LAPPEENRANTA UNIVERSITY OF TECHNOLOGY
Faculty of Technology
LUT Metal
Laboratory of welding technology and laser processing
Master’s Thesis

*Matti Manninen*

**DESIGN AND MANUFACTURE OF A MICRODISTILLATION COLUMN**

Examiners: Professor Antti Salminen
Professor Ville Alopaeus

Supervisor: Lic. (Tech.), M.Sc. (Tech.) Heidi Piili

## ABSTRACT

Lappeenranta University of Technology
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Matti Manninen

**Design and manufacture of a microdistillation column**

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69 pages, 46 figures, 6 tables and 4 appendices

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Keywords: microdistillation, distillation column, design, manufacture, laser processing

Aim of this thesis was to design and manufacture a microdistillation column. The literature review part of this thesis covers stainless steels, material processing and basics about engineering design and distillation. The main focus, however, is on the experimental part.

Experimental part is divided into five distinct sections: First part is where the device is introduced and separated into three parts. Secondly the device is designed part by part. It consists mostly of detail problem solving, since the first drawings had already been drawn and the critical dimensions decided. Third part is the manufacture, which was not fully completed since the final assembly was left out of this thesis. Fourth part is the test welding for the device, and its analysis. Finally some ideas for further studies are presented.

The main goal of this thesis was accomplished. The device only lacks some final assembly but otherwise it is complete. One thing that became clear during the process was how difficult it is to produce small and precise steel parts with conventional manufacturing methods. Internal stresses within steel plates and thermal distortions can easily ruin small steel structures. Designing appropriate welding jigs is an important task for even simple devices.

Laser material processing is a promising tool for this kind of steel processing because of the flexibility, good cutting quality and also precise and low heat input when welding. Next step in this project is the final assembly and the actual distillation tests. The tests will be carried out at Helsinki University of Technology.

**TIIVISTELMÄ**

Lappeenrannan Teknillinen Yliopisto
Teknillinen tiedekunta
Konetekniikka

Matti Manninen

**Mikrotislaimen suunnittelu ja valmistus**

Diplomityö

2009

69 sivua, 46 kuvaa, 6 taulukkoa ja 4 liitettä

Tarkastajat: Professori Antti Salminen
Professori Ville Alopaeus

Hakusanat: microdistillation, distillation column, design, manufacture, laser processing

Työn tarkoitus oli suunnitella ja valmistaa pienikokoinen mikrotislain. Työn kirjallisessa osassa käsitellään ruostumattomia teräksiä ja niiden hitsausta, materiaalin työstöä lasereilla ja perusteita teknisestä suunnittelusta ja tislauksesta. Työn varsinainen painopiste on kuitenkin kokeellisessa osassa.

Kokeellinen osa on jaettu löyhästi viiteen osaan. Ensimmäisessä osassa esitellään tislain ja sen komponentit. Toinen osa on työn tärkein osa; siinä suunnitellaan laite osa osalta ja askel askeleelta. Suunnittelu tässä työssä koostuu lähinnä laitteen yksityiskohtien ja valmistettavuuden suunnittelusta, koska tislain keksittiin Teknillisessä korkeakoulussa ja myös ensimmäiset piirustukset luonnosteltiin siellä jo ennen työn aloitusta. Kolmas osa käsittelee laitteen valmistusta jälleen osa osalta. Laitteen lopullinen kokoonpano on jätetty työn ulkopuolelle. Neljännessä osassa suoritetaan laitteen valmistukseen liittyviä koehitsauksia ja analysoidaan hitsausten tuloksia. Lopuksi esitetään joitain ideoita jatkotutkimuksiin liittyen.

Työn tärkein päämäärä saavutettiin: tislain on kokoonpanoa vaille valmis. Työn aikana selveni esimerkiksi se, miten vaikeaa on valmistaa pieniä ja tarkkoja teräsosia perinteisillä työstömenetelmillä. Teräsaihioiden valmistuksessa syntyneet sisäiset jännitykset ja työstön aikana ilmenevät lämpövaikutukset pilaavat helposti kappaleen. Hitsauskiinnittimien järkevä suunnitteleminen on tärkeä vaihe pientenkin kappaleiden valmistuksessa, tosin joskus kappaleet voidaan toki suunnitella niin, ettei kiinnittimiä tarvita.

Lasertyöstö on lupaava työkalu esimerkiksi tässä työssä vaadittavaan materiaalin työstöön, koska se on joustava menetelmä, laserleikkausjälki on laadukas ja laserhitsaus on erittäin hyvä pienten kappaleiden liittämismenetelmä tarkasta ja vähäisestä lämmöntuonnista johtuen.

# ACKNOWLEDGMENTS

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*Omistan työni Ukille. Matti on tosiaan diplomi-insinööri ny.*

In Lappeenranta 15.10.2009

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Matti Manninen

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# LIST OF ABBREVIATIONS AND SYMBOLS

Abbreviations:

| | |
|---|---|
| AISI | American Iron and Steel Institute |
| $CO_2$ | Carbon dioxide |
| $Cr_{eq}$ | Chromium equivalence |
| FABTech | Fabrication Technology |
| FN | Ferrite Number |
| HAZ | Heat Affected Zone |
| HUT | Helsinki University of Technology |
| LUT | Lappeenranta University of Technology |
| Nd:YAG | Neodymium: Yttrium Aluminum Garnet |
| $Ni_{eq}$ | Nickel equivalence |
| nm | Nanometer |
| min | Minute |
| mm | Millimeter |
| $R_a$ | Surface roughness term |
| TEKES | Finnish Funding Agency for Technology and Innovation |
| TIG | Tungsten Inert Gas |
| WRC | Welding Research Council |
| µm | Micrometer |

Symbols:

| | |
|---|---|
| c | Specific heat |
| J | Energy per distance |
| kg | Kilogram |

| | |
|---|---|
| l | Liter |
| L | Latent heat of melting |
| m | Mass |
| m | Meter |
| P | Power |
| Q | Energy |
| $Q_{max}$ | Maximum energy |
| $\Delta T$ | Temperature difference |
| W | Watt |
| Ø | Diameter |

# LITERATURE REVIEW

## 1 INTRODUCTION

This thesis was written at LUT Laser (Laboratory of Welding Technology and Laser Processing) at Lappeenranta University of Technology and is part of the FABTech project - project for development of laser assisted manufacturing of micro/milli-scale devices for chemical processes. This three-year project started at the beginning of 2009 and is funded by the Finnish Funding Agency for Technology and Innovation (TEKES). The idea for a microdistillation column came from the Chemical Engineering research group at the Department of Biotechnology and Chemical Technology at Helsinki University of Technology (HUT).

The goal of this thesis is to design and manufacture a microdistillation column. It is called microdistillation because the distillated volumes are measured in milliliters and dimensions in millimeters or less. The distillation tests that will be carried out with the product are not discussed in or otherwise any part of this thesis.

There are two main reasons for a small distillation unit. Firstly, it could theoretically be used in production, maybe in series with many such units following the number-up principle rather than scale-up. The number-up principle means that the scale and the critical dimensions of the product do not change when the product proceeds from laboratory scale to industrial scale production, but that it is designed so that many units can be used in series to increase and control the yield. The principle often takes advantage of modular constructions. A small distillation column could be used for example for hazardous or expensive materials, or in any case for applications which do not require large volumes to be distilled. More importantly, however, it could be used to test the distillation process cycle in micro-scale. Process development could then move from laboratory scale straight to industrial scale without need for expensive pilot plants.

The literature portion of the thesis consists of separate topics pertaining to different important aspects of designing and manufacturing. Laser welding and cutting are given special importance since they are well-suited manufacturing processes for this application.

In the experimental part of the thesis the microdistillation column is designed and manufactured step by step. The design process in this thesis did not start from scratch; the preliminary drawings and dimensions had already been designed by researchers at HUT. The designing in this thesis consisted of refining those existing drawings, always bearing in mind that the device would have to be easy to manufacture also.

# 2 STAINLESS STEELS

Stainless steels are heavily alloyed steels which have superior corrosion resistance compared to the carbon and conventional low-alloy steels because they contain relatively large amounts of chromium. Other elements may also increase corrosion resistance, but their usefulness in this respect is limited. [2]

Stainless steels are an important part of this thesis, because the main part of the distillation device has to built from a heat, water and solvent resistant alloy, and stainless steels are the most obvious choice. The choice between different types of stainless steels is more difficult. In addition to corrosion resistance, weldability and manufacturability has to be considered as well.

Generally, stainless steels contain at least 10 % of chromium, with or without other elements. This is the minimum amount of chromium necessary to form a stable, passive chromium oxide film, which is the basis for the corrosion resistance of all stainless corrosion-resistant alloys. In the United States, however, steels that contain as little as 4 % of chromium can be classified as stainless. Together these steels form a family known as stainless and heat-resisting steels, some of which possess very high strength and oxidation resistance. Some contain more than 30 % of chromium or less than 50 % of iron. [2, 5]

## 2.1 Types of stainless steels

In a broad sense, stainless steels can be divided into four different groups based on their microstructure: austenitic, ferritic, martensitic, and austenitic-ferritic (duplex). In each group there is one composition that represents the basic, general purpose alloy. The other compositions are derived from this basic alloy, with specific variations in composition made to obtain specific properties. [2]

### 2.1.1 Martensitic stainless steels

Of all stainless steels, only martensitic steels can be quenched and hardened. Their chemical composition is designed to make them strong, yet still moderately corrosion resistant. Martensitic stainless steels often contain at least 12 % of chromium to enhance the corrosion resistance and enough carbon to allow the steel to attain high toughness when hardened. Too much chromium makes it impossible to reach purely martensitic structure, and too much carbon creates carbides that bind chromium and in so doing weaken the corrosion resistance. [3]

Usually martensitic stainless steels contain 0.2-0.5 % of carbon and 12-14 % of chromium and small amounts of other alloying elements, like manganese, silicon, sulfur and phosphor. In some cases this basic composition is not wear resistant or strong enough and some variations have been created. For high toughness applications, low carbon martensitic steel is preferred, $C \leq 0.15$ % and $Cr = 11.5 - 13$ %. Chromium content is lower to ensure fully martensitic structure and carbon content is lower to ensure even distribution of chromium throughout the steel. It has a somewhat weaker resistance to corrosion but its impact strength and fatigue durability are excellent. This kind of steel is used in valves, pump parts, bearings, ovens, superheater components etc. [3]

In order to further improve the corrosion resistance of basic martensitic stainless steel small amount of nickel is added, the composition of these steels is $C < 0.2$ %, $Cr = 16\text{-}18$ %, $Ni \approx 2$ %. Nickel serves two purposes: It improves corrosion resistance, and makes the austenitic region larger, which ensures that the structure can be made martensitic regardless of the high chromium content. It can not reach as high hardness as the base steel but it has the best corrosion resistance of all martensitic stainless steels. [3]

High carbon content martensitic stainless steels are used when high hardness and abrasive wear resistance is required. Their composition is usually $C = 0.6\text{-}1.2$ %, $Cr = 16\text{-}18$ %. Due to high carbon content, a lot of carbides remain in the structure after hardening. Because these carbides bind chromium, chromium content has to be raised to ensure enough chromium remains in the structure to form the passive oxide film. These kinds of steels are very hard but not as tough or as corrosion resistant as the rest of the martensitic stainless steels. [3]

In addition to martensitic stainless steels being the only kind of stainless steel that can be hardened, they are also cheap, since there are less expensive alloying elements. On the other hand, its corrosion and heat resistances are not as good, and only one type (AISI 410S) can be welded since they develop brittleness very easily. Table 1 shows the compositions and typical uses of AISI (American Iron and Steel Institute) standard martensitic grades. [3, 4, 5]

**Table 1.** Martensitic stainless steels. [6]

| Grade | C | Mn | Si | Cr | Mo | P | S | Comments/Applications |
|---|---|---|---|---|---|---|---|---|
| **410** | 0.15 | 1 | 0.5 | 11.5-13 | - | 0.04 | 0.03 | The basic composition. Used for cutlery, steam and gas turbine blades and buckets, bushings |
| **416** | 0.15 | 1.25 | 1 | 12-14 | 0.6 | 0.04 | 0.15 | Addition of sulfur for machinability, used for screws, gears etc. |
| **420** | 0.15-0.4 | 1 | 1 | 12-14 | - | 0.04 | 0.03 | Dental and surgical instruments, cutlery |
| **431** | 0.2 | 1 | 1 | 15-17 | 1.25-2 | 0.04 | 0.03 | Enhanced corrosion resistance, high strength |
| **440A** | 0.6-0.75 | 1 | 1 | 16-18 | 0.75 | 0.04 | 0.03 | Ball bearings and races, gauge blocks, molds and dies, cutlery |
| **440B** | 0.75-0.95 | 1 | 1 | 16-18 | 0.75 | 0.04 | 0.03 | As 440A, higher hardness |
| **440C** | 0.95-1.2 | 1 | 1 | 16-18 | 0.75 | 0.04 | 0.03 | As 440B, higher hardness |

### 2.1.2 Ferritic stainless steels

Ferritic stainless steels typically contain more chromium and/or less carbon than the martensitic grades. In addition to better corrosion resistance, both changes aim to stabilize ferrite, so much so that it is stable at all temperatures. On the other hand, ferritic grades are not as strong and the high chromium content makes the steel susceptible to several embrittlement phenomena at higher temperatures. In general, ferritic stainless steels are susceptible to brittle failures at the following temperatures: 400-500 ºC, 650-800 ºC and above 950 ºC. Therefore, ferritic stainless steels are not usually used at temperatures of 400-800 ºC. [4]

The chromium content in ferritic stainless steels is between 14 and 30 % while the carbon content is kept below 0.12 %. Typical applications may include appliances, automotive and architectural trim (decorative purposes), as the cheapest stainless steels are found in this family (type 409). Table 2 shows typical ferritic grades. [4]

**Table 2.** Ferritic stainless steels. [6]

| Grade | C | Mn | Si | Cr | P | S | Comments/Applications |
|---|---|---|---|---|---|---|---|
| **405** | 0.08 | 1 | 1 | 11.5-14.5 | 0.04 | 0.03 | 0.1-0.3 Al |
| **409** | 0.08 | 1 | 1 | 10.5-11.75 | 0.045 | 0.045 | (6xC) Ti min |
| **429** | 0.12 | 1 | 1 | 14-16 | 0.04 | 0.03 | |
| **430** | 0.12 | 1 | 1 | 16-18 | 0.04 | 0.03 | |
| **446** | 0.20 | 1.5 | 1 | 23-27 | 0.04 | 0.03 | 0.25 N |

### 2.1.3 Austenitic stainless steels

Austenitic stainless steels are chromium-nickel steels, that have been heat treated so that their microstructure is permanently austenitic. They are by far the most often used grade of stainless steels, especially AISI SS304 and SS316 types (and their low carbon varieties). The austenitic grades are non-magnetic in the annealed condition, but can become slightly magnetic after cold working due to some of the microstructure becoming ferritic. They can not be hardened by heat treatment, only by cold-working, and combine good corrosion and heat resistance with decent mechanical properties over a wide temperature range. The basis for austenitic stainless steels is a Fe-Cr-Ni composition even though many additional elements are also alloyed in small quantities to form the final compositions. The basic, general purpose composition is widely known as 18-8 (Cr-Ni), and is the basis for over 20 different variations (most of which are shown in table 3). These variations can be categorized as follows [2]:

- The chromium-nickel ratio is modified to change the forming characteristics

- The carbon content is decreased to prevent intergranular corrosion
- Niobium or titanium have been added to stabilize the structure
- Molybdenum is added or the chromium and nickel contents have been increased to improve corrosion or oxidation resistance.

**Table 3.** Austenitic stainless steels. [4]

| Grade | C max. | Si max. | Mn max. | Cr | Ni | Mo | Ti | Nb | Al | V |
|---|---|---|---|---|---|---|---|---|---|---|
| **301** | 0.15 | 1 | 2 | 16-18 | 6-8 | | | | | |
| **302** | 0.15 | 1 | 2 | 17-19 | 8-10 | | | | | |
| **304** | 0.08 | 1 | 2 | 17.5-20 | 8-10.5 | | | | | |
| **310** | 0.25 | 1.5 | 2 | 24-26 | 19-22 | | | | | |
| **316** | 0.08 | 1 | 2 | 16-18 | 10-14 | 2-3 | | | | |
| **321** | 0.08 | 1 | 2 | 17-19 | 9-12 | | 5xC min | | | |
| **347** | 0.08 | 1 | 2 | 17-19 | 9-13 | | | 10xC min | | |
| **E 1250** | 0.1 | 0.5 | 6 | 15 | 10 | | | | | 0.25 |
| **20/25-Nb** | 0.05 | 1 | 1 | 20 | 25 | | | 0.7 | | |
| **A 286** | 0.05 | 1 | 1 | 15 | 26 | 1.2 | ~1.9 | ~0.18 | ~0.25 | |
| **254SMO** | 0.02 | 0.8 | 1 | 18.5-20.5 | 17.5-18.5 | 6-6.5 | ~1.9 | ~0.18 | ~0.25 | |
| **Al-6XN** | 0.03 | 1 | 2 | 20-22 | 23.5-25.5 | 6-7 | | | | |

## 2.1.4 Duplex stainless steels (austenitic-ferritic)

Duplex steels, shown in
Table 4, usually contain 50 % austenitic and 50 % ferritic phase. This mixed phase structure leads to a more refined grain size of both the austenite and ferrite. Together with the presence of ferrite, this makes the material about twice as strong as common austenitic steels. They contain only half as much nickel as austenitic steels and are thus less expensive and less sensitive to the price of nickel. With the high chromium concentration they have good pitting and crevice corrosion resistance, and also a slightly better stress corrosion resistance than austenitic steels. [4]

**Table 4.** Duplex stainless steels. A219 is a superduplex alloy. [4]

| Type | Cr | Ni | C | Mn | Si | P | S | Other |
|---|---|---|---|---|---|---|---|---|
| **Type 329** | 28 | 6 | 0.1 | 2 | 1 | 0.04 | 0.03 | 1.5 Mo |
| **Type 326** | 26 | 6.5 | 0.05 | 1 | 0.6 | 0.01 | 0.01 | 0.25 Ti |
| **2RE60** | 18.5 | 4.5 | 0.02 | 1.5 | 1.6 | 0.01 | 0.01 | 2.5 Mo |
| **IC378** | 21.8 | 5.5 | 0.03 | 1.38 | 0.4 | 0.03 | 0.01 | 3 Mo 0.18 Cu 0.07 V 0.14 N |
| **IC381** | 22.1 | 5.8 | 0.02 | 1.92 | 0.48 | 0.03 | 0.01 | 3.2 Mo 0.07 Cu 0.13 V 0.14 N |
| **A219** | 25.6 | 9.4 | 0.03 | 0.7 | 0.6 | 0.01 | 0.01 | 4.1 Mo 0.27 N |

The main disadvantage of duplex steels is that they are very susceptible to many embrittlement phenomena, such as Sigma, Chi, and Alpha Prime, at higher temperatures. These phases can form rapidly, for example in 100 seconds at 900 ºC. Furthermore, even shorter exposure may decrease toughness. Because of this, the safe temperature range for duplex stainless steels is about -50 – 280 ºC. [9]

The superduplex stainless steels have a higher chromium and molybdenum concentration to enhance pitting corrosion resistance, which is balanced by a higher nickel and nitrogen concentrations in order to maintain equal amounts of ferrite and austenite. [9]

## 2.2 Welding of 304L and 316L stainless steels

AISI types 304L and 316L (L for low carbon content of 0.03 wt% maximum) stainless steels are iron based austenitic stainless steels, which have adequate Ni (>8%) to be fully austenitic with adequate Cr (>18%) for corrosion resistance. The low carbon content is to avoid the formation of chromium-rich carbides during processing and especially during welding. If the carbon content is high, chromium rich carbides may precipitate on grain boundaries in the weld heat affected zone, which reduces the free chromium content and makes the heat affected zone (HAZ) susceptible to intergranular corrosion. This is called sensitization, and in addition to chromium depletion, it also reduces fracture toughness. [14]

The depletion of chromium due to the formation, growth, and precipitation of chromium rich carbides in the grain boundaries occur mostly in the temperatures from 450 ºC to around 850 ºC and is most notable in the weld HAZ. By reducing the amount of carbon available for the reaction with chromium, the L grades have an enhanced resistance to weld HAZ sensitization. [14]

In general, types 304L and 316L can be readily welded by both arc and beam welding processes. They are, however, susceptible to the formation of two distinct weld defects: solidification cracking and lack of penetration. In case of deep and narrow laser blind welds (welds that do not penetrate through the full thickness of the joint), root porosity is also a common defect. This happens, because the material outgasses and the bubbles do not have enough time to escape from the root before the keyhole closes. [11, 14]

Nickel equivalence ($Ni_{eq}$) is the term used for the cumulative effects of austenite stabilizing elements. They are expressed as the weighed summation of the concentration levels of the austenite stabilizers, which provides a measure of tendency for austenite formation. The chromium equivalence ($Cr_{eq}$) is a similar term used for the cumulative effects of ferrite stabilizers. The ratio of these two terms – equivalence ratio $Ni_{eq}/Cr_{eq}$ – can be used as a quantitative indicator for predicting the primary mode of solidification for welded 300 series stainless steels. A small amount of ferrite (3-4 vol. %) in the finished weld is desired, weld pool solidification as primary ferrite is preferred to prevent hot cracking during welding. A calculated $Cr_{eq}/Ni_{eq}$ ratio of 1.52 to 1.9 is recommended to control the primary mode of solidification and prevent solidification cracks in type 304L while the $Ni_{eq}/Cr_{eq}$ ratio of 1.42 to 1.9 is recommended for type 316L stainless steel. Figure 1 illustrates the effect of $Ni_{eq}/Cr_{eq}$ ratio on cracking susceptibility. Welding Research Council (WRC) 1992 equivalences were conceived by Siewert and Kotecki and can be calculated from equations

$$Cr_{eq} = Cr + Mo + 0.7Nb \tag{1}$$

$$Ni_{eq} = Ni + 35C + 20N + 0.25Cu \tag{2}$$

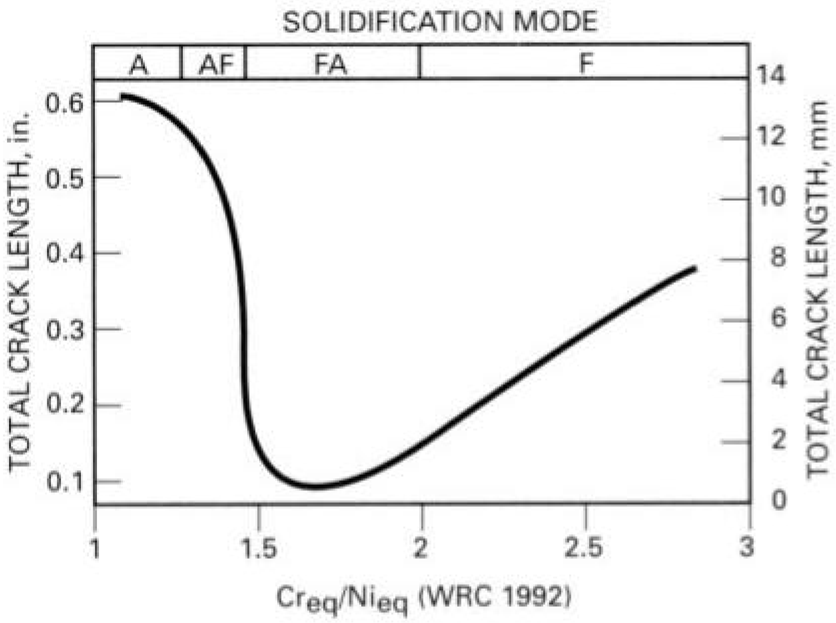



**Figure 1.** Cracking susceptibility based on WRC-1992 Cr and Ni equivalence. [14]

Generally, in absence of phosphorus and sulfur considerations, a minimum of 4 % of ferrite (Ferrite Number FN 4) and a maximum of about 21 % are required to prevent solidification cracking. At very low FN, welds solidify as primary austenite and have significant cracking tendencies, while at very high FN the weld will solidify fully as ferrite and exhibit some of the same cracking tendencies seen in materials with FN less than 4. [14]

In addition to Cr and Ni equivalences, the effect of sulfur and phosphor on cracking and weld penetration need to be considered. Small amounts of sulfur (0.005-0.030 %) have been associated with improved weld penetration; very low sulfur steels (less than ~50 ppm) exhibit poor or intermittent penetration and unstable weld pool control. Lower than 0.005 % sulfur levels may be used with lower $Cr_{eq}/Ni_{eq}$ ratios although low sulfur steels may suffer from lack of penetration. The effect of phosphorus is important to the avoidance of fusion zone cracking but has little effect on weld puddle control or penetration effects. The cracking tendencies of P and S tend to be combined. [14]

# 3 LASER MATERIAL PROCESSING

Laser light has some unique properties compared to regular light. It is almost collimated, that is, one-directional. It is not completely collimated because usually the back mirror of the resonator is slightly concave to enhance the stability of the resonator. This slight widening of the beam is called beam divergence. Laser light is also monochromatic, meaning it has one narrow range of wavelengths (for most practical purposes, one wavelength). The industrial processing lasers use wavelengths between 0.15 and 10.6 µm. The third unique property of laser light is its coherency, meaning the light waves are in the same phase. [1]

## 3.1 Laser systems

The laser systems that are most feasible for the kind of material processing required in this thesis are fiber lasers and $CO_2$ lasers. Other laser types are excluded.

### 3.1.1 Fiber laser

In a fiber laser the beam is generated inside an active optical fiber. An active fiber in this context means optical fiber that works as an active medium, as opposed to a passive fiber, which is used only for beam guidance. The fiber is the active gain medium and is doped with rare-earth elements, such as erbium, ytterbium, neodymium etc. A diode laser is used as the pumping energy source. Fiber lasers usually use a double-clad fiber wherein the gain medium forms the core of the fiber, which is surrounded by two layers of cladding. The lasing mode of the beam propagates in the core, while a multimode pump beam propagates in the inner cladding layer. The outer cladding keeps this pump light confined. Figure 2 shows the working principle of a fiber laser and figure 3 shows some fiber laser modules. The laser active core can be made very small, which makes the diameter of the beam also very small and a very good beam quality can be achieved. High power is attained by adding several fiber laser modules together and in this case beam is called multimode. Single mode is formed in one laser module. Single mode beam has excellent beam quality whereas the quality of multimode beam suffers in mixing of several single modes. The wavelength of fiber lasers depend on the doping substance, but typically used erbium doped lasers emit wavelength of 1530-1560 nm and ytterbium doped lasers 1055-1085 nm. [1, 15]

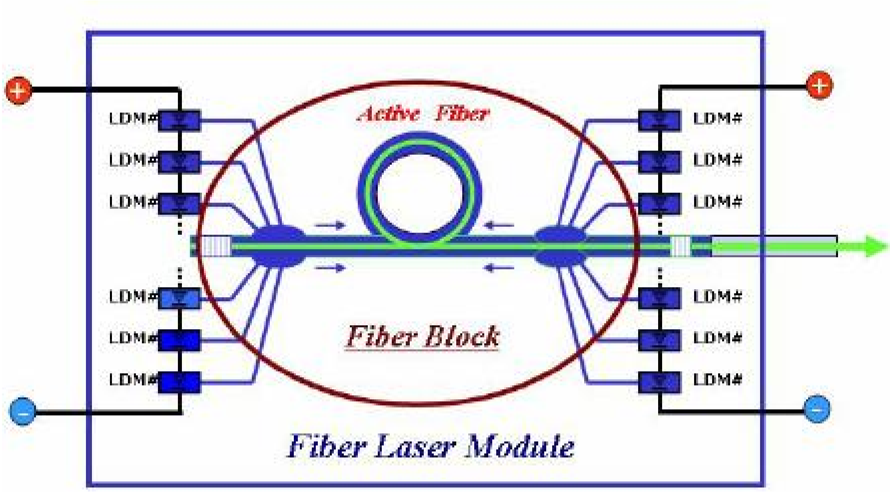



**Figure 2.** The working principle of a fiber laser. LDM = laser diode module. [15]


**Figure 3.** Fiber laser modules. [15]

### 3.1.2 $CO_2$ laser

The $CO_2$ laser is the most common heavy industrial laser. It is a gas laser in which a mixture of helium (60-85 %), nitrogen (13-35 %) and carbon dioxide (1-9 %) gases is the active medium. The medium is excited by an electrical current, and the lasing happens in the carbon dioxide atoms. Nitrogen aids in this while helium effectively cools the gas mixture. $CO_2$ lasers emit wavelength of 10.6 µm. [1]

Most of the industrial $CO_2$ lasers are fast-axial, in which the lasing gas mixture flows parallel with the optical axis of the resonator and the beam. High flow speed cools the system effectively. Output powers from this type of laser range from 700 W to 15 kW. [1]

Other possibility is the cross-flow configuration, where the gas flow is perpendicular to the optical axis of the resonator. The gas flows slowly through the resonator and into the heat exchanger. The main disadvantage compared to the fast-flow laser is its poorer beam quality, but on the other hand higher maximum powers are available. [1]

The newest version is the diffusion cooled, so-called slab laser, where the excitation and beam formation happens between two large copper electrodes. The short distance between the electrodes and water cooling allow for fast heat removal from the resonator. The advantages are a compact and durable structure and very low gas consumption. [1]

## 3.3 Laser cutting

Along with welding, laser cutting is the most popular laser material processing method. Especially in Finland cutting is by far the most common process because of the strong role of heavy metal industry. [16]

### 3.3.1 Basics

Melt shearing using inert or active gas is the most widely used cutting method. The inert gas melt shearing is based on the formation of a narrow penetrating cavity that melts the surrounding material, which is subsequently removed by the shearing action of a coaxial jet of inert assist gas. It is used with materials that can be readily melted. Nitrogen is a common choice of gas when no oxidation can be tolerated and argon or helium may be used for materials such as titanium that form deleterious brittle nitrides. Inert gas cutting is limited to steel thickness of approximately 8 mm because of the instabilities that begin to occur. A high quality cut is often achieved but cutting speeds are relatively low compared to active gas cutting. The edge of laser cut features a regular pattern of striations. [17]

If the inert assist gas is replaced by a reactive gas, such as oxygen or air, additional process energy may be generated via an exothermic chemical reaction. Cutting speeds can then be increased, but the mechanism still relies on the formation of a penetrating cavity so the beam must be focused to produce the required power density. Temperatures are higher than in inert gas cutting, which can lead to edge charring in carbon-based materials and poorer edge quality. The term ‘active gas’ is relative to the material: for example air is considered active when cutting aluminum but inert when cutting alumina. [17]

Vaporization cutting is a technique normally used with pulsed lasers, and for continuous wave cutting of some non-metallic materials. A higher power density of around $10^6$ W/mm$^2$ is used, which is 100 times more than inert gas melt shearing uses. Material is rapidly heated to vaporization temperature before much melting occurs (for those materials that melt). Some woods, paper and polymers are cut with vaporization cutting. [17]

### 3.3.2 Advantages of laser cutting

Laser cutting has a significant role in manufacturing sheet metal products. It has been used to replace conventional cutting methods to reduce manufacturing costs. In these cases only the cutting technique is changed, and the product shape, material and other aspects remain the same. While this approach takes advantage of many of the possibilities of laser cutting, there are plenty of other advantages that should be understood already when designing a new product. [1]

Laser cutting is a fast and accurate manufacturing method which can replace previously machined parts. The freedom of shape and size of the part is also typical for laser cutting: feature to be cut can be a straight line, a corner or an arc. Same tool can fabricate all these. The size of the part is only restricted by the limits of the working area or the billet. This advantage can be realized in the assembly phase, since the parts can be designed so that it is easy and risks for mistakes are minimized. Figure 4A illustrates conventional thinking where dimensions and manufacturing have been designed as easy as possible; and figure 4B shows an example of the same construction designed for easier installation utilizing the possibilities of laser cutting. [1]

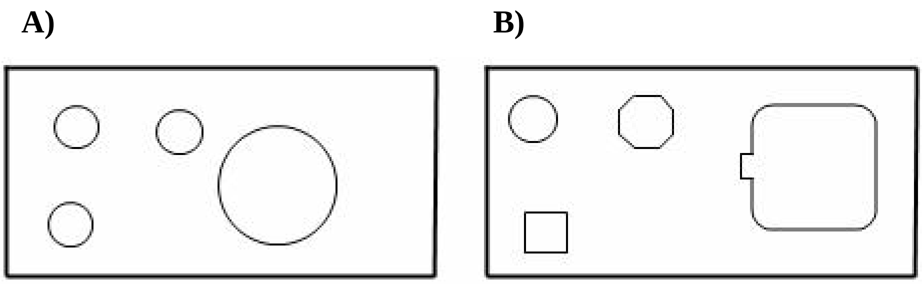



**Figure 4.** Conventional structure (A) and a structure showing the benefits of laser cutting (B). [1]

The parts can be designed to include shapes, protruding parts, slots, openings and similar shapes which help with positioning. They can help with welding assembly and jigs since the parts can be designed to fit better with each other. [1]

Laser cutting is a non-contact process meaning no tool wear, no tool storage costs or tool setup time and no deformation of the cut surface. The process is also environmentally friendly since it is quiet, permits the most efficient use of materials, and restricts harmful fumes to a well-defined interaction zone remote from the operator that can be ventilated efficiently. In comparison to conventional cutting methods, laser cutting has some distinct advantages, see table 5. [1, 17]

**Table 5.** Comparison of different cutting techniques. [17]

| | **Laser cutting** | **Plasma cutting** | **Flame cutting** | **Water jet cutting** |
|---|---|---|---|---|
| **Materials** | All homogenous | Metallic | Metallic | All |
| **Max. thickness (steel, mm.)** | 30 | 50 | 300 | 100 |
| **Kerf width, mm** | 0.15-0.2 | 1-1.5 | 3.2 | 0.8-1.2 |
| **Heat affected zone, mm** | 0.05 | 0.5 | 2 | - |
| **Relative heat input** | 1 | 10 | 100 | - |
| **Edge quality (relative)** | Square, smooth | Bevelled | Square, rough | Square, smooth |
| **Edge roughness ($R_a$, µm)** | 1-10 | - | - | 2.0-6.5 |
| **Relative productivity** | High | Medium | Low | Medium |
| **Relative capital cost** | 1 | 0.1 | 0.01 | 1 |

# 4 LASER WELDING

Laser welding can be either keyhole or conduction limited. It is most often keyhole welding, shown in figure 5, where the power density at the surface of the workpiece is high enough to produce a vapor cavity in the weld, which is then filled with molten material. There is no keyhole in conduction limited welding; the welding is similar to conventional welding where the heat is transferred by conduction and stirring inside the weld. This process produces a shallower and wider weld than keyhole welding, but its preparation tolerances are more lenient. [10]

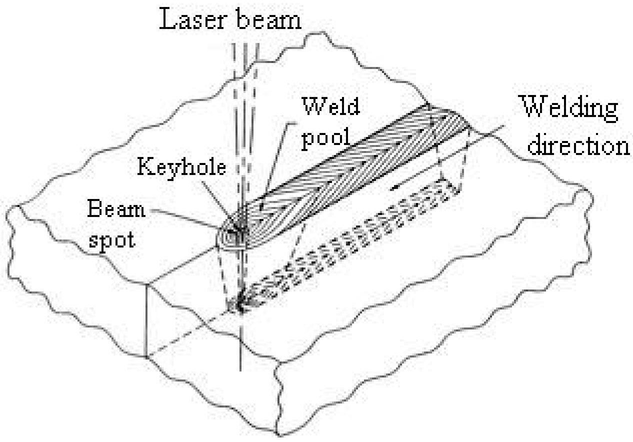



**Figure 5.** Keyhole welding. [10]

## 4.1 Advantages

Laser welding has many advantages over conventional welding processes: deep, narrow weld, low heat input, flexibility and freedom in joint configurations, repeatability/ease of automation and high rate of production. In single-pass welding the penetration depth is only limited by laser power. This sometimes eliminates the need for V-groove weld preparations or filler materials. In some cases the deep penetration makes laser welding the only viable option. One example in ship building is the seam welds between plates and stiffeners. The deep penetration also allows several layers of material to be lap welded in a single pass. [1, 10]

High welding speeds and precise heat input minimize diffusion of heat into the surrounding material. The heat affected zones are narrow and there is less chance of thermal damage to nearby features or components. Thermal distortions are typically only a fraction compared to conventional welding processes. This is one of the reasons why – often pulsed – laser welding is used in a large number of hermetic sealing applications. Metal cases can be welded shut without significant damage to the electronic devices inside. One example is the welding of air bag inflators, where the device containing highly reactive chemicals is welded. Low heat input of laser welding ensures the temperature does not rise above the ignition threshold of the chemicals. Also, precise welding of medical devices and automotive industry take advantage of the small heat distortion of laser welding. Minimal distortion also reduces the need for post-processing. [1, 10]

High production rate is a combination of several factors: welding speed can be many meters per minute, laser welding is easy to automate and robotize and beam can be directed into many work stations in turn. [1, 10]

Another advantage is that welds can be usually placed exactly where they are needed, which is particularly important if it allows the lines of stress in a device to pass through the joint smoothly. In a joint made with a partial penetration butt weld, or with a fillet weld, the lines of stress are bent in their passage through the joint producing stress concentrations that reduce fatigue strength. [1, 10]

Laser beam can often access joint positions that are inaccessible to other welding techniques, such as butt joints between gears. The accessibility also enables components to be fabricated where access is only available or practical from one side. [11]

## 4.2 Disadvantages

The tiny, focused spot size of the laser beam requires close fitting joints unless it is a lap joint or filler wire is used. Otherwise a large portion of the beam energy will be lost through the gap between the joint faces. Furthermore, because the fusion zone width of laser welds is so narrow, care has to be taken in aligning the laser beam with the joint line. [11]

Workpieces with good dimensional quality as well as precise laser beam manipulation equipment is necessary for laser welding. Not only to achieve acceptable beam and joint line alignment, but also to control the beam focus position and welding speed and hence the energy input into the workpiece. [11]

Most often welding lasers are not portable; they can not be taken on site. Lasers are sometimes mounted on robot arms and gantry systems, but they are still workshop or production line based machines. There are at least three main reasons for this: 1) Ideally, lasers need to be mounted on a stable base to maintain the optical alignment of the resonators, 2) To operate they need large electrical power supplies and cooling water systems, 3) Safety issues. Lastly, in comparison to arc and many other types of welding machines, lasers with the necessary accessory equipment are expensive. On the other hand, new generation lasers are not so rigid and inflexible anymore but instead can be portable and taken on site. [11, 20]

### 4.3 Design considerations

According to Dawes, there are eight important design aspects when laser welding is considered [11]:

1. Can the materials be readily welded, with an acceptable tolerance?
2. Will the weld adequately fulfill its service requirements?
3. Are the joint configurations and sizes practical for laser welding?
4. Can the required joint fit tolerances be achieved?
5. Is the potential number of the products, or weld length, high enough to justify equipment and processing costs? If not, is the product suitable for a laser job shop, or are there other products that could share the same equipment?
6. Will multiple workstations sharing one laser be more practical than individual lasers with single stations?
7. Will laser welding enable a simpler or more practical design to be adopted, possibly leading to material savings, elimination of unnecessary up- or downstream engineering requirements or improved product quality?
8. Has all welding and joining techniques been considered?

## 5 PROCESS OF ENGINEERING DESIGN

Engineering is the creative process of turning abstract ideas into physical representations. These may be either products or systems that meet human needs. What distinguishes design from other types of problems is nature of both the problem and solution. Design problems are open ended in nature, which means they have more than one solution unlike, for example, analysis problems such as determining the maximum height of a snowball given an initial velocity and release height. [7]

In this thesis the designing is a rough combination of all three methods described in this chapter. However, the main designing principle, which this thesis will try to follow, is the five-step method. The two other methods are explained because they describe other aspects of designing and complement the five-step process.

## 5.1 Design process according to Ohsuga

Solving design problems is often an iterative process: as the solution to a design problem evolves, the design has to be continually refined. While implementing a solution, the designer may find the solution unsafe, too expensive or just not working and has to go back a step and think of a better solution. The design process model shown in figure 6, created by Ohsuga in 1989 illustrates this process. [8]

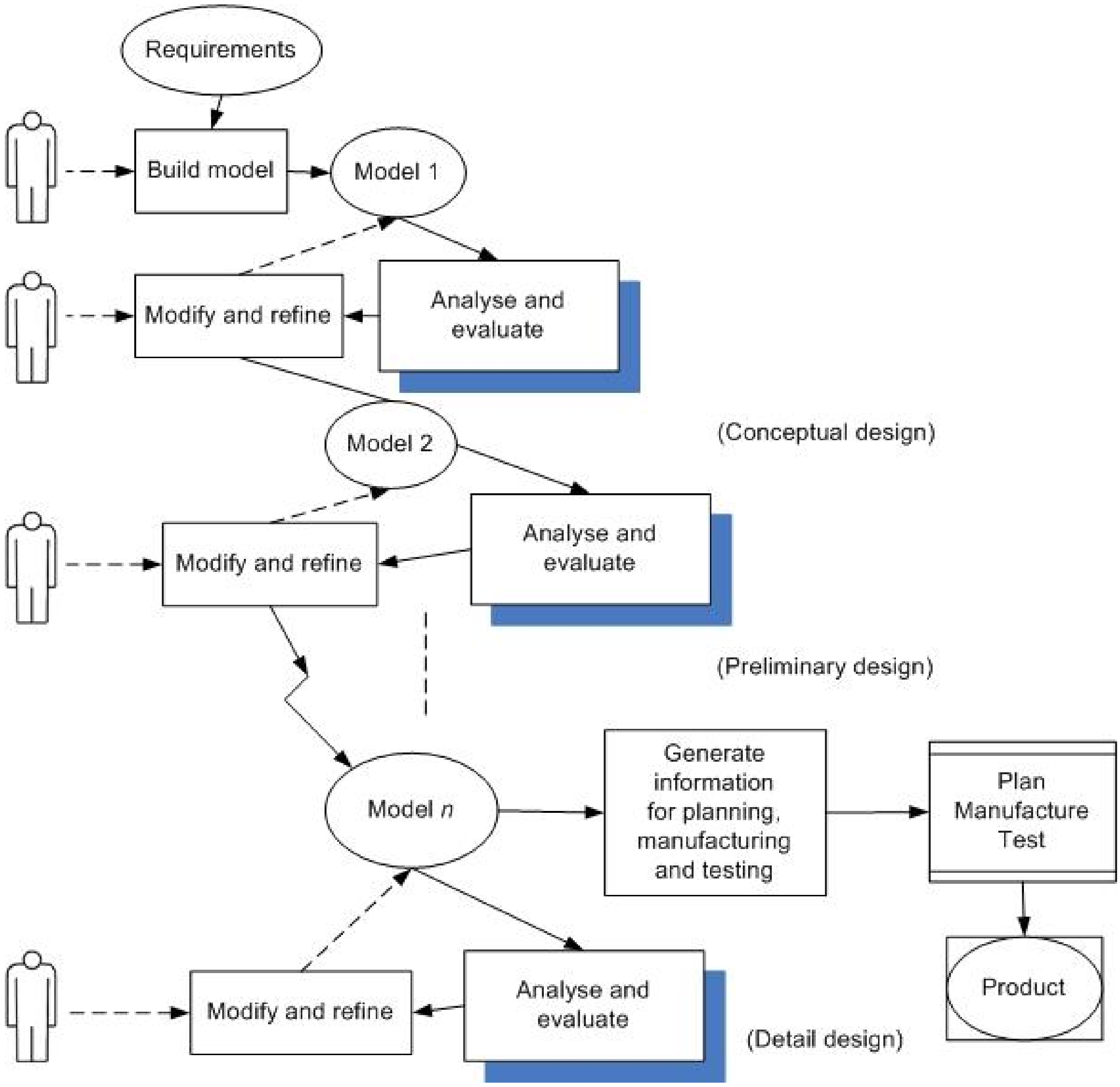



**Figure 6.** The design process according to Ohsuga. [8]

Ohsuga describes design as a series of stages, progressing through conceptual design and preliminary design to detail design. The various stages are generalized into a common form in which models of the design are developed through a process of analysis and evaluation leading to modification and refinement of the model. In the early stages, a tentative solution is proposed. This is evaluated from a number of viewpoints to establish the fitness of the proposed design in relation to the given requirements. If the proposal is unsuitable, it is modified and the process repeated until the design is at a point where it can be developed in more depth, and the preliminary design stage will start. In this stage the process of refining and modification is repeated at a greater level of detail. Finally the design proceeds to complete the definition of the design for manufacture. [8]

## 5.2 Five-step design process

The second design process model, complementing that of Ohsuga, is the basic five-step design process, which is usually used in problem solving, and works for design problems as well. The first step is problem definition. It usually contains a listing of the product or customer requirements and specially information about product functions and features. In the next step, relevant information for the design of the product and its specifications is obtained. A survey of the availability of similar products in the market should be performed at this stage. Once the details are identified, multiple alternatives to meet the goals and requirements of the design are generated. Considering cost, safety and other criteria, the more promising alternatives are selected for further analysis. In detail design and analysis step the solutions are tested and the final design is selected. Following this, a prototype is constructed and functional tests are performed to verify and possibly modify the design. [7]

### 5.2.1 Define the problem

The solution to a problem starts with a clear, unambiguous definition of the problem. A design problem often starts as a vague, abstract idea, and may evolve through a series of steps or processes as the designer develops a more complete understanding of the problem. The actual problem definition statement is a result of first identifying a need for a new product, system or a machine. Once a need has been established, that need is clearly defined in terms of an engineering design problem statement. For example the statement “Design a better mousetrap” is not an adequate problem definition because it is too vague. Further research would be needed to identify what is wrong with the current mouse trap designs. A better definition could be “Design a mousetrap that allows for a sanitary disposal of the trapped mouse”. The problem statement should specifically address the real need yet be broad enough not to preclude certain solutions. [7]

Next step in defining the problem is establishing criteria for success, that is, the specifications a design solution must meet or the attributes it must possess to be considered successful. At this point, however, the criteria are preliminary and they might need to be redefined or modified as the solution develops. [7]

### 5.2.2 Gather pertinent information

In the next step the designer should collect all available information relating to the problem. This information can reveal facts about the problem that result in a redefinition of the problem. It may help discover mistakes and false starts made by other designers. Information gathering begins by asking the following questions [7]:

- Is the problem real and its statement accurate?
- Is there really a need for a new solution?
- What are the existing solutions?
- What is wrong with and right about the way the problem is currently being solved?
- What companies manufacture the existing solution to the problem?
- What are the economic factors?
- How much will people pay for a solution to the problem?
- What other factors are important to the problem solution (safety, aesthetics, environmental issues etc.)?

Sources of information include books, publications, scientific encyclopedias, technical handbooks, electronic catalogs, indexes and the internet. Manufacturers, professional and trade organizations, suppliers etc. have valuable information on their websites. [7]

### 5.2.3 Generate multiple solutions

The next step is the creative process of generating new ideas that may solve the problem. First step is to start with existing solutions, tear them apart and find out what is wrong with those solutions and focus on how to improve their weaknesses. Then the designer consciously combines new ideas, tools and methods to produce a unique solution. There are no rigid rules to follow to experience creativity, just the designer's own willingness to consciously think and act creatively, try new things and take risks. At this stage, brainstorming is often a team effort in which people from different disciplines are involved in generating multiple solutions to the problem. [7]

### 5.2.4 Analyze and select a solution

Analysis is the evaluation of the proposed designs. The designer applies her technical knowledge to the proposed solutions and uses the results to decide which solution to carry out. Before deciding which solution to implement, each alternative solution needs to be analyzed against the selection criteria in step 1. Every design problem is unique and requires different types of analysis. The following is a list of analysis that may need to be considered [7]:

- Functional analysis
- Industrial design/Ergonomics
- Mechanical/Strength analysis
- Electrical/Electromagnetic
- Manufacturability/Testability
- Product safety and liability
- Economic and market analysis
- Regulatory and compliance

After analyzing the solutions, the best solution needs to be decided and documented. This solution is then refined developed during the later stages of the design process. At this stage the designer needs quantitative basis for judging and evaluating each design alternative. One widely used method is the decision matrix. It is a mathematical tool one can use to derive a number that specifies and justifies the best decision. [7]

The first step in creating the matrix is to rank, in order of importance, the desirable attributes or criteria for the design solution. These attributes can include factors such as safety, manufacturing considerations, the ease of fabrication and assembly, cost, portability, compliance with government regulations etc. Then, to each attribute or criteria, is assigned a value factor related to the relative importance of that attribute. For example, suppose safety is thought to be twice as important as cost. A value factor of 20 would be assigned to safety and 10 for cost (in a scale of 0-100). [7]

Next each design is evaluated against the stated criteria. A rating factor is given to each solution, based on how well that solution satisfies the given criterion. As much as possible information is needed to make an accurate evaluation. Analysis phase results, as well as computer models and prototypes yield valuable information which can provide a basis for evaluation. In most cases, the designer has to use her engineering judgment, and the decision is subjective. [7]

### 5.2.5 Test and implement the solution

Implementation refers to the testing, construction and manufacturing of the solution to the design problem. Several methods are considered, such as prototyping and concurrent engineering, as well as distinct activities that happen during implementation, such as documenting the design solution and applying for patents. [7]

## 5.3 Pahl and Beitz model

The Pahl and Beitz model is shown in figure 7. Here the design process is described by a flow diagram comprising four main phases, which can be summarized as [8]:

1. Clarification of the task, which involves collecting information about the design requirements and the constraints, and describing these in a specification
2. Conceptual design, which involves establishment of the function to be included in the design, and identification and development of suitable solutions
3. Embodiment design, in which conceptual solution is developed in more detail, problems are resolved and weak aspects eliminated
4. Detail design, in which the dimensions, tolerances, materials and form of individual components of the design are specified in detail for subsequent manufacture.

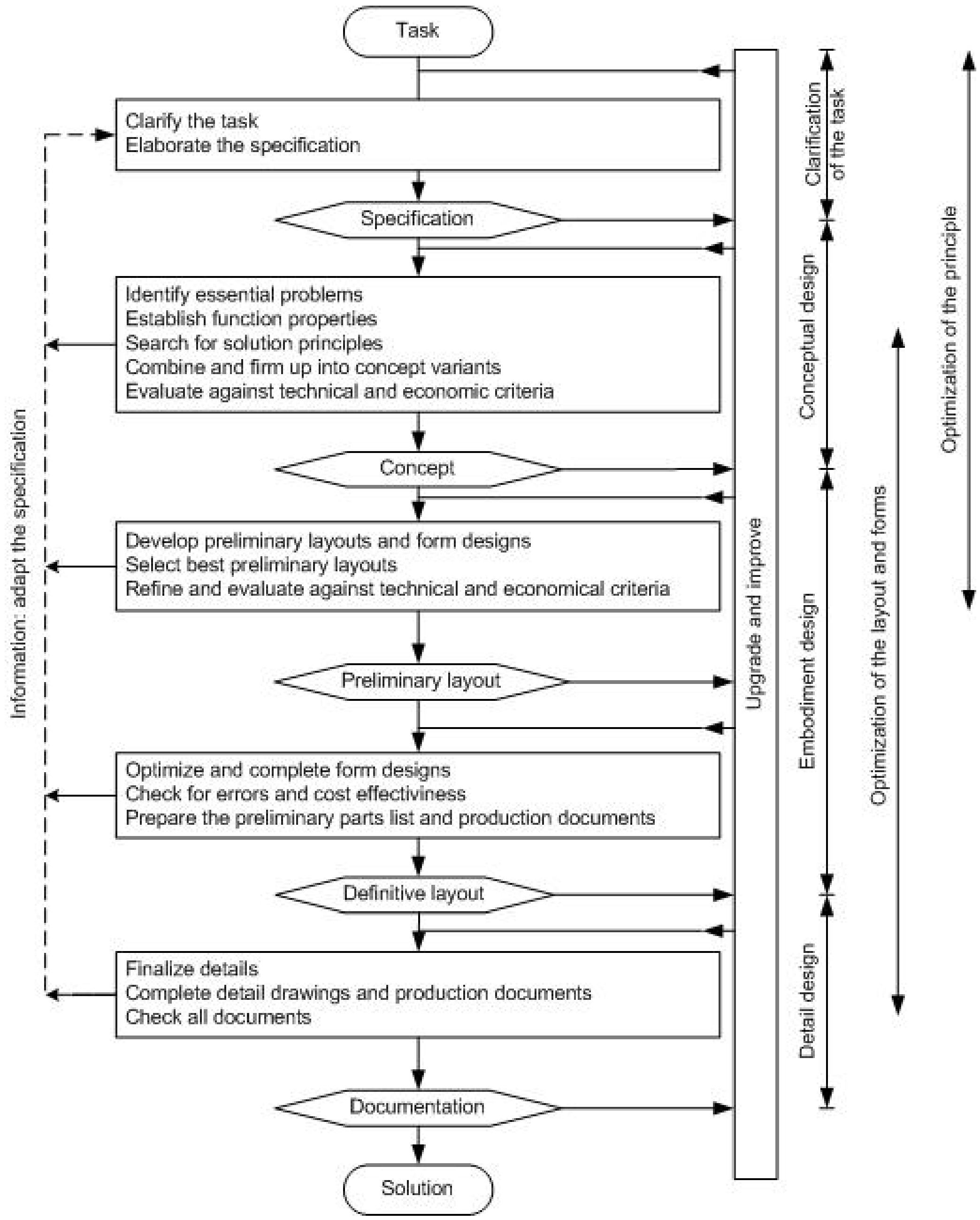



**Figure 7.** Pahl and Beitz design concept. [8]

Although figure ?? presents a straightforward sequence of stages through the process, in practice the main phases are not always so clearly defined and there is invariably feedback to previous stages and iteration between stages. [8]

# 6 DISTILLATION COLUMNS

Distillation is a physical method or process for separating a liquid mixture into its constituents. It was noted that when such a mixture is vaporized, the vapor normally has a composition different from that of the residual liquid. The term comes from a distillate product liquid, which is formed in the distillation process as the vapor condenses. The residual liquid is often called the bottoms product. [18]

During the past centuries, distillation has become the key separation method in the chemical processing and related industries, having little competition today as a simple, effective and economical way to separate on a commercial scale. Early distillations were batch type, sometimes called simple distillation or differential distillation. A batch of liquid mixture was vaporized from a still, or a still pot, by adding heat and the product vapor condensed into one or more fractions. Hence the term fractional distillation became associated with any distillation operation designed to make separations into defined or specified constituent fractions. [19]

Another important discovery was that a simple distillation, operated continuously (flash vaporization), could have its effect multiplied through the use of staging, that is, operating several flash vaporizations in series with liquid residue in countercurrent. By combining the stages into a vertical, cylindrical vessel, with addition of liquid reflux at the top and vapor from a heated still pot at the bottom, a vapor-liquid countercurrent contacting operation called rectification was established. [19]

## 6.1 Components and operation of a distillation column

Figure 8 shows a schematic diagram of a typical, vertical distillation column. The feed, which contains the components to be separated, enters the column at around the middle of the column. It can be in any state from a cold liquid to a superheated vapor. Liquid and vapor are in countercurrent contact throughout the column as the liquid flows down and the vapor rises up. [6]

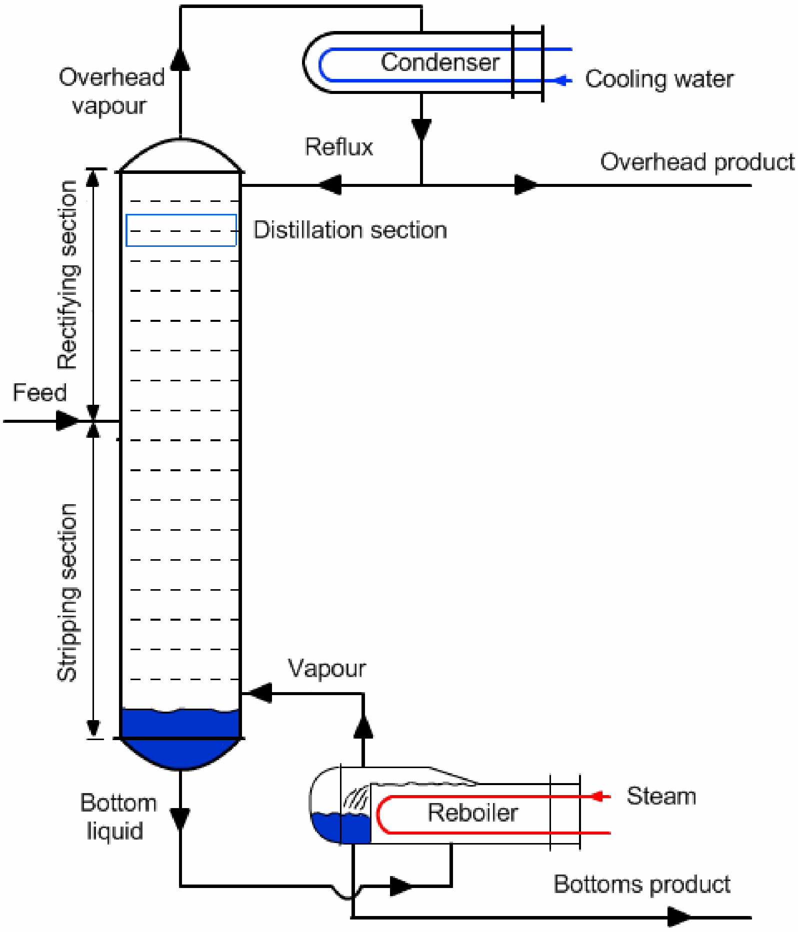



**Figure 8.** A schematic diagram of a distillation column. [6]

At each stage (represented with dash lines) some of the vapor moving up is condensed, which in turn evaporates some of the liquid moving down. If there are two components in the feed, then a greater amount of the less volatile component will condense at each stage and a greater amount of the more volatile component will evaporate. [6]

Rectifying section is the name given to the stages above the feed point, where the concentration of the more volatile component increases in both the liquid and the vapor towards top of the column. In stripping section the concentration of the more volatile component decreases in both the liquid and the vapor towards bottom of the column. [6]

The overhead vapor in the top of the column moves to the condenser, and in this heat exchanger cooling water or air or some other medium is used to condense the vapor to liquid. The liquid is split into two parts: 1) the reflux is fed back to the column where it falls down the column in countercurrent flow with the vapor from the reboiler. 2) The overhead product contains liquid with a composition specified in the design of the column. The ratio of the reflux flow rate to distillate flow rate is called the reflux ratio, which is an important parameter in the designing and operation of any distillation column. [6]

The bottom liquid contains the less volatile components in the feed and flows from the base of the column to the reboiler. In the reboiler, steam is usually used to vaporize some of the liquid; the vapor then rises up the column in countercurrent flow with the liquid falling down. The amount of heat fed to the reboiler determines the vapor flow. The bottoms product has a specified composition and is fixed during the design of the column. It is the second product stream from a distillation column. [6]

The column can also be specified differently, for example by concentration or recovery degree. Generally, every component of every stream can not be specified. The separation efficiency requirements are determined by the specifications. These requirements are the basis for the number of stages and/or the height of the column, and the degree of recovery is the basis for the ratio of reflux. Adding more stages and increasing the flow of process streams are alternative ways to improve separation efficiency. Often there is an optimum economical efficiency between these. [21]

## 6.2 Microdistillation columns

There are two problem areas in process development: scale-up and the effect of recycle. Previously, the first stage after laboratory experiments was a pilot plant, which solved the recycling problem as well. Nowadays, however, the rapid transfer of laboratory results to processes in industrial scale has become important. The idea is to go straight from laboratory to build a production plant capable of supplying the entire world market without having to build time- and money-consuming pilot plant. [12]

However, the complexity of the processes involved mean that a miniplant has to be built, that is, a plant which comprises all the essential unit operations and recycle loops of the process, but is based on the elements of a laboratory set-up. The main reason for operating a miniplant is the study of recycling problems. Simply put, during the distillation process impurities might enrich into the reflux stream and to be able to research and analyze the effect of these enrichments on the process the system has to be operational for a time which is dependent on the size of the distillation unit. The microplant in this thesis is still much smaller than conventional miniplants. [12, 13]

In contrast to typical industrial distillation columns being tens of meters high, huge devices, microdistillation columns are small systems whose dimensions are measured in millimeters and volumes in milliliters. Testing the recycling problem with such a small system could take as little time as a week and would indicate if there was a risk with the actual production plant. [13]

# EXPERIMENTAL PART

## 7 INTRODUCTION AND OBJECTIVES

The reason for physical models is that distillation is a complex process, and while the dimensions and the structure can be designed to work in theory, practical prototypes need to be produced to help simulate the distillation process in microscale. The purpose of the experimental part of this thesis is to first design and then manufacture a microdistillation column. This case was conceived by the Chemical Engineering research group at the Department of Biotechnology and Chemical Technology at Helsinki University of Technology and is part of TEKES-funded FABTech project.

## 8 STRUCTURE OF THE MICRODISTILLATION COLUMN

The part of the microdistillation column which is relevant to this thesis consists of three distinct components: the reboiler, the separation unit and the distillation chamber. The one remaining important component would be the condenser, but since it is designed and manufactured at HUT it will not be included in this thesis. This chapter introduces the different components and explains their roles and how they relate to each other.

### 8.1 Reboiler

The purpose of the reboiler is to transfer thermal energy to the liquid inside the distillation chamber. The liquid is then vaporized and leaves the boiler and enters the separation unit in countercurrent with the liquid flow. In small heaters the heat can be either transferred directly to the liquid with an electric heater or through a medium (the body of the reboiler in this case). A bare electric heater is often used in glass distillation units, but due to size of this microdistillation column it can not be used. Furthermore, heating through a medium is safer since a bare heater can overheat and explode if the fluid level descents too low.

In this case the reboiler – shown in figure 9 – is made of heat-conducting material block (for example aluminum, brass, copper). One or more cartridge type heaters are inserted inside to heat the block. The heating power is adjusted according to the temperature measured from a separate hole. The block surrounds the distillation chamber and heats the liquid through the walls of the chamber. The main requirement for the reboiler is to have a good thermal contact between the cartridge heater and the distillation chamber.

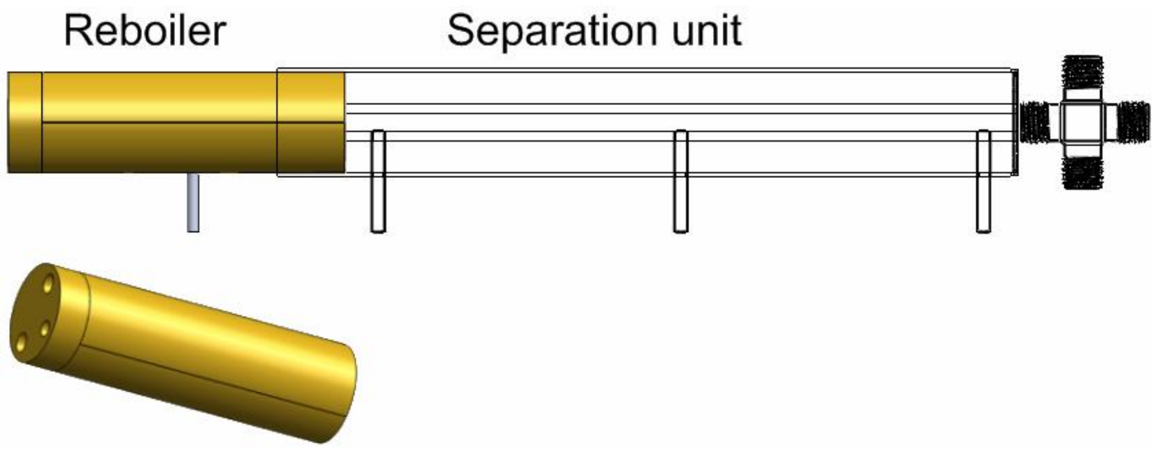



**Figure 9.** Final design of the microdistillation column, reboiler highlighted.

## 8.2 Separation unit

In conventional distillation systems the separation unit consists of layers of either vertically packed columns or plate columns. The choice between these depends largely on the scale of the device; the plate column construction needs to be spacious enough for working inside the device so it is not used when the dimensions are below few meters. In microscale conditions the only working choice at the moment is horizontally packed column.

The separation unit is shown in figure 10. Its most important role in the system is to separate the components in the liquid and vapor mixtures. Ideally it would be completely adiabatic (no heat is gained or lost by the system), which is usually not feasible, especially in laboratory conditions, because of heat losses. The distillation chamber inside is covered with insulation material, such as fiberglass, which is contained by the outer steel tube shown in figure 10. The heat losses should be sufficiently low with this kind of a structure. At the hot end (attached to the reboiler) the temperature should be equal to the boiling temperature of the heavier element and at the cold end equal to the boiling temperature of the lighter element. The outer tube will be heated with heating cable wrapped around it, on top of which comes a small layer of insulation. The three tubes are feed and extraction tubes soldered or otherwise attached to the distillation chamber.

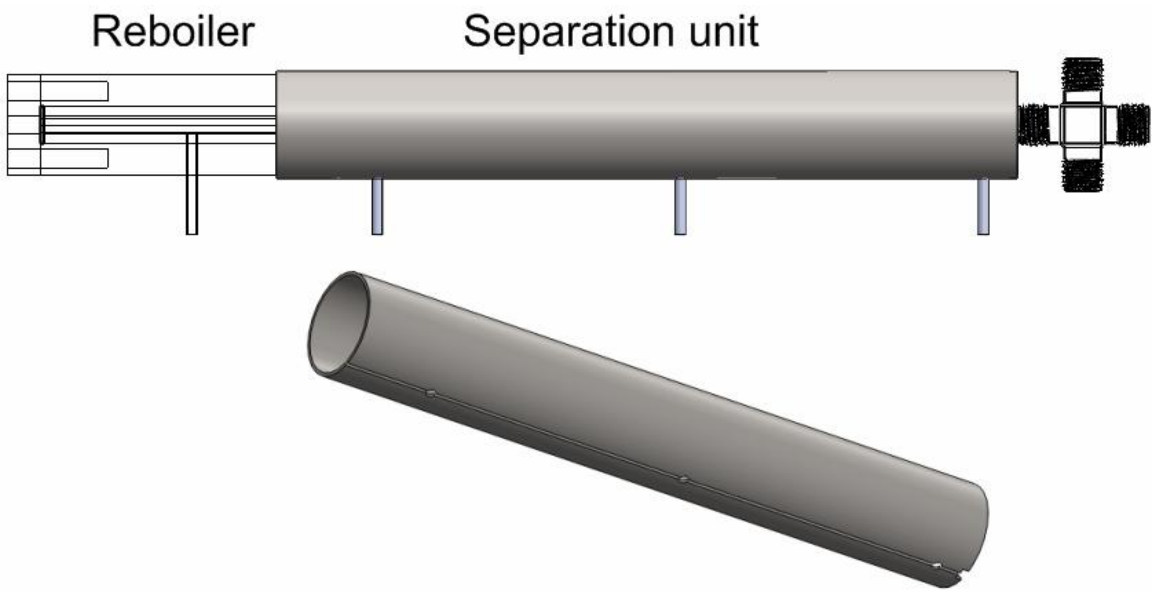



**Figure 10.** Final design of the microdistillation column, separation unit highlighted.

## 8.3 Distillation chamber

Distillation chamber is a square, stainless steel tube that goes through both the reboiler and the separation unit, as shown in figure 11. The liquid to be vaporized in the reboiler flows inside this chamber, inside metal foam which is welded or otherwise inserted inside the chamber. Vapor rises above the metal foam and flows to the cold end of the separation unit, in countercurrent with the liquid. The cross pipe fitting is welded onto the chamber and connects the condenser to the system. Later in this thesis, the hot end of the chamber refers to the end which is inside the reboiler and the cold end refers to the end which is attached to the cross fitting.

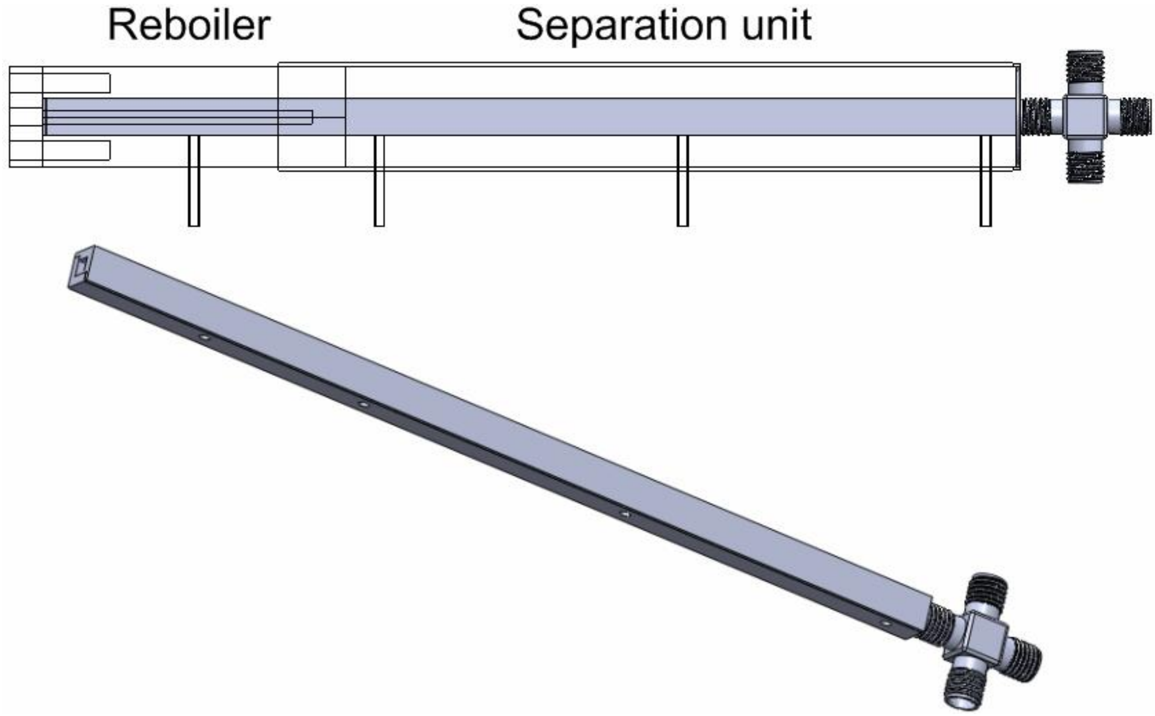



**Figure 11.** Final design of the microdistillation column, distillation chamber highlighted.

Metal foam is required for the liquid to be able move horizontally inside the foam, by capillary force alone, towards the reboiler. As such, it has certain requirements it must fulfill in this system:

- It has to be able to withstand solvents and water even in high temperatures (>300 $^{o}$C)
- It has to have good wetting properties for both organic substances and water
- The pressure loss in liquid stream must be low and the stream mixed in radial direction
- The capillary force has to be enough to prevent the liquid from flowing the wrong direction with the vapor current.

# 9 DESIGN OF THE MICRODISTILLATION COLUMN

The first design drafts were drawn, and the basic principle conceived by the Chemical Engineering research group at the Department of Biotechnology and Chemical Technology at Helsinki University of Technology. Most of the groundwork in terms of design for this device had already been conducted; the topic of this thesis was already the fifth generation of the same product. Thus, the designing in this thesis consists of refining those drawings and thinking of how the device could be easiest and most efficiently manufactured. All the 3D models in this thesis were created with Solidworks 2008 (Service Pack 4), some sketches were also drawn with Paint.NET version 3.01.

The main requirements throughout the designing process were that the device could be easily assembled and disassembled and that it would be easy and cheap to manufacture. The crucial dimensions (given in each design chapter) were not and could not be altered, which somewhat restricted the creative process. The designing still mostly followed the five-step designing method explained in chapter 5.2, and it was definitely iterative as explained by Ohsuga. Except whereas in Ohsuga's principle the models are conceptual, this particular distillation unit is already the fifth physical generation of the same idea.

In addition to these there are many established design methods and practices whose aim is to help designers and manufacturers, marketing and others involved to work together in making of the product. However, many of these are much too convoluted to work in this case due to relative simplicity of the product and the academic working conditions. The most feasible approach to this case is the five-step method to designing. That is not to say other methods are useless, two other methods are covered in the literature part, but they should and can not be followed to the letter in this case. Simple design problems can become time-consuming if complex and rigid methods are applied to them so, for example, the actual microdistillation column design process here will follow a very improvised and simplified version of the design methods covered in the literature part. Yet while it did not strictly follow any procedure, it was a flexible yet thorough process during which the author was in constant contact with the manufacturing department as well as the chemistry experts.

In the five-step design method the first step is to define the problem as unambiguously as possible. In this case the problem statement could be “Design and manufacture a small-scale distillation column capable of effective separation of solvent liquid mixtures." The next step – gathering pertinent information – was already done by the researchers at HUT so this thesis will exploit that knowledge and start designing the actual device and generating solutions to the problems found during said design process. The next step in the five-step process is the creation of a matrix and evaluation of each solution by assigning each solution values and deciding the best solution. In this thesis this process is simplified to selecting the best solution by comparing the solution to the requirements and deciding if the solution works. It is more an iterative process than a process of generating multiple solutions and then deciding the best.

## 9.1 Reboiler

The first draft of the reboiler, seen in figure 12, is already very similar to the final version even though it lacks some crucial details and dimensions. As can be seen from the drawing it is a solid block of brass with holes for both the chamber and the cartridge heater as well as a hole for the 1/8" tube. The main problems from the engineering point of view here were: how to machine the holes and how to attach the tube to the chamber so that the joint would be air-tight.

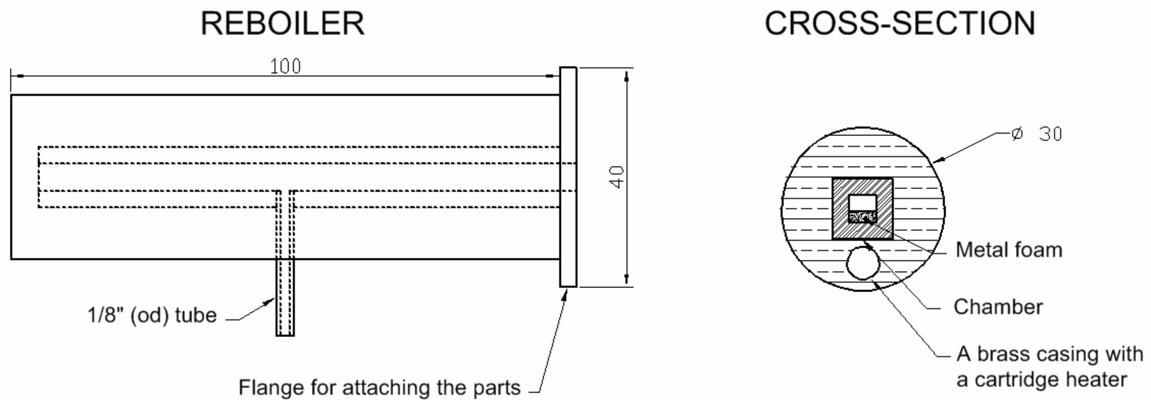



**Figure 12.** First drawing of the reboiler.

One idea was not to make this from a single block of metal, but to fill an empty 30 mm in diameter tube with metal powder, which would transfer the heat from the cartridge heater. It would be relatively easy to put together, with an added benefit of not having to worry if the heater or the chamber will fit in since the powder is just used to fill the gaps. It does not solve the second problem, though, and its heat conduction capability is questionable. Since an effective heat transfer was the most critical requirement, this construction was ultimately scrapped and it was decided that the reboiler would be manufactured as first proposed: from a single piece of brass.

### 9.1.1 Second draft

The second draft of the reboiler (see figure 13) featured two cartridge heaters placed symmetrically below the chamber such that heating would be uniform along the whole length of the chamber floor. However, one was deemed enough as the heat would surely conduct effectively enough that the uneven heat input would not be a problem. Furthermore, it would be difficult to manufacture this design since the holes are too deep to be mechanically drilled (to be more precise, the holes could be drilled but they would not be straight enough) and more laborious methods would have to be applied, such as electrical discharge machining.

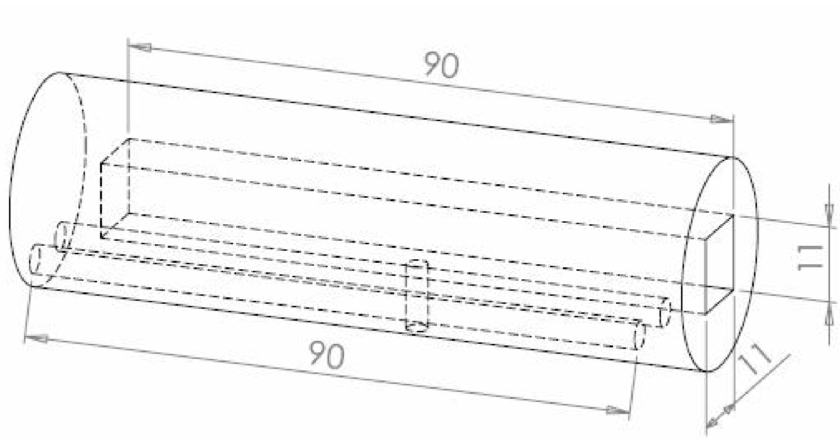



**Figure 13.** Second design of the reboiler.

### 9.1.2 Third draft

After it was decided that one cartridge heater was enough, the question whether the distillation chamber needed to go inside the reboiler at all arose. If the dimensions of the square hole were 5 x 5 mm, the chamber would not have to go inside at all, but would be attached to the reboiler with some kind of hermetic flange system, figure 14 illustrates this.

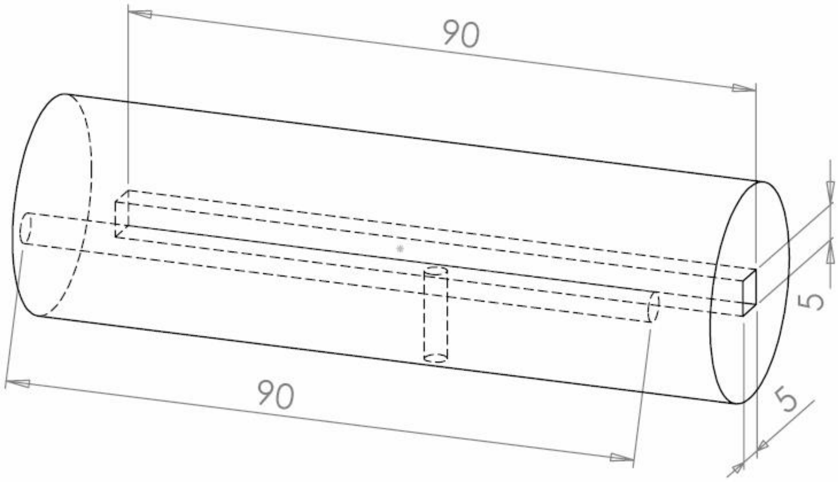



**Figure 14.** Third reboiler design.

This design would have three significant advantages: the chamber length would be reduced making it easier to manufacture, the reboiler could be made smaller (this might not be feasible due to rapid heat losses when the reboiler is too small) and it would make the reboiler easily replaceable in case different kind of heating methods were wanted. Full length chamber would restrict some possible reboiler configurations. The disadvantages are that manufacturing the device as a whole would become more cumbersome because the joint between the chamber and the reboiler would have to be figured out; and that the wall material of the chamber would change as the vapor and liquid enters and leaves the reboiler, possibly affecting the distillation process. In the end disadvantages were deemed greater than the advantages and this design was scrapped.

### 9.1.3 Fourth draft

Manufacturing the boiler was difficult because both the circular and the rectangular hole are very deep. The most feasible solution to this was to move the hole for the heater next to the chamber hole and machine it from two 100 mm rods of brass. In theory it could be made from a single rod sawed in half, but then there would be some material loss from the sawing which would slightly change the profile of the boiler.

The boiler itself consists of three parts: a lower part, an upper part and a plate at the end, which holds them all together, all seen in figure 15.

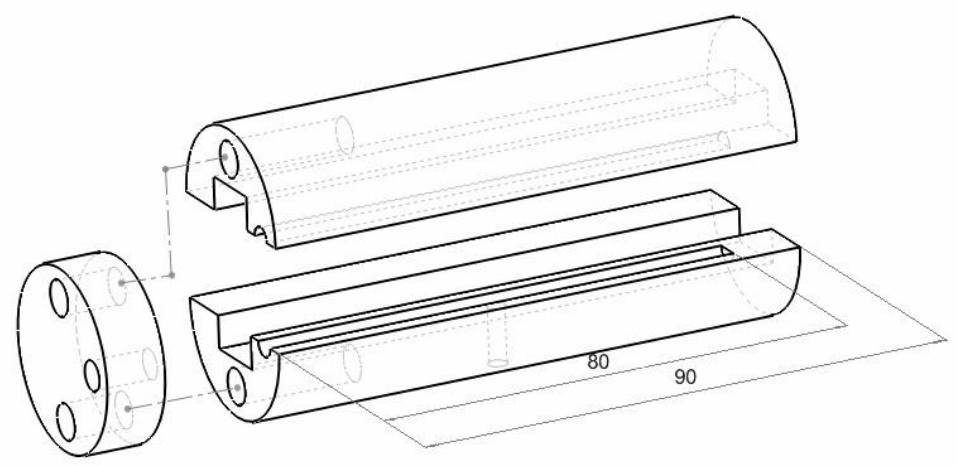



**Figure 15.** Final design of the reboiler.

Since the heater is in the middle of the joint, it should heat both halves equally and the joint should not be problematic. This design is easy to assemble, and relatively easy to manufacture.

## 9.2 Separation unit

The separation part of the distillation column is basically just a tube inside which is the distillation chamber; insulating material is used to fill the gap between them. It has three holes for the small 1/8” feed and extraction pipes. The first drawings for it are shown in figure 16. The drawings also show flanges at both ends and the distillation column inside but in this thesis those are separate parts and not considered to belong to separation unit.

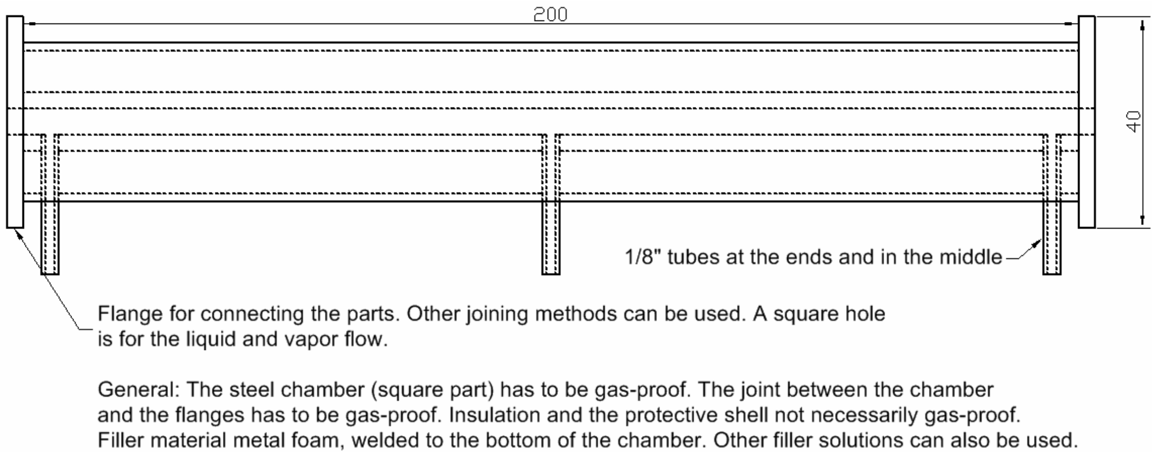



**Figure 16.** First drawings for the separation unit.

This part is very simple and virtually the only problem from the manufacturing perspective was which material it should be. It was later decided it should be common structural steel, which should be able to withstand the slightly raised temperature and some stresses without weakening. The fact that the operators should be able to assemble and disassemble the device with ease posed more serious problems. Figure 17 illustrates the problem: The 1/8” pipes cross through both the chamber and the separation unit, making the assembly challenging.

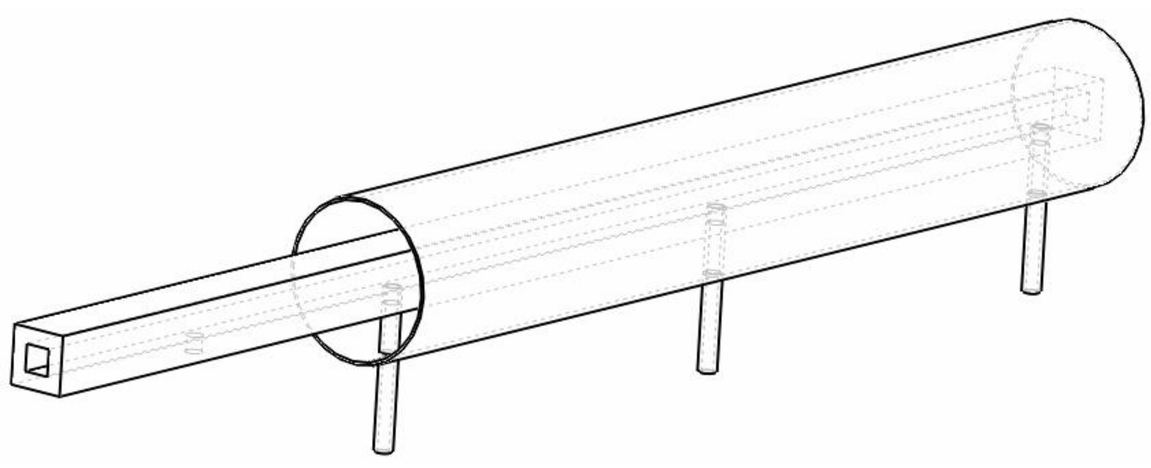


**Figure 17.** Separation unit with the distillation chamber.

The pipes have to be welded or soldered to the chamber before they are inserted inside the separation tube, which means the tube has to be designed so that it can be opened somehow and the chamber along with the pipes pulled out. A simple solution is to cut the tube open along the holes as shown in figure 18, after which the tube can be slightly spread and the chamber is released. If this solution proves too stiff, the thickness of the tube wall can be reduced or small layer of steel machined from the other side of the tube, in comparison to cutline, where most of the stress is accumulated when the tube is spread. Other possibility would be to assemble the separation unit from two halves with or without a kind of a hinge system.

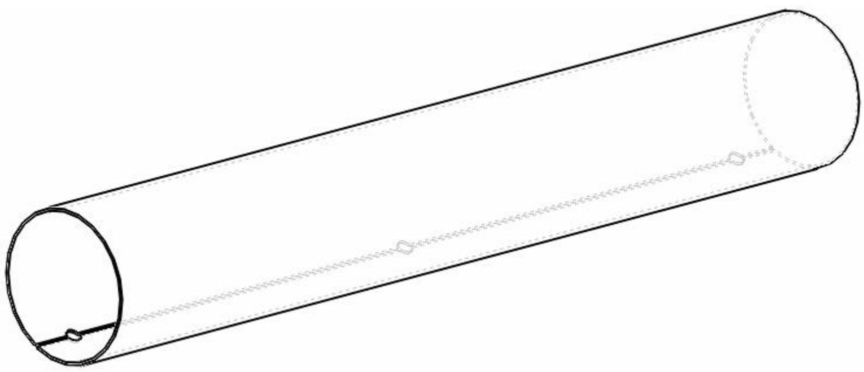


**Figure 18.** Separation unit that allows for the chamber to be removed.

Another problem regarding this part is how to support the chamber and enclose the cold end of the separation unit while keeping the device as easy to disassemble as possible. A simple solution would be a steel plate which has a hole for the chamber, and some brackets to hold it in place. Figure 19 illustrates this idea. Cutting this kind of normally a rather complex shape from steel should not be a problem for a flexible laser cutting system.

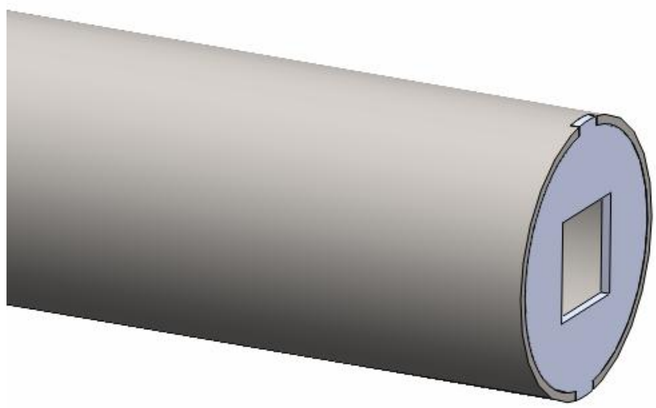


**Figure 19.** Cold end of the separation unit.

## 9.3 Distillation chamber

The first draft and some dimensions of the distillation chamber can be seen in figure 20. The main difficulty is that the cross-sectional dimensions are very small compared to the length of the chamber. It is difficult to cut long, straight parts from any steel because the internal stresses tend to bend steels. Fortunately, the straightness requirements are not very stringent, and neither do the walls need to be exactly uniform in thickness. This allows – if needed – the chamber to be machined to fit the dimensional requirements after it has been otherwise fabricated, even if it bended slightly during the manufacturing process. Since the channel height can be anything from two to ten millimeters, five millimeters was chosen for the sake of symmetry and ease of manufacture.

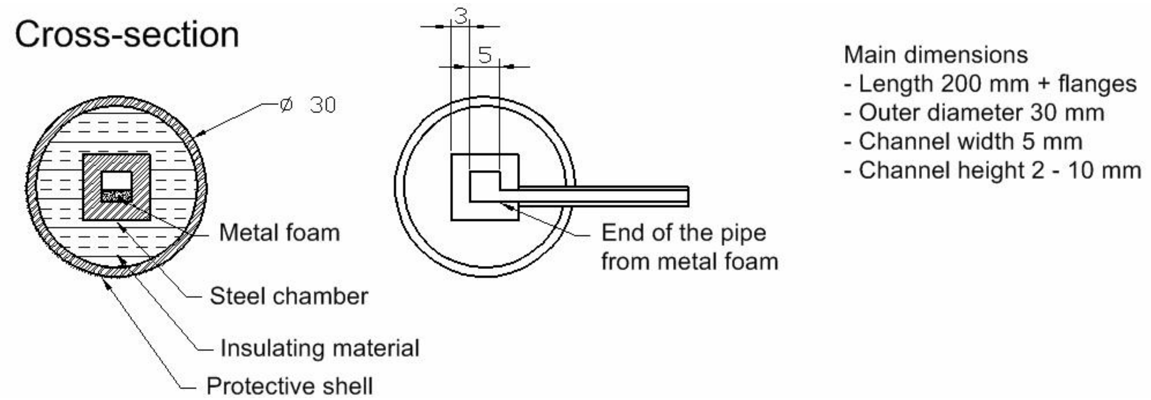



**Figure 20.** First drawings of what the chamber should be like. The outer shell and insulation represents the separation unit.

### 9.3.1 Second design

The first idea was to weld four slides of stainless steel to form the chamber (see figure 21). Possible expected problems with this kind of design involved the small dimensions of these slides as the heat input when welding and cutting as well as the internal stresses would surely bend the slides.

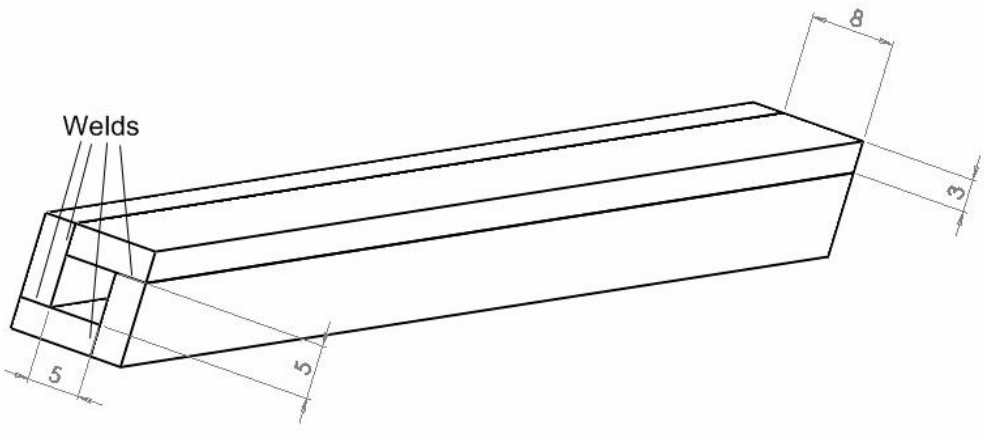



**Figure 21.** A theoretically feasible way to manufacture the chamber.

After cutting the slides with a $CO_2$ laser, it was noted that the thermal effect of the cutting was negligible, but the internal stresses within the initial billet were enough to bend the slides in two dimensions. It was not critical, though, and with a proper clamping system, these slides could well still be welded.

The welding experiments were carried out at the Manchester University with their millisecond-pulsed Nd:YAG laser. Due to its low average power, not deep enough penetration could be achieved, but it was enough to weld the pieces together and conclude that this was not the most feasible way to manufacture the chamber. Small dimensional errors in cutting and positioning of the workpiece several times accumulated to a point where the resulting accuracy of the dimensions was finally unacceptable.

### 9.3.2 Profile configurations

Despite the apparent simplicity of the distillation chamber, there are still many other ways it could be manufactured: with thinner walls it could perhaps be bought ready from a steel profile supplier, and if that is not possible different design could be used. Possible other profile designs are shown in figure 22.

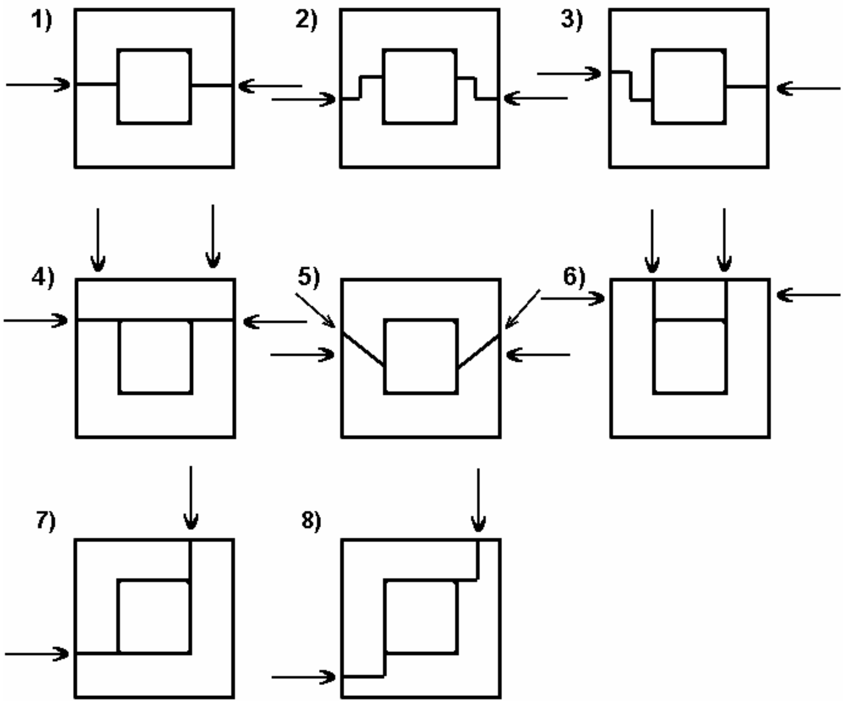



**Figure 22.** Different chamber profiles. Arrows denote possible welding directions.

Options 1, 4 and 6 would probably be problematic because of the difficulties in aligning the parts. On the other hand, the joints are placed such that positioning and aligning errors might be irrelevant (because the joint would not be in the corner) as long as the chamber can be machined to fit inside the reboiler. Option 2 shows a self-positioning design; option 3 is a simpler version of it. Option 5 would be difficult to manufacture but is an interesting possibility nonetheless. Options 7 and 8 are the most interesting due to simplicity of manufacture and ease of alignment of the parts.

### 9.3.3 Third design

The improved second version only has two parts which would be welded together. The profile configuration shown in figure 23A already eliminates many of the problems with the first draft, but positioning would still remain a problem as that kind of design would require a welding rig to be designed and assembled. This problem was solved by developing the chamber walls further so that it would be self-positioning and welding the two pieces together could be accomplished with simple clamping, see figure 23B (also option 8 in figure 22). These parts would be machined.

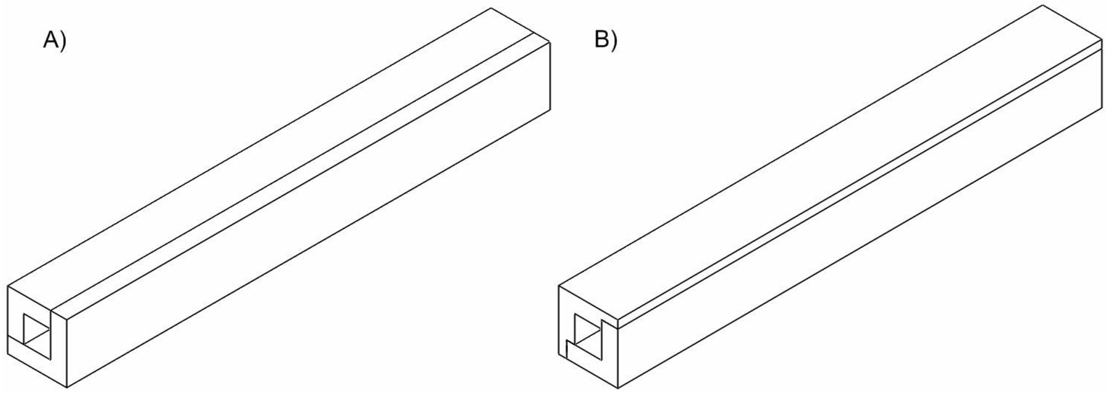



**Figure 23.** A) Second, improved design. B) Self-positioning design of the chamber.

The self-positioning version has quite low material thicknesses, which makes conventional welding risky since a lot of heat is introduced. Laser welding on the other hand is a very gentle process and thus suitable for this application. The technical drawings for the design shown in figure 23B are given in appendix IV.

At this point it was also decided that the chamber would consist of just one continuous channel through both the reboiler and the separation unit instead of two separate chambers, that is, one for both. This will simplify the design as well as ensure smoother operation of the distillation unit because the channel does not have any joints which might affect the countercurrent flow of liquid and vapor inside. The downside is that if a different kind of reboiler was required, the chamber would have to be cut at the joint between the separation unit and the reboiler. Changing the reboiler would be easier if the chambers were separate.

### 9.3.4 Metal foam

One part of the distillation chamber design is also how to attach the metal foam inside the chamber. At first it was decided that a 3 mm thick and 289 mm long (length of the chamber) slide of foam would be welded onto the bottom of the chamber.

One interesting thought, however, was to manufacture the chamber such that the metal foam would be mechanically bound in place, the principle shown in figure 24.

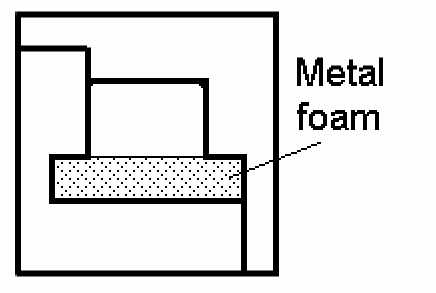



**Figure 24.** Distillation chamber cross-section, foam mechanically bound.

Problems with this kind of a design involve difficult mechanical machining steps and that critical dimensions have to be altered slightly. Also, proportionally larger portion of the fluid would not be in contact with the vapor current since the volume of the foam would increase. If the chamber itself was 5 x 5 mm, the foam would have to go at least 1 mm inside the walls, which might have a significant effect on the process. As an advantage, no welding would have to be done, the foam would stay in place perfectly, and the foam could be changed if needed, for example to test different metal foams.

Later on it was decided that whole of the chamber should be filled with foam. This was due to tests carried out by the Chemical Engineering research group at HUT, which showed that the distillation efficiency would be greatly enhanced if the chamber was filled with foam.

### 9.3.5 Hot end of the chamber

The chamber was designed to be open at both ends, yet the end facing the reboiler has to be sealed shut. It was decided that a simple plate would be welded at the end, but there is a simpler way to do it: the plate could be included already in the chamber design. The idea is shown in figure 25. The hot end could then be sealed by welding it shut. Only problem in this design is that it might be difficult to machine since the corners inside need to be relatively sharp.

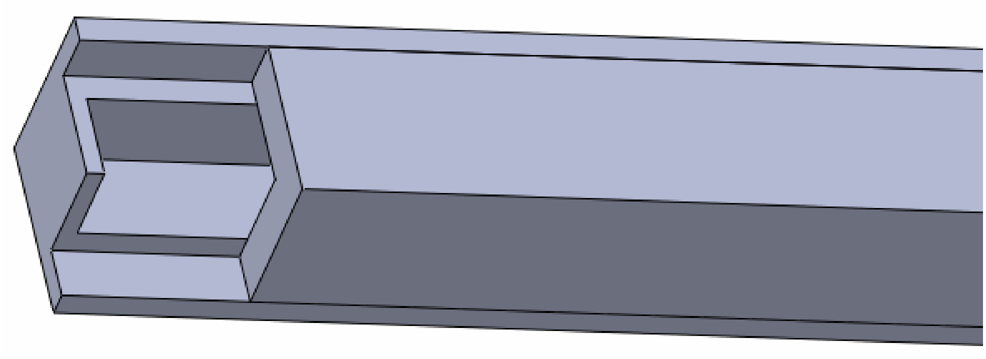


**Figure 25.** Possible hot end of the distillation chamber.

## 9.4 Joint design

There are three distinct problems regarding assembly: How to ensure that the 1/8” pipes are positioned correctly, the joint between the reboiler and the separation unit, and the connection between the separation unit and the condenser.

First problem is best solved by smart assembly. The hole in reboiler is used to mark the hole position onto the outer wall of the chamber, after which the hole is drilled and the pipe soldered or welded in place. Some clearance in the pipe hole in the reboiler ensures the pipe fits through. The pipes for the separation unit can not be positioned this way, but since the whole part is nothing more than a containment tube for the insulation, the dimensional requirements and accuracy are not stringent and the holes can be drilled large enough to compensate for the small positioning errors that might occur during manufacture and assembly.

### 9.4.1 Reboiler and the separation unit

At first, the idea was to build the distillation chamber from two parts, one inside the reboiler and one in the separation unit. The chambers would be welded onto flanges which would also hold the reboiler and the separation unit together, see figure 26. The hole inside the flanges would be filled with copper seal that would make the seal hermetic (air tight).

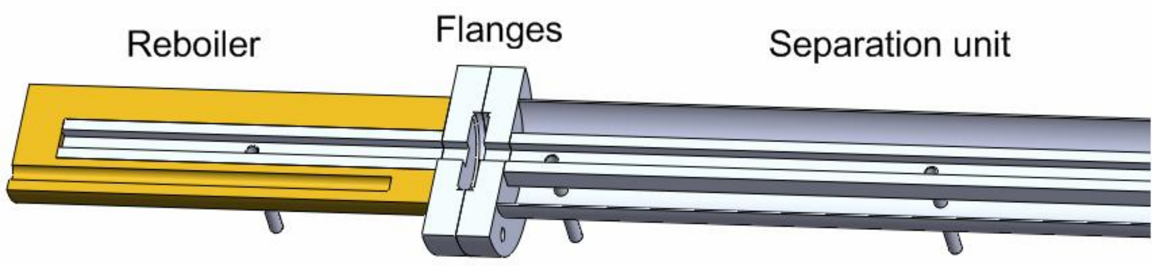



**Figure 26.** Joint between reboiler and separation unit.

It was later decided that the chamber would be one continuous channel throughout the whole system, which simplifies the assembly quite a lot. At first flanges were still thought to be a good way to join the two parts, but since there would be no need for copper seals, the flanges could have easily been cut by laser from sheet metal. This gave an opportunity to try to manufacture a different kind of flange, which would be welded onto the separation unit but would still allow it to be opened and disassembled by spreading, as described previously. This design is shown in Figure 27.

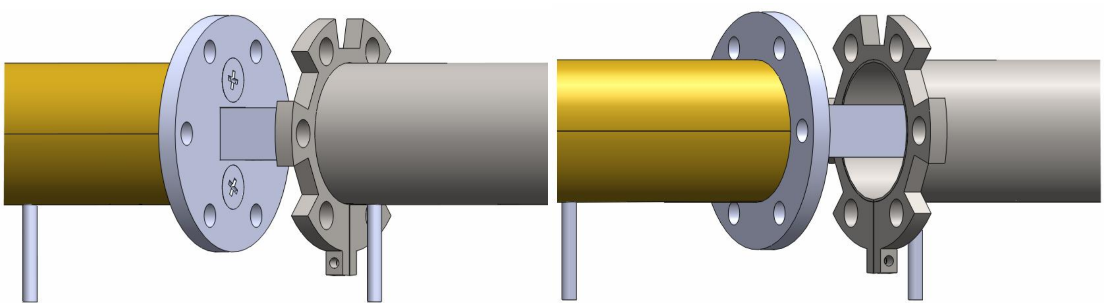


**Figure 27.** A possible way of joining the reboiler and the separation unit.

However, since the chamber is continuous, the question of whether flanges would be needed at all arose. If there was an easier way of joining the parts without a slightly complicated system of flanges it would simplify the already quite simple construction even further. The first obvious solution was to try a simple steel shaft collar; the collar could be screwed around the parts tight, which would keep them conjoined. Figure 28 shows the initial idea.

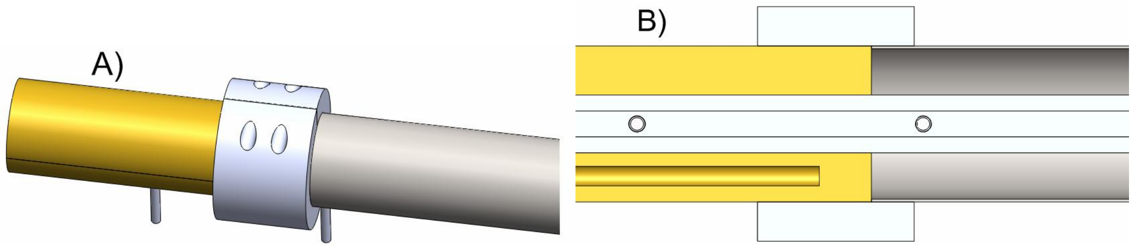



**Figure 28.** Clamping the parts together with a shaft collar. A) Outside view. B) Top view, cut horizontally.

The problem with this kind of clamping is the hot end 1/8" pipe in the separation tube which is blocking the shaft collar and most likely preventing a rigid, stable joint. To counter this, and to make the joint simplest possible, the separation unit was lengthened to partly cover the reboiler. This not only makes the joint easy to assemble, fasten and disassemble but also makes it so simple that it only requires one extra machining step when manufacturing the reboiler. Figure 29B illustrates this. The joint will probably not hold without some kind of clamping device, but that is a minor problem which is easily solved by, for example, a robust hose clamp that can withstand the heat around the joint. As an alternative to the solution shown in figure 29B, the inner dimension of the separation tube could match the outer dimension of the reboiler (shown in figure 29A), which would allow for the separation tube to be removed by spreading it a little and gliding it over the reboiler (instead of the other way, which would be problematic because the connection to the condenser would not allow it). It would also eliminate the one extra machining step required for this perhaps aesthetically more pleasant solution.

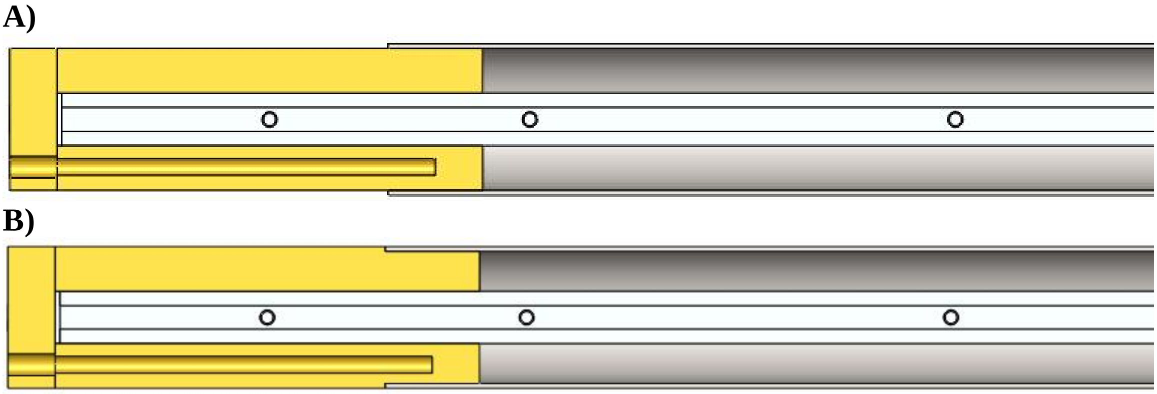



**Figure 29.** Overlapping joint between the reboiler and the separation unit. Cut horizontally, top view.

### 9.4.2 Separation unit and the condenser

The condenser will be designed and manufactured separately so this thesis will only focus on how to attach the cold end of the separation unit to the condenser. A cross or a tee pipe fitting will be welded to the cold end of the distillation chamber, which will connect it and the rest of the distillation column to the condenser, joint shown in figure 30. Various inlets and outlets of the condenser will be attached to the other inserts of the cross fitting. One idea was to machine threads inside the chamber so the pipe fitting could be screwed in and thus positioned accurately prior to welding, but this was deemed too time-consuming and unnecessary since the joint does not need to be especially accurate. For this welding, laser welding would not be the best choice due to difficulties in preparing the parts for welding, TIG (tungsten inert gas) welding with as low heat input as possible would be a more feasible choice.

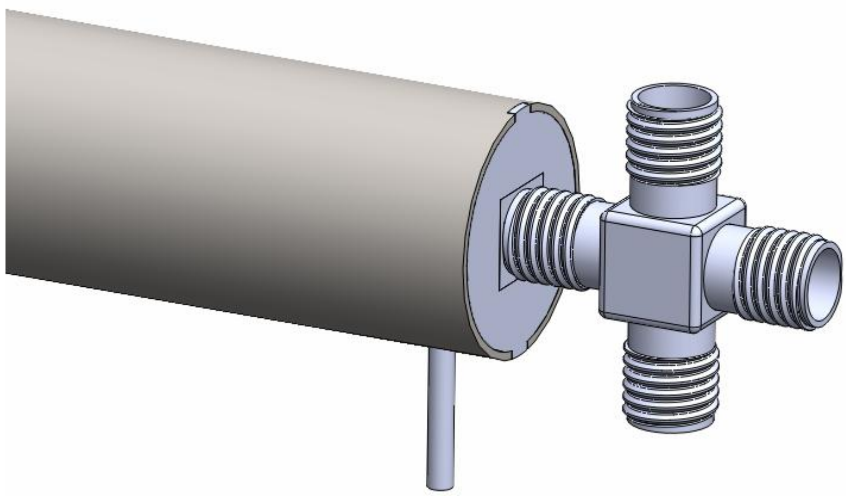


**Figure 30.** Joint between the separation unit and the condenser.

# 10 MANUFACTURING

All the parts – except the cold end plate, which was cut in the Laboratory of Welding Technology and Laser Processing – were manufactured at the Department of Mechanical Engineering at the Faculty of Technology at Lappeenranta University of

Technology. Laser welding was done at the Laboratory of Welding Technology and Laser Processing.

## 10.1 Used equipment

For manufacturing the products, two mechanical milling machines were required: a universal milling machine shown in figure 31 and a manual center lathe shown in figure 32. Welding was conducted with an IPG five kilowatt fiber laser, shown in figure 33. Laser cutting was done by a 2.5 kilowatt $CO_2$-laser, which is part of the Finnpower sheet metal machining center at the Laboratory of Welding Research and Laser Processing, seen in figure 34. The metal foam was cut with an IPG 200 W fiber laser, shown in figure 35. In addition to these smaller equipment was used for, for example, manual grinding.

**Figure 31.** Universal milling machine, MAKETEK Oy, model FA 3 C.

**Figure 32.** Manual center lathe, model CU 500M.

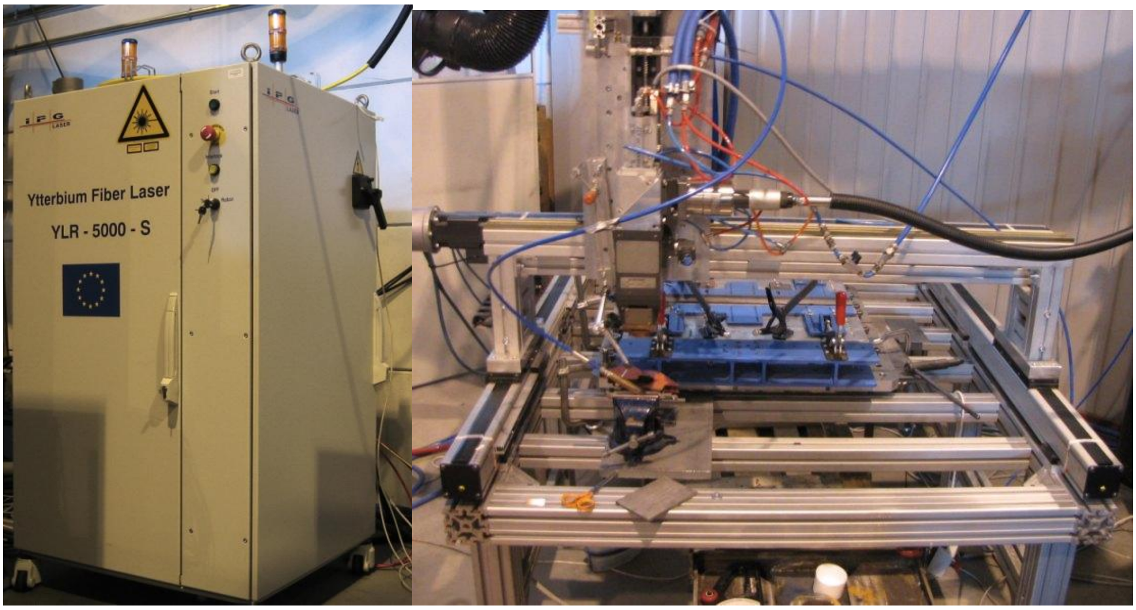



**Figure 33.** IPG 5 kW fiber laser and the work station for the chamber test welds.

**Figure 34.** Finnpower sheet metal machining center. Triangular part is the laser resonator.

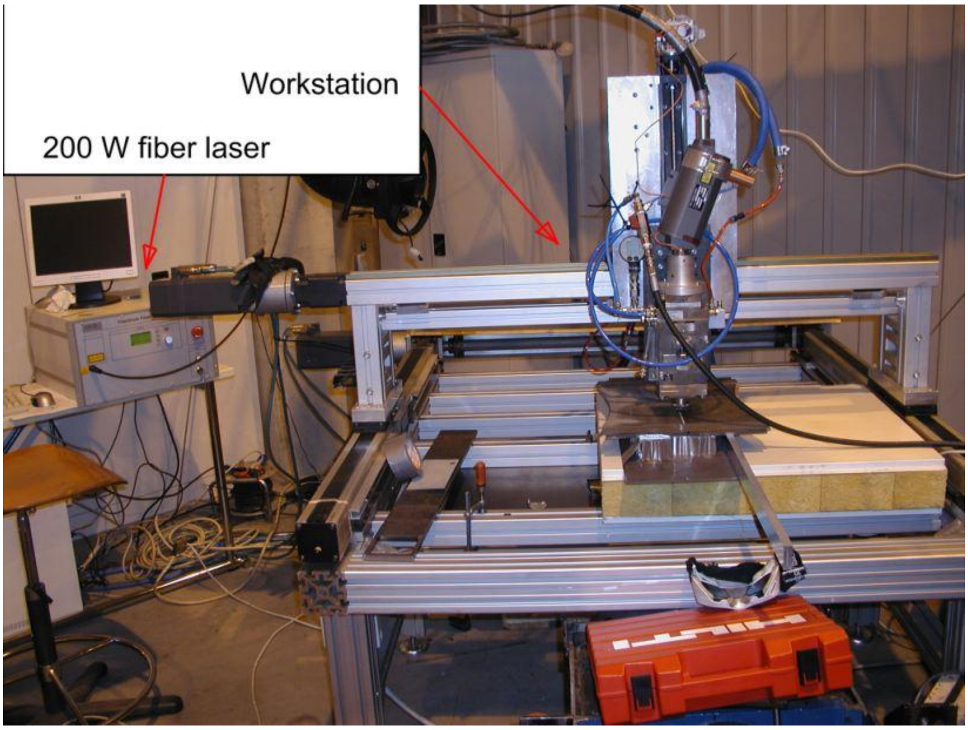



**Figure 35.** 200 W fiber laser and the workstation for metal foam cutting.

## 10.2 Reboiler

As mentioned in the design chapter, the reboiler was manufactured from two rods of brass to avoid material losses that would have occurred had it been made from a single rod sawed in two. The diameter of the rods was initially correct so the lathe was used only to cut them to correct length (90 mm). In the next step half of both rods were machined off, and the rectangular slot was milled to both halves with the universal milling machine. Next the halves were pressed together and all the holes were drilled. Finally, the threads were carved with a hand-held threading tool, shown in figure 36.

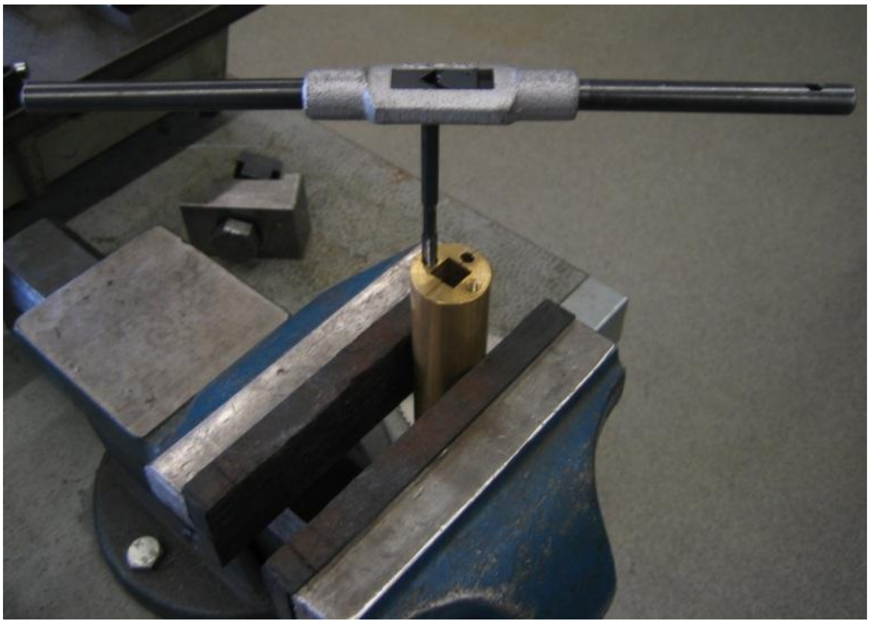


**Figure 36.** A hand-held thread carving tool.

The end plate was cut from the initial rod with the lathe and the holes drilled with the milling machine. Figure 37 shows the ready, disassembled reboiler; in figure 38 the reboiler is assembled. The technical drawings for this part are shown in appendix II.

**Figure 37.** Disassembled reboiler.


**Figure 38.** Assembled reboiler.

## 10.3 Separation unit

The initial pipe for the separation unit had a 30 mm inner dimension and 38 mm outer dimension. The lathe was used to first cut it to correct length and then to remove the excess wall thickness so that the final outer dimension was 32 mm. Next, the milling machine was used to drill the holes, after which the tube was cut axially. A shaft of solid steel was used inside the tube while the holes and the cut were machined to preserve the round shape during clamping. Figure 39 shows the ready separation unit, the cold end plate and the hose clamps used to tighten the tube around the reboiler. The plate was manufactured with $CO_2$ laser cutting at the Laboratory of Welding Technology and Laser Processing. The technical drawings for these parts are shown in appendix III.

**Figure 39.** The separation unit.

## 10.4 Distillation chamber

Whereas the first distillation test chamber was made from SS304L, the actual distillation chamber was manufactured from rods of SS316L stainless steel, shown in figure 40. The difference between these materials is explained in chapter 2.

**Figure 40.** Rods of SS316L austenitic stainless steel.

After cutting the rods to correct length with the lathe (already cut in figure 40), the milling machine was used to machine these according to the dimensions as depicted in the technical drawings. The finished parts are shown in figure 41 and the technical drawings shown in appendix IV. The three holes for the 1/8” pipes were drilled as second to the last step in the process. Last step was drilling the fourth hole for the 1/8" pipe in the reboiler. To ensure the hole was properly positioned it was drilled only after the distillation chamber was fit to the reboiler and the position marked through the hole in the reboiler.

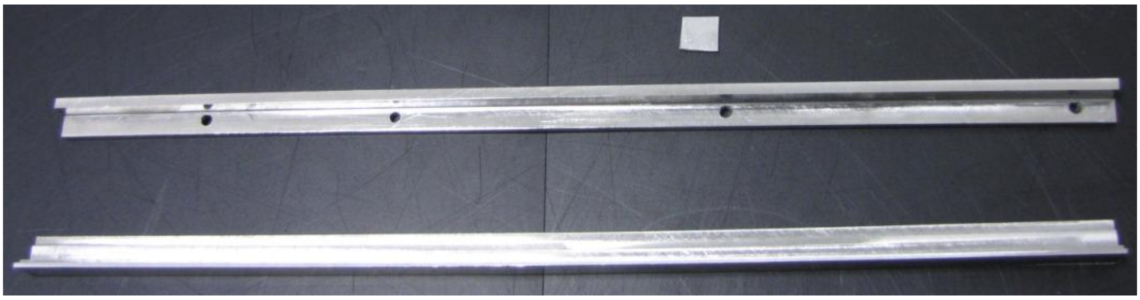


**Figure 41.** Parts for the distillation chamber, waiting to be welded.

The only problem that occurred during the manufacture was that of the internal stresses with the initial billet bending the parts after the machining. This was expected, however, and clamping the pieces together and tack welding them carefully will most likely be enough to overcome the slight bending. If not, a welding jig could be constructed. The chamber will be slightly grinded afterwards to make it fit inside the reboiler.

Before the final welding of the chamber, the metal foam (type Recemat RCM-NC-4753.016) needed to be cut to right dimensions and shape. The foam was cut with a 200W fiber laser and is shown in figure 42. Two slides of foam can be seen, one is 3 mm thick and the other 2 mm. The 2 mm thick slide was manufactured by cutting one millimeter off of another 3 mm thick slide. Used laser power was 200 W, focal point was 2 mm below the surface, and cutting speed was 1 m/min for the first cut and 0.8 m/min for the cutting of 1 mm off of the 3 mm slide. In this case laser cutting is a superb method, again because of its low and precise heat input. The cut edges of the foam are undisturbed and straight, and the pores have not clogged since the edge has practically not melted at all, or, like in case of mechanical cutting, flattened.

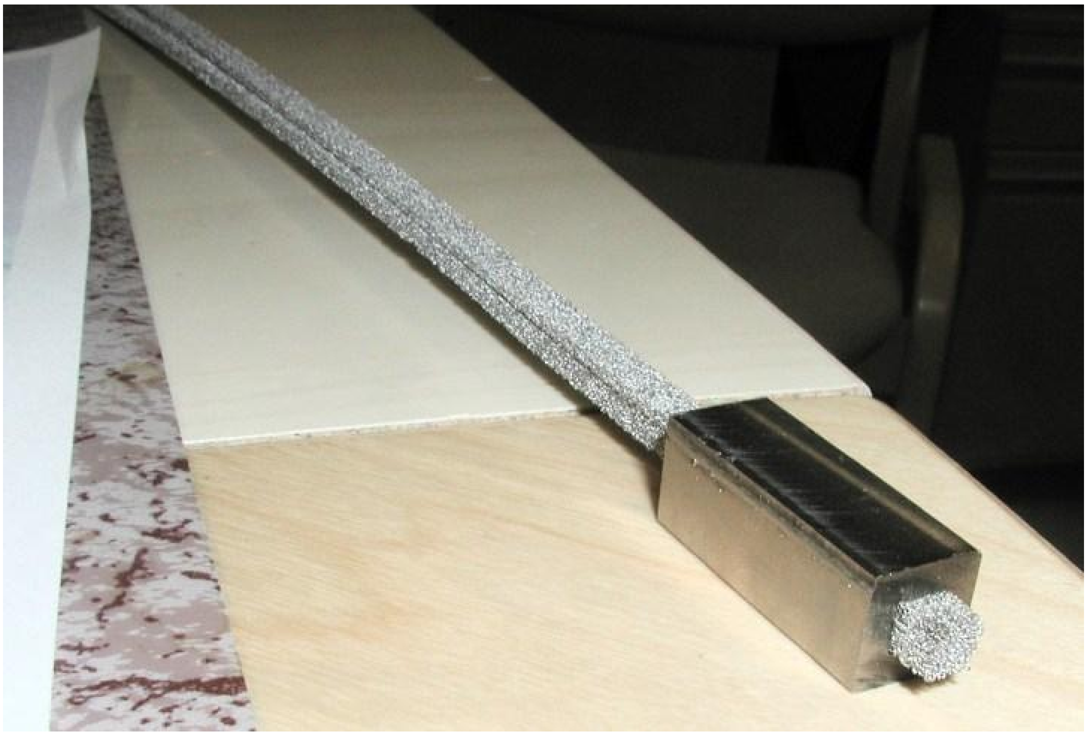


**Figure 42.** Metal foam inside the test chamber, cut with a 200W fiber laser.

# 11 ASSEMBLY

The assembly itself is not included in this thesis but the device will be assembled in the following order:

1. The chamber and the plate that seals the hot end of the chamber are welded
2. Foam is inserted in place (no welding needed since the foam will fill the whole chamber)
3. The 1/8" pipes are soldered to the chamber. Prior to soldering, the hose clamp which tightens the separation unit and reboiler together is placed between the first and second 1/8" pipe (counting from the reboiler)
4. The cross pipe fitting is welded to the cold end of the chamber. Prior to welding, the chamber is fitted through the plate that seals the cold end of the separation unit
5. The device is now otherwise easy to assemble.

# 12 TEST WELDING

Since the welding of the distillation chamber was such a crucial (as well as rather interesting) moment in manufacturing, the results of the test welds are covered here in more detail than might otherwise be prudent given the aims of this thesis. First welding tests were carried out on a 90 mm long test chamber, to insure the eventual assembly welding was successful. It was imperative that the penetration was sufficient and that there were not any excessive heat distortions. In chapter 4.3 was listed eight very well thought-out considerations for laser welding. In this case, particularly points one through to four and seven are why laser welding was chosen. No other method could provide as high-quality weld with such low heat input.

## 12.1 Results

The test welds were conducted with the 5 kW fiber laser. The parameters were chosen by experienced laboratory technicians and the first test already resulted in an acceptable weld. Used laser power was 1.5 kW, welding speed 3 m/min, focal point was on the surface and the gas was argon with a 25 l/min gas flow. After the welding, the chamber was cut into four pieces and macrosections were prepared for a closer examination of the quality of the welds in three places: at the start of the welding, in the middle and in the end. Start and end points were chosen to be around 5 mm from the edges of the chamber. Figure 43 shows macro images from these three points along the length of the chamber.

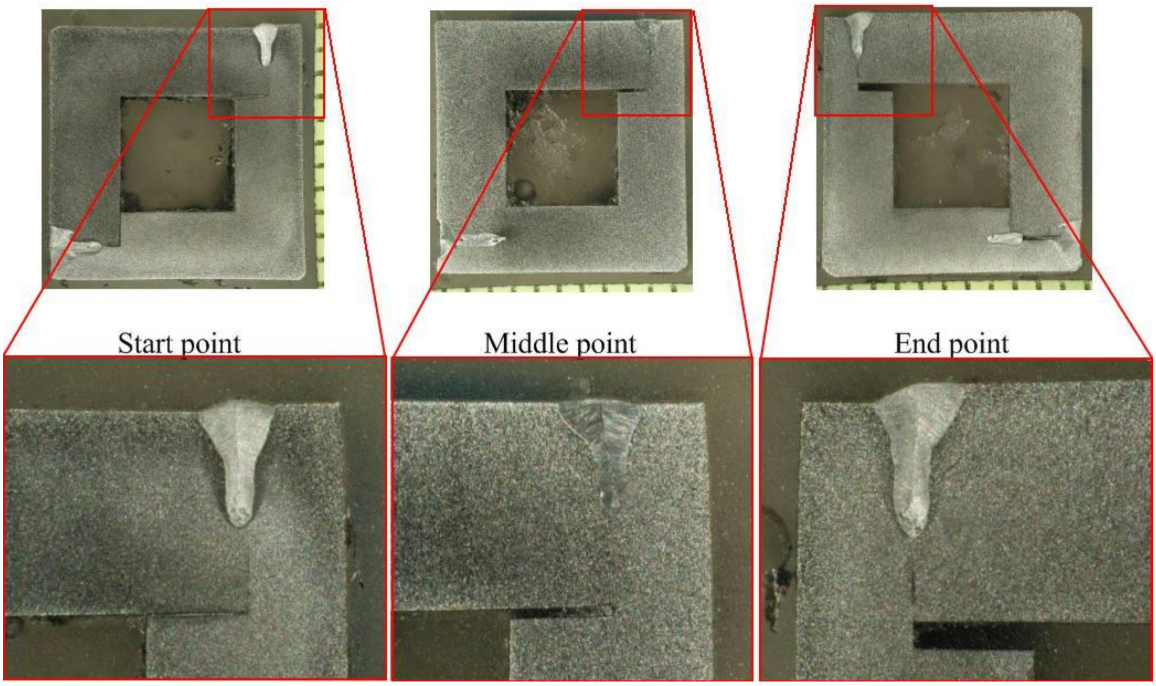



**Figure 43.** Macro images of the cross-sections of the welded distillation test chamber.

Due to positioning error during the first welding (best seen in the bottom right image in figure 43 as the gap), the second welding proved more difficult since the parts could not be pressed together as tight as they should be, resulting in an air gap between the parts. As expected, the air gap was too wide, so that most of the beam passed through and melted and welded the bottom of the groove, shown in figure 44. An oscillating beam mechanism was used for second pass to solve this problem. A scanning mechanism oscillated the beam slightly 200 times a second, which melted the edges of the groove and produced a good-quality weld, shown also in figure 44.

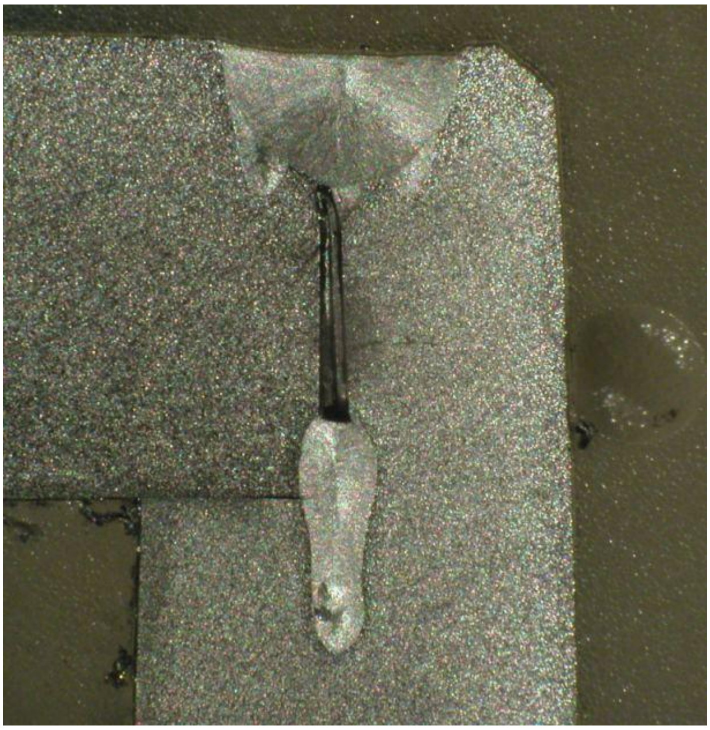


**Figure 44.** Cross-section of a corner of the distillation chamber (end point). The wide, semicircular weld at the top was achieved with an oscillating laser beam. The deep, narrow weld without oscillation. Welding was performed in two separate stages.

## 12.2 Test weld heat input analysis

The dimensions and area of the better weld (shown earlier in figure 42) are given in table 6. From these values (the start, middle and end point refer to figure 42), and the mechanical and physical properties of SS304L stainless steel, the amount of heat spent in melting the steel can be calculated. In this case 1457 joules of energy was spent in melting the steel, while 2700 joules of energy was introduced to the piece. The exact calculations are given in appendix I. According to the calculations 54 % of the introduced heat was spent in melting the steel. The gap width gives an idea about the scale of the positioning error that occurred during the welding.

**Table 6.** Dimensions and cross-sectional area of the weld in three points.

| | Penetration, [mm] | Weld width, [mm] | Weld root width, [mm] | Weld cross-sectional area, [mm$^2$] | Gap width, [mm] |
|---|---|---|---|---|---|
| **Start point** | 1.78 | 1.17 | 0.42 | 1.18 | - |
| **Middle point** | 1.55 | 1.26 | 0.44 | 1.06 | 0.15 |
| **End point** | 1.91 | 1.14 | 0.44 | 1.15 | 0.35 |

# 13 CONCLUSIONS

The aim of this thesis was to design and manufacture a microdistillation column. It was part of the FABTech project, funded by TEKES. The idea as well as the first drawings for this kind of a distillation device came from the Chemical Engineering research group at the Department of Biotechnology and Chemical Technology at Helsinki University of Technology.

These drawings were then refined and the idea developed during this thesis. Lastly, all the parts were manufactured. The final assembly was not included to the scope of this thesis due to time restrictions. The final product looks very much like the preliminary drawings, only major changes were: flanges were not needed and the distillation chamber is a continuous chamber instead of two parts split at the joint between the reboiler and the separation unit. The design process mostly consisted of detail problem solving and trying to come up with solutions to the manufacturing problems.

Next step in utilizing the benefits of the product are the assembly, after which come the actual distillation test runs to see if the device works and how effective is the separation. These tests will be carried out by the Chemical Engineering research group at HUT.

Honing and refining the drawings for the device was a challenging yet not too difficult task which let some room for creativity also, even if the preliminary drawings were partly finished already. Manufacturing the designed parts did not present any unforeseen challenges or difficulties once the best manufacturing method was found.

One thing that was underestimated at the beginning was how much the internal stresses of the billet tend to bend long and thin machined steel parts. Because of this, the first idea of fabricating the distillation chamber from steel slides did not succeed. Combining such parts into a working device requires very precise and rigid jigs for welding, and even then it might not be possible. An improvement could be reached with active cooling of the fixture.

Since this thesis was written at LUT Laser, a good assortment of laser equipment was available for the manufacturing. During the thesis various lasers ($CO_2$ and two different fiber lasers) have been used for producing the required parts, and thus the advantages as well as disadvantages of laser processing became much more evident. For example some welding during assembly is very unsuitable for laser welding mainly because it would require complicated system to be created for holding the workpiece, whereas with manual welding (tungsten inert gas (TIG) or metal inert or active gas (MIG/MAG) welding) those welds are easy and fast. The main advantages with laser processing were low and precise heat input when welding the chamber and cutting the parts, and the freedom of shape to be cut as demonstrated when the cold end plate of the separation unit was cut.

One thing that was very clear almost from the beginning was that expertise of many people was required even for creating this rather simple device. It does not include any electronic parts, nor does it include any other really complicated applied science technology (such as hydraulic systems etc.). Despite the simplicity, many experts were needed and involved, which only reinforces the fact that people need to work together to make something worthwhile happen.

# 14 FURTHER STUDIES

No design is ever perfect; the designers of the product know this the best. Small details in design or maybe the material choice can be fine-tuned to no end. On the other hand, the line has to be drawn somewhere, compromises made and product built. Still, it is always beneficial to think of things that could be improved or changed in the future.

## 14.1 Welding jig

This first version, or prototype, of this device was welded by clamping the pieces together and carefully tack welding them. If larger batches were required, (as in actual production) a welding jig would be needed to make the assembly quicker and to control quality. An idea for such a jig is presented graphically in figure 45.

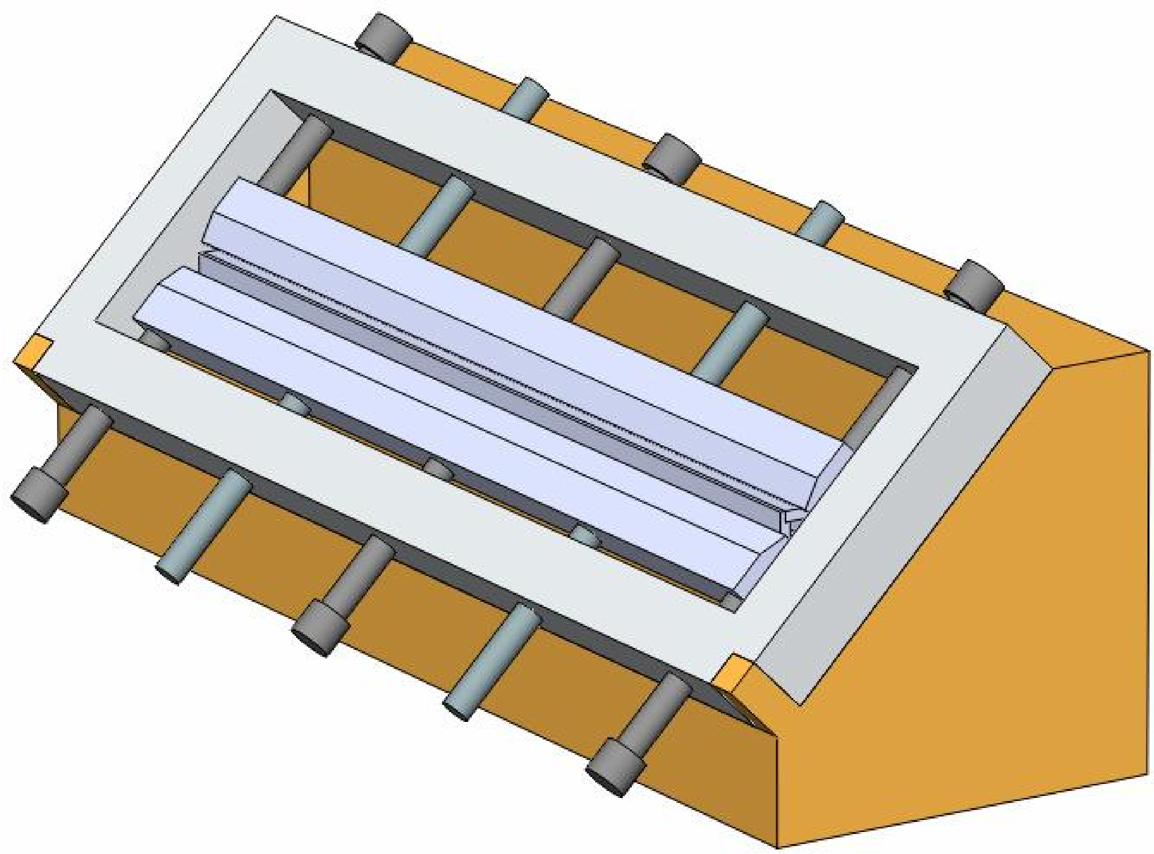


**Figure 45.** Welding jig model for the chamber.

One possible problem with this kind of a jig is that the welding head of the laser might not have enough room since it has to be very close to the surface unless remote welding system is used. If not, and if there is not enough room, some solution could be easily found by tweaking the clamping system.

## 14.2 Different separation unit

Since the only real function of the separation unit is to confine the insulating material, there are many possible ways it can be built. In theory, there does not even have to be a tube for it, just enough common tin foil wrapped around enough insulation would do. The one designed in this thesis is perhaps still too complex and time-consuming to manufacture given the simple requirements for the part.

## 14.3 Modular design

In this case one interesting possibility is to try to make the structure as modular as possible. As it is, the construction is modular in a sense that several of these could be put together and the product streams combined to increase the volume to an extent. However, since modularity was not the number one priority, it has not been seriously considered so far. For example, by slightly altering the shape of both the reboiler and the separation unit it is possible to produce a truly modular microdistillation column, shown in principle in figure 46. This kind of design follows the number-up principle, so the critical dimensions of the design are not altered. If higher volumes are needed, more of these are working in series instead of scaling up a single unit. However, it needs to be noted that since the volume of a single chamber is very small, even a high number of them is not going to result in very large volumes.

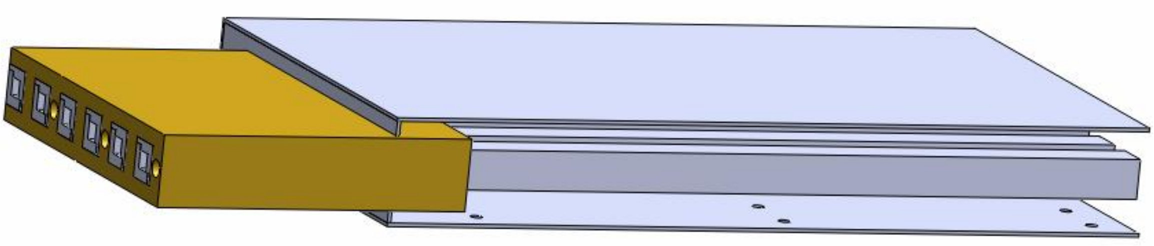


**Figure 46.** One possible modular design.

Dimensions, joint design and the general construction would of course have to be tweaked; for example the reboiler might require thicker walls or the separation unit more room for the insulation. Also, a cheaper and easier way to manufacture the distillation chamber would have to be found, although it would still not be economically feasible to manufacture thousands of these for a process that requires large volumes to be distilled.

# REFERENCES


1. Kujanpää V., Salminen A., Vihinen J., Lasertyöstö, 2005, Teknologiateollisuus ry, ISBN 951-817-876-3, pp. 68
2. Horton H. L., Jones F. D., Oberg E., Ryffel H. H., Machinery's Handbook (27$^{th}$ edition), 2004, ISBN 978-0-8311-2711-4, pp. 439-440
3. Lindroos V., Sulonen M., Veistinen M., Uudistettu Miekk-ojan METALLIOPPI, 1986, Teknillisten tieteiden akatemia, ISBN 951-666-216-1, pp. 440-471
4. Bhadeshia H.K.D.H., Sourmail T., Stainless steels, From: http://www.msm.cam.ac.uk/phase-trans/2005/Stainless_steels/stainless.html, referenced 12/05/2009
5. Kelly J., Stainless steels, In: Mechanical Engineers' Handbook – Materials and Mechanical Design (3$^{rd}$ Edition), 2006, pp. 49-53, 439-440
6. Lee J., Distillation Design Tutorial, From: http://lorien.ncl.ac.uk/ming/distil/dist-tut.htm, referenced 22/06/2009
7. Khandani S., Engineering Design Process – Education Transfer Plan, 2005, Industry Initiatives for Science and Math Education (IISME), 24 pp.
8. Browne J., Mcmahon C., CADCAM : principles, practice, and manufacturing management; 2$^{nd}$ edition, 1998, Pearson Education Limited, ISBN 0-201-17819-2, pp. 4-12
9. Dyson J., Duplex stainless steels, From: http://www.gowelding.com/met/duplex.html, referenced 29/06/2009
10. Duhamel R., F., Farson D., Taking advantage of laser welding: Use increases as laser welding becomes more familiar, The Fabricator, web article May 15$^{th}$, 2001.
11. Dawes C., Laser welding: A practical guide, 1992, Abington Publishing, ISBN 1 85573 034 0, 258 p.
12. Wörz O., Process development via a miniplant, Chemical Engineering and Processing, 1995, Volume 34, pp. 261-268

13. Discussions with M.Sc. (Tech.) Aarne Sundberg and D.Sc. (Tech.) Petri Uusi-Kyyny
14. Korinko P.S., Malene S.H., Considerations for the Weldability of Types 304L and 316L stainless steel, Journal of Failure Analysis and Prevention, 2001, Volume 1, Number 4, pp. 61-68
15. Grupp M., Laser Cutting of Sheet Metals with High-Power Fiber Lasers, 69th JLPS Conference, Tokyo, December 10th – 11th 2007
16. Kujanpää V., The Finns are into heavy metal, Industrial Laser Solutions Magazine for Manufacturers, 2006, Volume 21, Issue 5. Also available at: http://www.industrial-lasers.com/display_article/254871/39/none/none/Feat/The-Finns-are-into-heavy-metal.
17. Ion, J.C., Laser Processing of Engineering Materials, 2005, Elsevier Butterworth-Heinemann, ISBN 0 7506 6079 1, 556 pp.
18. McKetta J. J. Jr., Distillation, Encyclopedia of chemical processing and design, 1982, Marcel Dekker Inc., ISBN 0-8247-2466-6, pp. 42-82
19. Ruthven D. M., editor. Distillation, Encyclopedia of separation technology, 1997, John Wiley & Sons Inc., ISBN 0-471-16124-1, pp. 584-625
20. Discussions with Professor D. Sc. (Tech.) Antti Salminen
21. Discussions with Professor D. Sc. (Tech.) Ville Alopaeus

## APPENDICES

# Appendix I Distillation chamber test welding heat input calculations

Here is calculated the energy needed to melt the amount of stainless steel that was molten during the first preliminary welding of the distillation chamber. Area dimension for mass calculations were derived from macroscopic images of the weld cross-section (shown in figure 43).

Density of SS304 stainless steel = 8030 $kg/m^3$
Mass m = 0.00000113 $m^2$ x 0,09 m x 8030 $kg/m^3$ = 0.00082 kg
Latent heat of melting (fusion) L = 300 kJ/kg
Specific heat c = 500 J/kg.$^{o}$C,

The energy required to heat the steel from 20 $^{o}$C to about 1500 $^{o}$C (energy required to melt stainless steel) can be calculated from equation

$Q = mc\Delta T + mL$,

where m is mass, c specific heat for stainless steel, $\Delta T$ temperature difference and L latent heat of melting. Thus,

Q = 0.00082 kg x 500 J/kg.$^{o}$C x 1480 $^{o}$C + 0.00082 kg x 300000 J/kg ≈ 1457 J

Next is calculated the amount of energy introduced to the workpiece during welding:

Laser power P = 1500 W
Welding speed s = 3 m/min. = 0.05 m/s

Heat input can then be calculated from equation

$J = P/s$,

where P is the laser power at the workpiece and s welding speed. Thus,

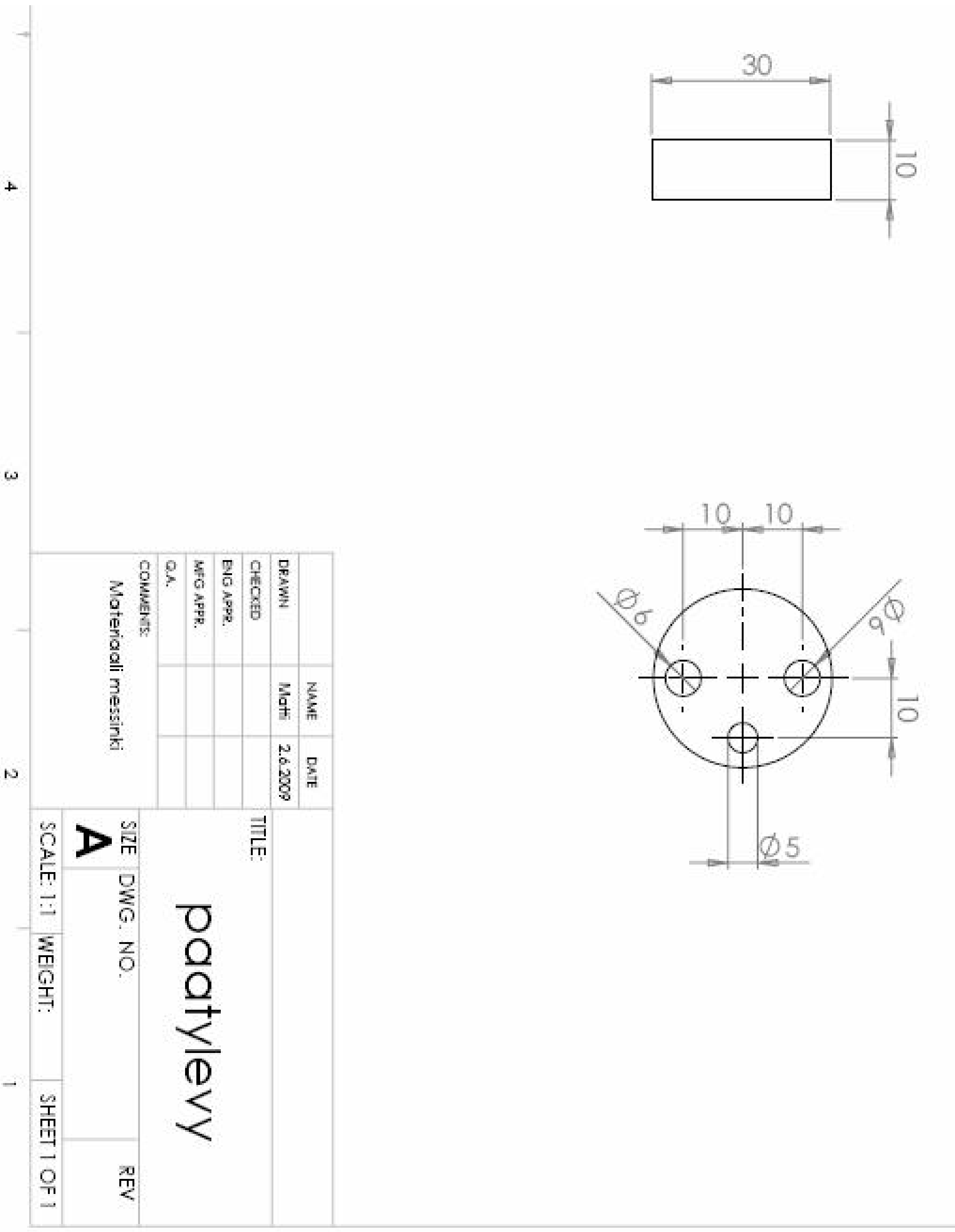
J = 1500 W / 0.05 m/s = 30000 J/m.

The weld is 90 mm long, therefore the maximum heat input is

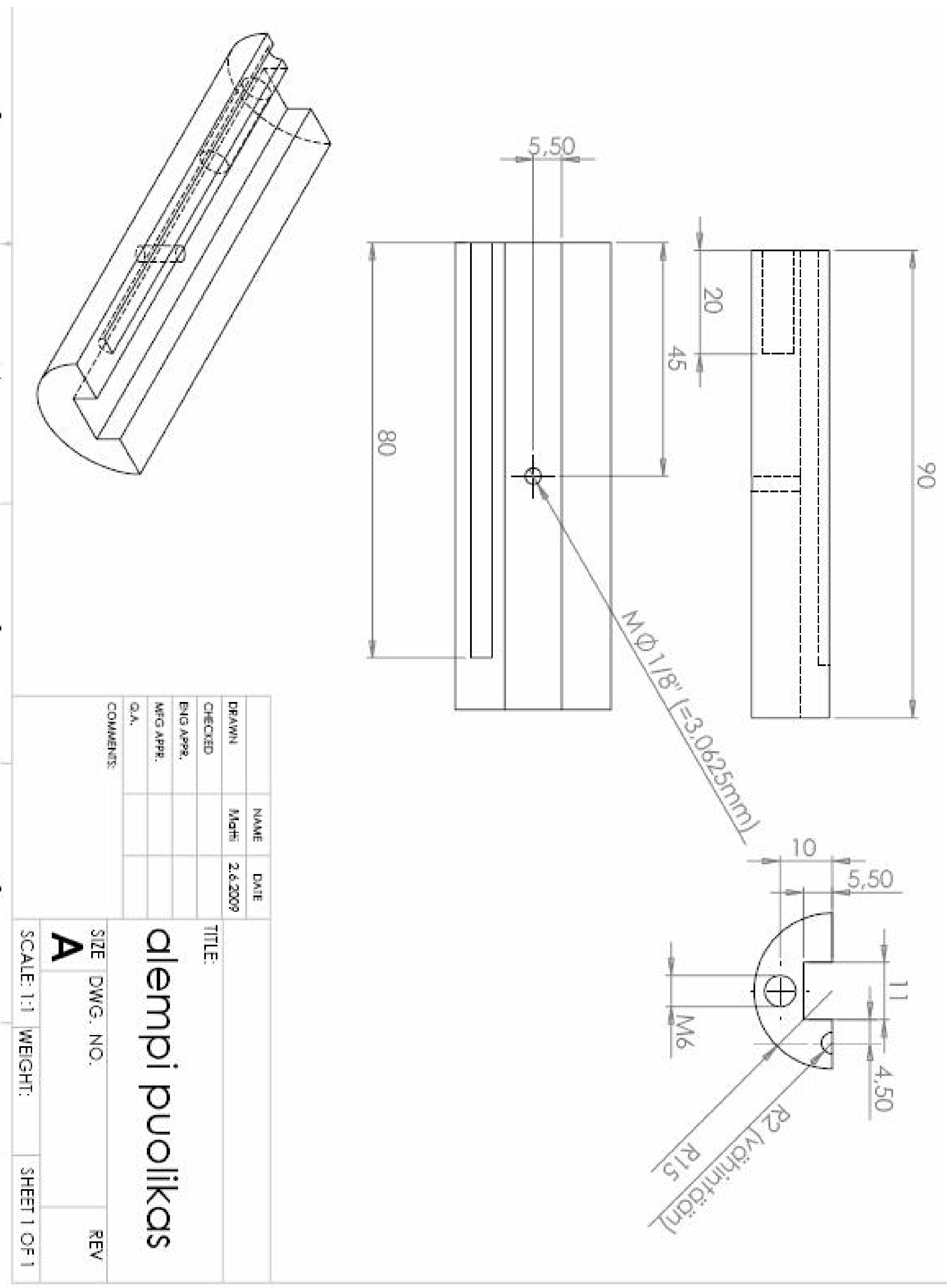
$Q_{max}$ = 30000 J/m x 0.09 m = 2700 J.

The ratio of thermal energy spent in melting the steel and the maximum amount of energy is Q / $Q_{max}$ = 1457 J / 2700 J = 0.54. Thus, 54 % of the laser beam energy was spent in heating and melting the steel.

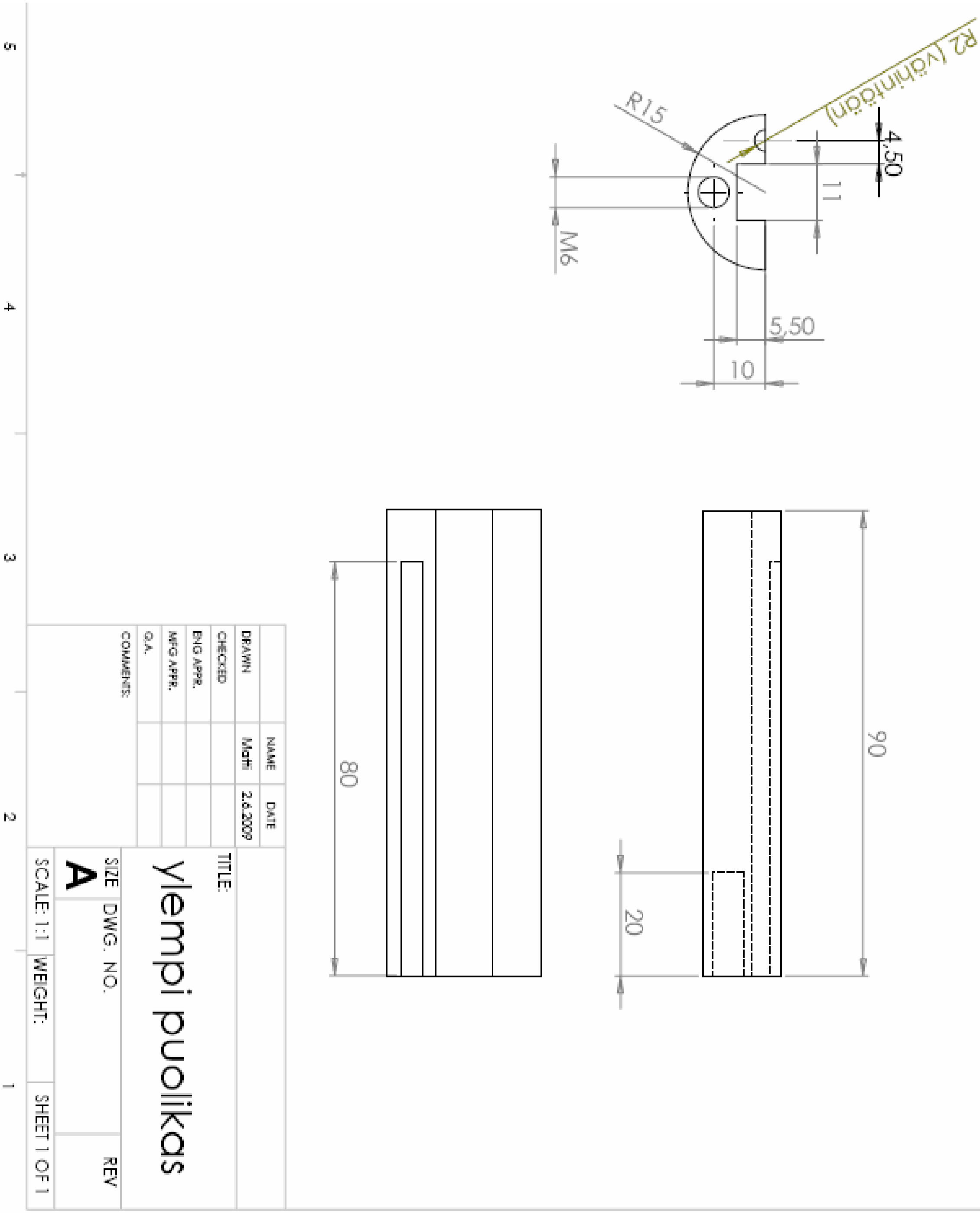
Appendix II Technical drawing for the reboiler – end plate



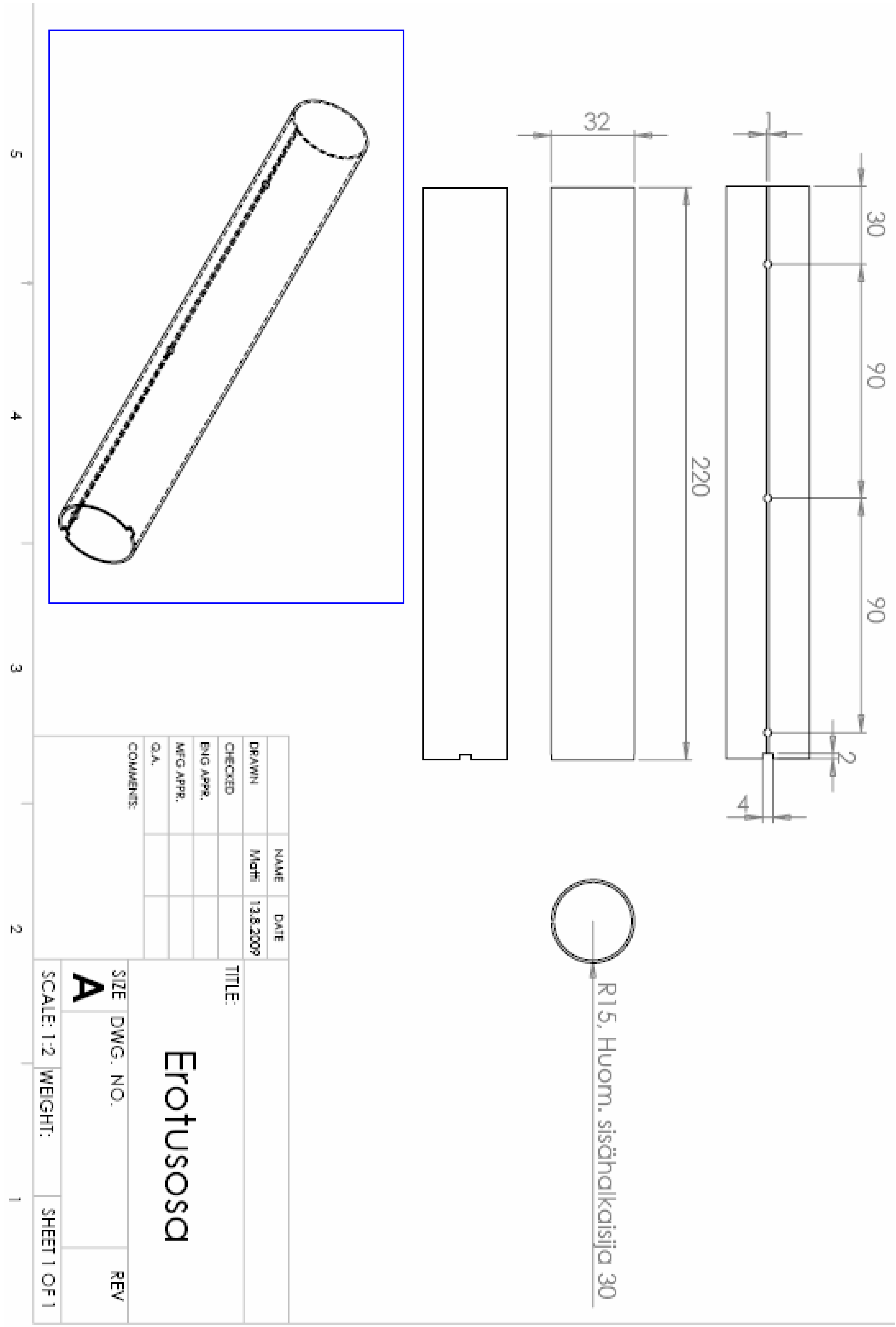
Appendix II Technical drawings for the reboiler – lower part



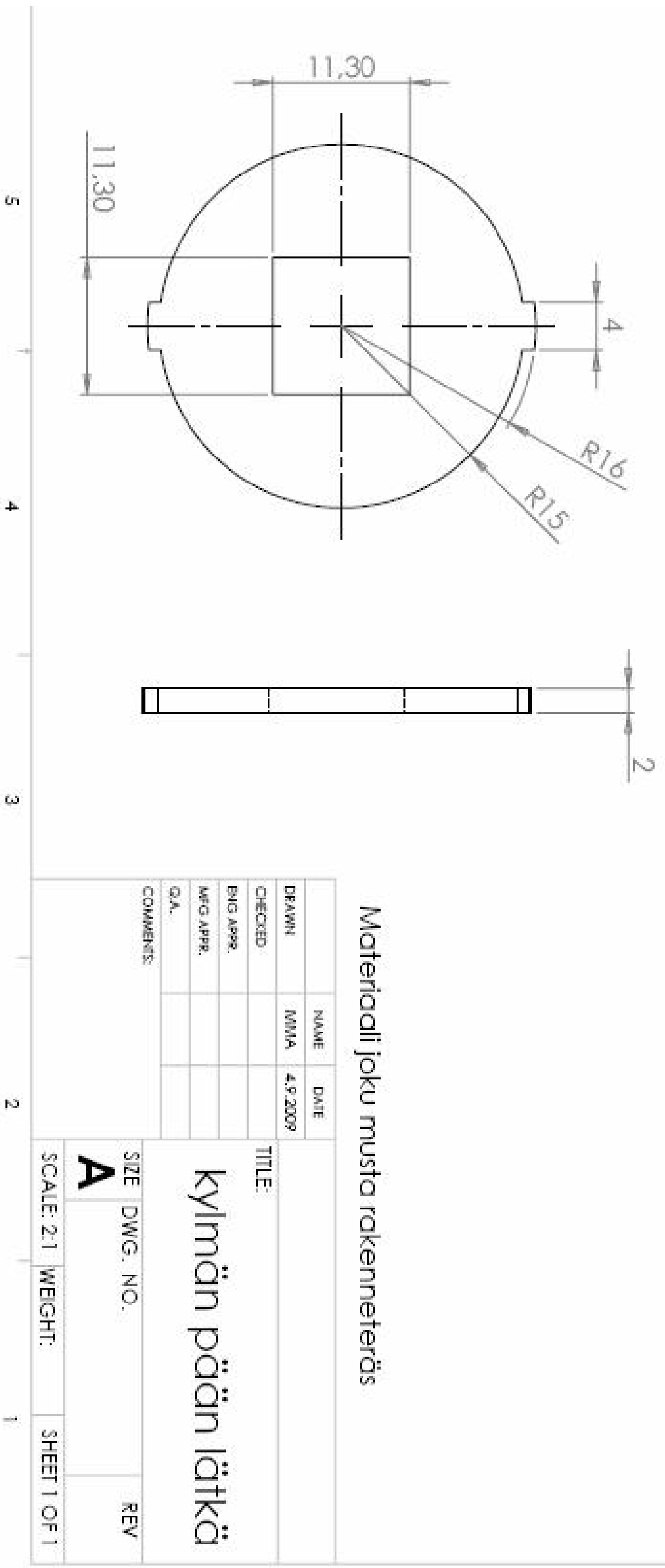
Appendix II Technical drawings for the reboiler – upper part



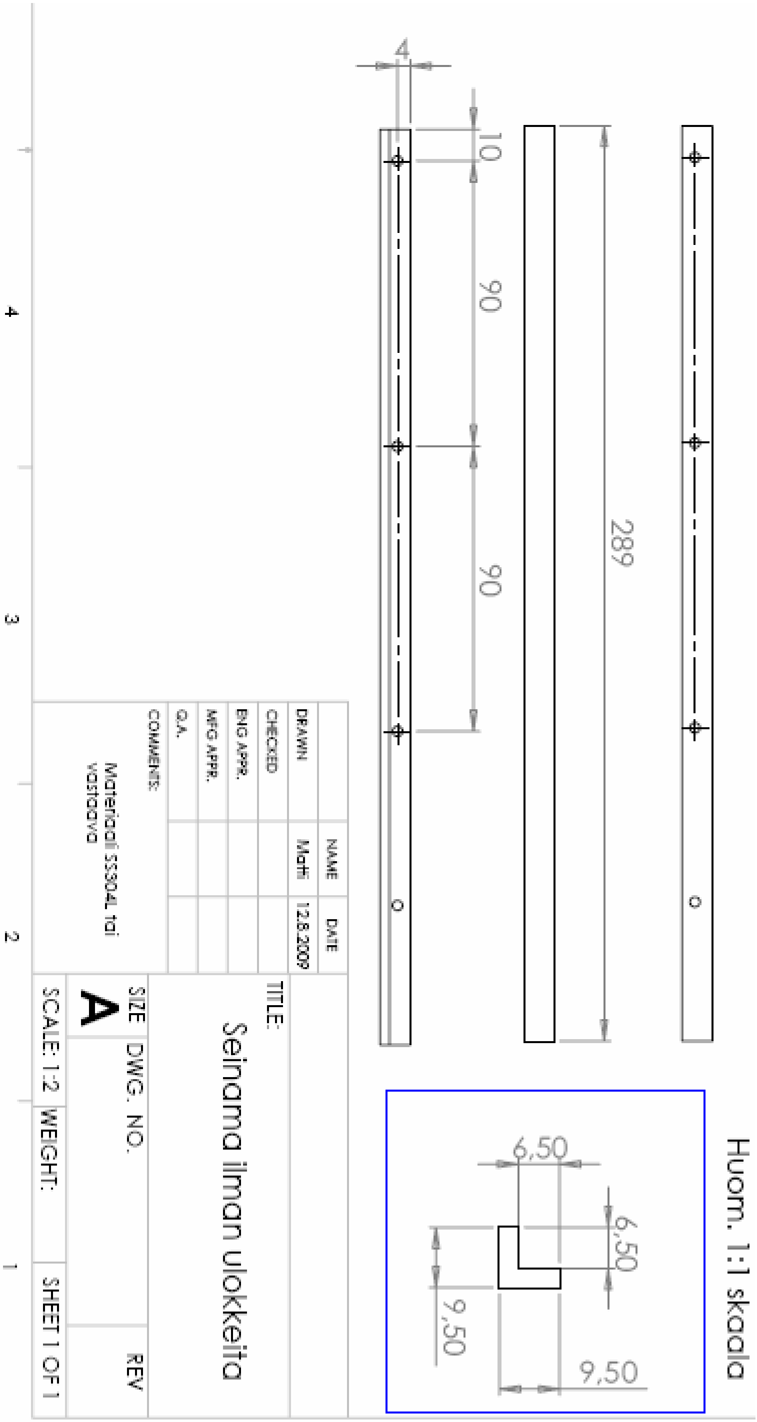
Appendix III Technical drawings for the separation unit



# Appendix III Technical drawings for the separation unit – cold end plate



# Appendix IV Technical drawings for the distillation chamber



Appendix IV Technical drawings for the distillation chamber