### Name of Process: Autoclaving

#### Applicable POPs wastes:
- PCB's and not for other POPs

#### Status:
Autoclaving is a technology that has been around for many years now and is well proven. In general for PCBs, only the oil and transformer components such as ceramics, cardboard and wood are incinerated. After decontamination, the various metals such as copper, steel and aluminium are sent to the metals recycling industry.

In fact, the autoclaving cannot be compared with the other technologies dealt with, as it does not treat the PCB itself, but its objective is the clean-up and re-use of the with PCB contaminated materials, which is a completely different purpose as the other technologies. One has to consider that 98% of the contaminated oil is not going into the autoclaving but directly to the incinerator!

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#### Technology description:
Autoclaving is a solvent decontamination process that extracts PCBs from contaminated material. The process is most often used in projects in conjunction with high temperature incineration.

For capacitors the process involves shredding and placing all the material into the autoclaving chamber and by vacuum extraction with solvent remove the PCB. The condensers solid materials, after sawing and decontamination, are totally treated in the incineration plant through the solid burden way, and only the ceramics can then be landfilled. The extracted oil and PCBs is sent for HTI incineration, Alkali metal reduction or other alternative technologies.

Transformers on the other hand are drained, completely disassembled including the core and windings and the casing and all components are placed in the autoclave chamber and decontaminated (SBC, 2002).

In the early 1980’s it was found that the conventional approach to cleaning PCB transformers, namely the method whereby the units were rinsed (3 to 6 times) and soaked with oil or solvent, could not effectively bring concentrations down to 50mg/kg of PCBs, unless initial PCB concentrations were very low. The usual rinse and soak procedure used previously could be adequate for the decontamination of casings only. It was not adequate for the decontamination of transformer internal components. Concentrations of 0.5% to 4% of PCBs remained associated with the core and windings after 18 hours of soaking and rinsing.

These findings influenced the design of equipment and operation methods so that the solvent could reach into internal transformer components. A as a result, it has become possible to process materials and meet likely government guidelines. This work has resulted in yet more advanced decontamination procedures which are now the standard for this system.

The technology is also used to decontaminate a wide variety of PCB contaminated equipment and materials, including: electro-magnets, breakers, relays, ballasts, cables, radiators, drums, piping, vessels, valves and debris. Various other equipment and materials are evaluated on a case basis.

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![Diagram of Autoclave System](image)

**Figure 1: Autoclave system**
Figure 2: Autoclaves Process Flow Sheet example France
**Description of Flow sheet:** example France for 9 autoclaves

Type: front door autoclave, material: stainless steel AISI 316. The autoclave door is opened by means of a hydraulic pump. The autoclave area is set on a concrete area whose dimensions were calculated in order to prevent any eventual leakage of oil and solvent. This retention capacity is lined with appropriate epoxy paint for corrosion protection.

**Process basis**

The property of certain solvents to solubilize PCBs is used to extract these contaminants from casings, cores and windings. This allows transformer and capacitor metal components to be recuperated. The extraction solvent disrobes and solubilizes the PCBs from casing wall surfaces and from internal cores, coils and windings. The efficiency of decontamination is a function of induced cycles of phase changes of the extraction solvent within the material containing the PCBs. The solvent is regenerated through distillation in a concurrent process step. The distillation process minimizes the volume of the extracted PCBs requiring storage or disposal. The clean distilled solvent is not flammable and is reused. The decontamination methods used depend on the type of internal components of transformer, being processed and on the size of the equipment.

**Description of the different process steps**

Transformers to be decontaminated are usually delivered as they were when they have been shut down i.e. filled with PCB. Transformers are weighed full, drained and weighed empty. They are subsequently stored in the storage area.

- **Draining**
  
  After unloading, transformers are moved to the draining stand which consists of grids and metallic platform. Liquids are drained by means of vacuum pump to storage tanks and then sent to the incineration plant. Draining will recover up to 98% of PCB contents. Transformers can then be moved to a temporary storage prior to disassembly.

- **Material separation**

  After draining, transformers are dismantled in different types of materials:
  - Cores are taken out of the transformer casings.
  - The ceramics, bolts and piping material are removed and stored in baskets of different sizes to await decontamination.
  - The remaining draining oil collected in the retention platform is drained by means of vacuum pump to storage tanks.
  - The different components, cores, casing and miscellaneous material stored in baskets are then introduced into the autoclave decontamination unit.

- **Decontamination**

  The washing up process cycles are monitored under control of a PLC which can be recalled by the operators and implemented according to the type and quantity of material to be treated. There are two decontamination cycles:
  - Long cycles for core decontamination.
  - Short cycles for casings decontamination and miscellaneous material (piping, bolts, ceramics…..)

- **Cycle description**

  Both the long and short cycles include:
  - Loading the components for decontamination into the autoclave
  - Drawing a vacuum and charging the solvent
  - Heating to eliminate water
  - Decontamination and drainage of PCB – loaded solvent
  - Drying and cooling the charge and final drainage
  - Breaking the vacuum and unloading

  PCB transfer is handled under vacuum with no pump-down.

**Solvent regeneration and recycling**

Solvent is regenerated by distillation. As PCB and solvent boiling points are far apart, simple distillation suffices. Distillation takes place under vacuum. Vapours condense on a closed-circuit water-cooled heat exchanger. Incondensable gases are strained through activated carbon filters prior to being evacuated. Distillates (PCB + distillated + residual solvent) are drained off and sent to storage for incineration.
PART I: Criteria on the Adaptation of the Technology to the Country

A. Performance:

1. Minimum pre-treatment:
   - Draining of contaminated oil from the materials.
   - Dismantling for transformers.
   - Sawing for capacitors.
   - Core, casing, ceramics and piping materials separation.

   Each of the three categories of items will be treated separately.

2. Destruction efficiency (DE):
   DE values of greater than 99,999% have been reported for all chlorinated compounds. DRE's greater than 99,999 % of recent treatment are given in the Annex in Table 2.

3. Toxic by-products:
   Vacuum pump air exhaust is treated by active carbon filters. Thus, no toxic by-products are noted.

4. Uncontrolled releases:
   None.

5. Capacity to treat all POPs:
   Compounds treated: only PCB's no other POPs

6. Throughput:
   Autoclaves may either be fixed or mobile. If sufficient quantities exist in the country then a mobile autoclave can be considered. Obviously the unit can be moved about according to location of PCB stock. It is also possible to build a fixed autoclave plant in the origin country.

6.1 Quantity [tons/day, L/day]
   The treatment capacity of an autoclave can reach 50 t / day on cores.
   The overall capacity of the plant is 12 000 t / year (including PCB and metallic material).

6.2 POPs throughput : [POPs waste/total waste in %]
   Does not apply here (see under Part II, F)

7. Wastes/Residuals:
   This issue is dealt with in detail under Part II, F. Output/generation waste

7.1 Secondary waste stream volumes:

7.2 Off gas treatment:
   The off gas is treated via an activated carbon filter. The flow of gas is 125 Nm$^3$ / h, conform the EU emission standards.

7.3 Complete elimination:
   Detailed information and treatment examples:
   9 full scale treatment examples can be found in France.

In the separate Annex the following information is given:
Table 1: Technology overview-Summary Technical Details
Table 2: Some recent examples of decontaminations
Table 3: Utilities required
Table 4: Client References for Autoclaving
### PART II: Criteria on the Adaptation of the Country to the Technology

**Note:** This part has to be filled in every time the “suitability” of the technology has to be examined for a certain country situation!!

| **A. Resource needs:** | Data on the example of France are given for the 9 autoclaves together.

Large amounts of waste are needed to justify location in origin country (in excess of 2000 t). If sufficient quantities exist in the country then a mobile autoclave can be considered. Obviously the unit can be moved about according to location of PCB stock. It is also possible to build a fixed autoclave plant in the origin country. For developing countries option is interesting as only the oil is sent overseas for destruction thus reducing the amount of material, weight, space and danger in the shipment. The copper, steel and aluminium are recycled in origin country. Decontamination of PCBs and PCB contaminated equipment is to be preferred over complete incineration. It is not sustainable to incinerate transformer coils, windings and tanks.

For autoclaving as a fixed plant in a country consideration must be given to utility supplies such as energy, compressed air, water, trade waste etc. The data given in the following part are collected for 9 autoclaves together. See further details in Annex in Table 3.

#### 1. Power requirements:

- **Electrical supply:** 1000 kVA
- **Total gas heating power installed:** 1260 kW

A compressor needs to be installed to deliver compressed air, a 7 bar pressure for the general plant circuit process and for utility (pneumatic tools)

Those values are for 9 autoclaves working together.

#### 2. Water requirements:

- **Water is used in a closed – loop circuit. The volume flow is about 100 m³/h, but is directly rejected after use.**

Thus, no real water consumption can be noted. However, if a well is not available, a cold water unit has to be supplied.

#### 3. Fuel volumes:

- All on natural gas, see details Annex, Table 2

#### 4. Reagents volumes:

- **Perchlorethylene is used as solvent. The amount needed is around 40 m³; This quantity is recycled and reused. The overall consumption of solvent is 200 t/y.**

#### 5. Weather tight buildings

- The samples have to be prepared before HPLC analysis.

#### 7. Sampling requirements/facilities:

- **The samples have to be prepared before HPLC analysis.**

#### 9. Laboratory requirements:

- **HPLC and normal standard laboratory equipment.**
- **HPLC method able to measure very low concentration of PCBs.**

#### Requirements in country:

- Depending on the requirements of the concerned authorities. From the side of the autoclave operator strict rules are that all incoming transformers received, have to be analysed before treatment independent of the local or national requirements.

#### 11. Number of personnel required:

- **16:** consisting of 2 shifts of 8 h.

#### 11.1 Number of Technicians required (skilled labour):

- Complex plant requires expertise to run in origin country.

#### 11.2 Number of Labourers required (unskilled labour):

- **6**
**8. Costs:**
Autoclaving is very cost effective given the correct circumstances. Installations as used in France report that at least stocks of 2000 tonnes justify establishment. For large onshore origin country stocks of contaminated equipment then mobile or fixed autoclave plant can offer excellent opportunities for cost reduction compared to sending all the material offshore. The costs are comparable with incineration but have none of the attendant cost of packaging and transportation. It has to be taken into account that the recycling of the materials will often produce a positive cost result.

Excellent decontamination standard (to NDT) and recovery of metals often contributes to reducing the overall cost of autoclaving. In some cases the recycling revenue exceeds the autoclaving costs. One has to consider that 98% of the contaminated oil is not going into the autoclaving but directly to the incinerator!

For the cost estimate an example of Autoclave, not the installations in France but a mobile unit in Argentina has been taken: Total costs ca 1 Mio US$. Treatment cost between 1 and 1.5 USD/kg

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**C. Impact:**

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<th>1. Discharges to air:</th>
<th>2. Discharges to water:</th>
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<td>125 Nm³ / h of gas treated according to the EU standards.</td>
<td>None</td>
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<th>3. Discharges to land:</th>
<th>4. Soil impact (noise etc):</th>
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<td>Ceramics: 400 t / y of non-contaminated neutral material are landfilled</td>
<td>Conform to the EU norms.</td>
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**D. Risks**
Perchloroethylene has toxicological issues. Workers wear individual tracing equipments in order to avoid too high exposure to the solvent.

**2. Risks of technology:**

Although very limited, due to operation under vacuum condition, the following safety and fire considerations have been applied:

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<th>1. Fire prevention</th>
<th>2. Fire fighting equipment</th>
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<td>As perchloroethylene and askarel are being used, i.e. chlorinated products, and in particular solutions of polychlorobiphenyl in trichlorobenzene, in case of fire these could create conditions for the formation of dioxin, products which are toxic.</td>
<td>Although the PCB products chlorobenzene and perchloroethylene are inflammable, these products the unit is nevertheless equipped with powder extinguishers located at easily accessible points particularly those closest to storage area of solvent, PCB + oil, paper, woods and plastics.</td>
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</table>

However, the materials used in the system are mainly constituted (95% min.) by inflammable liquids: steel, aluminium, copper, and the operating temperatures are always much lower than the ignition temperature of the few combustible materials such as paper, wood, rubber, mineral oil and plastic materials (5% max.)

As far as regards the risk of fire of combustible materials inside the autoclave, the system is equipped with very strict temperature controls for the working solvent (perchloroethylene) so that it is impossible under any circumstances for the solvent molecules to reach temperatures approaching the cracking point, which is in the order of magnitude of 140°C.

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<th>3. Operational risks:</th>
<th>Mechanical risks due to manipulation of heavy metallic components.</th>
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**E. Constructability:**

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<th>1. Ease of installation/construction of plant:</th>
<th>2. Ease of shipping/transit:</th>
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<td>Complex plant requiring expertise to run in origin country.</td>
<td>The mobile autoclave in Mexico, for example, requires 3 x 20 ft containers, and one 40 ft container for transport. Autoclaves of the size of those installed in France are not mobile, of course, and have to be assembled and built completely in situ.</td>
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<th>3. Ease of operation:</th>
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F. Output/generation waste: Dates are based on 12,000 t/y transformers and capacitors. Care has to be taken on the determination of below-listed generated and deposited waste, as this system is not as many of the other technologies, a destruction system of the contaminants and as such are these factors not comparable with this other technologies!

![Flowchart of Autoclaving Process]

**Figure 3: Autoclaves process, Mass Balance**

1. Generated waste (% of input waste) 2. Deposited waste at landfill (% of input waste)

400 t/12000 t = 3.3%

3. Waste quality properties (pH, TCLP)

*Note: This Technology Specification and Data Sheet (TSDS) does not certify any particular technology, but tries to summarise the state of the art of the concerned technology on the basis of data delivered by the companies or other sources, which have been made available to the author and refers the reader to original documents for further evaluation. Without the efforts below listed technology suppliers it would not have been possible to set up this TSDS. Date 01.09.2008

 Technology suppliers that have contributed to this TSDS:
TREDI - Groupe Séché, France

References:

POPs Technology Specification and Data Sheet