three parts each of ammonium hydrogen sulphate and fluorite. The mixture is heated on a magnesia furrow with the oxidizing flame of a butane torch. The hydrofluoric acid liberated by the mixture attacks the mineral, forming volatile boron trifluoride BF_3 . This compound gives a green flame coloration, which is usually of only momentary duration.

A test with turmeric paper may be used to screen water soluble or acid soluble minerals for any boron content. Turmeric paper is moistened with a dilute hydrochloric acid solution of the mineral, and then dried at 100°C on the outside of a test tube containing boiling water. In the presence of boron the paper assumes a reddish-brown colour, and this is changed to inky-black by moistening with ammonia. The test is only disturbed by ferric compounds, Ti, and Mo which cause a brown colour already in acid solution, but these elements are usually absent in minerals which are soluble in dilute hydrochloric acid. However, the brown colour produced by these compounds, unlike that due to boric acid, does not change to bluish black on the addition of alkali.

Ba barium and Sr strontium

Both alkaline earth elements are quite common in crustal rocks with 400 ppm Ba and 460 ppm Sr. They are similar in their chemical behaviour, but most barium compounds are less soluble. The barium or strontium content of a mineral has to be converted to carbonate for an assay. This is done by the fusion of a 1:1 mixture with soda on charcoal or on a magnesia

furrow. Dark minerals should be fused with potassium carbonate instead of soda. The resulting bead or residue is treated with hot water in a clay dish or the depression of a spot test plate. The water is soaked off with filter paper. This procedure is repeated twice, the residue contains any barium or strontium as carbonate.

To test the flame coloration this residue is dissolved with some drops of hydrochloric acid and the solution is brought to the flame by a platinum wire loop. Strontium chloride is more volatile than the barium compound. Sr produce a crimson colour, barium causes a yellowish green. It is not possible to detect minor amounts of Ba in this way, since the flame coloration can be seen only if at least 15 mg Ba are present per millilitre. The use of a spectroscope is recommended; Ba shows green lines at 524.2 nm and 513.7 nm, together with other green and red lines; Sr shows many red lines, an orange line at 604.5 nm and a blue one at 460.8 nm.

In order to detect such minor amounts of barium the fusion residue is dissolved in hot acetic acid in a small test tube. Now add some solid sodium acetate and some drops of a concentrated potassium dichromate solution. A yellow precipitate of BaCrO_4 is formed if Ba is present. The hot mixture is filtrated into another test tube and some drops of a saturated solution of ammonium sulphate are added to the clear solution. The presence of Sr is indicated if any white precipitate of SrSO_4 is produced or a distinct turbidity is seen by looking into the tube in the axial direction.

Be beryllium (French: glucinium)

With 2 ppm in the earth's crust Be is one of the rarer elements. Beryl is the main occurring mineral, but many other minerals, especially silicates containing Be are known. - Any soluble Be-compounds are poisonous, they are formed by fusion reactions.

The finely powdered mineral is mixed with the same amount of soda and transferred to charcoal. Moisten with one drop of water and melt the mixture. This melt is well ground and treated with a flake of NaOH and 3 ml water in a test tube for some minutes without heating. Add some drops of a freshly prepared solution of quinalizarin to the filtered solution. The violet colour changes to blue if Be or Mg is present. If only Mg is present this blue vanishes on the addition of bromine water. - To prepare the quinalizarin solution please touch some solid quinalizarin with a clean spatula, hold it vertical and dissolve the tiny amount adhering to the spatula in ammonia solution. - The bromine water is prepared in the following way. Dissolve a pinhead of potassium bromide KBr and a pinhead of potassium bromate KBrO_3 in some drops of water in a normal test tube, add some drops of hydrochloric acid and close the tube by a cork stopper soaked with molten paraffin. Bromine is evolved by the reaction:

$$\text{BrO}_3^- + 5 \text{Br}^- + 6 \text{H}^+ \Rightarrow 3 \text{H}_2\text{O} + 3 \text{Br}_2$$
 Now add some water and a spatula of solid sodium acetate. This preparation should be done only in a fume-cupboard or in open air if a good ventilation is ensured! To destroy any bromine add a spatula of sodium thiosulphate to the solution before discarding it.

Grains or small fragments of beryl can be identified without fusion. They should be etched with hot NaOH solution in a test tube, washed with water, followed by boiling with an aqueous solution of quinalizarin. The surface of beryl achieves an intense blue colour.

Bi bismuth

With a value of only 0.2 ppm in the earth's crust bismuth is a rare element, but it is concentrated in sulphide ores and their oxidation products.

Any considerable amount of Bi produces a white to yellow zoned sublimate on charcoal by heating with an oxidizing flame. Only high amounts cause a brittle, reddish white bead on heating with soda. A 1:1 mixture of sulphur and potassium iodide is placed on the sublimate and ignited, followed by a short repeated application of the oxidizing flame. The sublimate changes its colour to red due to the formation of bismuth iodide. Any yellow sublimate occurring during this treatment points to the simultaneous presence of lead.

Br bromine

Bromine is only found in salt minerals or brines. Seawater contains approximately 65 ppm. Br is sometimes considerably enriched in salt lakes like the Dead Sea which contains 3 %. For a sensitive test, place some crystals of potassium permanganate and a small crystal of copper sulphate in a small beaker or on a spot test plate and add two drops of the slightly acidified solution to be tested. Warm this arrangement without boiling. Free bromine is liberated and will blue wetted iodide-starch paper placed atop the vessel. The test fails if more than traces of iodide are present.

C carbon

Carbonates are dissolved by hot diluted HCl in a test tube, calcite and aragonite already dissolve in the cold acid. To detect the CO₂ evolved by effervescence hold a glass rod with a drop of a saturated solution of Ba(OH)₂ or Ca(OH)₂ over the tube. A white colour of the drop indicates CO₂. The colour is best seen if the end of the glass rod is coloured black or red by coating with sealing wax. The CO₃-content of some members of the scapolite series may not be detected by this method.

Any organic matter is destroyed by heating to a mild red glow with access to air, graphite needs a somewhat higher temperature and diamond is oxidized only at temperatures beyond 800° C. Carborundum SiC may be found sometimes in placer mineral concentrates after a century of intense industrial use of this mineral. It is not oxidized by oxygen even at high temperatures; but it is readily decomposed by the fusion with KOH on a stainless steel spoon with access to air:



Ca calcium

The presence of Ca in acid soluble minerals like zeolites or phosphates can be identified by the following procedure. Place a small grain on a microscopic slide and wet it with a drop of 30% sulphuric acid. Now place a cover glass upon the grain. By means of a microscope or a good lens it is possible to observe the slow precipitation of gypsum; needles, twinned crystals and sometimes well shaped crystals are formed. In most cases the grain resembles the shape of a sea urchin after some time.

Minerals which are insoluble in acids should be fused after grinding with soda 1:1 on charcoal. The residue is treated with hot water in a clay dish or on a spot plate. The liquid is soaked with filter paper. Treating with water and soaking off the dissolved matter is repeated twice. The remaining carbonate is dissolved with some drops of diluted HCl. If neither Sr or Ba is present, this solution imparts a yellowish-red colour to the flame if it is brought to it by a platinum wire loop or a magnesia rod. The use of a spectrometer is recommended: Ca produces a red line at 622 nm and a green one at 533 nm, both lines in an equal distant to the

yellow sodium line if a prism spectrometer is used. - For a wet identification NH_4OH is added to the solution of the carbonate in HCl . After filtration a part of the solution is tested for Ba and Sr by the addition of ammonium sulphate. If no precipitate occurs calcium oxalate is precipitated in the hot solution by the addition of ammonium oxalate. If Ba or Sr is present it is thrown down by the addition of some solid ammonium sulphate, now boil the solution and filtrate after some minutes. Again precipitate Ca as the oxalate in this filtrate. Mn, Co, and the rare earth interfere since they may produce a precipitate under these conditions as well. But the presence of Ca can be assured by dissolving the filtrated oxalate in HCl and looking for the flame coloration.

Cd cadmium

Cadmium is a rare element with an average content of 0.2 ppm in the earth crust. The main amount of Cd is isomorphically incorporated in zinc ores; e.g. sphalerite often contains 1 to 0.1 % Cd. Nevertheless pure cadmium ores sometimes occur especially in veins of low hydrothermally formed deposits.

Since Cd is more volatile than Zn any zinc ore should be treated first only shortly with a reducing flame on charcoal. In this way it is possible to detect even trace amounts of Cd by the typical darkbrown to yellow changing sublimate and the peacock-like iridescence of the outer part of the sublimate. Try the first assay with a small fragment only, and try again with a mixture of soda and charcoal powder if no clear result is achieved.

Ce cerium, the rare earth elements, and Th thorium.

Scandium, yttrium, lanthanum, and the 14 elements following lanthanum are known as the rare earth elements (REE). Only these 14 elements with the atomic numbers 58 to 71 are called lanthanides. All REE are very similar in their chemical properties and hence difficult to separate from each other. They are all trivalent, only cerium can achieve a tetravalent state. It should be mentioned that europium and samarium may enter the divalent state only under strongly reducing conditions, while praseodymium oxide enters the tetravalent state if it is glowed in the presence of air. Thorium is the first element of the actinide group. Its chemical behavior is very similar to the REE, though Th only occurs in the tetravalent state. The name REE is somewhat misleading: the average amount of cerium in the earth's crust is similar to copper (45 ppm), even the less abundant heavy lanthanides each reach about 1 ppm. However, a considerable part of the overall amount of the rare earth elements is incorporated as mixed crystal ions in some main rock-forming minerals such as micas, feldspars, and pyroxenes. - The REE and thorium also form special minerals, but these minerals are mostly disseminated as fine grains in plutonic rocks. Therefore useful mineral deposits are only found in pegmatites or as placer minerals.

Two subgroups are divided, the cerium group with mainly Ce, La, Nd, Pr with the minerals monazite CePO_4 , allanite which is a member of the epidote group (sometimes called by the old name orthite), cerite is a Ce,Ca-silicate, fergusonite, samarskite and pyrochlore are oxides containing niobium, tantalum, and titanium; while bastnaesite and parisite are fluorine-containing carbonates.

The yttrium group comprising Y and the heavier lanthanides are found especially in the minerals xenotime YPO_4 , gadolinite which is a silicate containing Fe and Be, and in some oxides together with Nb, Ta, Ti, Fe, like euxenite and yttrantalite. Many of these minerals also contain some Th or U. Any such trace amounts of Th and U are unfavourable because of

radiation safety conditions in plant processing. Florencite $CeAl_3(OH)_6(PO_4)_2$ and other REE bearing minerals of the crandallite group are mainly associated with the weathering horizon of carbonatites. - Scandium is concentrated in Thortveitite $(Sc,Y)_2Si_2O_7$, a pegmatitic mineral and in bazzite, a Sc-analogue of Beryl.

All rare earth elements and Th form oxalates which are insoluble in weak mineral acids if no alkali ions are present. In this way REE and Th can be separated and identified using the wet method. The following procedures may be used to obtain a solution appropriate for the oxalate precipitation. Finely powdered specimens should always be used.

Carbonates/fluorides are heated with some drops of concentrated sulphuric acid till the formation of white vapours of sulphuric acid ceases. The test tube or clay dish used is somewhat etched by the formation of HF, but this does not matter. A drop of diluted HCl is added to the cooled substance, which is now dissolved in approximately 8 ml of distilled water.

Phosphates are mixed with five parts of dry sodium carbonate and melted in a clay dish. The dish with this melt is transferred to a small beaker and boiled with 10 ml of water for some minutes. The residue is isolated by filtration and washed twice with hot water. The filtrate is discarded, it contains sodium phosphate and the surplus of sodium carbonate. The residue is dissolved in 2 ml of diluted HCl.

Silicates can be dissolved in a mixture of conc. HCl and conc. HNO_3 in a test tube. Evaporate to a nearly dry state, add some drops of HCl and gain a clear solution by filtration. Already glowed silicates and bazzite are insoluble in aqua regia, they may be handled like phosphates.

The clear solutions achieved in one of the ways described above can be used for oxalate precipitation, any turbid solutions should be filtrated beforehand. Now dilute this solution with twice the volume of distilled water and add some solid oxalic acid. Mix thoroughly and heat for some minutes without boiling. All rare earth elements and Th are precipitated as white oxalates, which should be isolated by filtration.

Oxides like yttrantalite and pyrochlore are mixed with eight parts of $KHSO_4$ and melted on a clay dish or on a magnesia furrow. The melt is dissolved in approx. 10 ml of cold water plus some drops of diluted HCl. Ammonia solution is now added till the well stirred solution gives a basic reaction. The precipitate is isolated by filtration and washed twice with water. A suspension of this precipitate is treated with a warm oxalic acid solution till only a white heavy precipitate of the REE oxalates remain.

Special tests for different REE elements:

- A part of the oxalates is carefully heated on a magnesia furrow to a low red glow. If more than 1% of Pr is present, a more or less brown colour is obtained. Pure CeO_2 has a yellowish tinge, but most REE oxides are white.

- Another part of the oxalate precipitate is dissolved in some drops of concentrated nitric acid. The solution is yellow only in the presence of Ce. This yellow colour vanishes on the addition of hydrogen peroxide. Now add some solid ammonium acetate and heat the solution. The formation of a brown suspension indicates the presence of cerium.

- Being radioactive, thorium may be detected using a Geiger counter if available. If not, some drops of potassium iodate solution are added to a part of the hydrochloric solution used for the precipitation of the oxalates. A white precipitate of $\text{Th}(\text{JO}_3)_4$ points to the presence of Th.

- A content of La can be detected in the following way. Part of the oxalate precipitate is glowed on a magnesia furrow and then boiled with diluted acetic acid in a test tube. After filtration add some drops of a solution of iodine in potassium iodide solution. Now add ammonium hydroxide dropwise till the colour is only faintly brown. The presence of lanthanum is indicated by the formation of a blue solution or a blue precipitate on heating.

Some lanthanide elements show strong absorption bands in the visible range. These are the elements neodymium, praseodymium, samarium, europium, dysprosium, holmium, erbium, and thulium. If a solution of these elements is held before the slit of a spectroscope directed towards a strong light, dark bands may be seen interrupting the continuous spectrum. The clear fusion solution may be used for this purpose. A small, cellphone-sized, portable spectroscope adapted for such inspection is still on sale at a price of approx. 360 €. Prominent absorption bands are located about the middle of the green, and on the border between yellow and red.

For some decades now, there has been an increasingly brisk demand for many rare earth elements owing to a multitude of technical applications. Most spectacular was the rise of the price of gadolinium during the 1980s, due to its extreme neutron capture ability.

Cl chlorine

Seawater contains 1.9% chlorine. Only few rock forming minerals show Cl as a lattice element, like scapolite, sodalite or apatite; but fluid inclusions are mostly filled with chloride brines. Chlorine is often a member of minerals of the exogenic cycle. There are three rather sensitive tests for Cl.- Take care to avoid any contamination with sweat!

- The fine ground mineral is mixed with the third part of copper oxide, wetted with distilled water to produce a slurry and now spread on charcoal. Blow gently with a fanning flame to dry the mixture, then heat the dry mixture as high as possible and watch the flame colour. Disregard any green colour, only an azure-blue colour indicates the presence of Cl!

- The mineral powder is fused with KHSO_4 in a small test tube. The escaping HCl is indicated by the white fume of NH_4Cl which is formed in the vicinity of a drop of ammonia solution held atop. A folded strip of filter paper may be placed on the rim of the test tube and wetted with the ammonia solution.

- The mineral powder is mixed with some pyrolusite and heated with some drops of concentrated sulphuric acid in a test tube. Free chlorine is thus formed and a wetted iodine starch paper placed atop will change to blue.

Chlorargyrite AgCl produces a positive result only with the first test. - Any bromides or iodides, with the exception of AgBr and AgI , produce a mixture of the free element and SO_2 on fusion with KHSO_4 .

Co cobalt

With a content of 23 ppm in the earth's crust cobalt is a common element which is mainly found in sulphide ores. - Make a bead of NaPO_3 on a magnesia rod. Take up some of the roasted mineral powder by the hot bead and glow intensively with the oxidizing flame. The presence of cobalt is indicated by a smalt-blue colour, this colour changes towards violet if Mn or large amounts of Fe and Ni are present.

Another assay can be made on a spot test plate. A pinhead of the mineral powder is dissolved in nitric acid in a depression and dried. Approximately the same amount of solid NH_4SCN ammonium thiocyanate and sodium thiosulphate is added and intensively rubbed with a glass rod. If Co is present a blue colour evolves; any Ni present will produce a greenish tinge. If a drop of distilled water is added, the colour vanishes, but returns on warming the mixture.

Cr chromium

The chromium content of 200 ppm in the earth's crust marks Cr as a very common element. It is largely removed from a magma in the early stage of crystallization as chromite FeCr_2O_4 , which is the main mined mineral. A considerable number of other Cr-bearing minerals are known, many of them show a peculiar green or red to pink colour. In minerals chromium is normally trivalent, it enters the hexavalent state only at high temperatures in an alkaline environment and in the presence of oxygen. Such natural conditions - for instance, burning bituminous limestones - are quite rare; therefore minerals containing the chromate ion CrO_4^{2-} are also rare.

Mostly Cr is changed to yellow sodium chromate Na_2CrO_4 by the fusion of Cr bearing minerals with a mixture of soda and potassium nitrate on a magnesia furrow or a magnesia rod. The reaction was described already in the general part. If manganese is also present the melt is green. In such rare cases the melt is dissolved in a small beaker in a few millilitres of distilled water by boiling. Now acetic acid is added till the solution is acid and boiling is repeated. Wait till any precipitate has settled and pour the clear solution into a clay dish. Put a pinhead-sized crystal of lead acetate in this solution and stir with a small glass rod. Yellow lead chromate settles at the bottom. The precipitate is isolated by decanting the solution and washing once with water. By melting with sodium metaphosphate or microcosmic salt a green colour is achieved.

Some silicates and oxides do not react with the molten soda-nitre mixture. Such minerals are melted beforehand with a surplus of a 1:1 mixture of soda + borax on charcoal. The powdered bead is molten with the same amount of nitre KNO_3 . - Spinel and pyrope coloured red by their Cr content turn opaque black on heating; during cooling the colour changes via yellowish green back to red. The same is true for silicates with a red colour due to the presence of Cr + Fe. Do not try this test with any valuable minerals or gems, the specimen may crack or stay opaque!

Cs cesium and Rb rubidium

The amount of both alkali metals in the earth's crust is 1 ppm for Cs and 120 ppm for Rb. Due to its large ionic radius Cs is concentrated in pegmatites where the rare mineral pollucite $(\text{Cs},\text{Na})_2\text{Al}_2\text{Si}_4\text{O}_{12}\text{H}_2\text{O}$ is sometimes found. The considerable amount of Rb enters the

position of the K^+ ion in feldspars and micas, in pegmatites, thus the ratio Rb/K is somewhat increased, but no minerals with Rb as a main constituent are formed.

Both elements have to be identified by their flame emission spectra using a spectroscope: a red line at 789 nm and a violet one at 421.5 nm indicates Rb; Cs shows a blue line at 458 nm.

Cu copper

Nearly all copper minerals can be dissolved by melting with a mixture 1:2.5 of ammonium chloride and ammonium nitrate on a magnesia furrow. This melt is dissolved in a rather concentrated solution of ammonium carbonate, a deep blue colour indicates the presence of copper. Even small amounts are detected in this way if the solution is transferred to a small test tube; by looking in the axial direction from top to bottom the colour perception is independent of the height of the solution column. Identification is impaired by the presence of Ni, because Ni also causes a light-blue coloured solution with ammonium carbonate. To avoid any uncertainty some of the blue solution is brought to an acid reaction by acetic acid, and a solution of potassium ferrocyanide $K_4Fe(CN)_6$ is added. A brown precipitate indicates the presence of copper. Zinc ferrocyanide paper may be used instead.

It should be mentioned that many secondary copper minerals are soluble in ammonium carbonate solution, e.g. malachite, azurite, all basic sulphates, phosphates, chlorides, and the copper silicates chrysokolla and diopside. The dissolution rate of Cu-silicates is enhanced by the addition of ammonia solution.

F fluorine

Fluorine is a very common element with 700 ppm in the earth's crust. In igneous rocks considerable amounts of F enter the position of OH ions, especially in micas, amphiboles and in apatite. Fluorite is an ubiquitous mineral in hydrothermal veins.

A test for fluorine by heating with sodium phosphate was already described in the general part. - For another test, place 1 ml concentrated sulphuric acid in a new test tube, add some minute crystals of potassium chromate or potassium dichromate. On boiling, the mixture runs evenly over the glass surface. Only now a few grains of the mineral are added. On rotating the tube, an uneven flow of the sulphuric acid, with formation of unmoistened areas, indicates the presence of fluorine. Silicic and boric acid strongly diminish the sensitivity of the test. - The test tube can be further used for any other test after rinsing with plenty of water, but not for this test for F! Firstly pour off the cooled acid into plenty of water, then rinse the test tube!

Fe iron

Sulphides are molten with a 1 : 2.5 mixture of ammonium chloride and ammonium nitrate, oxides are molten with potassium hydrogen sulphate, but silicates have to be fused with the same amount of soda on charcoal. Any residue of the soda melt is dissolved in HCl, the other fusion melts are dissolved in water, and two drops of nitric acid are added. On the addition of potassium ferrocyanide $K_4Fe(CN)_6$ a blue precipitate is formed. - The test is impaired by fluoride ions, but sulphide minerals do not contain fluorine and any fusion with potassium hydrogen sulphate expels the HF. Therefore disturbance is only likely to occur if F-containing silicates are tested for Fe, e.g. amphiboles. In such rare cases the residue of the soda melt may be fused with potassium hydrogen sulphate before dissolution in water.

In minerals iron may enter the bivalent state called ferrous iron, as well as the trivalent state called ferric iron. If both ions Fe^{2+} and Fe^{3+} are present in the same lattice of an oxide or a silicate, the crystal assumes a dark to black colour; such minerals are called mafic minerals, e.g. biotite, magnetite, augite, and many amphiboles. The presence of ferrous iron in a mineral can be detected in the following way. Mix the finely powdered mineral with three times its volume with dry sodium tetraborate $\text{Na}_2\text{B}_4\text{O}_7$ in a large closed glass tube and fuse the mixture. While hot, crack the glass containing the fusion by touching it with drops of water, break off the end, and transfer to a test tube containing 3 ml concentrated HCl. Boil for about a minute, then dilute with 5 ml water. Divide the solution into two parts and test one for ferrous iron with a solution of the red salt of potassium ferricyanide $\text{K}_3\text{Fe}(\text{CN})_6$. A blue precipitate of Turnbells blue is decisive for Fe^{2+} . The other part may be tested for Fe^{3+} with a solution of the yellow salt of potassium ferrocyanide $\text{K}_4\text{Fe}(\text{CN})_6$; this blue precipitate is called Prussian blue.- The tests are very decisive, any oxidation resulting from contact with air during the fusion and the dissolution, which cannot be wholly avoided, are so trifling that practically they may be disregarded.

Hg mercury

The mercury content of the earth's crust is 0.5 ppm, the native element and HgS cinnabarite is found in hydrothermal deposits of very low temperature origin. Small amounts of Hg are incorporated in some sulphides containing Sb and As.

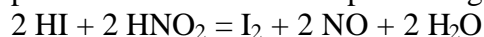
Any considerable Hg content can be detected by heating the mineral powder with six parts potassium oxalate in a closed tube. Metallic mercury is deposited at the cold parts, use a lens for observation. The sensitivity of this test is considerably enhanced if you put a tiny piece of gold foil adhering to an iron wire in the upper part of the closed tube. The yellow colour of the gold is turned white by any mercury vapour formed during the heating.

To detect even traces of Hg the mineral is fused with an 1 : 2.5 mixture of ammonium chloride and ammonium nitrate and this melt is dissolved with a small amount of water. Disregard any white precipitate. Now a small piece of aluminium foil is rubbed partly with a fine abrasive paper, washed with distilled water and etched with concentrated NaOH on a watchglass until a uniform evolution of hydrogen occurs at the roughened place. After rinsing with water and rubbing with a piece of cotton wool moistened slightly with very diluted NaOH, the foil is dried. A drop of the test solution is placed on the prepared aluminium foil, after 5 minutes the liquid is taken up with filter paper. After some minutes, a white growth of aluminum hydroxide appears on the foil if mercury is present.

I iodine

Seawater contains 0.05 ppm iodine, higher amounts are sometimes found in salt lakes and brines. Small amounts may occur in volcanic exhalations. NaIO_3 sodium iodate and NaIO_4 sodium periodate were reported from the sodium nitrate deposits of the Atacama Desert in Chile. Considerable amounts of iodine compounds are incorporated into organisms.-

A pinch of starch is mixed with cold water, this suspension is poured into 200 ml boiling water. Soak filter paper with the cooled solution and dry it. Such prepared paper is treated in succession with single drops of diluted acetic acid, test solution, and diluted solution of potassium nitrite. A blue spot or ring shows the presence of iodine, due to the reaction:



K potassium

Acid soluble minerals are brought to the flame as a slurry with concentrated hydrochloric acid with the magnesia rod or the platinum wire loop. Use a cobalt glass. A violet flame indicates K, but any Li impairs the detection.

Acid insoluble silicates are mixed with the same amount of CaF_2 and a drop of concentrated sulphuric acid, this slurry is taken up with the Pt wire loop. The use of a spectroscope is recommended, a double red line at 766 nm and 770 nm indicates K.

For a wet method the mineral is mixed with the same amounts of soda and borax, and molten to a clear bead on charcoal or a platinum wire loop. This bead is dissolved in a small amount of diluted HCl. A drop of the clear solution or the filtrate is placed on a microscopic slide and treated with a minute amount of solid sodium cobaltinitrite $\text{Na}_3\text{Co}(\text{NO}_2)_6$. A yellow precipitate or a turbidity indicates the presence of potassium. Unfortunately, much lithium may also produce a precipitate!

Li lithium

With a 30 ppm content in the earth's crust lithium is not a rare element. Due to its small ionic radius Li is only partly incorporated in the lattice of rock forming minerals where it enters the lattice position otherwise filled by magnesium. Remaining amounts in the magma form individual minerals like lepidolite, spodumene, amblygonite, and petalite in some pegmatites.

Lithium chloride is the most volatile of all alkali and earth alkali chlorides. The immediate formation of a carmine red colour on the border of the flame is characteristic for the presence of lithium. If sodium is present the flame should be observed through a cobalt glass. Phosphates can be wetted with concentrated HCl and then tested for the flame coloration, in such cases the pale blue-green colour typical for phosphoric acid is observed simultaneously.

Acid insoluble silicates should be tested after mixing with calcium fluoride and concentrated sulphuric acid. - It is another possibility to mix the mineral powder with BaCl_2 and to melt this mixture on a platinum wire loop, the red colour of Li will appear only at the tip of the flame. - Normally the use of a spectroscope is not necessary; Li causes a red line at 670.8 nm.

Mg magnesium

Admittedly, the identification of low amounts of Mg in minerals by means of a quick assay is a difficult task. A lot of elements disturb almost any test, these elements have to be separated by precipitation at first. Nevertheless there are two reactions which might fit at least for some frequent Mg-minerals.

- Diphenylcarbazide is a dyestuff which gives a deep red-violet coloration to magnesium hydroxide. Many other elements also react with the reagent, therefore the reaction is done as a staining reaction on the solid sample. Only more or less white minerals can be used, e.g. carbonates, oxides, phosphates. An alcoholic solution of diphenylcarbazide is prepared: three pinches of diphenylcarbazide are dissolved in 5 ml ethanol and 1 ml of water and a flake of NaOH is added. The solution can be stored in a plastic bottle for some weeks. Two drops each

of this solution are placed in two depressions of a spot test plate. Now the test plate is slightly warmed and a pin-head sized sample is placed in each depression. One of the mineral specimens has previously been heated to a red glow while held by a forceps. After 5 minutes the solution is soaked away with filter paper and replaced by hot water, and this method of washing is continued till no more colouring matter goes into the water. Magnesite and slightly soluble minerals like brucite are stained red-violet. Dolomite and other carbonates bearing considerable Mg are stained only if they have been preheated.

- White silicates like chrysotile and talc are mixed with sodium carbonate and potassium carbonate and fused on a platinum wire loop. The bead is dissolved in hydrochloric acid on a spot plate or a clay dish and a drop of NaOH is added. Now add a drop of the following solution: a tiny amount of p-nitrobenzeneazo- α -naphthol is dissolved in 10 ml water together with 3 flakes KOH. A blue precipitate or colour, compared with a blank, indicates magnesium. Bear in mind that aluminium and iron seriously interfere with this test.

A somewhat laborious method can be used for mafic minerals. Acid soluble minerals are dissolved in concentrated HCl; acid insoluble minerals are molten with a flake each of NaOH and KOH on a stainless steel spoon. Only after complete cooling the solid melt is dissolved with as little water as possible, any remnants sticking to the spoon are dissolved with nitric acid. All liquids are combined and 1 ml conc. HCl and a drop of diluted sulphuric acid are added. Now add a pinch of solid ammonium chloride and ammonia solution till a basic reaction is assured. Dissolve half a pinch each of solid sodium sulphide and ammonium carbonate in this solution and boil it. From the hot filtrate of this mixture Mg is precipitated by adding some drops of an ammonia solution and a hot solution of $\text{NH}_4\text{NaHPO}_4$ (microcosmic salt). A crystalline, quick settling precipitate of struvite $\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$ indicates magnesium. The precipitate is insoluble in conc. NH_4OH and should not be coloured brown by the addition of hydrogen peroxide.

Mn manganese

With 1% of the earth's crust manganese is a very common element. In igneous rocks Mn^{2+} mostly replaces ferrous iron. Under surface conditions it enters quickly the tri- and tetravalent charge and insoluble oxides are formed.

The mineral powder is mixed with 4 parts of soda and 1 part of KNO_3 . Melt this mixture on a clay dish. A green to blue-green colour indicates Mn; the test is very sensitive, there is no disturbance by Cr, which turns to chromate by this oxidation melt. Add some water and pour the green liquid into a small test tube. On acidifying with acetic acid the solution turns violet due to the formation of permanganate, simultaneously brown flocculi of MnO_2 settle. After some time the violet colour ceases and the brown precipitation increases. If any Cr is present, the remaining liquid shows a yellow colour.

Black manganese oxides are easily dissolved by sulphurous acid H_2SO_3 . The black streak of such oxides on a streak plate is wetted with a drop of HCl and a very small crystal of sodium thiosulphate is added. The black streak vanishes almost immediately.

Mo molybdenum

Molybdenum is a rare element, the average amount in the earth's crust being 1 ppm. The most frequent molybdenum-containing mineral is molybdenite MoS_2 , it is easily identified by its low hardness, its high lustre and the colour. A flake held to the oxidizing flame imparts a yellow-green colour to it. Molybdenite dissolves in hot concentrated nitric acid; it is also possible to dissolve it by melting with a mixture of ammonium chloride with ammonium nitrate.

Other Mo-minerals like molybdates may be dissolved in the same way. For further identification a drop of the test solution and a drop of ammonium thiocyanate solution are placed on filter paper previously moistened with dilute hydrochloric acid. Only in the presence of iron does a red fleck appear which disappears on adding two drops of stannous chloride solution. In its place a brick-red colour of the Mo(III)-thiocyanate complex appears, if Mo is present. The stannous chloride solution is prepared by dissolving a pinch of SnCl_2 in 1 ml concentrated HCl and adding 2 ml water.

Na sodium

The yellow flame coloration of sodium is extremely sensitive, only a longlasting intense yellow flame colour indicates a reasonable amount of Na. - Acid insoluble silicates are mixed with CaF_2 and a drop of concentrated sulphuric acid or ammonium hydrogen sulphate for the flame test.

Nb niobium, also called columbium, and Ta tantalum

The average amounts in the earth's crust are 24 ppm Nb and 2 ppm Ta. Both elements are almost invariably associated, because they are isomorphous with one another, and their chemical behaviour is very similar. However, the Nb/Ta ratio may vary in a wide range in different deposits. They are found in granite pegmatites constituting the acid-forming elements in some oxides, like columbite, fergusonite, samarskite, and euxenite. Pyrochlore is often associated with carbonatites. All these minerals have a hardness of $\cong 6$, the streak colour is brown to black. Other lattice elements are Fe, Mn, Ti, and very often Y, Sn, U, and Th. Moreover, niobium is occasionally found in some silicate minerals in nepheline syenites, e.g. eudialyte.- Ores containing columbite/tantalite are sometimes called "coltan" in Africa.

Oxides are fused with 8 parts of potassium hydrogen sulphate in a test tube. When the decomposition is complete, as shown by the disappearance of black particles, the tube is inclined and turned while cooling, causing the melt to solidify as a thin crust on its sides. In this way it is more readily dissolved on subsequent treatment with cold water, which requires considerable time. The white residue of niobic and tantalic oxides is collected on a filter and washed with diluted HCl, followed by washing with water and finally with ammonia solution. A portion of this residue is boiled in a test tube with conc. HCl and granulated tin, a blue colour indicates Nb. If much Nb is present the blue colour remains if water is added, if the colour fades, only traces of Nb are present. In such cases the mineral may contain mainly Ta. - Some textbooks call the fading of the blue on the addition of water a test for Ta; in fact it is not possible to reduce Ta oxide in this way. But indeed minerals containing Ta mostly also have some traces of Nb. -

Silicates must be fused with the fivefold amount of borax on a magnesia rod. Make two or three beads and, with a hammer blow, crush the beads together with the broken tips of the

magnesia rods, wrapped in paper. This powder is dissolved with 2 ml concentrated HCl in a test tube till the solution is nearly clear; of course the magnesia rod pieces will remain. If Nb is present, a blue colour will appear on the addition of tin to the boiling solution. If Ti is present as well as Nb, the colour turns violet first and later blue.

White Nb_2O_5 can be converted quickly into the dark brown Nb_2O_3 with a reducing flame. This change in colour is best observed if a water suspension of the filter residue is dried on the margin of a magnesia furrow and then heated strongly. The flame is then directed on the mass in such fashion that the inner, reducing cone impinges on the solid. The darkening is easily seen with even small particles; it disappears completely or at least partially when the flame is taken away because contact with the air brings about reoxidation.

Ni nickel

80 ppm Ni in the earth's crust mark nickel as a common element. The divalent element is concentrated in orthomagmatically formed sulphide ores such as pentlandite and pyrrhotite as well as in hydrothermal veins where Ni is often associated with arsenic and cobalt. Some amount is incorporated by silicates like olivine. On weathering Ni-silicates such as garnierite are formed.

Fuse the mineral with a mixture 1 : 2.5 of ammonium chloride and ammonium nitrate in a small test tube, after cooling add some ammonia solution. Disregard any white turbidity or a brown precipitate. A drop of this solution is placed on a dry strip of filter paper impregnated with an alcoholic solution of dimethylglyoxime. The paper is then immersed in a bath of dilute ammonia, and kept in gentle motion. A red fleck indicates the presence of Ni. In the presence of much Mn the paper turns brown, in this case the test is repeated using ammonium carbonate as the bath liquid. - Use a slightly warmed, saturated solution of dimethylglyoxime in acetone or a 1% solution in alcohol to impregnate the paper.

P phosphorus

When heated with the oxidizing flame many phosphates impart a pale bluish-green colour to the flame, while others show the reaction only if moistened with concentrated sulphuric acid or ammonium hydrogen sulphate solution and then heated. The colour is not very marked and may be masked by other elements which produce a flame colour.

The test already described by the reduction with metallic magnesium is rather sensitive; the mineral powder used for this test should first be heated to a low red glow on a magnesia furrow. This expels any water and OH content that might impair reduction.

For a wet indication dissolve a pinhead-sized amount of the mineral in a test tube by melting with ammonium hydrogen sulphate. After cooling add some distilled water. Now prepare a solution of a pinhead-sized ammonium molybdate in 5 ml diluted nitric acid and add some drops of the test solution at room temperature. A yellow precipitate indicates the presence of phosphoric acid, it usually takes some minutes to form the precipitate! If arsenates are present they will form a yellow precipitate if the solution is warmed! Silicic acid produces a yellow colour.

Pb lead

The earth's crust contains 15 ppm Pb. Lead minerals are mainly found in hydrothermal veins where galena PbS is an ubiquitous mineral. A lot of other lead minerals are formed in the oxidation zone of hydrothermal deposits. Lead is divalent in minerals with the exception of plattnerite PbO₂.

If Pb is a main constituent, it is easily detected by heating a 1 : 1 mixture of the mineral with soda on charcoal, an addition of charcoal powder or potassium oxalate is recommended.

For a wet indication dissolve a tiny amount of the mineral in diluted nitric acid or melt it with a mixture of ammonium nitrate and ammonium chloride on a magnesia furrow. Add a tiny amount of KJ potassium iodide to the solution or the moistened melt. The yellow colour of PbI₂ indicates lead. - Cerussite PbCO₃ is coloured yellow if it is wetted first with a saturated solution of oxalic acid and then, after a minute, with potassium iodide solution.

If a piece of pyromorphite Pb₅Cl(PO₄)₃ is heated on charcoal it melts easily, on cooling it crystallizes to a faceted sphere. The name of the mineral is due to this effect. Mostly the charcoal assumes a thin white coating of PbCl₂.

Pt platinum and the platinum group elements

Ruthenium Ru, rhodium Rh, and palladium Pd, are called the light platinum elements, while osmium Os, iridium Ir, and platinum Pt are the heavy platinum elements. They are very rare with a content of 0.01 ppm Pd, 0.005 ppm Pt, and only 0.001 ppm each for the other elements of this group in the earth's crust. The elements are mainly disseminated in orthomagmatic basic and ultrabasic igneous rocks as elements, only Pt occurs sometimes as sperrylite PtAs₂. Mineable deposits are preferably alluvial placer deposits.

Platinum and palladium are readily dissolved by a mixture of concentrated HNO₃ + HCl, but there is no attack by any acid for rhodium, iridium and alloys with much osmium + iridium. - Ru and Os are dissolved by a melt of KOH and KNO₃ forming green and violet compounds, respectively. Rhodium is dissolved slowly by potassium pyrosulphate at a red glow only.

Chloride solutions of platinum form yellow crystals of (NH₄)₂PtCl₆ on the addition of NH₄Cl. On microscopic observation the crystals show the morphology of octahedra. The test is not very sensitive, since the solubility of the precipitate is 1:100.

Platinum salts, like those of gold, palladium and osmium, in acid solution, are reduced to the metal by stannous chloride. This can be used for a screening test: a drop of the solution is placed on filter paper and a drop of stannous chloride SnCl₂ in strong hydrochloric acid is placed upon it. Any stain indicates the presence of the above mentioned noble metals, only As and Sb may produce a stain, too. If the result is positive, you may proceed in the following way.

For a sensitive Pt-test the solution is made basic with ammonia and some oxalic acid is added. On gentle warming any gold salts are reduced to the metal. Now combine a clear drop of this solution with a drop of potassium iodide; a brown-red colour indicates the presence of platinum.

For a sensitive test for palladium, filter paper is bathed in a cold saturated alcohol solution of dimethylglyoxime. After drying, the paper is placed in a concentrated solution of a nickel salt that has been made barely ammoniacal; red nickel dimethylglyoxime precipitates. After thorough washing with water, the paper is bathed briefly in alcohol and dried. Now add some solid sodium acetate to the test solution and place a drop of it on the red paper. After gentle drying bathe the red paper in dilute HCl until the surface surrounding the drop position becomes white. Then wash the paper in cold water. A pink to red spot remains, depending on the quantity of palladium present. - Palladium and palladium alloys produce a brown-black stain if a drop of an alcoholic solution of iodine is placed on the metal surface and allowed to evaporate in the air. The staining withstands washing with alcohol, the stain disappears on heating to redness. No stain is given by platinum, but any silver in the alloy may produce a yellow stain which is readily dissolved by a concentrated solution of sodium thiosulphate.

S sulphur

At least part of any sulphur in a mineral is converted to sodium sulphide by melting a mixture of the mineral powder with soda on charcoal. Add some charcoal powder if sulphates are tested. The bead is treated with two drops of water on a clean sheet of silver. A black stain of silver sulphide indicates the presence of sulphur, but Se and Te may give the same result. - Place a drop of the dissolved bead on filter paper and add a drop of a diluted solution of $\text{Na}_4\text{Fe}(\text{CN})_5\text{NO}$ sodium nitroprusside. A violet colour indicates the presence of sulphur, Se and Te do not interfere.

Acid soluble exogenic sulphate minerals are dissolved in HCl, the solution is filtrated and BaCl_2 solution is added. A white precipitate sometimes only forms after the inner wall of the test tube has been rubbed with a glass rod; it indicates the presence of the sulphate ion SO_4^{2-} . Barite and celestine should be molten with a surplus of soda on a stainless steel teaspoon, this melt is dissolved in water. After filtration this solution is acidified with HCl and then again barium chloride solution is added. - On melting the following reaction has occurred: $\text{BaSO}_4 + \text{Na}_2\text{CO}_3 = \text{Na}_2\text{SO}_4 + \text{BaCO}_3$.

- Yellow native sulphur is easily detected by touching with the flame, a brown melt is formed which burns to SO_2 with its typical pungent odour.

The presence of acid insoluble sulphide minerals may be detected in the following way. Some mineral powder is mixed with diluted HCl in a test tube. Add a piece of zinc and warm gently. A small amount of hydrogen sulphide H_2S is formed together with the hydrogen. This is detected by the blackening of a wet lead acetate paper held atop.

Sb antimony

Antimony is a rare element with a 0.2 ppm content in the earth's crust. It is mostly concentrated in hydrothermal veins of low temperature origin, often as stibnite Sb_2S_3 .

Most antimony compounds, when heated on charcoal in the oxidizing flame, yield a dense white sublimate of Sb_2O_3 . It is deposited quite near to the heated part and it is volatile. This sublimate may be deposited on a cover glass placed on the charcoal at an appropriate place. This cover glass plus sublimate can be used for the following tests:

- Dissolve the white sublimate by a drop of diluted HCl and place this drop on a clean sheet of silver. Add a piece of granulated tin to the liquid, a black spot of metallic Sb forms on the silver.
- The white sublimate turns to red by heating with solid sodium thiosulphate in a test tube. Use a broken piece of the cover glass and a small amount of the sodium thiosulphate. Put cotton wool into the upper part of the test tube and heat in a nearly horizontal inclination, a lot of water is given off at first.

Many sulphantimonites produce a characteristic sublimate of the oxysulphide of antimony $\text{Sb}_2\text{S}_2\text{O}$ only on a rather intense heat in a closed tube. It appears black when hot, but changes on cooling to a reddish-brown.

Se selenium

The amount of selenium in the earth's crust is only 0.09 ppm. Selenium occurs mostly together with sulphides in hydrothermal veins, selenites and selenates within the oxidation zone are extremely rare.

When a substance containing selenium is heated on charcoal, a curious odour can be observed resembling rotten radishes. This rather sensitive test was first described by Berzelius.

For a wet test dissolve the mineral with a minute amount of nitric acid, add 1 ml concentrated HCl and boil. Now solve a pinhead-sized crystal of potassium iodide KJ on a watchglass in a drop of concentrated HCl and soak this solution with a filter paper. A drop of the test solution is placed in the middle of the moist part. Any black-brown stain that develops is completely decolorized by a drop of sodium thiosulphate solution if no selenium is present; otherwise a red-brown stain of Se is left.

Si silicon

Approximately 0.4 g of the mineral powder is mixed with only a quarter of this amount of calcium fluoride and placed in a plastic tube like those used for tablets. One millilitre of concentrated sulphuric acid or a concentrated solution of NH_4HSO_4 is added and the tube is closed with the plastic lid, in which a hole with a diameter of 2 - 4 mm has previously been made by means of a hot nail. The hole is covered with a wet black fabric, and the fabric must be kept wet by placing some wet cotton wool on the lid. Now heat the lower part of the plastic tube gently by dipping it into hot water for 10 minutes.

Any SiF_4 , evolved in the reaction of $\text{CaF}_2 + \text{H}_2\text{SO}_4 + \text{silicate}$, is decomposed by the water according to the formula: $3 \text{SiF}_4 + 2 \text{H}_2\text{O} = \text{SiO}_2 + 2 \text{H}_2\text{SiF}_6$. After opening the lid the black fabric should have turned white in the presence of any silicate in the tested mineral. - This test is impaired by the presence of a considerable amount of boric acid, and it may fail if too much calcium fluoride has been used. In such cases only HBF_4 , or H_2SiF_4 is formed; both acids are soluble in water.

It is also possible to proceed in the following way. Melt a pinch of the mineral powder with a flake of NaOH on a stainless steel spoon. Only after complete cooling add one millilitre of distilled water to the solid melt and dissolve it at least partly. This solution is poured into a plastic beaker with one millilitre of ammonium molybdate solution, and drop by drop HNO_3 is added till a weak acid solution is achieved. Now dissolve a pinhead sized amount of SnCl_2

and a flake of NaOH in some drops of water in a plastic vessel. After complete cooling pour this solution into the beaker with the test solution. A blue colour indicates the presence of silicate; the colour fades after some minutes. Arsenic and phosphoric acid must be absent in the mineral.

Another method is to dissolve the NaOH melt completely in water, to pour it into a test tube, then cautiously adding an equal volume of concentrated nitric acid, and to evaporate to dryness. Be careful towards the end of the operation, do not allow the tube to become very hot. Moisten the residue after cooling with 3 ml HCl, boil for a few seconds and add 5 ml distilled water. Heat again to boiling and remove the insoluble silica by filtering. This silica should be white after washing with water. It is tested for purity by puncturing the filter and washing the silica by means of a jet of water into a clean test tube. Then add a little KOH and boil; if pure, the silica will go wholly into solution. In rare cases when Nb-containing silicates such as eudyalite are tested, a turbidity of niob oxide may remain.

Sn tin

Tin is a rare element with an average amount of 3 ppm in crustal rocks. This sounds past belief since bronze, its alloy with copper, was the main material for tools and weapons for millenniums in the history of mankind. Even in the present electronic age tin is an important material for soldering. Fortunately the occurrence of tin bearing minerals is nearly exclusively associated with the pneumatolytic, pegmatitic, and hydrothermal stage of granite plutons. The mineral chiefly found in it is cassiterite SnO_2 , a hard, heavy, and extremely weathering resistant mineral. It is insoluble in any acid or mixture of acids and therefore collected as a placer mineral in the sands which have resulted from the disintegration of weathered granite plutones. Moreover, Sn scarcely enters the lattice of the main rock forming minerals and other tin minerals like stannite $\text{Cu}_2\text{FeSnS}_4$ are quite rare. Minor amounts of tin are found in some pegmatite minerals like columbite. Tin is tetravalent in minerals, but in solution it can be reduced to the divalent state.

Prospectors for tin are mainly searching for cassiterite as a placer mineral. The mechanical corroded grains of cassiterite are difficult to distinguish from other placer minerals. They are placed on a sheet of zinc and a drop of diluted HCl is added. Within a few seconds SnO_2 grains are covered with a dull-grey skin of metallic tin. Flat thick-walled dishes made from cast zinc are often used for this purpose. The test may fail if the grains are covered with iron oxides. In such cases the iron oxide coating can be removed by warm HCl saturated with oxalic acid, followed by washing with water.

To detect tin in other minerals the tin must be present as SnCl_2 in solution. This is achieved in the following way. Sulphides are molten with a 1 : 2.5 mixture of $\text{NH}_4\text{Cl}/\text{NH}_4\text{NO}_3$, for oxides and silicates the fusion with KOH on a stainless steel spoon is recommended. In both cases the cool solid melt is dissolved in a little water, and the same amount of concentrated HCl is cautiously added. Add to this solution a few granules of stick zinc, after some seconds all tin is reduced to the divalent state. There are two ways to detect the presence of tin:

- The test solution is stirred with a test tube filled with cold water. If the portion of the test tube which has been dipped into the solution, is held in a nonluminous Bunsen flame, a characteristic blue flame mantle forms around the test tube. This flame colour is specific for tin only, the test fails only in the presence of As.

- For a wet method mix some drops of ammonium molybdate solution with a surplus of $\text{NH}_4\text{NaHPO}_4$ on a spot test plate or a clay dish, add a drop of nitric acid and warm gently. A yellow precipitate of ammonium phosphomolybdate is formed. Soak the liquid with paper and wash the precipitate twice with water. Adding a drop of the test solution will reduce the yellow precipitate to molybdenum blue. Both tests are rather sensitive, antimony does not interfere.

For a reduction on charcoal mix the mineral powder with one part of charcoal powder and two parts of soda and make into a paste with water. Globules of tin are achieved by a fusion on charcoal only if tin is a major component; if heated intensively and for a considerable time, sufficient tin may volatilize to give a white, non-volatile coating of SnO_2 on the charcoal.

Sr strontium, see Ba barium

Ta tantalum, see Nb columbium

Te tellurium

Tellurium is a very rare element with only 0.002 ppm content in the earth's crust. Tellurides or the native element associated with sulphides are mostly found. Oxidation products such as salts of tellurous and telluric acid very rarely occur in gossans.- A very delicate test for Te is made by heating the mineral powder with concentrated sulphuric acid, when the latter assumes a reddish-violet colour. The cautious addition of water, after cooling, causes the colour to disappear, and a grayish-black precipitate of Te is produced.

Ti titanium

4.4 weight % of the earth's crust is made up of titanium, indeed it is the ninth in the rank of abundance of elements. It is found in combination with oxygen in oxides and silicates, predominantly in the tetravalent, but sometimes in the trivalent state. Rutile TiO_2 , ilmenite FeTiO_3 , titanomagnetite, and titanite CaTiSiO_5 are the most common titanium minerals. They are present as accessory minerals in most igneous rocks. Trace amounts of Ti are nearly always incorporated in the lattice of pyroxenes, amphiboles, and many micas. Regarding its physical and chemical properties rutile is in fact a mineral apt to be concentrated in placer deposits. Unfortunately, it mostly occurs in igneous rocks as fine needles or it is intergrown with quartz. For this reason only a few mineable placer deposits of rutile are known, but these few are very large.

For a wet test the mineral powder is fused on charcoal with the sixfold amount of soda, and the melt is dissolved with concentrated HCl. Boil the solution, filter if necessary, then add a little granulated tin or a piece of zinc. On boiling, a violet colour indicates Ti owing to the formation of TiCl_3 . The colour is seen best when the liquid becomes cold, and the hydrogen formation ceases. This test may fail if the tested matter contains less than 3% titanium oxide.

Another highly sensitive test works only in a sulphuric acid solution. The above-mentioned melt with soda is treated with sulphuric acid in a test tube and heated. After cooling to room temperature, some water and a tiny amount of $\text{NaNH}_4\text{HPO}_4$ is added, then some hydrogen peroxide. A yellow colour indicates Ti. If vanadium is present it will cause a red colour, in case of any doubt, add a small amount of NaF. The yellow colour due to Ti will vanish, while the red colour of V stays. - Some Ti minerals like oxides can be dissolved by fusion with

KHSO_4 in a test tube, and dissolving the melt in cold water. Such solutions can be used for the hydrogen peroxide test also.

U uranium

The uranium content of the earth's crust is 2 ppm. U-bearing minerals are often associated with granite plutons. The minerals uraninite UO_2 , pitchblende, brannerite $\text{U}(\text{Ti},\text{Fe})_2\text{O}_6$, and coffinite USiO_4 occur in pegmatites and hydrothermal veins. Small amounts of U are often incorporated in the crystal lattice of zircon and monazite. Uranium is tetravalent in all these minerals of the endogenic cycle. On contact with oxygen and water all these uranium minerals are quickly oxidized, and the hexavalent uranium forms the uranyl ion UO_2^{2+} . This particular ion is dumb-bell shaped and cannot be incorporated into the lattice of any other mineral. As a result, uranyl-minerals are formed. More than 100 of such uranyl-minerals are known. Most prominent are the uranium micas. These minerals are not silicates because the sheet layers are made up by PO_4^{3-} and UO_2^{2+} ions. Cations and water are arranged between the layers. - Within the exogenic cycle, trace amounts of U are often found in lignites and phosphorites.

All crystalline uranyl compounds, with the exception of such minerals that contain transition elements, show a fluorescence in ultra-violet light. Already borax beads which contain uranium exhibit an appreciable green fluorescence, but sodium fluoride beads light up to a deep yellow colour if they contain any U. This may be used in the following highly sensitive test:

Sodium fluoride is fused to form a bead in a loop of platinum wire (NaF melts at 992°C). When cold, the bead appears only slightly violet in ultraviolet light. Now the bead is wetted with the test solution. After careful evaporation, fuse the bead again for a short time. After cooling examine the bead in ultraviolet light. As a source of UV the use of a cheap pocket money tester is recommended. Only Nb and Be give a very weak similar fluorescence.

Nearly all uranium minerals can be dissolved in nitric acid, but zircon, monazite, and some oxides should be taken up by a hot soda bead on a platinum wire. After melting, take up some potassium nitrate and melt again. This bead is dissolved in diluted HCl.

- For a wet test, some drops of this solution are placed on a spot plate or a clay dish. Add a tiny amount of sodium thiosulphate, stir and then add a drop of a solution of potassium ferrocyanide $\text{K}_4\text{Fe}(\text{CN})_6$. A brown precipitate indicates U; when a flake of NaOH is added the precipitate turns yellow.

It should be mentioned that most minerals containing the uranyl group are soluble in a solution of ammonium carbonate, which thereby assumes a yellow colour.

V vanadium

Vanadium makes up 110 ppm of the earth's crust, so it is no rare element. However, the main mass of vanadium is widely distributed in igneous rocks, where the trivalent vanadium takes up the position of trivalent iron in mafic minerals. During weathering of these mafic minerals, the vanadium enters the pentavalent state, and it is widely redistributed because alkali vanadates are readily water soluble. Vanadium minerals form preferably in the outcrop of lead deposits. Vanadinite $\text{Pb}_5\text{Cl}(\text{VO}_4)_3$ and descloizite $\text{Pb}(\text{Zn},\text{Cu})(\text{OH})\text{VO}_4$ are the most common vanadates. Insoluble minerals also form together with the uranyl ion. Among those, carnotite $\text{K}_2(\text{UO}_2)_2(\text{VO}_4)_2 \cdot 3\text{H}_2\text{O}$, which possesses a structure similar to uranium micas, is the best known. V may also enter the tetravalent state, but minerals which contain tetravalent vanadium are extremely rare. Patronite VS_4 was only found together with asphalt in two

mines in Peru; for a long time, this was the main mined ore. Roscoelite, a green vanadium(III)-bearing mica from sandstone bands, was also mined for this element.

All vanadates can be dissolved in strong acids. If vanadates are boiled with concentrated HCl, the oxychloride of tetravalent vanadium VOCl_2 is formed due to the reaction:

$\text{V}_2\text{O}_5 + 6 \text{HCl} = 2 \text{VOCl}_2 + 3 \text{H}_2\text{O} + \text{Cl}_2$. The chlorine gives a blue colour to wet potassium iodide-starch paper held atop of the test tube. The colour of the solution changes to green and finally to a light blue on dilution with water, but this colour may be masked if Cu or U is present. Ensure a good ventilation due to the formation of chlorine! Please mind that any black manganese oxides will also cause the evolution of chlorine.

For a more sensitive wet test you need a solution of pentavalent V in sulphuric acid. Vanadates can be dissolved in sulphuric acid or may be molten with KHSO_4 , followed by dissolving in few drops of diluted sulphuric acid. Silicates must be melted with a mixture of soda with some KNO_3 on a magnesia furrow. The melt is dissolved in water and brought to a strong acidic state by the addition of sulphuric acid. Filter this solution if any precipitate occurs and treat some drops with a tiny amount of solid NaF on a spot test plate, a clay dish, or a watch glass. Now add, only drop by drop, a very diluted solution of hydrogen peroxide. If vanadium is present, a red to pink coloration appears. It is a pitfall of this test that too much hydrogen peroxide converts the red precipitate of peroxovanadium sulphate yellow. Therefore the diluted hydrogen peroxide should be added only in very small amounts. The addition of NaF is only necessary if Ti or Fe^{3+} is present.

The amber colour of a bead of phosphorous salt in the oxidizing flame is a quick but quite reliable test for minerals where V is a main constituent.

W tungsten (German: Wolfram)

The content of tungsten in the earth's crust is approximately 1 ppm. The element is always hexavalent in minerals. It does not enter the lattice of igneous rock forming minerals; thus, it concentrates as tungstates in pegmatites and hydrothermal veins. Only three tungstates are widespread and important: wolframite $(\text{Fe,Mn})\text{WO}_4$, scheelite CaWO_4 , and stoltzite PbWO_4 . On weathering of these minerals some extremely rare minerals are sometimes formed. Tungsten is also found in small quantities in a number of columbites and tantalites.

Scheelite and stoltzite dissolve in hot concentrated HCl, while forming a yellow precipitate of WO_3 . If boiling is continued after the addition of granulated tin, a blue colour results. The colour may change to brown on further reduction. Wolframite and columbite are to be fused with KOH on a magnesia furrow. Dissolve the solid melt in water only after complete cooling. Add again tin to the filtered solution. On acidifying with HCl and boiling, the blue reduction test is obtained. The following test may be carried out on filter paper: a drop of the aqueous solution of the KOH melt is placed on filter paper; then, on the opposite side of the paper a drop of concentrated HCl is added. When treated with a solution of SnCl_2 in HCl the yellowish colour changes to blue due to the formation of partially reduced tungsten oxides.

Zn zinc

Zinc is a common element in the earth's crust with 65 ppm. It is mostly concentrated in hydrothermal veins. The principal mineral is sphalerite, ZnS , but several other sulphides contain Zn in small quantities as a constituent or as intergrown admixtures of ZnS .

Smithsonite ZnCO_3 , and a considerable number of other zinc-bearing minerals such as silicates, phosphates, arsenates, vanadates, and zinkite ZnO are formed in the oxidation zone.

It is recommended to mix the mineral powder with half the amount of soda, to add some charcoal powder and to make into a paste with water. Zinc volatilizes when this mixture is heated on charcoal and is detected by the non-volatile coating of ZnO , which is of a pale canary-yellow colour when hot and white when cold. - If the mineral contains aluminium or tin, some borax should be added and intense heating is necessary. This is especially recommended if gahnite ZnAl_2O_4 or stannite $\text{Cu}_2\text{SnFeS}_4$ with intergrown ZnS is tested. The white sublimate of ZnO may be captured by a cover glass placed in the proper position on the charcoal. There is also a wet detection technique: Dissolve the white coating in hot acetic acid. The solution gives a white precipitate with a solution of potassium ferrocyanide $\text{K}_4\text{Fe}(\text{CN})_6$. - Another way to confirm that the white coating is ZnO consists of wetting the sublimate with a considerably diluted solution of cobalt nitrate, then heating again. The green colour of Rinmann's green indicates Zn.

The following procedure is recommended only if traces of zinc are to be detected in silicates or technical products: Melt the powder with a flake of NaOH on a magnesia furrow. After complete cooling dissolve in a few drops of water. This solution is mixed on a watch glass with a few drops of a green solution of 1 mg diphenylthiocarbazone (dithizone) in 10 ml carbon tetrachloride. Now the carbon tetrachloride is evaporated by blowing while stirring with a glass rod. The solution assumes a raspberry-red colour in the presence of zinc. A precipitate of any colour should be disregarded. A fume cupboard should be used, carbon tetrachloride smells agreeable, but it is poisonous if it is inhaled!

Zr zirconium

Zirconium makes up 160 ppm of the earth's crust. Due to its tetravalent charge and its size, the Zr^{4+} ion is not incorporated into the lattice of the main rock-forming minerals; almost invariably, zircon ZrSiO_4 is formed and finely dispersed in igneous rocks, especially in the late stages of crystallization. Some complex Zr-silicates occur only in nepheline syenites. Pure zircon is the most weathering-resistant silicate, therefore it is concentrated in placer deposits. Zircon often captures tetravalent uranium and thorium during crystallization from the magma. The radioactive decay of these elements over geological time disturbs the short-range order of the crystal lattice; such minerals are called metamict. Minerals altered in this way exhibit a dark colour. Syenites containing Zr-silicates which have been subject to weathering may form baddeleyite ZrO_2 as the weathering product. Thus formed baddeleyite layers are the main mineable zirconium ores.

Zr minerals are decomposed by the fusion with NaOH on a stainless steel spoon or a silver dish, or by melting the mineral powder with a mixture of soda and borax on charcoal. The crushed bead or the cold and solid NaOH melt is treated with water in order to dissolve the water soluble compounds. The water is discarded, either by filtering or by soaking with filter paper. The remaining insoluble zirconium oxide hydrate is dissolved in a few drops of diluted HCl . A small part of this solution is treated with hydrogen peroxide on a watch glass, from a near neutral solution a white precipitate forms. If the solution turns yellow, Ti is present. If no Ti is present, a drop of the hydrochloric solution of the melt residue is placed on turmeric paper; if it turns orange, Zr is present. As the colour is not very marked, the following test is better.

Place some drops of the hydrochloric acid solution in a clay dish or a spot test plate and evaporate slowly to dryness only, do not overheat. Add two drops of an alcoholic alizarin solution, and boil once. Then add a drop of dilute hydrochloric acid. Only the red-violet zirconium lake remains unaffected. Be, Al, Ti, and Th do not interfere. - The alcoholic alizarin solution is prepared by dissolving alizarin in ethanol, treating it drop by drop with highly diluted HCl until a pure yellow colour develops; this solution is diluted with an equal volume of ethanol and filtered.

No simple identification tests can be recommended for the heavy elements of the third main group of the periodic table of the elements: **Ga** gallium, **In** indium, and **Tl** thallium, the same is true for **Ge** germanium. With average amounts in crustal rocks of 15 ppm gallium, 2 ppm germanium, 1 ppm thallium, and 0.1 ppm indium, only the last-named may be called a rare element. No indium minerals are known, Tl minerals are extremely rare and Ge and Ga minerals are only rarely found, mainly in the oxidation zone of a few sulphide ore deposits. For technical use all these elements are mainly yielded as by-products during the purification of raw zinc. - Indium and thallium each produce a single line in flame emission spectra: Tl 535 nm green, and In 451 nm indigo-blue. The names of both elements are based on their flame coloration. - Thallium is a very poisonous element!

The preparation of test papers and reagents

Not all test papers and reagents are on sale. Moreover, it is much cheaper to prepare them, especially for only occasional use. The wet papers should be pinned to a string by means of a clothes peg and dried in a dust-free atmosphere. All papers should be stored in airtight plastic vessels in a dark place. Always use distilled water.

Dimethylglyoxime paper is used for the identification of Ni and Pd. - Filter paper is soaked with a saturated alcoholic solution of dimethylglyoxime and dried. The reagent paper is stable for a long time.

Iodide-starch-paper is turned blue by the action of strong oxidizing compounds, especially by free chlorine and bromine. - Three pinches of starch are mixed with some water and poured into 150 ml boiling water. After cooling, approximately one gram of potassium iodide is dissolved in this liquid, followed by soaking and drying of the filter paper in the usual way. The wetted paper is held atop the solution to be proved; take in mind that nitrogen dioxide NO_2 also is able to turn the paper blue, this compound may be produced on boiling strong acid solutions containing nitrates.

Lead acetate paper is used to identify the presence of hydrogen sulphide H_2S . - 2.5 g of potassium acetate is dissolved in 100 ml water. The filter paper is soaked and dried as usual.

Pyrolusite paper serves to detect the presence of SO_2 . - Three pinches of potassium permanganate KMnO_4 are dissolved in 100 ml water. Filter paper is soaked with this dark violet solution and dried. Soaking and drying is repeated twice and the brown paper is finally well washed with water before the last drying. The dry paper is stable for 1-2 years.

Turmeric-paper is used for the identification of boron and zirconium as well as a test paper for alkalis. - Turmeric is an Indian plant, *curcuma longa*; the powdered rhizome of this is used as a dye-stuff and as a spice, especially in curry. The active content curcumin is on sale, but not the test paper. The easiest way is to shake half a teaspoon of turmeric powder with 10 ml acetone, and, after the powder settles, to soak a white filter paper with the yellow solution.

The dried paper is stable for years if it is stored in an air-tight container in a cool and dark place. (Turmeric is called curcuma in French and German.)

Zinc sulphide paper can be used as a screening reagent to detect Ag, Cu, Pb, Sb, Bi, Hg, trivalent As, Sn, and Cd. Antimony causes a red stain, cadmium and arsenic a yellow one, all other elements a dark brown staining. Stains caused by mercury compounds turn black. - Filter paper is soaked with a solution of 2 g zinc sulphate (or another soluble zinc salt) in 100 ml water and dried. This paper is again soaked with a solution of 2 g sodium sulphide or potassium sulphide in 100 ml water. After drying it is again soaked with the zinc solution, well washed and dried. The paper is stable.

Zinc ferrocyanide paper is used for the rapid detection of iron and copper. - The paper is soaked with a solution of 2 g of a soluble zinc salt, like the sulphate, in 100 ml water and dried. The dry paper is soaked with a solution of 2 g potassium ferrocyanide $K_4Fe(CN)_6$, a yellow compound, in 100 ml water. After drying the paper is treated again with the zinc solution, well washed with water and dried. - A brown stain results in contact with a solution containing Cu^{2+} ions, a blue stain is caused by Fe^{3+} ions. The very faint staining by solutions containing Mn^{2+} (pink), Ni^{2+} (green), and Co^{2+} (blue) cannot be recommended as conclusive tests.

An **iodine solution** can be prepared from potassium iodide in the following way. Heat a mixture of two pinches each of potassium iodide KI and potassium hydrogen sulphate $KHSO_4$ in a test tube till a sublimate of free iodine is formed on the inner wall of the test tube. After complete cooling blow a stream of air into the tube till no odour of sulphur dioxide is smelled. Now scratch the iodine from the walls by means of a spatula while holding the tube in a slanting position. Transfer the iodine to a watch glass. It can be dissolved either in alcohol or in a potassium iodide solution. - The solution is used to identify lanthanum or to convert the white oxide sublimates of Pb and Bi on charcoal into the yellow or red coloured iodide sublimates. The solution is stable if it is stored in a glass bottle with a ground glass stopper or a cork stopper soaked with paraffin.

Bromine water dissolves metallic gold, platinum and palladium and it is used to test for beryllium and magnesium. - A pinhead sized amount of each potassium bromide KBr and potassium bromate $KBrO_3$ is dissolved in some drops of diluted HCl, 5 ml water and a pinch of sodium acetate is added. Always ensure good ventilation, it is dangerous to inhale any of the brown vapours! The solution should be freshly prepared for use. Add a surplus of sodium thiosulphate to any remaining liquid before flushing with much water in the sink.

Ammonium molybdate solution is used to detect phosphates, arsenates and silicates. - One gram of ammonium molybdate in a finely powdered state is mixed with 10 ml water, approximately 10 ml concentrated nitric acid is added drop by drop and stirred intensively till any white precipitate is dissolved and a clear solution results. The solution is stable, it should be stored in a plastic bottle.

Stannous chloride solution is used as a reducing agent in some tests. - 0.5 g $SnCl_2$ is dissolved in 6 ml water + 4 ml concentrated hydrochloric acid.- The solution is stable.

Staining tests for carbonates

Aragonite is a modification of CaCO_3 . It is stable only at high pressures, but is often formed metastable if CaCO_3 is precipitated in the presence of Mg^{2+} ions and in the temperature range of 29°C to 260°C . Both calcite and aragonite effervesce on contact with cold diluted HCl and even with vinegar, but aragonite scratches calcite; this is a simple test if a crystal of calcite with a flat face is available. However, sometimes a staining test to differentiate between calcite and aragonite is required. - Boil the carbonate with a 10% solution of cobalt nitrate, only aragonite assumes a pink colour, while calcite remains unstained. Any remaining cobalt nitrate solution can be used for further tests.

Calcite and dolomite may be distinguished in the following way: A small fragment of the mineral is immersed for 30 seconds in a solution made from 10 ml distilled water and 1 ml concentrated HCl; now the specimen is immersed for 5 minutes in a solution of 100 ml distilled water + two pinches of alizarin sulphonic acid + 10 drops of concentrated HCl. Finally the fragment is washed with distilled water till the washing water stays colourless. Calcite, aragonite, and witherite are stained red, while ankerite, strontianite, and cerussite may turn purple. No staining is observed for dolomite, magnesite, siderite, smithsonite, gypsum, and anhydrite.

It is only rarely necessary to differentiate between magnesite and dolomite, since dense polycrystalline magnesite mostly shows a characteristic white colour. An appropriate staining test was already described, see Mg.

To differentiate between dolomite, pure calcite, and magnesium containing calcite, shake 0.2 g alizarin sulphonic acid with 25 ml methanol and add 15 ml of 30% NaOH. Boil the mineral for 6 minutes with this mixture. Dolomite and the Mg-containing calcite turn purple, pure calcite remains unstained.

All dye-stuff solutions and etching acids should be used only once and then discarded.

In rare cases it may be necessary to isolate small amounts of minerals or fossils from calcareous sediments like limestone. A 0.4% solution of monochloroacetic acid CH_2ClCOOH dissolves calcite, aragonite and dolomite, most other minerals including apatite remain unattacked. The dissolution is a very slow process, at least 24 hours and a large surplus of the acid is needed for fine powders. The disintegration of bigger lumps may take weeks!

UV fluorescence

Ultra violet radiation is invisible for the human eye, but most sources of UV also emit some light in the visible range. Some solid and liquid matter emits visible light by the absorption of UV light, normally only a small part of the visible spectrum. Therefore this fluorescence light is coloured. The effect is called phosphorescence if the emission of visible light continues for some time after stopping the UV source. In minerals fluorescence is mostly due to impurity atoms in the lattice. This is the reason why some mineral species always fluoresce, while other minerals exhibit the effect only sometimes in certain deposits. Depending on the conditions of formation of the mineral, the amount as well as the kind of impurity atoms may vary; such variations can cause changes in colour and intensity of the fluorescence of a mineral! It should be borne in mind that many organic materials also fluoresce e.g. teeth, some lichen, fungi on rotten wood, some kind of fabric, and naphtha. - Even thin coatings of iron and manganese oxides may suppress the effect.

Shortwave UV has a 185 nm to 300 nm wave length, longwave UV ranges from 300 nm to 400 nm. Take care to avoid any UV light catching your eyes, even the reflection from a light surface is dangerous; severe damage, even blindness, may result. Glass absorbs shortwave UV, but not longwave UV. Wear fabric gloves if you intend to handle minerals in UV light for longer than a few seconds. - Portable battery-operated UV light lamps operating on shortwave 254 nm and longwave 366 nm for field use are on sale with prices in the range from €92 to €360. Battery-operated money testers are much cheaper, they only work in the long wave, but the price range is in the order of €5 to €30. An EPROM eraser is a strong source for shortwave UV.

The following minerals always exhibit fluorescence

Mineral	shortwave UV	longwave UV
autunite $\text{Ca}(\text{UO}_2)_2(\text{PO}_4)_2 \cdot 10\text{-}12\text{H}_2\text{O}$ and many other uranyl-minerals	yellow	
cerussite PbCO_3	yellow	yellow
hauyne $(\text{Na,Ca})_{4\text{-}8}(\text{AlSiO}_4)_6(\text{SO}_4,\text{Cl})_{1\text{-}2}$		orange
hydrozincite $\text{Zn}_5(\text{CO}_3)_2(\text{OH})_6$	blue white	
malayaite $\text{Ca}_5\text{SnSiO}_5$	greenish yellow	
powellite CaMoO_4	yellow	
ruby $\text{Al}_2\text{O}_3 + 0.1\text{-}0.6\%\text{Cr}_2\text{O}_3$	red	red
scheelite CaWO_4	blue to yellow	
scapolite $\text{Na}_3(\text{AlSi}_3\text{O}_8)_3 \cdot \text{NaCl} / \text{Ca}_3(\text{Al}_2\text{Si}_2\text{O}_8)_3 \cdot \text{CaCO}_3$	yellow	orange-yellow
willemitte Zn_2SiO_4	green	
zircon ZrSiO_4	yellow to orange	yellow to orange

The following minerals sometimes fluoresce, depending on the origin:

Alkali feldspar, anhydrite, andalusite, aragonite, anglesite, apatite, barite, benitoite, chalcedony, calcite, colesmanite, cristobalite, danburite, diamond, dumortierite, eucryptite, fluoborite, fluorite, gypsum, halite, hyalite, jadeite, lapis lazuli, monazite, opal, petalite, periclase, rhodochrosite, sapphire, smithsonite, sphalerite, spodumene, strontianite, thomsonite, tremolite, topaz, turquoise, witherite, wollastonite. - No claim to completeness!

The solubility of some minerals

These files comprise only selected minerals and mineral groups. Tests should be made with a small amount of finely ground material.

The following minerals are insoluble in cold concentrated hydrochloric acid

albite $\text{NaAlSi}_3\text{O}_8$	paragonite $\text{NaAl}_2(\text{Si}_3\text{AlO}_{10})(\text{OH})_2$
amblygonite $(\text{Li},\text{Na})\text{Al}(\text{PO}_4)(\text{F},\text{OH})$	prehnite $\text{Ca}_2\text{Al}_2\text{Si}_3\text{O}_{10}(\text{OH})_2$
astrophyllite $(\text{K},\text{Na})_3(\text{Fe},\text{Mn})\text{T}_2\text{Si}_8\text{O}_{24}(\text{OH},\text{O})_7$	pseudobrookite Fe_2TiO_5
axinite $\text{Ca}_2(\text{Fe},\text{Mg},\text{Mn})\text{Al}_2\text{BOH},\text{O}(\text{Si}_2\text{O}_7)_2$	pyrite FeS_2
babingtonite $\text{Ca}_2(\text{Fe},\text{Mn})\text{FeSi}_5\text{O}_{14}(\text{OH})$	pyrochlore $(\text{Ca},\text{Na})_2\text{Nb}_2\text{O}_6(\text{OH},\text{F})$
barite BaSO_4	spodumene LiAlSiO_6
boracite $\text{Mg}_3\text{B}_7\text{O}_{13}\text{Cl}$	talc $\text{Mg}_3(\text{Si}_4\text{O}_{10})(\text{OH})_2$
chloritoid $(\text{Fe},\text{Mg},\text{Mn})_2\text{Al}_4\text{Si}_2\text{O}_{10}(\text{OH})_4$	titanite CaTiSiO_5
celestine SrSO_4	vesuvianite $\text{Ca}_{10}\text{Mg}_2\text{Al}_4(\text{Si}_2\text{O}_7)_2(\text{OH})_4$
hematite Fe_2O_3	xenotime YPO_4
ilmenite FeTiO_3	zircon ZrSiO_4
lawsonite $\text{CaAl}_2\text{Si}_2\text{O}_7(\text{OH})_2\text{H}_2\text{O}$	zoisite $\text{CaAl}_3(\text{SiO}_4)_3(\text{OH})$
lazulite $\text{MgAl}_2(\text{PO}_4)_2(\text{OH})_2$	<u>and most members of the following mineral groups: feldspars with the exception of anorthite, clinopyroxenes, orthopyroxenes, epidote, amphiboles</u>
marialite $3\text{NaAlSi}_3\text{O}_8\text{NaCl}$	
muscovite $\text{KA}_2(\text{Si}_3\text{AlO}_{10})(\text{OH},\text{F})_2$	
allanite $(\text{Ce},\text{Ca},\text{Y})_2(\text{Al},\text{Fe})_3(\text{SiO}_4)_3(\text{OH})$	

The following minerals are soluble in hot concentrated hydrochloric acid

analcime $\text{NaAlSi}_2\text{O}_6 \cdot \text{H}_2\text{O}$	meionite $3\text{CaAl}_2\text{Si}_2\text{O}_8 \cdot \text{CaCO}_3$
anorthite $\text{CaAl}_2\text{Si}_2\text{O}_8$	melilite $(\text{Ca},\text{Na})_2(\text{Mg},\text{Al},\text{Fe})\text{Si}_2\text{O}_7$
apatite $\text{Ca}_5(\text{F},\text{OH},\text{Cl})(\text{PO}_4)_3$	monticellite CaMgSiO_4
apophyllite $\text{KCa}_4(\text{Si}_4\text{O}_{10})_2(\text{F},\text{OH})8\text{H}_2\text{O}$	nepheline $\text{KNa}_3(\text{AlSiO}_4)_4$
augite $(\text{Ca},\text{Na})(\text{Mg},\text{Fe},\text{Al},\text{Ti})(\text{Si},\text{Al})_2\text{O}_6$	olivine $(\text{Mg},\text{Fe})_2\text{SiO}_4$
brucite $\text{Mg}(\text{OH})_2$	periclase MgO
chlorite $(\text{Mg},\text{Fe})_3(\text{OH})_2/\text{Si}_4\text{O}_{10}/(\text{Mg},\text{Fe})_3(\text{OH})_6$	pectolite $\text{Ca}_2\text{NaH}(\text{Si}_3\text{O}_9)$
chondrodite $(\text{Mg},\text{Fe})_5(\text{OH},\text{F})_2(\text{SiO}_4)_2$	rhodonite $\text{CaMn}_4\text{Si}_5\text{O}_{15}$
chrysotile $\text{Mg}_3(\text{Si}_4\text{O}_{10})(\text{OH})_8$	saponite, a Mg-rich mineral of the smectite group
colemanite $\text{Ca}_2\text{B}_6\text{O}_{11}5\text{H}_2\text{O}$	
datolite $\text{CaBSiO}_4(\text{OH})$	sodalite $\text{Na}_8(\text{Cl}/(\text{AlSiO}_4)_6)$
eudialyte, a red cyclosilicate with Na, Ca, Fe, Mn, Ce, Zr occurring in nepheline syenites	wöhlerite $\text{Ca}_2\text{NaZr}(\text{F},\text{OH},\text{O})_2/\text{Si}_2\text{O}_7$
fluorite CaF_2	epidote after glowing
gadolinite $\text{Y}_2\text{FeBe}_2(\text{O}/\text{SiO}_4)_2$	garnets after melting, except uwarovite
gehlenite $(\text{Ca},\text{Na})_2(\text{Mg},\text{Al},\text{Fe})\text{Si}_2\text{O}_7$	all carbonates
glaucanite, a green iron-rich mica	all calcium silicates
goethite FeOOH	all calcium silicate hydrates
ilvaite $\text{CaFe}_3(\text{O}/\text{Si}_2\text{O}_7/\text{OH})$	most zeolites
leucite KAlSi_2O_6	andesine, labradorite, and bytownite are imperfectly soluble
ludwigite $\text{Mg}_2\text{Fe}(\text{O}_2/\text{BO}_3)$	most phosphates
Mn oxides like cryptomelane, and pyrolusite evolve chlorine on dissolution!	

The following minerals are dissolved by hot concentrated sulphuric acid

amblygonite $(\text{Li,Na})\text{Al}(\text{F,OH/PO}_4)$	ilmenite FeTiO_3
alunite $\text{KA}_3(\text{OH})_6(\text{SO}_4)_2$	monazite CePO_4
anatase TiO_2	allanite (orthite) a Ce-rich epidote
anglesite PbSO_4	perovskite CaTiO_3
anhydrite CaSO_4	phlogopite $\text{KMg}_3(\text{Si}_3\text{AlO}_{10})(\text{F,OH})_2$
baddeleyite ZrO_2	pyrophyllite $\text{Al}_2(\text{OH})_2(\text{Si}_4\text{O}_{10})$
barite BaSO_4	rutile TiO_2
biotite dark mica	titanite $\text{CaTiSiO}_4/\text{O}$
böhmite AlOOH	topaz $\text{Al}_2(\text{F,OH})_2\text{SiO}_4$
chloritoid $\text{FeAl}_2(\text{OH})_2/\text{O}/\text{SiO}_4$	vermiculite $(\text{Mg,Fe,Al})_3(\text{Si,Al})_4\text{O}_{10}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$
cryolite Na_3AlF_6	zircon ZrSiO_4
gibbsite $\text{Al}(\text{OH})_3$	most members of the crandallite group

The following minerals are soluble in hot 20% KOH

alunite $\text{KA}_3(\text{OH})_6(\text{SO}_4)_2$	realgar As_4S_6
anglesite PbSO_4	stibnite Sb_2S_3
chalcedony SiO_2	sulfur S
opal $\text{SiO}_2 \cdot n\text{H}_2\text{O}$	tridymite SiO_2
orpiment As_2S_3	wavellite $\text{Al}_3(\text{PO}_4)_2(\text{OH,F})_3 \cdot 5\text{H}_2\text{O}$

A 10% solution of **ammonium carbonate** dissolves most copper ores, with the exception of the sulphides and oxides; the dissolution rate is sometimes enhanced by the addition of ammonia solution. The solution turns deep blue.

Most uranium minerals containing hexavalent U are dissolved by ammonium carbonate solution; the solution turns yellow. Minerals containing tetravalent U are only slowly attacked by a mixture of hydrogen peroxide and ammonium carbonate solution.

Miscellaneous hints

The **Mohs' hardness** of a mineral measures its resistance to being scratched, but the hardness may be considerably different on different faces. Large mineral specimens are pressed on to and drawn across a smooth surface of a standard. Small grains are best determined by placing the grain on the smooth surface of the standard, it is then pressed firmly into that surface with a flat wooden implement, such as the blunt end of a pencil, and steadily drawn across it. The presence of a scratch, which must be an elongated indentation and not just a linear accumulation of powder, must be controlled by a lens! It is preferable to use flat pieces of the standard minerals calcite (H=3), fluorite (H=4), apatite (H=5), orthoclase (H=6), and quartz (H=7). Obsidian or window pane glass may be used instead of apatite which is often not available. Some textbooks recommend the use of metal sheets: copper (H≈3), brass (H≈3.5 to 4), soft iron (H≈4 to 5), and annealed steel (H≈5.5 to 7). Such sheets must be controlled before use by minerals with known hardness! - It is very important not to confuse the true hardness of a single mineral grain with the brittleness, or lack of granular cohesion, of a fine-grained mineral aggregate. Furthermore, inclusions or surface coatings of a soft mineral may be harder, e.g. hematite on calcite or soft fibrous minerals which are silicious. For preliminary tests a sewing-machine needle can be used. Its blunt end should be mounted in wood; for transport its sharp end may be inserted into a small piece of cork. Most times the hardness of such a device is in the range of 5.5 to 6.

The **density** of a mineral is its mass per unit volume and is given as g cm^{-3} . To determine the density of a mineral you need a balance with a sensitivity of 0.001 g. If such a device is available you can record the weight of the mineral sample and the volume of water which it displaces. The mineral piece is tied by a very thin copper wire and held into a vessel with water on a balance. The surplus of weight after immersing the mineral gives the mass of the displaced water. Litz wire from an electric cable may be used.

Another possibility is to use a pycnometer, that is a small bottle fitted with a long ground-glass stopper, which is pierced along its length by a small-diameter hole. With four readings it is possible to calculate the density:

The dry pycnometer = P, the mineral + the dry pycnometer = M, the pycnometer + the mineral filled with water = T. Eventually the pycnometer is emptied and filled only with water = W. - The mass of the mineral is $M - P$, the volume of the mineral is $W + M - P - T$.

Only non-porous minerals can be used! Many minerals are members of a mixed crystal series, that means their density may vary considerably. Therefore it makes no sense to calculate more than the first decimal place.

It is possible to use heavy liquids to measure the density by the swim-sink method. Such liquids are:

Tribromomethane (bromoform) CHBr_3 , $D = 2.90$; methanol is used for dilution

Tetrabromoethane $\text{Br}_2\text{CHCHBr}_2$; $D = 2.96$; methanol is used for dilution

Diiodomethane CH_2I_2 , $D = 3.32$; xylene is used for dilution

Clerici solution is a concentrated solution of thallium formate and thallium malonate, $D = 4.25$; distilled water is used for dilution.

All these liquids are extremely poisonous already on contact with skin, most gloves are attacked by the halogen-containing hydrocarbons. The health risks caused by such vapours are seriously increased by smoking during handling; high temperatures may convert such vapours to more toxic compounds! Any use of such heavy liquids should be restricted to use in scientific laboratories in well ventilated fume-cupboards and by well-trained staff!

There is one exception: a saturated solution of sodium polytungstate in water reaches densities of 2.9 to 3.0 and this compound is classified as a less poisonous compound. The solution is very expensive, 100 ml cost approx. € 120, but the above-mentioned poisonous liquids are also expensive.

If you have determined the main elements of a mineral you have to compare this result with physical properties like colour, lustre, hardness, streak colour, cleavage, and density in order to establish **which mineral** it is. Nobody knows all minerals by heart, so you need a good textbook, preferably one with appropriate tables. For instance two books can be recommended which contain such tables for approximately 200 of the most abundant minerals:

John Sinkankas: "Mineralogy for amateurs" - Van Nostrand Reinhold Company
New York - Cincinnati - Toronto - London - Melbourne 1964

which is unfortunately out of print, or

Meurig P. Jones: "Applied Mineralogy - A Quantitative Approach", 1987
Graham & Trotman Ltd. London, ISBN 0-860-10511-3

Another data source must be used for rare minerals or if no clear result is obtained. In such cases search the internet <http://www.webmineral.com> ⇒ Mineralogy database ⇒ subgroups "Chemical composition" and "Determinative Mineralogy"

Another and possibly more user-friendly source can be found using the search engines HotBot or MSN Web Search:

<http://www.geonord.org/edu/minid.html> ⇒ Nomenklatur ⇒ alfabetisk och systematic mineral förteckning ⇒ mineral search ⇒ Boolean search database for given elements in formula

The **streak colour** is an important information especially for sulphides, oxides, carbonates, and phosphates. Large specimens should be rubbed on the streak plate, if the mineral is intergrown it is easier to rub the mineral with the edge of the streak plate. For this purpose it is convenient to cut a plate into small pieces of 15 to 20 mm width by means of a diamond saw or a grinder. Such pieces may be used also for acid fusions.

Spot test plates can be cut into small strips with only three depressions as well, such strips can be heated more easily.

Light coloured precipitates are better seen on a dark background. Watchglasses can be placed on dark paper or charcoal; clay dishes may be dark glazed: mix equal amounts of fine powdered basalt and borax and produce a slurry by adding drop by drop a solution of cobalt nitrate on stirring. On firing to 1000°C the dried slurry melts to a **bluish-black glaze**.

It is recommended to **clean the equipment** after any use. Pipe cleaners can be used for cleaning tubes. Use soap and a hard sponge or brush to clean streak plates, most remaining streaks are dissolved by melting a mixture of $\text{NH}_4\text{Cl}/\text{NH}_4\text{NO}_3$ on the plate. After cleaning,

rinse all items with distilled water. - Agate mortars should be cleaned by repeated grinding of fine quartz powder. Agate is a slightly porous matter, avoid wetting it with any solutions.

Colour perception may vary from person to person; some people have difficulties distinguishing red and green tinges. Such different colour perceptions may lead to misunderstandings in doing quick assays, but it is not an unconquerable handicap if you are aware of it. So consult an ophthalmologist if you distinguish stop and go at traffic lights by their position rather than by colour!

For the detection of ferromagnetic minerals a small, but strong **magnet** is needed, especially if the mineral is intergrown in a rock, e.g. pyrrhotite in gabbro. A larger strong magnet is suitable to remove magnetite from panning residues or from sand and gravels. To prevent small particles clinging to the magnet, it may be wrapped in a plastic bag. Tightly adhering small particles can be removed from the magnet by means of a freshly used chewing gum! - A magnetic field may disturb or destroy the information stored on credit cards or electronic devices like pocket computers! This danger is at least minimized if you wrap the magnet in a thin steel sheet, cut from a cola can, then again in cardboard, and place this package in a lidded tin made of iron, such as a candy box. Take care to pack the magnet well away from watches, credit cards and electronic devices in your luggage.

Comminution is an important part of sample preparation. Cracking mineral pieces wrapped in paper or plastic by a hammer blow is recommended as a first step. An appropriate anvil of 10 mm thickness and 20 to 35 mm diameter may be turned and polished by a lathe from a steel rod. If possible it should be annealed. It can be also used to determine hardness and for striking small chisels. Small amounts of mineral powder are obtained by means of a sapphire or diamond file, but for very hard minerals a very small steel mortar serves a useful function. Such a device can be built in the following way: Search for a tool trading company delivering semifinished material and order a guide bush (French: matrice cyclique, German: Führungshülse) DIN 9845 with 8 mm inner diameter, 12 mm outer diameter, 10 mm length and a round punch (French: poinçon, German: Schneidstempel) DIN 9861, with 8 mm diameter, 70 mm length. Cut the round punch in the middle by means of a grinder. Insert one half into the guide bush up to just one third of its height and fix this position with rubber tubing. This arrangement serves as a small steel mortar, the other part of the round punch is used as the pestle. - Cutting-pliers are useful for detaching and breaking up small fragments of minerals.

A mortar made from sintered corundum is an acceptable substitute for an agate mortar. The former is harder and non-porous, but slightly more brittle; the price is similar.

Alkaline fusions done on stainless steel are always contaminated by iron and very often by Cr and Ni. It is possible to avoid such contamination if the fusion is done on a silver dish. Any goldsmith is able to prepare such a dish of pure silver 20 mm diameter, 7 mm height and 0.4 mm wall thickness with a small handle for holding by a forceps at a price of approximately €15. Only melts containing alkali hydroxides should be prepared on such a device! Never use it for acid fusions or for borate melts! Place it on a clay dish on charcoal during heating. Pure silver melts at 960.5°C, therefore heat very cautiously!

During wet analysis the use of a **water bath** is often useful. To obtain a cheap, dismantlable water bath, place a tea light on a piece of plywood of appropriate size and drill three holes into the plywood forming an equilateral triangle which just surrounds the tea light. Now insert three pieces of iron wire \approx 2 mm thickness and 70 mm length into these holes. Place a tin of 55 mm diameter, 35 mm height on this tripod; e.g. 70 g double concentrated

tomato paste from Italy is sold in such tins. The lower part of a small cola can may be used as well, see figure 5, page 10.

Some boreholes in a piece of wood or polystyrene-foam may serve as a **test tube rack**. Any such test tube rack should first be tested for tilting by means of a test tube filled with water and fitted with a small funnel.

A stiff paste of waterglass and fine powdered talc may be used as a considerably **heat-resistant glue** for joining ceramic, glass, and iron.

It is useful to keep small pieces of the equipment in small labelled boxes for **storage and transport**. Match boxes are useful for clay dishes and other small items; aluminium cigar tubes may be used for magnesia rods, furrows, and other elongated items. Ask for such aluminium tubes at a tobacco shop. For transport, the entire equipment should be stored in a shockproof box made of stiff cardboard, plastic or tin. Glass items may be wrapped in bubble foil or crumpled paper, don't forget to fill any voids with such material. The use of soft plastic foam cannot be recommended, it may be converted to a sticky mass by the influence of heat and chemicals after some time.

The use of a **spectroscope** is recommended for the identification of alkali metals and alkaline earth metals. Spectroscopes using a prism for dispersion are expensive, with prices ranging from \approx €160 to €300. A cheap, but useful alternative is to use spectroscopes which only contain a slit and a replica diffraction grating. Such instruments need a slightly oblique look to the ocular side and are supplied by *Ward's Natural Sciences*, New York or *Astromedia*, Germany at prices from €6 to 24. On watching flame coloration the position of the burner as well as the spectroscope should be fixed and the flame and the slit should be parallel. - Prism spectroscopes show a non-linear wave length scale; the refraction index of the prism glass depends on the wave length, but this dependence is not linear. As the result the spectrum seems to be compressed in the blue, shortwave range. Spectroscopes with a diffraction grating show a linear wave length scale, such spectra are called normal spectra. Normal flame emission spectra of the alkali metals are shown in the appendix.

Most chemicals are stored in **plastic vessels**, this is cheap and useful especially for field use. Please remember that nearly all kinds of plastic have a limited lifetime, after some years the material becomes brittle and any squeezing may cause cracks. Some liquids enhance this development considerably, like all oxidizing materials, ketones, halogen-containing hydrocarbons, and many liquid organic compounds with the exception of methanol and ethanol. Bottles and vials are mostly made of polyethylene, the high density kind marked PE-HD is superior to the low density type marked PE-LD. Polypropylene PP is comparable to PE-HD, it is used for funnels and beakers since it withstands boiling water for short term use. For heavy duty field use by exploration companies bottles are available that are made of polytetrafluoroethylene PTFE, teflon perfluoroalkoxylalkane PFA, or tetrafluoroethylene-hexafluoropropylene FEP. Such bottles are inert to all chemicals suggested in this book, including all concentrated acids and they withstand mechanical shocks. With prices in the range of €20 to €30 for 100 ml items they are expensive, but they may avoid a lot of severe trouble during field work. - Suitable for similar reasons are a 50 ml beaker made from PFA or ETFE ethylene-tetrafluoroethylene copolymer at a price of \approx €10; such item can be used in a boiling water bath and is inert to all used chemicals. It should not be heated by an open flame. - The lower part of a plastic pill tube can be used as a small plastic beaker at temperatures up to 70°C.

The assays presented in this booklet have been selected in order to avoid the use of very poisonous compounds, such as alkali cyanides, mercury salts, and benzidine, as well as compounds that may cause severe risks during handling and transport, such as concentrated hydrofluoric acid and sodium superoxide. If you always follow all safety instructions cautiously and use only the small amounts recommended there is no **health risk** in laboratory work. A greater risk may be represented by some minerals, especially those containing Hg, As, U, and Cd. Mercury and arsenic are very toxic elements if they are incorporated and both elements are volatile on heating. So test all minerals that may contain these elements first by heating in a closed tube with soda + charcoal powder. If such a test is positive any heating on charcoal should be done only if an excellent ventilation is ensured. Collectors should store cinnabar specimens in air-tight polystyrene boxes; often native mercury in very small droplets is present and the mercury vapours may cause a chronic intoxication. Minerals containing arsenic are not volatile at room temperature but should be stored off limits for children.

Cadmium is very toxic, too; it is considerably volatile by heating on charcoal, but the danger is much less. - The chemical toxicity of uranium is similar to that of mercury, the radiation risk is additional, especially for U-containing minerals which are in radioactive equilibrium with their decay products. No volatile products are formed by the recommended tests for U. But all uranium-containing minerals of a collection should again be sealed in air-tight plastic or polystyrene cases; the radioactive decay continuously produces radon ^{222}Rn with a half-life period of 3.82 days and radon ^{219}Rn with a half-life period of 3.9 sec; this noble gas may enter the air. Since radon is fairly soluble in water it may be incorporated by inhalation!

Tellurium is a less poisonous element, no health risk, but a social risk may be encountered by the inhalation of traces of TeO_2 fumes during heating of Te-bearing minerals on charcoal. At least a part of the Te is converted by metabolism into dimethyltellurium $(\text{CH}_3)_2\text{Te}$ which is emitted by respiration and this compound has a very strong garlic-like smell. The same holds true for Se-bearing minerals, but the inhalation of more than traces of SeO_2 may cause an irritation of the mucous membrane of your nose. This so-called selenium nose-cold ceases within days, but the garlic smell of sweat and breath is additional.

Commercial and scientific laboratories should store any waste containing more than trace amounts of heavy metals like Cd, Pb, Cu, Ni, Co, Cr, Ba, Sr, Hg, U, Th, and As in a break-proof vessel. A near-neutral reaction of the solution can be provided by the addition of ternary calcium phosphate and aluminium hydroxide. Under such conditions most toxic elements enter the precipitate.

A cheap method for the unambiguous identification of transparent minerals is the determination of the **refractive index** and of birefringence. Only mineral grains in the size of 20 to 30 μm are needed for the Becke line method if a polarizing microscope and a set of refracting index liquids are available. It is also possible to use a **gem refractometer**, but in this case a mineral specimen with a flat or polished surface of approximately 2 mm^2 is needed.

A **polariscope** is useful for the inspection of transparent single crystals and gems in the size range from one mm to several centimetres. The specimen is illuminated through a polarization filter and is viewed through a second polarization filter, both in crossed position. The field of view always stays black for glass and cubic crystals, like garnets or spinels, but if birefringent crystals are turned between the filters they usually change from bright to extinction. Synthetic spinels grown by the Verneuil technique show a typical, undulated extinction. Such cheap

synthetic spinels can be coloured nearly any colour during production by doping with different metals, thus imitating precious stones! - A century ago tourmaline-tongs were used for such purpose. Two thin plates cut parallel to the c-axis of a green tourmaline crystal were mounted parallel to one another by means of spring-like wire tongs. One of the mountings was rotatable.- Today it is more convenient to use two linear polarizing filters for cameras or cheap polarizing foil. It is a simple arrangement to build a cube from stiff cardboard, one side open. The filters are inserted in appropriate cuttings in two opposite squares next to the open side. Any skilful hobby worker may invent other arrangements. Such a device may be useful for the inspection of placer mineral concentrates containing raw gemstone minerals.

United Nations reports estimate the number of people depending on **wild gold digging** at one million, thus dispersing approximately 500 tons of mercury per year into the environment. This caused serious contamination, especially in the Philippines and Brazil. Wild gold diggers are called garrimpeiros, they use the mercury to dissolve the fine gold powder gained by the washing process. The mercury is evaporated by heating with a strong flame on an iron base. In that way a massive piece of gold is achieved, but the mercury is spilled into the environment. Unfortunately some mercury - especially in the aquatic ecosystem - is transformed by the metabolism of microorganisms to much more poisonous compounds such as dimethylmercury. At the end of the food chain the mercury is concentrated in fish and shell-fish.- Commercial companies sometimes also use mercury for collecting gold from fine slimes, but they recycle the mercury by distillation in iron retorts. Mercury dissolves all metals with the exception of iron. - The use of mercury can be avoided by a very old process: molten lead with a melting point of 327.4°C readily dissolves gold, silver, and most other metals. The lead and all other metals besides the noble metals can be oxidized to the oxides by heating with an oxidizing flame. PbO melts at 890°C, this melt was soaked up by a base of slightly compressed fine powdered bone ash in a clay dish. This process was considerably improved early in the 19th century by heating the Pb-Au alloy together with some borax in a clay dish. The borax melt dissolves all oxides immediately, and a noble metal bead is formed which only contains gold, silver and any metals of the platinum group originally present in the starting material. Any borax melt residues can be removed by boiling with water or diluted sulphuric acid. Admittedly, this process is somewhat less simple than the amalgam process, but the use of lead + borax is cheaper. The price for 1 kg Hg is ≈ €75, the use of pure lead + borax instead may cost ≈ € 25. - Lead from batteries contains considerable amounts of antimony and is less suitable for this purpose. Never try to use soldering tin for dissolving noble metals, the gold may be taken up by the SnO₂ resulting in oxidation!

Beginners may have difficulties finding **supply sources** for chemicals and lab ware. Generally local suppliers should be preferred, since you save postage and packing costs. Try the Yellow Pages and ask at the next pharmacy. Butane torches and stainless steel forceps are mostly sold in DIY stores.- If no local supplier is available try the internet:

Australia: (lab ware) Crown Scientific <http://crownsscientific.com.au>

(chemicals) Sigma Aldrich Pty Ltd. ausmail@sial.com

North America: <http://www.coleparmer.com> or info@coleparmer.com (lab ware)

aldrich@sial.com (chemicals) or canada@sial.com

For cheap replica grating spectrometers and sets of refractive liquids please contact: e-mail: customerservice@wardsci.com

III Appendix

A basic kit of chemicals and equipment:

soda, dry Na_2CO_3 , sodium carbonate
 borax, dry $\text{Na}_2\text{B}_4\text{O}_7$, sodium tetraborate
 phosphorous salt $\text{NH}_4\text{NaHPO}_4 \cdot 4\text{H}_2\text{O}$ sodium ammonium hydrogen phosphate
 or
 sodium meta phosphate NaPO_3
 ammonium hydrogen sulphate NH_4HSO_4
 a mixture of ammonium chloride and ammonium nitrate $\text{NH}_4\text{Cl}/\text{NH}_4\text{NO}_3$ 1:2.5
 a mixture of dry soda and potassium nitrate $\text{Na}_2\text{CO}_3/\text{KNO}_3$ 3:1
 tin Sn, granules or foil
 sodium perborate $\text{NaBO}_3 \cdot 4\text{H}_2\text{O}$ or denture cleaning tablets
 fluorite CaF_2
 gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$
 silver Ag, sheet or coin
 pH paper
 cotton wool
 magnesium ribbon Mg

butane burner
 charcoal in rectangular pieces
 magnesia rods
 magnesia furrows
 closed glass tubes
 open glass tubes
 wooden clothes peg (spring type)
 test tubes, small and normal size with cork stopper
 forceps, stainless steel
 spatula, stainless steel
 microscopic slides and cover glasses
 sapphire file
 a small mortar, made from agate or corundum, alternatively a porcelain mortar
 filter paper for analytical use, 50 mm diameter
 glass rods
 water colour brush
 streak plate
 cobalt glass

hydrochloric acid, HCl 15%
 acetic acid, CH_3COOH 20%
 ammonium hydroxide, NH_4OH 15%
 methanol CH_3OH or ethanol $\text{C}_2\text{H}_5\text{OH}$

Annex I, comprises all chemicals and equipment mentioned in the General part:

potassium hydrogen sulphate KHSO_4
 oxalic acid $(\text{COOH})_2 \cdot 2\text{H}_2\text{O}$
 Zn, in granules
 cobalt nitrate $\text{Co}(\text{NO}_3)_2$
 tin chloride SnCl_2
 sodium thiosulphate $\text{Na}_2\text{S}_2\text{O}_5 \cdot 5\text{H}_2\text{O}$
 ammonium carbonate $(\text{NH}_4)_2\text{CO}_3$

ammonium heptamolybdate $(\text{NH}_4)_6\text{Mo}_7\text{O}_{27} \cdot 4\text{H}_2\text{O}$
 potassium oxalate $\text{K}_2(\text{COO})_2$
 lead acetate $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ for preparing lead acetate paper
 barium hydroxide $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ or calcium hydroxide $\text{Ca}(\text{OH})_2$
 ammonium hypophosphite NH_4HPO_2
 potassium permanganate KMnO_4 for preparing pyrolusite paper
 sodium hydroxide NaOH
 potassium hydroxide KOH
 dimethyl glyoxime $\text{CH}_3\text{C}(\text{NOH})\text{C}(\text{NOH})\text{CH}_3$
 nitric acid 15% HNO_3
 Pt wire loop
 a small beaker, 10 or 25 ml
 dropper
 stainless steel spoon
 small porcelain dish
 glass funnel, top diameter 35 mm
 plastic funnel
 clay dishes

Annex II, comprises all chemicals and equipment mentioned in the Special part:

Some chemicals are only used for the determination of one or a few elements, this is indicated by the relevant element symbols in brackets.

ammonium sulphate $(\text{NH}_4)_2\text{SO}_4$ (Ca)
 sodium acetate CH_3COONa
 potassium carbonate K_2CO_3
 potassium nitrate KNO_3
 potassium nitrite KNO_2 (I)
 potassium bromide KBr (only used for the preparation of bromine water)
 potassium bromate KBrO_3 (only used for the preparation of bromine water)
 potassium iodate KIO_3 (Th)
 potassium dichromate $\text{K}_2\text{Cr}_2\text{O}_7$ (F, Pb)
 ammonium oxalate $(\text{COONH}_4)_2 \cdot \text{H}_2\text{O}$ (Ca)
 ammonium thiocyanate NH_4CNS (Co, Mo)
 sodium nitroprusside $\text{Na}_2\text{Fe}(\text{CN})_5\text{NO} \cdot 2\text{H}_2\text{O}$ (S in presence of Se or Te)
 sodium cobaltinitrite $\text{Na}_3\text{Co}(\text{NO}_2)_6$ also called sodium hexanitritocobaltate(III) (K)
 sodium fluoride NaF (U)
 calcium hypochlorite $\text{Ca}(\text{ClO})_2$ (Au)
 ammonium acetate $\text{CH}_3\text{COONH}_4$
 sodium sulphide Na_2S
 potassium ferrocyanide $\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}$
 potassium ferricyanide $\text{K}_3\text{Fe}(\text{CN})_6$ (Fe^{2+})
 5-(4-dimethylaminobenzyliden)-rhodanine $\text{C}_{12}\text{H}_{12}\text{N}_2\text{OS}_2$ (Ag, Au, Pt, Pd)
 diphenylcarbazide $\text{C}_{13}\text{H}_{14}\text{N}_4\text{O}$ (Mg)
 aurintricarboxylic acid ammonium salt $\text{C}_{22}\text{H}_{14}\text{O}_9 \cdot 3\text{NH}_3$ (Al)
 quinalizarin, 1,2,5,8-tetrahydroxy anthraquinone $\text{C}_{14}\text{H}_8\text{O}_6$
 also sold as chinalizarin (Be, Mg)
 Na alizarin sulphonate $\text{C}_{14}\text{H}_7\text{O}_7\text{SNa}$ also sold as alizarin red S
 p-nitrobenzeneazo-a-naphthol $\text{O}_2\text{NC}_6\text{H}_4=\text{NC}_{10}\text{H}_6\text{OH}$
 also called 4-(4-nitrobenzoylazo)-1-naphthol or magneson II (Mg)
 diphenylthiocarbazid $\text{C}_6\text{H}_5\text{NHNHCSN}:\text{NC}_6\text{H}_5$ also called dithizon (Zn)
 sulphur S
 copper sulphate $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (Br)
 copper oxide CuO (Cl)

potassium iodide KI
barium chloride $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ (SO_4^{2-})
silver nitrate AgNO_3 (As)
turmeric paper (B, Zr)
KI/starch paper
ZnS paper
zinc ferrocyanide paper
hydrochloric acid, conc. HCl 30%
nitric acid, conc. HNO_3 68%
sulphuric acid H_2SO_4 100%

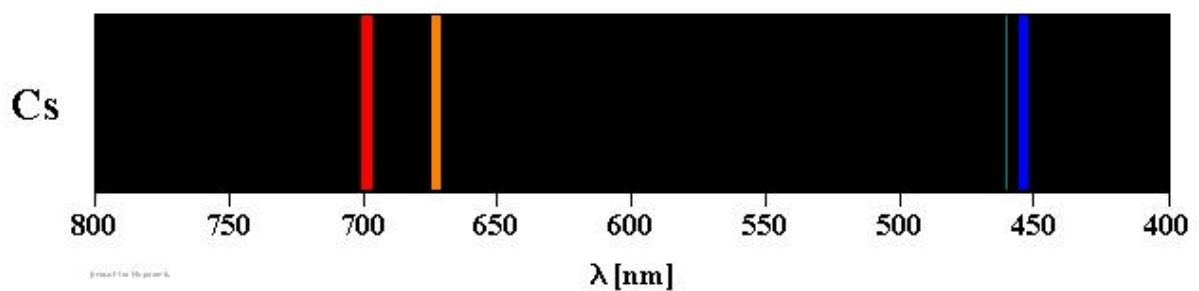
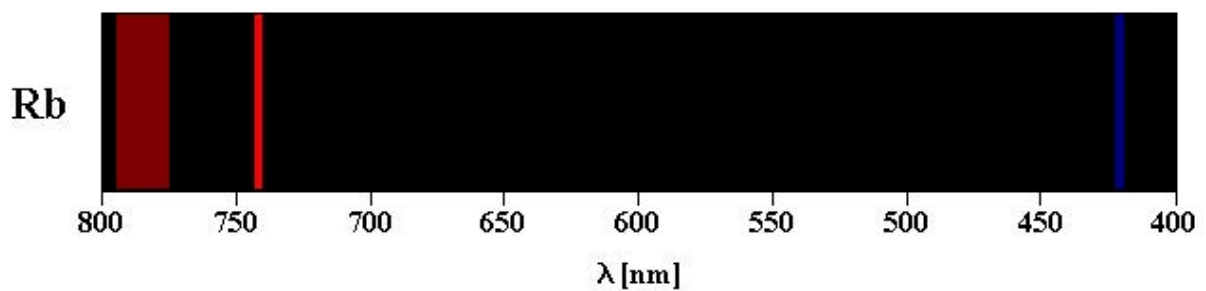
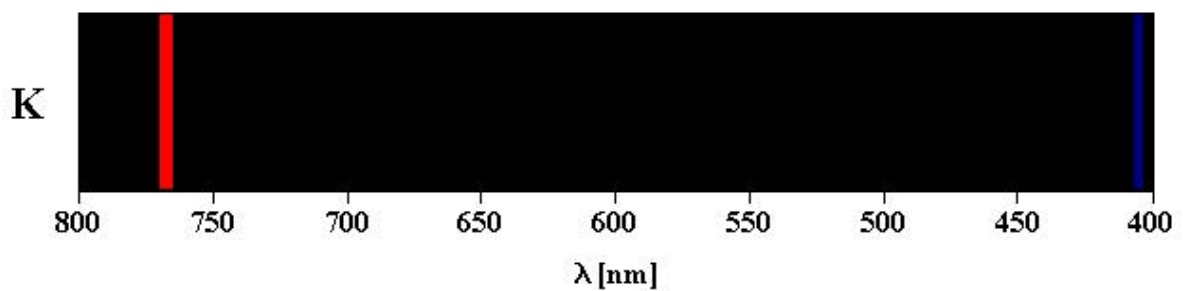
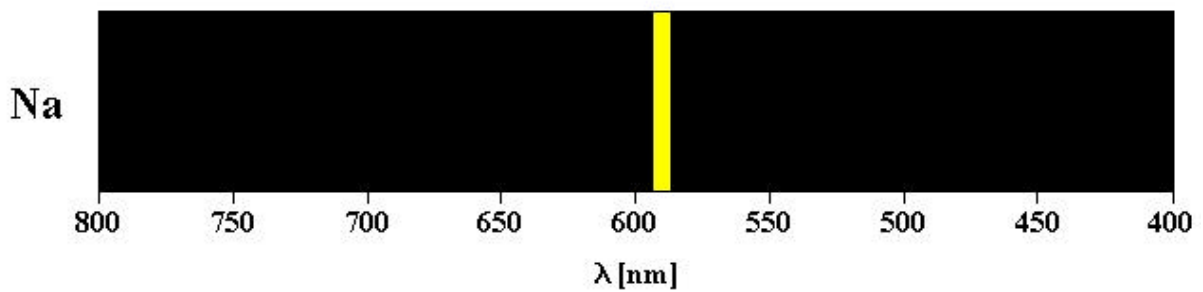
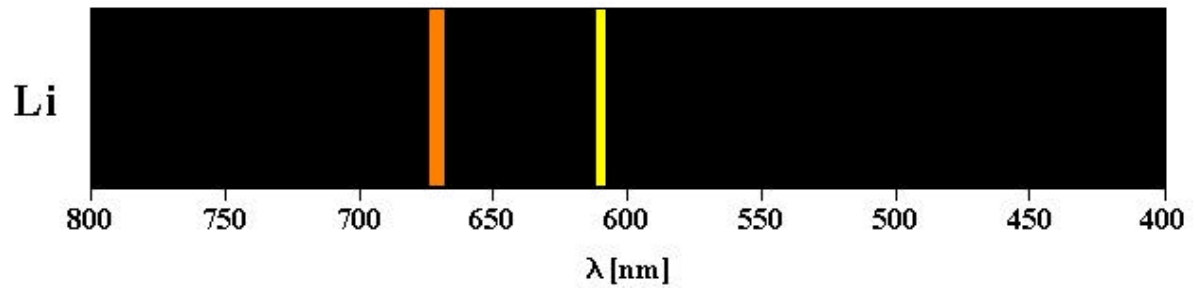
spot test plate
test tube rack
silver dish
plastic beaker
water bath/ tea light
SiC grinding paper

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Normal spectra of alkali metals



Glossary

carbonatite

A calcitic or dolomitic rock of apparently magmatic origin.

Earth's crust

The outmost layer of the Earth constitutes less than 0.1% of the Earth's volume. The continental crust of continents and continental shelves has an average thickness of 35 km, while the oceanic crust underlying the deep sea is only 5 to 10 km thick.

endogenic cycle

All geological events happening within the Earth's crust, mainly under reducing conditions.

exogenic cycle

Geological events on the Earth's surface, mainly under oxidizing conditions like weathering, transport, and deposition of sediments.

gossan

An iron-bearing weathered zone overlying many sulphide deposits. It contains mainly hematite and limonite, but sometimes residual minerals like gold and often oxidation products such as sulphates, phosphates, arsenates, and vanadates. *German: Eiserner Hut*

hydrothermal deposit

Deposits formed from aqueous fluids by the crystallization of ore and gangue minerals in the temperature range of 50° C to 400° C, mostly they occur in former fractures and faults.

ICP spectrometer

The substance is dissolved; this diluted solution is sprayed into the plasma of an inductively coupled plasma torch. The extremely high temperature of the plasma causes a wavelength-characteristic emission of visible light for nearly all present elements. A special combination of prisms, lenses, and detectors allows the simultaneous identification of many elements.

igneous rock

A rock that solidified from a wholly or partially molten material, a magma.

isomorphism

Two or more different crystalline compounds with very similar crystal lattices which form mixed crystals.

mafic minerals

Dark coloured minerals, mainly ferromagnesian, occurring in igneous rocks.

metamict

A mineral whose short-range order has been disrupted by radiation damage after crystallization. The radiation is due to uranium or thorium. Most metamict crystals show a dark colour, e.g. zircon.

metamorphic rocks

A rock derived from a sediment or other rock by the formation of new crystalline phases due to considerable changes in temperature and pressure, generally at depth in the Earth's crust.

nepheline syenite

Plutonic rock, mainly composed of Na-rich alkali feldspar and nepheline together with amphiboles or pyroxenes. Rare minerals are often frequent in minor amounts.

orthomagmatic

The stage during which the main mass of a magma crystallizes.

outcrop

Rocks and minerals that are exposed and visible at the surface.

oxidation zone

The part of a sulphide deposit that is chemically altered by the reaction with water and air.

pegmatite

A coarse-grained igneous rock at the upper margin of granites, mostly within the overlying rocks. During crystallization of a granite the residual melt is enriched with water and all elements which are not incorporated in the main minerals quartz, feldspars, and biotite. Thus such enriched trace elements themselves form minerals in the pegmatite.

placer deposit

A surficial mineral deposit which is formed by the mechanical concentration of weathering debris by tides or rivers. Typical placer minerals have a density higher than quartz, they are considerably weathering-resistant and mostly have a hardness higher than 5.

E.g. cassiterite, rutile, zircon, diamond, gold. *German: Seifenlagerstätte*

pneumatolysis

Alteration of a rock by a super-critical fluid derived from a magma.

plutonic rock

Such rocks have been formed at considerable depth by the very slow crystallization of a magma, therefore medium to coarse grained crystals are dominant. E.g. granite and gabbro.

silicates

Compounds with a crystal structure containing SiO_4 tetrahedra. The tetrahedra can be joined through one or more of the oxygen atoms to form groups (*neso*, *soro*), chains (*ino*), rings (*cyclo*), sheets (*phyllo*), or three-dimensional (*tecto*) structures together with other cations. -

Scanning electron microscope (SEM)

The specimen is sputtered with gold or graphite to ensure electric conductivity; it is positioned in a vacuum chamber. An electronic beam is focused on the specimen surface and scanned across it in a raster pattern of parallel lines. The electron impact at the surface causes the emission of secondary electrons with energies of a few tens of eV, and high energy backscattered electrons. The intensity of both emissions is very sensitive to the angle between primary beam and surface.

The emitted secondary electrons are collected and amplified; this signal is used to vary the brightness of a cathode ray monitor scanned in synchronism with the primary electron beam. Thus the SEM image shows the surface topography of the specimen at a magnification of 40 to more than 10,000 x.

Suitable detectors for backscattered electrons can produce also a backscattered image, in this case the intensity is dependent on the average atomic number of the specimen, as well as the surface topography: local variations in chemical composition result in variations in the contrast of the image.

Moreover, X-ray photons are emitted with wavelength and energy characteristic of the elements in the specimen. This spectrum can be used for quantitative chemical microanalysis. *French: microscope electronique a balayage - German: Rasterelektronenmikroskop*

transition elements

All elements with atomic numbers from 21 to 30, from 39 to 48, from 57 to 80, or 89 and higher. They are all metals.

volcanic exhalations

Gases produced by a volcano; they always contain water and carbon dioxide; H₂S, HCl, fluorides and chlorides are also frequently present.

volcanic rocks

Rocks that have been formed by the very quick crystallization of a magma at or near the surface. Crystals are generally very fine-grained, some glass may occur. E.g. basalt.

X-ray fluorescence spectrometer

The sample is either melted with lithium borate to produce a glass disc, or very finely powdered and pressed to a flat pellet. A characteristic X-ray spectrum of the elements is produced by directing an X-ray of very short wavelength at this sample. This is due to specific electronic transitions within the atoms of the substance. The emitted specific X-rays are either separated by a monochromator and detected (wavelength dispersive spectrometer) or determined by a detector which measures the energy of the X-ray quantum (energy dispersive spectrometer).

X-ray powder diffraction

A monochromatic X-ray beam is directed towards a flat, fine-powdered crystalline sample. The random orientation of the tiny crystals ensures all possible orientations between the X-ray and crystal lattice. Appropriate rotations of the sample and the detector allow all diffracted beams to be recorded consecutively (X-ray powder diffractometer).

The Debye-Scherrer method places the powder sample in a thin capillary of a Li-Be-borate glass. This capillary is turned around in the axis of a cylinder, the primary X-ray beam hits the sample perpendicular to the axis, and the diffracted beams are recorded by a film which is fixed to the inner surface of the cylinder.

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