Which set of ionic compounds?

Introduction
Teachers who have not used the problems before should read the section Using the problems before starting.

Prior knowledge
Tests for the identification of ions including flame tests, precipitation reactions and electrolysis. A detailed knowledge is unnecessary as students are encouraged to consult textbooks and data books during the exercise.

Resources
Data books and inorganic textbooks should be available for reference.
Any four of the possible 13 compounds could be issued, but the following four are recommended the first time the problem is attempted – barium nitrate, anhydrous copper(II) sulphate, sodium carbonate and potassium iodide. The compunds should be supplied at the start of the exercise in unlabelled, numbered bottles.
Students can request apparatus and chemicals during the practical session and these should be issued if they are safe to use. In particular, flame test equipment and electrolysis apparatus may be requested but should not be on view.

Group size
3.

Risk assessment
A risk assessment must be carried out for this problem.

Special safety requirements
The hazards associated with some of the chemicals eg barium nitrate should be noted.

Possible solutions
The following procedure is one systematic approach for identifying the compounds.
Stir each substance separately with water in a test tube.

1. Insoluble solids
Any insoluble substance must be barium carbonate, barium sulphate or copper(II) carbonate, but the latter is excluded as students were issued with white solids. Test any insoluble compound for the presence of carbonate by adding a strong acid such as dilute hydrochloric acid:
If the solid effervesces and dissolves, it is barium carbonate; if it does not, it is barium sulphate.

2. Soluble solids
a. If the solid dissolves to form a blue solution it contains copper(II) ions. The anion must be nitrate or sulphate as copper(II) carbonate is insoluble and copper(II) iodide does not exist. Test the solution for sulphate (barium chloride test) or for nitrate (brown ring test).
b. Test other soluble compounds as follows:
(i) for cations
- barium ions (sulphate test or flame test); and
- potassium and sodium ions (flame test).

(ii) for anions

— carbonate ions (strong acid);
— iodide ions (silver nitrate test, in the presence of nitric acid to avoid the precipitation of carbonates) or oxidation to iodine with bromine or chlorine water) ; and
— nitrate ions (brown ring test).

Other things that students could do include:
— adding a metal such as zinc or magnesium;
— electrolysis; and
— heating.

Suggested approach

During trialling the following instructions were given to students and proved to be extremely effective:
You can divide the work amongst yourselves but keep one another informed of your progress.

1. Working as a group, list all the compounds that the solid could be.
2. Again working as a group, devise as many different ways of identifying the solid as you can. Some methods will seem better than others – write down and discuss the advantages and disadvantages of each of the methods that you have devised.

Discussion can play a vital part in working out solutions to problems like this and sufficient time should be spent discussing the different methods and their advantages and disadvantages – perhaps 10 minutes initially with further discussion as required.

3. Devise a systematic procedure to identify the solid. Get this checked for safety.

Note – You should ask for the solid at this point.

4. Use your procedure to identify the solid.
5. Working as a group, prepare a short (ca 5-minute maximum) presentation to give to the rest of the class. If possible all group members should take part: any method of presentation (such as a blackboard, overhead projector, etc) can be used.

Outline the problem, explain how you selected possible compounds, explain the systematic procedure you devised to identify the solid, and describe how well it worked in practice. After the presentation, be prepared to accept and answer questions and to discuss what you did with the rest of the class.

Background information

A useful discussion of the colours of hydrated copper(II) compounds can be found in an article entitled Demonstration of ionic dissociation in aqueous solution, J. Chem. Ed., 1990, 67, 950.

For many years no anhydrous nitrates of transition metals were known. Heating the hydrated salts simply decomposed them because water is a stronger ligand than nitrate. Thus heating a hydrated nitrate prepared from aqueous solution will drive off the nitrate group rather than the water. Anhydrous nitrates have to be prepared without water rather than heating hydrated nitrates to try to remove water from them.

The preparation of anhydrous copper(II) nitrate was reported in 1957.4 Unlike anhydrous copper(II) sulphate, it exists as deep blue-green crystals. It is made by reacting copper metal with dinitrogen tetroxide5 in the absence of moisture. This gives $Cu(NO_3)_2.N_2O_4$, with structure $NO^+[Cu(NO_3)_3]^-$, which on heating to 90°C gives the anhydrous salt. The anhydrous nitrate sublimes in vacuo at 150–200°C producing deep blue-green crystals. The anhydrous nitrate is a monomeric vapour.

There are two different crystal forms of the solid:

— in one form, copper is 6-coordinate. There are six oxygen ligands from nitrates round each copper ion, and each nitrate is joined to two copper ions;

and

— in the other form, copper is 8-coordinate with infinite chains of copper and nitrate groups. There are two short (0.19 nm) copper-oxygen bonds in the chain, and the copper coordinates to six more oxygens in adjacent chains with longer (0.25 nm) copper-oxygen bonds. (See op. cit.)

Notes

1. Copper(II) iodide does not exist; coloured copper(II) compounds cannot be issued thus excluding copper(II) carbonate and copper(II) nitrate (see Background information).

2. Two useful articles detailing convenient laboratory preparations of chlorine water are: Methods for preparing aqueous solutions of chlorine and bromine for halogen displacement reactions, J. Chem. Ed., 1987, 64, 156. The preparation of halogen waters, J. Chem. Ed., 1991, 68, 932.

3. J. D. Lee, Concise inorganic chemistry, 4th ed., London: Chapman and Hall, 1991.

4. C. C. Addison and B. J. Hathaway, Proc. Chem. Soc., London, 1957, 19. Details of the laboratory preparation are given in G. Pass and H. Sutcliffe, Practical inorganic chemistry, London: Chapman and Hall,1968.

5. Dinitrogen tetroxide is uniquely useful as a non-aqueous solvent for preparing anhydrous metal nitrates. Much of its chemistry can be rationalised in terms of a self-ionisation equilibrium $N_2O_4 \rightleftharpoons NO^+ + NO_3^-$ although there is no physical evidence for this equilibrium in the pure liquid.

6. N. N. Greenwood and W. Earnshaw, Chemistry of the elements, Oxford: Pergamon Press, 1985.

Which set of ionic compounds?

Devise a procedure to identify the four solids and then use this to carry out the identification.

Four unnamed white solids are provided. Each solid is made up of one cation and one anion from the groups below. None of the solids contains an anion or cation that is used in any of the others.

Cations	Anions
Ba^{2+} Cu^{2+}	CO_3^{2-} I^-
K^+ Na^+	NO_3^- SO_4^{2-}

You should refer to any sources of information that you think might help such as your notebooks, textbooks and data books. Ask for assistance if you get stuck.

Safety

Normal safety procedures when handling chemicals should be adhered to and eye protection worn.

You must get your method checked for safety before starting on the practical work.