

## **Chlorate test**

This will have to be purchased from a lab supply store. It is added to some concentrated sulphuric acid. A red, orange or yellow colour indicates the presence of Chlorate in a Perchlorate sample. The test is fairly sensitive.

Mix about 0.1g of the phenylanthranilic acid with about 15cc concentrated Sulphuric acid to give a blackish/blueish solution. Some of the solid sample to be tested is put in the bottom of a small (preferable white) container and a few drops of the reagent added and put in contact with the sample. The colours above will appear if Chlorate is present. Be careful and do not use too much Perchlorate sample because if it contains a lot of Chlorate you may get splattering when the Chlorate reacts violently with the concentrated acid.

You can also use the test by adding the test reagent to a sample of dissolved Perchlorate to be tested. This can be problematic because if the test reagent is made up for a period of time it seems to give a yellow colour when added to pure water. The sensitivity of the test in solutions is thus:

A 0.4 g/l  $\text{KClO}_3$  solution gives a just barely visible discoloration. The best is to do the test both on clean water and on the unknown. If you then compare the colors of the precipitates side by side you can spot the yellow color more easily if you are testing low concentrations. For concentrations above 2 g/l or so this is not really necessary. The concentration of the indicator may also make a difference. 26.3 mg phenylanthranilic acid in 2.00 ml of 96% Sulfuric acid was used to prepare the indicator solution used here.

## **Perchlorate test**

The following test for Perchlorate is very useful when you are making Perchlorate by letting a Chlorate cell run and run until all the Chloride > Chlorate has been converted to Perchlorate. This test will tell you when Perchlorate has started to form. Methylene blue is used for staining specimens that are to be examined under a microscope. It may be possible to purchase it in a shop that sells dyes. It is sometimes sold in pet shops as a dilute solution for treating diseases of fish.

A 0.3% solution of Methylene blue is made by dissolving 0.3 grams Methylene blue in 100ml water. When a drop of this is put into a (liquid) sample from a cell that has Perchlorate in it the Methylene blue will form an insoluble purple precipitate. The solution should not be acid, if it is the purple ppt. will dissolve and you will not see it. The solutions must also be cold (important). It is fairly sensitive and will detect Perchlorate levels at one gram per litre or less. You should add the Methylene blue to the sample to be tested for to see the color change.

If a drop of the sample is put on a glass slide and a drop of Methylene blue added, this will make the test more sensitive. Persulphates and Dichromate (cell additives) also give a purple colour which is difficult to distinguish from the Perchlorate precipitate. The dilution of these substances will be fairly large and will probably not interfere with the test. Test a sample of your cell at the start of the run in order to see if the Persulphate or Dichromate is causing a purple colour. You will then be able to judge an increase in purple colour as being caused by Perchlorate formation.