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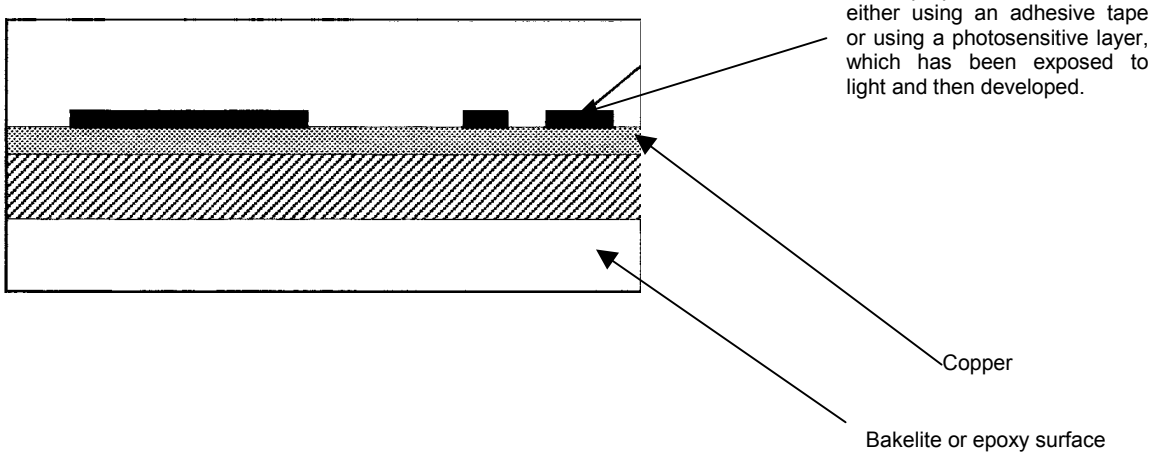
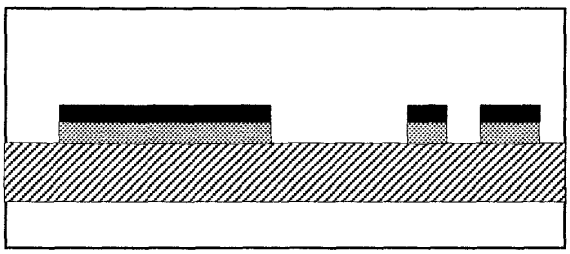
NEUTRALISING IRON PERCHLORIDE $FECl_3$ Réf AU55

BEFORE ETCHING



AFTER ETCHING



The iron perchloride has attacked all parts of the copper, which were not protected.
Note that the bakelite or epoxy surface as well as the opaque film have not been attacked by the iron perchloride.

WHAT HAPPENS DURING ETCHING ?

The plate to be etched is immersed in the $FeCl_3$ bath (iron perchloride) obtained by prior dilution of a concentrated trade solution. The perchloride gradually attacks the copper (for those who want to know everything : the reaction is as follows: $2Fe^{+++} + Cu \rightarrow 2Fe^{++} + Cu^{++}$)

COMMENTS:

Even without being used, the bath releases hydrochloric gas vapour (HCl), which is both corrosive and recognisable by its smell. For example we can observe that after a year, metals which are in the vicinity of the bath

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have been attacked and oxidised. It is, therefore, recommended to locate perchloride baths away from objects or devices, which can oxidise.

Conclusion : When we etch printed circuit boards, we attack the copper, which is on the printed circuit with iron perchloride, and we obtain a solution containing :

Ferric chloride which has not reacted	Ferrous chloride	Copper chloride	and Hydronium cations
$FeCl_3$	$FeCl_2$	$CuCl_2$	H_3O^+

When the bath is saturated (when it has to be renewed), it contains Cl^-, Fe^{3+}, Fe^{2+}, Cu^{2+} and H_3O^+ ions.

This saturated bath, if it were disposed in the natural environment would have a dual polluting effect :
* through its acidity (pH which is much lower than 7), it would attack and destroy the fauna and flora;
* washed out by rainwater, we would find the previously mentioned salts of iron and copper in groundwater.

How can we neutralise an iron chloride bath, which is saturated in copper (waste)?
* In order to eliminate the acidity :
  We simply have to add OH^- ions which characterise bases to the H_3O^+ ions which characterise bases (NaOH, or soda is a base) this can be expressed with the following equation :

$$H_3O^+ + OH^- \Rightarrow 2H_2O$$

* In order to avoid the copper and iron, which it should be remembered are very widely found in nature (copper being the most common metal) ending up in groundwater we have to transform their chlorides into insoluble (or not very soluble) in water compounds.

E.g. :
Whereas $CuSO_4$ and $CuCl_2$ are soluble in water,
copper hydroxide, $Cu(OH)_2$ is not very soluble at all (10 million times less than $CuCl_2$)

Whereas $FeCl_3$ and $FeCl_2$ salts are soluble in water,
$Fe(OH)_3$ and $Fe(OH)_2$ hydroxides dissolve very little in water.

NEUTRALISATION OF WASTE IRON PERCHLORIDE CAN BE CARRIED OUT IN THE FOLLOWING MANNER

$FeCl_3 + FeCl_2 + CuCl_2 + H_2O$ (waste perchloride) + NaOH (sodium peroxide)

$\Downarrow$

$Fe(OH)_2 + Fe(OH)_3$ + $Cu(OH)_2$ + $NaCl$ + H_2O

↓ virtually insoluble iron hydroxides

↓ virtually insoluble copper hydroxides

↓ sodium chloride or common salt

When the mixture is carried out correctly in the right proportions, in other words for the pH to be neither acidic nor basic, we should note that :

1. insoluble hydroxides of iron and copper will be precipitated during the reaction in the form of a red or brown sludge.
2. that the sodium chloride (sea salt that is used in cooking) is harmless and dissolves in the water.

NOW THAT WE HAVE NEUTRALISED THE WASTE IRON PERCHLORIDE, AND MADE IT HARMLESS, AND THAT THE VARIOUS COMPONENTS HAVE SEPARATED, ALL THAT WE NEED TO DO IS :

a) dispose of the filtrate (aqueous solution of NaCl) in the sink.
b) recover the harmless copper and iron hydroxides by filtration and dispose of them either in a landfill or spread them in the natural environment.

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PRACTICAL PROCEDURE FOR THE NEUTRALISATION AND ELIMINATION OF WASTE IRON PERCHLORIDE USING ANHYDROUS NAOH

Please note : this operation can only be carried out by an adult

Explanation:

NaOH : Sodium hydroxide is also called caustic soda since it is a very aggressive product and, therefore, requires considerable precautions to be taken when using.
In the form of white pellets (anhydrous) it can only be handled by people aged at least 18 years old (article R234-21 of the code of work). Indeed, the product is highly soluble in water and absorbs atmospheric moisture and is highly exothermic : when diluted in water it produces a temperature rise and there is risk of splashing on the skin and in the eyes.

As an aqueous solution, generally with a concentration in water of 0.325mol/l, sodium hydroxide is theoretically less dangerous (it attacks living tissue a little less since it is diluted) but it has the major disadvantage of requiring large quantities of solution to be handled. In fact 30 volumes of NaOH diluted to 0.325mol/l are required to neutralise 1 volume of aqueous $FeCl_3$ diluted at 3.25mol/l (usual dilutions). We will chose the solution that involves using the small anhydrous pellets, which makes it compulsory for the handling to be carried out by an adult. This requires the following precautions to be taken :

* Protecting the hands, clothing and above all the eyes against hazardous splashing.
* Handling small quantities at a time (maximum of 2 litres) in order to limit the temperature rise and avoid the reaction to "running away"

The operation that allows the neutralisation of perchloride using anhydrous NaOH requires the following equipment :

- a polyethylene (PE) bucket or one in polyvinyl chloride (PVC) with a volume of 2.5 litres,

- a stirrer (plastic off-cut) which is longer than the height of the bucket,

- goggles, protection gloves, overalls,

- a filter, a densimeter, pH paper, a plastic spoon.

HOW CAN WE FIND OUT WHETHER THE SOLUTION IS NEITHER ACIDIC NOR BASIC?

We know that the pH is neutral (neutralisation condition) when it is around a value of 7 : it is quite easy to obtain between 7 and 9 when, after correct mixing using a stirrer, the pH indicator paper shows a colour corresponding to 7<pH<9.

On the following pages you will find the procedure required to neutralise waste iron perchloride using anhydrous NaOH pellets described schematically.

PROPOSAL OF A DIFFERENT PROCEDURE ENABLING THE NEUTRALISATION OF PERCHLORIDE

Sodium carbonate, Na_2CO_3 (not very hazardous) can replace the caustic soda NaOH, it leads to the same products as the soda and can be used without hazard, but it has certain handling disadvantages.

In fact the reaction is as follows :

$$2\ Fe^{+++} + 3\ CO_3^{--} + 3\ H_2O \rightarrow 2\ Fe(OH)_3 \downarrow + 3\ CO_2 \uparrow$$

$$Cu^{++} + CO_3^{--} + H_2O \rightarrow Cu(OH)_2 \downarrow + CO2 \uparrow$$

Therefore we have precipitation of iron and copper hydroxides in the form of sludge as in the previous procedure, but there is also the release of carbon dioxide which produces a foam to such an extent that it tends to make the product overflow from the container.

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This solution is, therefore, only viable if we manage to wait for the disappearance of the foam between two additions of small quantities of sodium carbonate. For example, we have to add one teaspoon per hour of sodium carbonate over a 2-day period in order to have full neutralisation of 5 litres of waste iron perchloride

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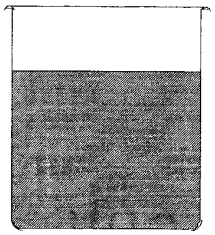
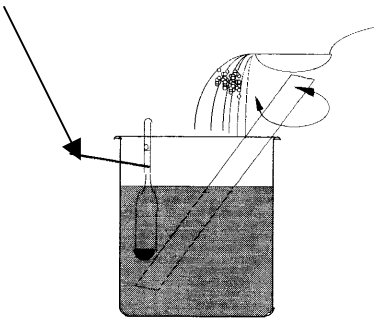
1)



Pour 2 litres of waste iron perchloride into the 2.5 litres plastic bucket. Place all of this in a sink for maximum safety.

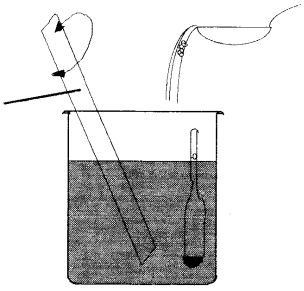
2)

Densimeter



Put in a densimeter and pour in 1 soupspoon of anhydrous sodium hydroxide. Stir with the plastic stirrer (which can be a plastic off-cut).

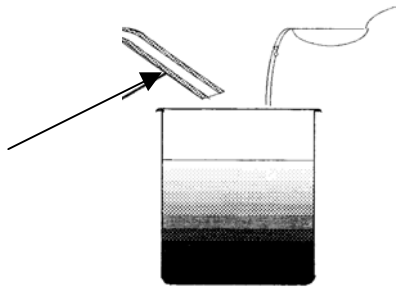
3)



Wait for 2 minutes then add the sodium hydroxide little by little whilst stirring until the densimeter shows a pH approximately equal to 7 (the "zero" graduation of the densimeter arrives at the liquid surface). Again wait for a few minutes.

4)

pH paper



Use the pH paper* to accurately check that the mixture is neutral. Add small quantities of sodium hydroxide whilst monitoring the change in colour of the pH paper. When the pH stabilises at around 7 after mixing, we can observe that a brown sludge is deposited at the bottom of the bucket, whilst the liquid becomes increasingly clear.

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*** TO CHECK THE PH USING PH PAPER :**

The paper must not change colour, the initial yellow/orange (7.0) must be stable. If the colour changes to become more red/blue, you have to add a little neutralising agent and repeat operation 4.

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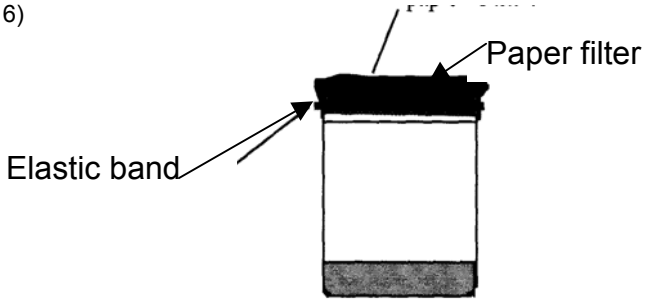
5)

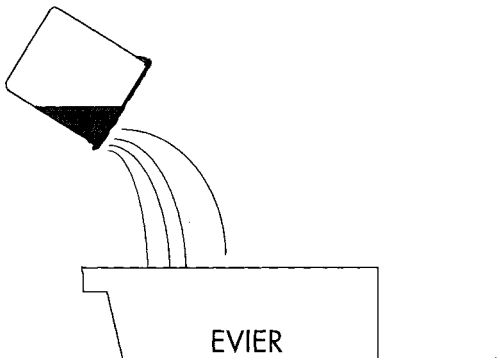
After 5 minutes, all of the sludge has deposited at the bottom of the bucket.

6)



Position and attach filter paper on the container so that the sludge remains in the container when it is turned upside down.

7)



SINK

Pour out the clear liquid into the sink (it is salt water and can be poured to waste). Put the sludge in a plastic container. This sludge can be thrown away either in a landfill or spread on a field.
In town, it may be disposed of using normal waste disposal procedures.

Please note : The procedure described is applicable for a saturated iron perchloride bath.
When the bath is half-saturated, more neutralising agent will have to be used (up to 3 times more).

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