CHAPTER I
INTRODUCTION

1.1 Background

Development in construction which increases each year is also followed by increases in the need of society. As a result, there is a great number of newly emerging industries at home which aim to meet the need of society as well as to reduce imports to satisfy it. One of the promising industries in the field of chemical engineering is chloroform. So far, Indonesia still has not built any chloroform plants. This makes development of chloroform plants in Indonesia have a prospect as it is very profitable and able to reduce dependence on imports of chloroform as well as to diversify products with high economic values which in turn add to state revenues and provide employment.

Molecular formula of trichloromethane or chloroform is CHCl$_3$. At normal pressure and temperature, it is a clear liquid with a typical smell. Even though the need for chloroform in Indonesia continues to increase over time, Indonesia remains highly relying on imports from abroad to satisfy the need.

Chloroform is popular for its use as an anesthetic, despite its wider use as a nonpolar solvent in laboratories or by industries [Amonette et al., 2009].

1.2 The Designed Production Capacity

The term production capacity can be defined as the maximum amount of production done within a particular unit of time. A to-be-established manufacturer should have an optimal production capacity in which the quantity and the type of the product manufactured should have a maximum profit with minimal costs.

Some factors taken into account to determine the designed capacity of a chloroform manufacturer are:

a. The demand of Chloroform in Indonesia

To meet the need of society for chloroform, Indonesia importing from other countries. The need for chloroform in Indonesia from 2006 to 2010 can be
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seen at the Table 1.1, and increases chloroform imports in Indonesia can be seen at figure 1.1.

Table 1.1  Import of Chloroform in Indonesia from 2006 to 2010

<table>
<thead>
<tr>
<th>Years</th>
<th>Chloroform import (ton)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2006</td>
<td>378.733</td>
</tr>
<tr>
<td>2007</td>
<td>317.820</td>
</tr>
<tr>
<td>2008</td>
<td>599.266</td>
</tr>
<tr>
<td>2009</td>
<td>593.659</td>
</tr>
<tr>
<td>2010</td>
<td>490.019</td>
</tr>
</tbody>
</table>

(Badan Pusat Statistik, 2011)

Figure 1.1 Need of chloroform in Indonesia

The increases in the imports of chloroform are consistent with the equation of a straight line:

\[ y = 0.0247x^2 - 49.349x \]
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Using the equation, it can be estimated that chloroform imports in 2017 will equal to 949,805 tonnes/year. Based on that estimated need for chloroform above, the plant capacity is then determined to reach 19,000 tonnes/year.

b. The demand of Chloroform Overseas
The need for chloroform abroad is also increasing. This is because this chloroform also serves another function, i.e. as a refrigerant, especially as raw materials in the manufacture of polytetraflouroetilene (PTFE) and flourinated etilenepropylene (FEP).

Table 1.2 The demand of Chloroform overseas at 2006 until 2010

<table>
<thead>
<tr>
<th>Years</th>
<th>Ton</th>
</tr>
</thead>
<tbody>
<tr>
<td>2006</td>
<td>331,791</td>
</tr>
<tr>
<td>2007</td>
<td>338,847</td>
</tr>
<tr>
<td>2008</td>
<td>345,909</td>
</tr>
<tr>
<td>2009</td>
<td>352,979</td>
</tr>
<tr>
<td>2010</td>
<td>360,055</td>
</tr>
</tbody>
</table>

c. Availability of Raw Materials
Acetone and bleaching powder are used as raw materials to make chloroform. Acetone has a molecular formula CH₃COCH₃ and can be produced from propanol dehydrogenation, while bleaching powder having a molecular formula CaOCl₂.H₂O is obtained from interaction between the gas emitted by bleaching powder and calcium hydroxide. The raw materials (acetone and bleaching powder) were obtained from PT. Smartlab Indonesia based in Serpong and PT. Asahimas Chemical based in Cilegon. The price of chloroform in the market can reach IDR 1,040,000. So doing, the construction of chloroform plants has a quite good prospect.
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d. Minimum Design Capacity

A number of chloroform plants have a minimum capacity of 18,000 tonnes/year and a maximum capacity of 90,000 tonnes/year. The minimum design capacity of the chloroform plant can be seen from the data on the capacity of the plants established abroad at the Table 1.3,

<table>
<thead>
<tr>
<th>Chloroform plant</th>
<th>Capacity (ton/years)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Dow Chemical Company, Freeport, Texas</td>
<td>60,750</td>
</tr>
<tr>
<td>2. Dow Chemical Company, Plaquemine, Louisiana</td>
<td>90,000</td>
</tr>
<tr>
<td>3. Hanlin Group Inc., Moundsville, West Virginia</td>
<td>18,000</td>
</tr>
<tr>
<td>4. Vulcan Materials Company, Geismar, Louisiana</td>
<td>40,500</td>
</tr>
<tr>
<td>5. Vulcan Materials Company, Wichita, Kansas</td>
<td>72,000</td>
</tr>
</tbody>
</table>

(Amonette et al., 2009)

Using above consideration and the projection based on the calculation of the increase in chloroform imports per year, the design production capacity in 2017 which amounts to 19,000 tonnes/year is then selected with the assumptions that it will:
1. Meet the domestic need for chloroform,
2. Reduce dependence on imported chloroform.

1.3 Site Selection for the Plant

It has been decided that the plant will be based in Cilegon, Banten. With the following considerations:
1. Sources of the Raw Materials

The plant of chloroform belongs to the process of weight reduction and thus the plant should be built near the sources of raw materials. The main raw
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materials include acetone from PT. Smartlab Indonesia in Serpong and bleaching powder from PT. Asahimas Chemical in Cilegon.

2. Product Marketing

Chloroform marketing is intended especially for raw materials for the manufacture of polymer, in which currently polymer plants are growing in Indonesia, particularly in Cilegon. Also, chloroform is used as raw materials for the manufacture of fungicide and germicide produced by the fertilizer manufacturer PT. Kujang. In addition, this area is also situated close to Cigading Port so that facilitates the marketing to areas outside Java and abroad.

3. Transportation

The means of transportation in this area are also quite good. To supply the raw materials, it can be carried out using land transportation which is close to Cilegon Highway. As for product marketing outside Java and overseas, sea transportation is used through Cigading Port which has adequate facilities.

4. Water Facilities

Cilegon is one of the industrial areas in Indonesia so that it’s main utility supply, which is water for processing and cooling, does not encounter problems since it is located near Cidanau River and if it is still unable to meet the need, Cilegon industrial areas also has a water supplier plant, which is PT. Krakatau Tirta, Indonesia with a production capacity of 2,000 per liters. Currently PT. Krakatau Tirta is acting as a water supplier in Krakatau Steel Group and the Regional Drinking Water Company of Cilegon Indonesia.

5. Labor Force

The labor force of the plants is recruited from Cilegon and its surrounding areas, in which the population in this area is high, making it a potential source of labor force.

6. Society

In relation to the social conditions, society here has already been familiar with the industrial environment so that establishment of a new plant is not the matter and the society can adapt easily and quickly.
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7. Waste Disposal
   Beside main products, waste in the form of solid and liquid is generated to transported using trucks to vacant land to be sold to manufacturers which use calcium as their raw materials.

8. Energy
   Energy supply is another thing which needs considering in the selection of the manufacturer location. To meet the electricity need, the supply is taken from the State-Owned Electricity Company (PLN) and the existing generators with a capacity of 700 kW.

9. Taxes
   The taxes to be paid may be lower and easier as Cilegon is an industrial area.

10. Costs of Construction
    The costs of construction may be cheaper because this Cilegon industrial area is located near a port so that the cost of transport to the location can be less expensive.
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Figure 1.2 Location of the plant
1.4 Literature Reviews

1.4.1 Types of the Processes

In general, there are four ways to make chloroform, namely:

1. Methane chlorination

This process is based on the reaction of methane chlorination with the assistance of alumina as the catalyst. The raw materials used consists of methane at a high purity level and a fixed bed catalytic reactor. The reaction takes place at a temperature of 275 to 450°C.

Reaction:

\[ \text{CH}_4 + \text{Cl}_2 \rightarrow \text{CH}_3\text{Cl} + \text{HCl} \] ..............................................(1.1)
\[ \text{CH}_3\text{Cl} + \text{Cl}_2 \rightarrow \text{CH}_2\text{Cl}_2 + \text{HCl} \] ..............................................(1.2)
\[ \text{CH}_2\text{Cl}_2 + \text{Cl}_2 \rightarrow \text{CHCl}_3 + \text{HCl} \] ..............................................(1.3)
\[ \text{CHCl}_3 + \text{Cl}_2 \rightarrow \text{CCl}_4 + \text{HCl} \] ..............................................(1.4)

Advantages:

- This is a thermal catalytic process in which heat can act as a catalyst so that no longer needs catalyst regeneration,
- The yield produced is quite high, ranging from 90 to 95%.

Disadvantages:

- The use of fixed bed reactors requires cantilever construction which is strong to support the catalyst and is made from materials which are resistant to heat liberation, considering the chlorination reaction is a high exothermic reaction. Those make the reactor costs quite expensive,
- This process is sensitive to the presence of impurities.

(Ketta & Cunningham, 1992)

2. Photochemical Chlorination

The chlorination process with the method of photochemical chlorination is based on the reaction of methane chlorination due to the activation of mass reactions and radiation. Separation of bleaching powder molecules into Cl radicals is performed by radiating the mass reaction and the light source whose radiation reaches 3000 A-5000 A. The raw material used is methane at
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a high purity level. The yield of this process reaches 90% and this process utilizes a photochemical reactor.

Advantages:
- To reduce impurities in the resulting chloromethane.

Disadvantages:
- Photochemical reactors must be made from a glass surface which is resistant to heat liberation. In addition, energy which produces radiation with a power level by 3000 to 5000 is necessary. As a consequence, the costs of reactor manufacture and maintenance are extremely high,
- since termination of the reaction chain occurs,
- The problem with light transmission to the reaction. Dirt or carbons on the glass surface or which the glass contains will be absorbed and thereby reducing the number of the absorbed components and wasting energy,
- The capacity per reactor is low,
- Accumulation often takes place in the reactor area which may lead to explosions.

(Ketta & Cunningham, 1992)

3. Reduction of Carbon Tetrachloride

Reaction:
\[ \text{CCl}_4 + 2(\text{H}) \rightarrow \text{CHCl}_3 + \text{HCl} \]
Yield 70-80% .................. (1.5)

The reduction of carbon tetrachloride with ethyl alcohol, at the top level, will produce chloroform. The reactions occur in a reactor with a temperature of 200° C for 25. However, these reactions only produce a small amount of chloroform and ethyl chloride. The UV radiation in carbon tetrachloride and alcohol produces chloroform with high conversion. Still, the reaction is very slow.

Advantages:
- The yield is quite high, i.e. 70 to 80%.

Disadvantages:
- The reaction is very slow.

(Faith & Keyes, 1959)
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4. Reactions between acetone and bleaching powder

Reaction:
\[ 2 \text{CH}_3\text{COCH}_3(s) + 6 \text{CaOCl}_2\cdot\text{H}_2\text{O}(s) \rightarrow 2 \text{CHCl}_3(l) + \text{Ca(CH}_3\text{COO)}_2(s) + 2 \text{Ca(OH)}_2(s) + 3 \text{CaCl}_2(s) + 6 \text{H}_2\text{O}(l) \ldots \ldots \ldots (1.6) \]

The reaction between acetone and bleaching powder produces crude chloroform and the product is purified through distillation. Chloroform reacts with the ratio of acetone to bleaching powder by 0.099 lb of acetone: 0.9986 of bleaching powder, the reaction temperature is maintained at about 50°C by using a cooling device.

Advantages:
- The reaction process is quite simple with a relatively low operating temperature,
- The yield generated is quite high, which ranges from 86 to 91%.

Disadvantages:
- The process belongs to the conventional process,
- The manufacturing process requires fairly huge costs.

Caused reaction between acetone and bleaching powder is simple and easy, so suitable for use production of chloroform.

(Faith & Keyes, 1959)

1.4.2 The Use of the Product

Currently, chloroform is used for the following purposes:

- As raw materials for the manufacture of polytetraflouroetilene,
- In the pharmaceutical field, as an extracting agent of penicillin,
- As raw materials for the manufacture of fungicide and germicide,
- In the field of medicine, as an anesthetic.
1.4.3 Physical and Chemical Properties

1.4.3.1 Raw Materials

A. The Primary Raw Materials

1. Acetone

   a. Physical properties:
      
      Molecular formula: \( \text{CH}_3\text{COCH}_3 \)
      Molecular weight: 58.08 g/mol
      Phase: colorless liquid
      Density: 0.79 g/cm\(^3\)
      Melting point: -94.9°C
      Boiling point: 56.53°C
      Critical point: 235.05°C
      Viscosity: 0.32 cp (20°C)
      Solubility: easily soluble in water

   (Kirk & Othmer, 1998)

   b. Chemical properties:
      
      • When acetone is reacted with hydrogen cyanide and sodium cyanide as well as dilute sulfuric acid, the reaction process will result in acetone cyanohydrins.

      Reaction:
      \[
      \text{CH}_3\text{COCH}_3 + \text{HCN} \xrightarrow{\text{NaCN}, \text{H}_2\text{SO}_4} \text{CH}_3 - \text{C} - \text{CH}_3 \ldots \ldots (1.6)
      \]

      • Acetone, when reacted with iodine and sodium hydroxide, will produce iodoform.

      Reaction:
      \[
      \text{CH}_3\text{COCH}_3 + 4\text{NaOH} + 3\text{I}_2 \rightarrow \text{CH}_3\text{COONa} + \text{CHI}_3 + 3\text{NaI} + 3\text{H}_2\text{O} \ldots (1.7)
      \]
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- Acetone can be reduced to 2-propanol through the reaction which is aided by lithium aluminum hydride.
  
  \[ \text{Reaction: } \text{CH}_3\text{COCH}_3 + 2 \text{(H)} \xrightarrow{\text{LiAlH}_4} \text{CH}_3 - \text{CH} - \text{CH}_3 \ldots \ldots \ldots \ldots \ldots (1.8) \]

  (Kirk & Othmer, 1998)

2. Bleaching Powder

a. Physical properties:

- Molecular formula: CaOCl$_2$H$_2$O
- Molecular weight: 145.01g/gmol
- Phase: white powder
- Density: 2.35 g/cm$^3$
- Melting point: 100 °C (decomposed)

Solubility in 100 mL of water: 21 g (20°C)

Can react with water and alcohol.

(Kirk & Othmer, 1998)

b. Chemical properties:

- is resulted from the interaction between bleaching powder gas and calcium hydroxide at a temperature of 40°C.
  
  \[ \text{Reaction: } \text{Ca(OH)}_2 + \text{Cl}_2 \rightarrow \text{CaOCl}_2 + \text{H}_2\text{O} \ldots \ldots \ldots \ldots \ldots (1.9) \]

- CaOCl$_2$ in bleaching powder is reacted with hydrochloric acid so as to produce calcium chloride and bleaching powder.
  
  \[ \text{Reaction: } \text{CaOCl}_2 + 2 \text{HCl} \rightarrow \text{CaCl}_2 + \text{Cl}_2 + \text{H}_2\text{O} \ldots \ldots \ldots \ldots \ldots (1.10) \]

- CaOCl$_2$ in bleaching powder is reacted with CO$_2$ and water to produce calcium carbonate, hypochlorous acid and calcium chloride.
  
  \[ \text{Reaction: } 2 \text{CaOCl}_2 + \text{CO}_2 + \text{H}_2\text{O} \rightarrow \text{CaCO}_3 + \text{CaCl}_2 + 2 \text{HOCl} \ldots (1.11) \]
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1.4.3.2 Product
A. Main Products
1. Chloroform
   a. Physical properties:
      Molecular formula: CHCl$_3$
      Molecular weight: 119.39 g/gmol
      Phase: clear liquid
      Boiling point: 61.2°C
      Melting point: -63.5°C
      Density: 1.48 gr/cm$^3$
      Critical temperature: 264°C
      Specific gravity: 1.489
      Viscosity: 0.57 cp (20°C)
      Heat capacity: 0.234 kal/g.°C, at 20°C
      Critical pressure: 53.8 atm
      Solubility in 100 mL water: 0.8 g (20°C)
      (Ketta & Cunningham, 1992)
   b. Chemical properties:
      • Chloroform, if reacts with air or light, it will have chloroform oxidized slowly into a toxic compound, i.e. phosgene (carbonyl chloride).
        Reaction:
        \[ \text{CHCl}_3 + \frac{1}{2} \text{O}_2 \xrightarrow{\text{air on light}} \text{COCl}_2 + \text{HCl} \]
        \[ \text{(1.16)} \]
      • Chloroform can be reduced using zinc and hydrochloric acid to form methylene chloride. If the reduction is carried out using zinc powder and water, it will generate methane.
        Reaction:
        \[ \text{CHCl}_3 + 2 \text{H} \xrightarrow{\text{Zn}} \text{CH}_2\text{Cl}_2 + \text{HCl} \]
        \[ \text{(1.17)} \]
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\[
\text{CHCl}_3 + 6 \text{H} \xrightarrow{\text{Zn, H}_2\text{O}} \text{CH}_4 + 3 \text{HCl} \quad \text{(1.18)}
\]

- Chloroform can react with concentrated nitric acid and form nitrous chloroform or chloropicrin.
  
  Reaction:
  \[
  \text{CHCl}_3 + \text{HNO}_3 \rightarrow \text{CCl}_3\text{NO}_2 + \text{H}_2\text{O} \quad \text{(1.19)}
  \]
  This chloropicrin is usually used as insecticide.

- Chloroform may undergo the process of chlorination due to bleaching powder if exposed to sunlight and produce carbon tetrachloride.
  
  Reaction:
  \[
  \text{CHCl}_3 + \text{Cl}_2 \xrightarrow{\text{Sun light}} \text{CCl}_4 + \text{HCl} \quad \text{(1.20)}
  \]

  \[\text{(Kirk & Othmer, 1998)}\]

B. By-Products

1. Calcium Acetate
   
   a. Physical properties:
   
   - Molecular formula: \(\text{Ca(CH}_3\text{COO)}_2\)
   - Molecular weight: \(158.17 \text{ g/mol}\)
   - Phase: white solid
   - Density: \(1.6 \text{ g/cm}^3\)
   - Melting point: \(160^\circ\text{C}\)
   - Solubility in water 100 mL: \(34.7 \text{ g (20}^\circ\text{C)}\)

   \[\text{(Kirk & Othmer, 1998)}\]

   b. Chemical properties:

   - If a dry distillation process is carried out on calcium acetate, it will generate calcium carbonate and acetone.

   Reaction:
   \[
   \text{Ca(CH}_3\text{COO)}_2 \xrightarrow{\text{Dry Distillation}} \text{CaCO}_3 + \text{CH}_3\text{COCH}_3 \quad \text{(1.21)}
   \]

   \[\text{(Kirk & Othmer, 1998)}\]
2. Calcium Hydroxide

a. Physical properties:

- Molecular properties: Ca(OH)$_2$
- Molecular weight: 74.1 g/gmol
- Phase: white powder
- Melting point: 580°C
- Density: 2.211 g/cm$^3$
- Specific gravity: 2.2
- Solubility in water 100 mL: 0.17 g (20°C)

(Perry & Green, 1999)

b. Chemical properties:

- If carbon dioxide gas is mixed with calcium hydroxide which has been dissolved in water, calcium carbonate is formed. If there is an excess of carbon dioxide gas which is mixed, it will not produce calcium carbonate. Instead, calcium bicarbonate will be produced.
  
  Reaction:
  \[
  \text{Ca(OH)}_2 + \text{CO}_2 \rightarrow \text{CaCO}_3 + \text{H}_2\text{O} \quad \text{(dissolve in water)} \quad \text{(1.22)}
  \]
  \[
  \text{Ca(OH)}_2 + \text{CO}_2 \rightarrow \text{Ca(HCO}_3\text{)}_2 + \text{H}_2\text{O} \quad \text{(excess)} \quad \text{(1.23)}
  \]

- Calcium hydroxide can react with the acid chloride to produce calcium chloride.
  
  Reaction:
  \[
  \text{Ca(OH)}_2 + 2 \text{HCl} \rightarrow \text{CaCl}_2 + 2 \text{H}_2\text{O} \quad \text{(1.24)}
  \]

- Calcium hydroxide which reacts with sulfuric acid will form calcium sulfate.
  
  Reaction:
  \[
  \text{Ca(OH)}_2 + \text{H}_2\text{SO}_4 \rightarrow \text{CaSO}_4 + 2 \text{H}_2\text{O} \quad \text{(1.25)}
  \]
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- Calcium hydroxide which reacts with nitric acid will form calcium nitrate.
  
  Reaction:
  \[
  \text{Ca(OH)}_2 + 2 \text{HNO}_3 \rightarrow \text{Ca(NO}_3)_2 + 2 \text{H}_2\text{O} \tag{1.26}
  \]
  (Kirk & Othmer, 1998)

3. Calcium Chloride
   a. Physical properties:
      Molecular formula : CaCl\textsubscript{2}
      Molecular weight : 110.98 g/gmol
      Phase : white powder
      Density : 2.15 g/cm\textsuperscript{3}
      Melting point : 772°C
      Boiling point : 193°C
      Solubility in water 100 mL : 74 g (20°C)
      (Kirk & Othmer, 1998)

   b. Chemical properties:
      - Calcium chloride which reacts with sodium hydroxide will form calcium hydroxide and sodium chloride.
        Reaction:
        \[
        \text{CaCl}_2 + 2 \text{NaOH} \rightarrow \text{Ca(OH)}_2 + 2 \text{NaCl} \tag{1.27}
        \]
      - Calcium chloride which reacts with sodium carbonate will form calcium carbonate and sodium chloride.
        Reaction:
        \[
        \text{CaCl}_2 + \text{Na}_2\text{CO}_3 \rightarrow \text{CaCO}_3 + 2 \text{NaCl} \tag{1.28}
        \]
        (Kirk & Othmer, 1998)
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4. Water

a. Physical properties:

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molecular formula</td>
<td>H₂O</td>
</tr>
<tr>
<td>Molecular weight</td>
<td>18.02 g/gmol</td>
</tr>
<tr>
<td>Phase</td>
<td>colorless liquid</td>
</tr>
<tr>
<td>Density</td>
<td>0.9982 g/cm³</td>
</tr>
<tr>
<td>Freezing point</td>
<td>0°C</td>
</tr>
<tr>
<td>Boiling point</td>
<td>100°C</td>
</tr>
<tr>
<td>Critical pressure</td>
<td>216.531 atm</td>
</tr>
<tr>
<td>Critical temperature</td>
<td>373.98°C</td>
</tr>
</tbody>
</table>

(Perry & Green, 1999)

b. Chemical properties:

- Water is a weak electrolyte which can conduct electricity because it is ionized into H⁺ and OH⁻ ions.
  
  Reaction:  
  \[ \text{H}_2\text{O} \rightleftharpoons \text{H}^+ + \text{OH}^- \]  
  (1.29)
- Water can break salts into acid and alkaline (salt hydrolysis),
- Water is neutral (pH=7),
- Water is a good solvent,
- Water is a polar covalent compound,
- Water which reacts with metal oxide will form alkaline hydroxide,
- Water which reacts with non-metal oxide will form acid.
  
  (Pudjaatmaka & Setiono, 1984)

1.4.4 General Process Overview

The process of chloroform production is actually the process of reacting acetone with bleaching powder, the reaction is given as follows:
2 CH₃COCH₃(ℓ) + 6 CaOCl₂H₂O(s) → 2 CHCl₃(ℓ) + Ca(CH₃COO)₂(s) + 2 Ca(OH)₂(s) + 3 CaCl₂(s) + 6 H₂O(l)  
\[ \Delta H = -305,634 \text{ KJ/mol} \]

The type of reactor used was the batch reactor which is equipped with a stirrer and a heating coil. The reactant comparison is 0.045 kg of acetone: 0.453 kg of bleaching powder. The reaction took place at a temperature of 50º C and 1 atm pressure in an exothermic reaction. Besides chloroform as the main products, the batch reactor also produces calcium acetate, calcium hydroxide and calcium chloride. In the light phase of the decanter, chloroform was produced which was then brought into the distillation tower for purification so that its purity level reaches 99%.

(Faith & Keyes, 1959)