# Guideline for the Minimisation of Mercury Emissions and Waste from Mercury Chlor-Alkali Plants

## Env. Prot. 13

## 3rd Edition

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# Euro Chlor

Euro Chlor is the European federation which represents the producers of chlorine and its primary derivatives.

Euro Chlor is working to:

- improve awareness and understanding of the contribution that chlorine chemistry has made to the thousands of products, which have improved our health, nutrition, standard of living and quality of life;
- maintain open and timely dialogue with regulators, politicians, scientists, the media and other interested stakeholders in the debate on chlorine;
- ensure our industry contributes actively to any public, regulatory or scientific debate and provides balanced and objective science-based information to help answer questions about chlorine and its derivatives;
- promote the best safety, health and environmental practices in the manufacture, handling and use of chlor-alkali products in order to assist our members in achieving continuous improvements (*Responsible Care)*.

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This reference manual is intended to give only guidelines and recommendations. The information is provided in good faith and has been based on the best information currently available. The information is to be relied upon at the user's own risk. Euro Chlor and its members make no guarantee and assume no liability whatsoever for the use and the interpretation of or the reliance on any of the information in this document.

Prior to 1990, Euro Chlor's technical activities took place under the name BITC (Bureau International Technique du Chlore). References to BITC documents may be assumed to be to Euro Chlor documents.

# RESPONSIBLE CARE IN ACTION

Chlorine is essential in the chemical industry and consequently there is a need for chlorine to be produced, stored, transported and used. The chlorine industry has co-operated over many years to ensure the well-being of its employees, local communities and the wider environment. This document is one in a series which the European producers, acting through Euro Chlor, have drawn up to promote continuous improvement in the general standards of health, safety and the environment associated with chlorine manufacture in the spirit of *Responsible Care*.

The voluntary recommendations, techniques and standards presented in these documents are based on the experiences and best practices adopted by member companies of Euro Chlor at their date of issue. They can be taken into account in full or partly, whenever companies decide it individually, in the operation of existing processes and in the design of new installations. They are in no way intended as a substitute for the relevant national or international regulations which should be fully complied with.

It has been assumed in the preparation of these publications that the users will ensure that the contents are relevant to the application selected and are correctly applied by appropriately qualified and experienced people for whose guidance they have been prepared. The contents are based on the most authoritative information available at the time of writing and on good engineering, medical or technical practice but it is essential to take account of appropriate subsequent developments or legislation. As a result, the text may be modified in the future to incorporate evolution of these and other factors.

This edition of the document has been drawn up by the Environmental Protection Working Group to whom all suggestions concerning possible revision should be addressed through the offices of Euro Chlor.

# Summary of the Main Modifications in this version

| Section | Nature |
|---|---|
| All | General review with simplification and update of the expected performance emission figures based on latest data gathered |

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# 1. INTERPRETATION OF THIS GUIDELINE

This document presents a statement of the best practices for chlorine production plants using the amalgam chlor-alkali electrolysis process, in relation to mercury emissions to air, to water and in products. It includes also information on the handling of solid wastes coming from normal operation, and has been based on the experience of existing plants whose current performance provides the best examples of mercury emission control (see ***Env. Prot. 11***).

Information on waste from maintenance of equipment and buildings and their demolition is contained in the document ***Env. Prot. 3***.

This document has been written to provide guidelines for the performance that can be achieved in each emission category expressed primarily as grams of mercury per tonne of chlorine capacity. Whilst some flexibility may be needed within an individual category, the guidelines lead to a maximum level of total mercury emission from all categories that are applicable to all plants. The guidelines are intended to represent the emission performance that will be achieved over the course of the year. They are not intended to be an instantaneous standard of performance which will be achieved by employing a particular technique, because there will be some variation depending for example on the work load of the plant or weather conditions, equipment being under maintenance, etc. The quoted value is the maximum level of total mercury emission achieved over the year if the techniques are applied as described.

Remark

Most of these principles can also be applied to the other electrolysis processes using mercury (production of alkoxides and dithionites).

# 2. INTRODUCTION

In the electrolysis of brine for the production of chlorine by the mercury process, the mercury is in intimate contact with the raw materials and some of the final products. It is thus inevitable that the untreated process streams contain mercury. A key element of operating this technology is to apply techniques that minimise any contamination in the final discharged streams and that, wherever possible, the mercury removed is recycled within the process. The purpose of this document is to describe these techniques.

# 3. GENERAL INFORMATION

Due to the process characteristics, mercury can be emitted from the process through air, water and in the products; solid waste sent to safe deposits can also contain some mercury.

## 3.1.Emissions from Gaseous Streams

Hydrogen and process exhaust streams are generally cooled (some mercury condensates as metal and can be directly recycled) and then treated in operations involving absorption or reaction of the mercury (for example calomel formation in a washing column, fixation on copper or carbon impregnated with sulphur or iodine). For some of these operations the mercury emerges in a liquid stream, which may be recycled to the brine system or treated as in paragraph 3.2. For other operations the mercury emerges in a solid stream, which may be treated as in paragraph 3.4.

Chlorine emerging from the cells contains only small amounts of mercury; the normal processes of gas cooling and washing reduce the mercury content down to insignificant concentrations. The mercury emerges from these operations in a liquid stream, which may be recycled to the brine system or treated as in paragraph3.2.

After the treatments described below (paragraph 4.6), the remaining total mercury emission from the gasses streams is expected to be lower than 0.1 g/t $Cl_2$ capacity.

## 3.2.Emissions from Liquid Streams

Mercury in liquid effluent streams can be removed by the following processes:

- Precipitation of mercury sulphide which is then filtered; the precipitate can be dissolved (solution containing active chlorine) and recycled in the brine loop or treated as solid waste (see paragraph 3.4). The use of thiourea or other special mercury binders like trimercaptotriazine is also possible.

- Absorption on ion exchange resins; the regeneration of those resins gives a mercury concentrated liquid that can be recycled in the brine.

- Treatment by a reducing agent in order to precipitate metallic mercury that is filtered and recycled.

The caustic solution produced is purified from mercury by passing through a granulated active carbon bed, giving rise to solid waste which may be treated as in paragraph 3.4.

Sometimes, the treated liquid stream is further ‘polished’ by filters. This can give rise to additional solid waste.

After the treatments described below (paragraph 4.1), the remaining total mercury emission from the liquid effluent streams is expected to be lower than 0.1 g/t $Cl_2$ capacity.

### 3.3. Other Sources of Mercury Emissions

Mercury emissions in the air of the cell room are influenced by its basic design (open or closed room, number of floors, natural or forced ventilation, direction of flow …), the design and area of the cells, the leak tightness, the type of decomposers, the accessibility of the cells and the construction materials …. These emissions are also influenced by the use of good operating and maintenance techniques which minimise the likelihood of mercury emissions (see ***Env. Prot. 5***).

Some accidental mercury spillage can occur during maintenance of cells or decomposers, e.g. opening the cells for anode changing or cleaning, assembling or dismantling equipment, or replacing defective pipes. If this mercury is not recovered quickly, it will continuously evaporate and cause supplementary emissions in the cell room.

A good maintenance and operating strategy can increase the lifetime of the cells parts and reduce the requirement to open them.

Mercury emissions are significantly reduced by good housekeeping practices. This topic is covered in detail in ***Env. Prot. 11.*** The remaining mercury emission from the cell room is expected to be lower than 1 g/t $Cl_2$ capacity.

### 3.4. Solid Waste

Depending on its composition, solid waste produced within the process may be treated chemically to re-dissolve ionic mercury in a liquid (then treated as a liquid stream) or can be retorted (the gaseous stream is treated like the other gaseous streams and the final solid waste ash is disposed of to secured landfill or underground mines).

Scrap pipes, vessels, pumps, building materials and rubber coating will contain some low residual concentration of mercury. This may be recoverable by the techniques described in ***Env. Prot. 3***.

According to the local legislation, some solid wastes may be suitable for disposal in secure landfill or underground mines without further treatment.

Generalised flow schemes are shown in Figures 1 and 2.

## 4. SPECIFIC TECHNIQUES

### 4.1 Brine and Water Systems

a) Technique

Three principal processes are used to purify depleted brine leaving the plant, and any other mercury-containing waste water.

They would normally include a first settling to remove large mercury droplets, followed, if necessary, by oxidation using hypochlorite, chlorine containing water/brine or hydrogen peroxide to dissolve metallic mercury.

- Precipitation of mercuric sulphide, followed by filtration

By adding sulphide or bisulphide, ionic mercury is precipitated as mercuric sulphide. The solid sulphide is filtered from the waste water on sand, candle or plate filters, which may involve using a filter aid.

The filter cake may be then

- dissolved in hypochlorite, chlorine containing water/brine or hydrogen peroxide, and recycled to the brine system, leaving the filter aid (if any) and other precipitated solids as a solid waste
- treated thermally with air for recovering Hg, taking care of the formation of $SO_2$; the gaseous stream is then treated like a process exhaust
- discharged in a secure landfill or salt mine

- Ion exchange to remove mercury from solution

Usually the resin is regenerated with hydrochloric acid giving a mercury-containing liquor which can be recycled to the brine system.

- Chemical reduction to metallic mercury for recycling in the cells

In the reduction process, an activated charcoal filter is used to separate the metal mercury obtained by adding a reducing agent (for example, hydroxylamine, $NaBH_4$ etc.) from the liquid effluent. After filtration the mercury obtained from this step can be directly recycled to the cells. Care must be taken to avoid recycling nitrogen compounds into the system ($NCl_3$ formation risk). Additional precautions with the hazardous properties of the reagents used have to be taken into consideration.

Other techniques that have been considered:

- Electrochemical reduction to metallic mercury for recycling in the cells

The reduction process is carried out on a fixed-bed carbon electrode to remove mercury from waste water. This technique was tested by some members but is not used due to difficulties in its industrialisation.

- Biochemical reduction to metallic mercury for recycling in the cells

This technique proposed by the University of Braunschweig (Germany) has been tested by some members, but its effectiveness was inferior to the principal techniques mentioned above.

b) Performances

Depending on the local situation, the specific flows …, these processes should reduce mercury in treated final water stream to lower than 0.1 g of mercury per tonne of chlorine of capacity.

c) Sampling and analysis

The concentration of the mercury in the liquid effluent leaving the plant should be checked at least as a daily average.

The analytical method has been defined in the Euro Chlor documents ***Analytical 3, Analytical 6 and Analytical 7***.

d) Remarks

The residual sludge from water and brine treatment may contain some mercury and can be treated as in paragraph 4.8.

## 4.2 Caustic Soda and Caustic Potash

a) Technique

There are a variety of filters used within the industry to remove mercury from sodium and potassium hydroxides. These include:

- Plate filters with carbon pre-coat
- Candle filters with carbon pre-coat
- Candle filters without pre-coat.

Although all types of filter can achieve very low levels of mercury in the product, the predominant technique is the plate filter with carbon pre-coat.

The efficiency of the filter depends on a number of factors:

- it must carefully be assembled according to the manufacturers' instructions so as to avoid leak paths
- the pre-coat deposition technique must give a consistent and homogeneous pre-coat layer
- the throughput, temperature and pressure of the caustic soda must be maintained within the design range and should be controlled to avoid rapid changes. As an example, a plate filter uses a pre-coat of 3-6 $kg/m^2$ of filter area, and operates with a throughput of 0.5-1.0 $m^3/m^2.h$ at a temperature of 50-70°C.

The use of a settling stage, e.g. a holding tank upstream of the filter, will assist filter efficiency and reduce the frequency of filter cleaning.

b) Performances

Such filters will reduce the mercury in caustic soda to about 0.05 mg/kg of 50% caustic soda liquor, which corresponds to about 0.1 g per tonne of chlorine capacity.

c) Sampling and analysis

The concentration of mercury in the product should be checked after filtration at least once per day. The sample point should be arranged to avoid contaminating the sample with mercury droplets, which may have settled in the pipework.

When a new pre-coat has been deposited, the concentration of mercury in the product should be checked several times per day until the required performance has been established.

d) Remarks

The mercury-laden filter material can be treated in a retort to recover the mercury for future use (see section 4.8).

Because of viscosity considerations, caustic is filtered hot. After filtration the caustic is cooled. This may result in further deposition of some mercury in caustic pipework and tanks. Where possible, this mercury is drained off and, after treatment (distillation) if necessary, returned to the mercury store. If this is not possible, the mercury remains in the tanks etc, until maintenance work or decommissioning is undertaken and the mercury is then recovered.

## 4.3 Hydrogen

It must be reminded that residual mercury air emissions from hydrogen burned must be considered as “process exhaust” and are not included in the emissions with the products.

a) Technique

Hydrogen leaves the decomposers saturated with water and mercury at a temperature usually between 90°C and 130°C. Mercury is generally removed in a multi-stage process.

In the first stage, the hydrogen is cooled by a heat exchanger mounted immediately above the cell or a cooler within the decomposer. The condensed mercury is recycled with water directly to the decomposer.

The second stage may involve either:

- Chilling or washing with chilled water
- compression and cooling
- scrubbing with hypochlorite

- use of a calomel reaction

In the 2 first options, mercury is recovered as metallic mercury; in the 2 last options, the liquid containing ionic mercury can be recirculated in the brine.

A third stage may involve either iodised or sulphurised charcoal, after sufficient increase of the hydrogen temperature (10 to 20°C) to avoid water condensation in the charcoal bed. The operating temperature is also important for the efficiency of the process, together with the granule size of the charcoal and the flow rate of hydrogen.

This third stage can also be carried out by reaction of mercury with copper on an alumina carrier at temperatures between 2°C and 20°C. The copper can be regenerated by heating up and the mercury recycled.

b) Performances

These techniques should reduce the mercury content to lower than 30 $\mu g/m^3$ of hydrogen at 1 bar, which corresponds to about 0.01 g per tonne of chlorine capacity.

If the hydrogen is compressed before treatment, then an even lower mercury concentration can be achieved (e.g. 0.003 g per tonne of chlorine capacity as yearly average, which corresponds to about 10 $\mu g/m^3$ of hydrogen at 1 bar).

c) Sampling and analysis

The concentration of mercury in the hydrogen leaving the plant as a product should be checked at least once per week. Sample points should be arranged to avoid contaminating the sample with solid material, e.g. dust from the charcoal bed, which may have settled in the pipework.

The analytical method has been defined in the Euro Chlor document ***Analytical 6***.

If purified hydrogen is used in the manufacture of hydrochloric acid by the reaction of hydrogen and chlorine, analysis of the resulting acid solution for mercury provides a satisfactory monitoring method for the mercury concentration in hydrogen. The sampling and analysis are easier here than with hydrogen gas, and the fact that chlorine contains virtually no mercury in chlorine as described below.

## *4.4 Chlorine*

a) Technique and performances

Chlorine leaves the cells at temperatures between 80°C and 95°C. The chlorine gas is usually cooled with chilled water to about 15°C (30 °C if no chilled water is available). No further mercury treatment is required for gaseous chlorine as it contains negligible traces of mercury (i.e. less than 0.001 g/t).

Note: due to the risk of solid chlorine hydrate formation, the temperature should be kept above 15 °C.

b) Sampling and analysis

The analytical method has been defined in the Euro Chlor ***Analytical 6***.

c) Remarks

The condensate from chlorine cooling will contain a small amount of mercury driven by the chlorine leaving the electrolysers and can be recycled in the brine.

## *4.5 Sodium hypochlorite*

When the chlorine is coming from the residual liquefaction gas, the mercury content of sodium hypochlorite will primarily reflect the residual mercury traces of the purified caustic soda. If the chlorine comes directly from the cells, washing with chlorinated brine can be beneficial.

No mercury removal processes are used for the hypochlorite produced. Typical mercury content is lower than 30 mg of mercury per tonne sodium hypochlorite product (cf. also hypochlorite risk assessment data).

## *4.6 Process Exhausts*

a) Description

Process exhaust refers to all gaseous streams by which mercury can be emitted to the atmosphere, apart from cell room ventilation air and hydrogen as product. Each plant may have of variety of streams but the typical streams which may have significant mercury content to require the use of a treatment process will include:

- Purge air from cell end-boxes
- Vents from wash water collection tanks
- Exhaust from any vacuum system used to collect spilled mercury (but some portable vacuum cleaner have their own mercury absorption system)
- Hydrogen burnt as a fuel, either by the company operating the chlorine plant or by another company to whom it has been sold as a fuel
- Hydrogen vented to atmosphere due to lack of downstream outlets
- Vents from caustic soda pumping tanks
- Vents from caustic soda filters
- Distillation units for mercury-contaminated solid wastes

- Vents from the mercury contamined waste storage (if not in closed recipients).

Safety considerations will almost certainly require separate units for different streams. For example, hydrogen streams should not be mixed with streams containing significant amounts of air to avoid formation of explosive mixtures.

Other streams are likely to contain some mercury, but in such low concentrations that they do not require treatment; they correspond to venting of:

- brine saturators
- brine filters and treatment tanks
- caustic soda stock tanks

b) Technique

Two principal single-stage processes are capable of achieving low levels of mercury in process exhaust:

- A chemical process, e.g. scrubbing with hypochlorite or with chlorinated brine, or use of a calomel reaction, which allows for direct recycling of mercury to the brine circuit
- Use of iodised or sulphurised charcoal. In this case, the mercury can then be recovered and recycled as indicated for the hydrogen in section 4.3 point b); the possible risk of carbon ignition has to be taken into consideration when oxygen or chlorine are present in the gas.

c) Performances

As the same techniques are used, the usual performances are quite similar to what can be obtained for hydrogen purification, about 30 µg Hg/m$^3$. Due to the wide range of process exhaust flows related to the chlorine production capacities, the corresponding emission can vary widely (see also section 4.7 point b).

d) Sampling and analysis

The concentration of mercury in the major gaseous streams should be checked at least once per week. The flow rate of each stream should be checked at least once per year, although more frequent measurements may be necessary to cover the range of the plant operating conditions. The analytical method has been defined in the Euro Chlor guideline ***Analytical 6***.

e) Remarks

It can be necessary to vent hydrogen at the cell room in emergency or upset conditions. Safety considerations dictate that no treatment system should be used on this stream, despite the mercury content, in view of the fact that differential pressure across the system would restrict hydrogen flow and compromise the safety feature.

Regardless of whether they include a treatment process or not, all mercury-contaminated streams must be taken into account in calculating the total mercury loss in process exhaust during the year. Releases during periods when a treatment process is shut-down must also be considered.

## 4.7 Cell room ventilation

a) Technique

Mercury cells are set up in very large buildings and a huge volume of ventilation air is needed to remove heat from the cells. Due to this huge volume, together with the fact that the ventilation flow escape to the atmosphere from many points, the use of mercury removal process is not practical. Mercury losses are influenced:

- by the basic design of the cell room and the cells, for example
    - number of the floors and cells,
    - area of the cells,
    - leak tightness,
    - type of decomposers (horizontal or vertical),
    - accessibility,
    - materials of construction,
- by use of operating and maintenance techniques which minimise the possibility for mercury emission;

by good housekeeping practices; the Euro Chlor document ***Env Prot 11 - Code of Practice - Mercury Housekeeping*** sets out in detail the good housekeeping practices which have been developed in the industry over the last 50 years.

## 4.8 Solid Wastes from Normal Operation

These consist principally of:

- Solids from brine purification
- Solids from caustic filtration
- Solids from water treatment
- Solids from sludge (sewer, traps, channels)
- Activated carbon from gaseous streams treatment

- Decomposer carbon from decomposer packing
- Residues from retorts.

The appropriate treatment depends on the form of the waste. If the mercury is predominantly in the inorganic state, treatment with aqueous chemicals should consider the quantity of contaminated water which will need to be treated.

Available techniques include:

(a) Mechanical/physical treatment

Suitable if significant quantities of metallic mercury are present and can be recycled. Care must be taken not to release mercury to the atmosphere. The final solid residue is disposed of properly (land-filled or stored in underground mines).

(b) Chemical treatment

Techniques include treating with hypochlorite, chlorinated brine or hydrogen peroxide. The liquid streams are treated as in 3.2, with the ionic mercury recycled to the brine system.

(c) Distillation

Distillation or retorting is carried out in specially designed units installed on site or at external facilities. The mercury is recovered as metallic mercury and can be stored for future make-up to the cells, if required. If not recycled, the mercury can be sent for stabilisation and disposal.

Special attention should be given to the treatment of the exhaust gases from these units. They should be treated as in 4.6 (Process Exhausts). The solid residue is land-filled or stored in underground mines.

A generalised table for handling solid wastes is shown in Figure 2.

These are covered comprehensively in ***Env.Prot. 3***.

# 5. SUMMARY

Whilst recognising that the particular circumstances of each plant will require some flexibility in the standards which can sensibly be set for individual emission categories, the application of the techniques described above will normally result in a maximum total emission of 1 g of mercury per tonne of chlorine capacity on an annual basis, based on the described guidelines for each potential emission category[1].

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[1] 1.2 g/t in the case of caustic potash and other specialties

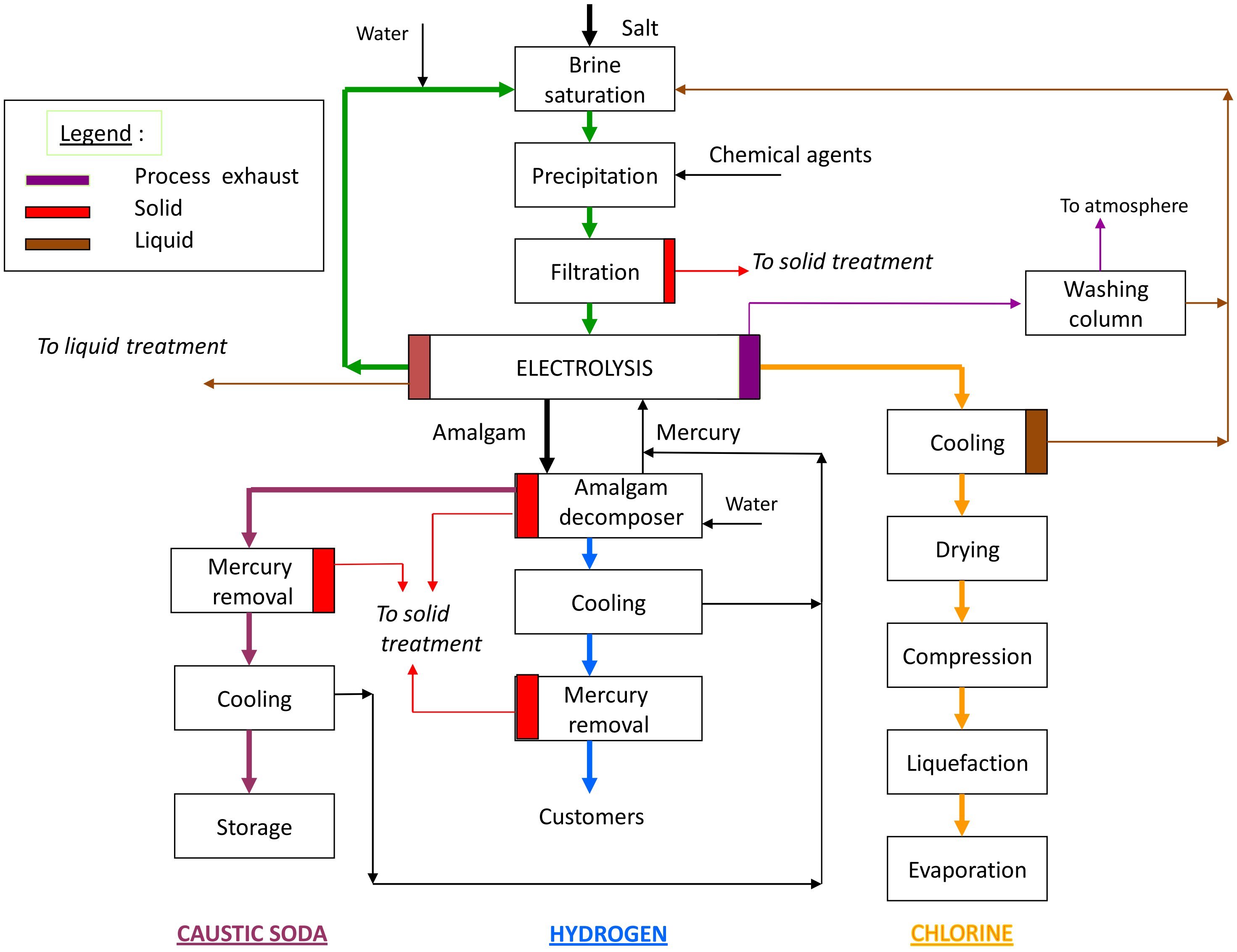
The industry recognises that control and abatement techniques cannot be applied for optimum effect at all sites and under all operating conditions.

Whilst, for reasons such as design, age and geography, not all plants may be able to achieve these figures, no individual plant should exceed a total mercury emission limit to air, water and in products of 1.5 g/t chlorine capacity.

# 6. REFERENCES

- ***Analytical 3 - Determination of Mercury in Solids***
- ***Analytical 6 - Determination of Mercury in Gasses***
- ***Analytical 7 - Determination of Mercury in Liquids***
- ***Env Prot 3 - Decommissioning of Mercury Chlor Alkali Plants***
- ***Env Prot 5 - Measurement of Air Flow and Mercury in Cell Room Ventilation***
- ***Env Prot 11 - Code of Practice - Mercury Housekeeping***

**Figure 1: Mercury Recycling in the Mercury Process**



# Figure 2: Waste Treatment Possibilities

| Materials typical contamination | | | Possible treatments | | |
|---|---|---|---|---|---|
| **Material** | **Typical Hg content %w/w** | **Physical state** | **Physical/mechanical treatment / Water washing** | **Chemical washing** | **Retorting** |
| Sludge from storage tanks and sumps | 10 - 30 | wet solid | ■ | | ■ |
| Sludge from settling catch pits, drains etc. | 2 - 80 | wet solid | ■ | | ■ |
| Sulphurised or iodised charcoal from hydrogen purification | 10 - 20 | dry solid | ■ | ■ | |
| Carbon from caustic filters | up to 40 | wet solid | ■ | | ■ |
| Graphite from decomposers | 2 | porous solid | ■ | ■ | ■ |
| Rubber/packing | variable | variable | ■ | ■ | |
| Brick work/concrete | 0.01-0.1 | dry solid | ■ | ■ | ■ |
| Ebonite-lined cell components (anodes covers, end boxes, side walls, pipework) | variable | inhomogeneous contamination | | | ■ |
| Steel (cells, decomposers, scrap components from baffles, H2 coolers, base plates, Hg pumps, pipework) | 0.001 - 1 | solid with surface contamination | ■ | ■ | ■ |

| Materials typical contamination | | | Possible treatments | | |
|---|---|---|---|---|---|
| **Material** | **Typical Hg content %w/w** | **Physical state** | **Physical/mechanical treatment / Water washing** | **Chemical washing** | **Retorting** |
| Plastic equipment | <0.1 | solid with surface contamination | | 🟪 | 🟪 |
| copper conductors | 0.04 | solid with surface contamination | | 🟪 | 🟪 (for multi sheets flexibles) |
| cell sealant (layers concrete) | 0.01 | | | 🟪 | 🟪 |
| asphalt | 1 - 20 % | non-homogeneous contamination | | 🟪 | 🟪 |
| concrete and subfloor | variable | non-homogeneous contamination | | 🟪 | 🟪 |
| wood | variable | contamination | | 🟪 | 🟪 |
| soil | variable | non-homogeneous contamination | | 🟪 | 🟪 |
| decomposer lagging (thermal insulation) | 0.03 | contamination | No treatment before disposing off | | |
| Retort residues | < 0.1 – 0.1 | dry porous solid | | | |
| Wooden floor boards | 0.05-0.08 | non-homogeneous contamination | | | |

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Details of membership categories and fees are available from:

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