Determining empirical formula

Posted by Anonymous on Mon, 2014-10-20 18:38

Determining empirical formula: Is it safe to heat copper sulfate powder as an alternative to Barium chloride to determine empirical formula? CLEAPSS tells me not to heat to dryness as decomposition produces toxic and corrosive fumes. What are they? Is it still ok to heat Barium chloride?

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Australian Curriculum:
Chemical Sciences
Year Level:
Senior Secondary
Laboratory Technicians:
Laboratory Technicians

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Answer by delese on question Determining empirical formula

Submitted by sat on 24 October 2014

While the heating of hydrated copper sulphate is a useful activity to demonstrate a chemical change, along with a colour change, it is not recommended as a method for determining an empirical formula. This is because of the difficulty in removing all of the water of crystallisation by heating, without also decomposing some of the copper sulphate. The use of

hydrated barium chloride is preferred, as it will not decompose under laboratory heating conditions. See below for further details and alternative reagents suited to empirical determination experiments.

Dehydration of copper sulphate pentahydrate

When copper sulphate pentahydrate is heated, the loss of water of crystallisation proceeds in a stepwise manner. From 30 to 110°C, copper sulphate pentahydrate loses two molecules of water to give the trihydrate, followed by the loss of two more water molecules to give the pale blue monohydrate. Further heating above 150°C gives white anhydrous copper sulphate.

$CuSO_4.5H_2O(s) \rightarrow CuSO_4.3H_2O(s) + 2H_2O(g)$

$CuSO_4.3H_2O(s) \rightarrow CuSO_4.H_2O(s) + 2H_2O(g)$

$CuSO_2.H_2O(s) \rightarrow CuSO_4(s) + H_2O(g)$

Decomposition of copper sulphate occurs at about 650°C. The products of decomposition are black copper (II) oxide and sulphur trioxide:

$CuSO_4(s) \rightarrow CuO(s) + SO_3(g)$

Sulphur trioxide is a highly corrosive substance, which is irritating to the respiratory system. Sulphur trioxide combines with water to form sulphuric acid.

The dehydration of copper sulphate pentahydrate would be safely carried out if the salt is heated in an oven. However, when heating is carried out over a Bunsen flame, it is not difficult to decompose at least some of the sample.

The dehydration of copper sulphate pentahydrate is more suited to a qualitative demonstration of a reversible reaction, where complete dehydration of the salt is not required.

Dehydration of barium chloride dihydrate

Barium chloride dihydrate loses its water of crystallisation when heated to 120°C. Its melting point is greater than 900°C and it therefore can withstand heating over a Bunsen flame. It is, however, more toxic than copper sulphate and its dehydration does not result in a colour change.

Dehydration of copper chloride dihydrate

Klingshirn *et al.* (2008) report a 'greener alternative' experiment for the determination of an empirical formula. Their method uses copper chloride dihydrate which loses its water of crystallisation at 110°C. Klingshirn *et al.* recommend dehydrating the salt in an oven, although the careful use of a hot plate on a low setting would also be appropriate. According to their method, the salt is placed in a crucible which is placed in an oven, with the crucible lid slightly ajar to allow water vapour to escape. The salt is heated for 30 minutes, removed from the oven, allowed to cool, weighed, and then heated for a further ten minutes. It is then removed from the oven, allowed to cool and reweighed to check that there is no further reduction in mass. The stoichiometry of the hydrated salt is then calculated.

The anhydrous salt can be rehydrated if left exposed to air overnight. However, the authors (Klingshirn *et al.* 2008), recommend that students carry out a steam rehydration of the copper chloride during lesson time as it adds a valuable and enjoyable aspect to the experiment. Steam rehydration can be achieved by placing the copper chloride on a piece of filter paper on a gauze mat which is supported by a ring and ring stand over a steam bath.

This reagent offers the advantage of a dramatic colour change from the blue of the copper chloride dihydrate to the light brown colour of the anhydrate. The experiment also does not generate waste as the copper chloride dihydrate can be re-used.

Oxidation of magnesium to magnesium oxide

The formation and reduction of metal oxides can also be employed in experiments to determine empirical formulae. Magnesium oxide can be generated from magnesium ribbon by heating the magnesium in a crucible over a Bunsen flame. The crucible is placed in a pipe clay triangle (with the lid slightly off-centre in order to expose the metal to air) and is heated until the contents turn grey-white, indicating the transformation of the magnesium to the oxide. The empirical formula of magnesium oxide is then calculated from the difference in the initial mass of the magnesium and that of the magnesium oxide produced. Comprehensive instructions for determining the empirical formula of magnesium oxide in this way can be found at http://www.webassign.net/question_assets/ucscgencheml1/lab_2/manual.html

Other empirical formula determinations can be made by observing the reduction of copper oxide with natural gas (Zidick and Weismann, 1973) or methane (Sanger and Geer, 2002). Sanger and Geer (2002) also report an interesting method for the reduction of copper oxide with aluminium foil in hydrochloric acid.

References

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