**NTNU – Trondheim**
Norwegian University of
Science and Technology

# Adsorptive Hydrogen Storage: Experimental investigations on thermal conductivity in porous media

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# NTNU

Norwegian University
of Science and Technology

Department of Energy
and Process Engineering

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## MASTER THESIS

for

Stud.techn. Jan Georg Henriksen

Spring 2013

Thermal conductivity measurements of porous materials

*Termisk konduktivitet for porøse materialer*

**Background and objective**

Hydrogen is a candidate as energy carrier for the future. The present project work efforts are focused on hydrogen storage material properties for on-board hydrogen storage technologies.

The most promising hydrogen storage methods are gas compression, liquefaction, chemical storage via metal hydrides and gas adsorption via physisorption (adsorption). Adsorption type storage systems are inexpensive alternatives that also have the potential to reach the energy density goals for on-board storage systems. Sorption type materials, like e.g. metal organic frameworks (MOFs) have been identified as a viable option. These are characterized by high porosity and high specific surface area.

However, the transient processes during charging and discharging of a storage system play an important role in the utilization of the hydrogen adsorption storage systems. Thermo-physical material properties such as thermal conductivity are crucial for determining the heat and mass transfer within the storage unit.

The main objective of this work is to install and verify a new characterization setup for thermal conductivity and specific heat capacity measurements. Furthermore, experimental investigation of porous materials provided by NTNU with an additional comparison with available literature data and prediction models.

**The following tasks are to be considered:**

1. A literature study on thermal conductivity of gas/solid porous media shall be performed, with emphasis on the transport mechanisms and predictive models. Also, the basic processes governing the thermal conductivity of the involved media (gases and solids) shall be presented.

2. Perform experiments to determine thermal conductivity of selected porous materials using the HotDisk transient measurement setup. The following tasks are involved:

   a. The setup shall be described, and a set of calibration/verification measurements shall be performed.

   b. A set of thermal conductivity measurements on selected hydrogen adsorption materials shall be performed, covering a range of temperatures and using different gases. If possible, thermal conductivity measurements shall also be performed using enhancement devices such as aluminum foam or wires.

    c.    An uncertainty analysis shall be performed.

    The measurements shall also be compared to predictions from the literature models/correlations described in part 1. The results shall be analyzed, presented and discussed.

3.    A model for heat distribution in a powdery material having aluminum foam or wires as enhancement device shall be developed. The model shall be presented and discussed. Predictions shall be compared to relevant measurements performed in section 2.

4.    Suggestions for further work shall be proposed.

- " --

Within 14 days of receiving the written text on the master thesis, the candidate shall submit a research plan for his project to the department.

When the thesis is evaluated, emphasis is put on processing of the results, and that they are presented in tabular and/or graphic form in a clear manner, and that they are analyzed carefully.

The thesis should be formulated as a research report with summary both in English and Norwegian, conclusion, literature references, table of contents etc. During the preparation of the text, the candidate should make an effort to produce a well-structured and easily readable report. In order to ease the evaluation of the thesis, it is important that the cross-references are correct. In the making of the report, strong emphasis should be placed on both a thorough discussion of the results and an orderly presentation.

The candidate is requested to initiate and keep close contact with his/her academic supervisor(s) throughout the working period. The candidate must follow the rules and regulations of NTNU as well as passive directions given by the Department of Energy and Process Engineering.
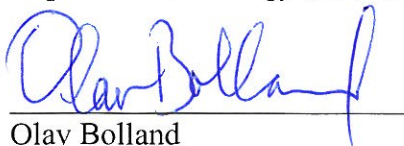
Risk assessment of the candidate's work shall be carried out according to the department's procedures. The risk assessment must be documented and included as part of the final report. Events related to the candidate's work adversely affecting the health, safety or security, must be documented and included as part of the final report. If the documentation on risk assessment represents a large number of pages, the full version is to be submitted electronically to the supervisor and an excerpt is included in the report.

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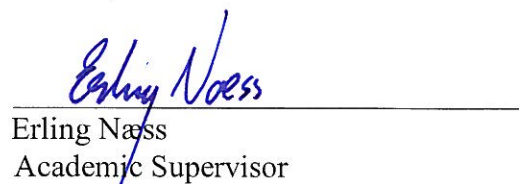
The final report is to be submitted digitally in DAIM. An executive summary of the thesis including title, student's name, supervisor's name, year, department name, and NTNU's logo and name, shall be submitted to the department as a separate pdf file. Based on an agreement with the supervisor, the final report and other material and documents may be given to the supervisor in digital format.

☒ Work to be done in lab (Water power lab, Fluids engineering lab, Thermal engineering lab)
☐ Field work

Department of Energy and Process Engineering, 14. January 2013

Olav Bolland
Department Head

Erling Næss
Academic Supervisor

Research Advisors: PhD student Christian Schlemminger, NTNU

# Preface

This work is the result of my master thesis at The Norwegian University of Science and Technology (NTNU), department of Energy and Process Engineering, located in Trondheim, Norway. This study allowed me to take part in an interesting research project on advanced hydrogen adsorption materials for hydrogen storage. The thesis revolves around the issues regarding the crucial filling and discharging process of a storage tank, determining the heat transport capabilities of the adsorption materials through lab experimentations.

During my work with this project I have learned a lot about research and working in a lab environment. The installation of the brand new Hot Disk TPS experimental setup took quite a long time, and due to equipment failure and delayed equipment deliveries the preliminary tests continued through Easter. However, playing around with the setup was also a lot of fun and provided me with useful insights that helped me at the later stages of the project. Also, my Co-supervisor, Christian Schlemminger, was always around ready to lend a helping hand, making sure that we were able to perform the necessary experiments.

Even though the laboratory tasks required a great amount of time and work, I feel that I have done good and precise work, and I am pleased with the final result. I found the work both challenging and exciting, and I am very happy to have been allowed to participate in this project.

I would like to thank my supervisor Erling Næss for guiding me on how to progress my work and providing very helpful insights at the end of the thesis. I also wish to express a special thanks to Christian Schlemminger for his cheerful nature and supportive behavior. He has inspired me into moving forward with my work, always giving me good advice and coming up with creative solutions.

My fellow students and friends outside university, and especially my girlfriend Astrid also need to be thanked; for making my days generally good, and for motivating and inspiring me after long working hours.

Trondheim, June 17th, 2013

Jan Georg Henriksen

# NTNU

Faculty of Engineering Science and Technology
**Department of Energy and Process Engineering**

Thesis title**:  Adsorptive Hydrogen Storage: Experimental investigations on thermal conductivity in porous media.**

Name: Jan Georg Henriksen
Date: 2013/06
Supervisor: Erling Næss and Ulrich Bünger
Co-supervisor: Christian Schlemminger

**Abstract:**

The objective of this work was to install and verify the Hot Disk TPS measurement setup for thermal conductivity measurements, and to carry out experiments on various porous materials. A literature survey on gas/solid porous media, with emphasis on the transport mechanisms and predictive models, was conducted. Special interest was taken in the widely-used Zehner/Bauer/Schlünder (ZBS) model for effective stagnant thermal conductivity of packed beds. Great care was shown in the determination of bed-properties such as porosity, because of the large effect it has on the effective thermal conductivity.

The porous materials investigated were the Metal Organic Framework (MOF) hydrogen adsorbents Cu-btc (HKUST-1) and Fe-btc-xerogel. Large ($\varnothing$1.395mm) and smaller ($\varnothing$0.38mm) glass beads served as a reference material for preliminary tests and setup validations. In a later stage the Cu-btc and Fe-btc was experimentally investigated. Thermal conductivity measurements were conducted on a packed bed with air, nitrogen ($N_2$) or helium (He) as fluid, in temperatures ranging from 243K$<T<$423K at an absolute pressure of zero to 0.5 bar.

The smaller glass beads ($\varnothing$0.38mm) were also tested together with an open-cell, high-porosity aluminum foam. The purpose of the metal foam in adsorption hydrogen storage is to increases the effective thermal conductivity of the bed. Experiments showed that applying the aluminum foam increased the magnitude of the effective thermal conductivity of a bed consisting of glass beads and air by a factor of 17 from 0.22 W/m$\times$K to 3.7W/m$\times$K at room temperature.

The preliminary experiments revealed a calibration error in the Hot Disk software, creating a discontinuity in the effective thermal conductivity in the range of 273K$<T<$283K. Outside that range, the Hot Disk measurement setup provides accurate measurements of the effective thermal conductivity of porous materials.

Hot Disk gives a measurement uncertainty of 5%. In addition to this comes the uncertainty of the theoretical model, due to the input of measured parameters such as porosity. An uncertainty analysis on the ZBS model gave an uncertainty of approximately ±10% for the glass beads and ±5% for the MOF, respectively. Adding the uncertainty of the ZBS model to the uncertainty of the experiments gives a total uncertainty of 15% for the glass beads experiments and 10% for the MOF.

Through a least square procedure, the solid conductivity of the MOF materials were fitted to the values of the ZBS model, determining temperature dependent functions for the solid conductivity yielding for each of the MOF's. The ZBS model proved to be a reliable estimate for the effective thermal conductivity in a packed bed, differing from the measurements with less than 10%.

# NTNU

Faculty of Engineering Science and Technology
**Department of Energy and Process Engineering**


Oppgavetittel**: Adsorbtiv Hydrogenlagring: Måling av termisk konduktivitet i porøse medier**
Navn: Jan Georg Henriksen
Dato: 06/2013
Veileder: Erling Næss og Ulrich Bünger
Med-veileder: Christian Schlemminger
**Sammendrag:**

Målet med dette arbeidet var å installere og verifisere et Hot Disk TPS måleoppsett for målinger av termisk varmeledningsevne i porøse medier, og å utføre eksperimenter på ulike porøse materialer. En litteraturstudie om gassfase/faste stoff- porøse medier, med vekt på transportmekanismer og prediktive modeller, ble gjennomført. Den mye brukte Zehner/Bauer/Schlünder (ZBS)-modellen for effektiv termisk konduktivitet i porøse medier ble tillagt spesiell vekt. Stor forsiktighet ble vist ved bestemmelsen av porøsitet, på grunn av den store effekten den har på varmeledningsevnen.

De porøse materialene som ble undersøkt var Metal Organic Framework (MOF) hydrogen adsorpsjonsmidlene Cu-btc (HKUST-1) og Fe-btc-xerogel. Store ($\varnothing$1.395mm) og mindre ($\varnothing$0.38mm) glassperler fungerte som referansemateriale for foreløpige tester og validering av måleoppsettet. På et senere stadium ble Cu-btc og Fe-btc eksperimentelt undersøkt. Målinger av termisk konduktivitet ble utført med luft, nitrogen ($N_2$) eller helium (He) som fluid, ved temperaturer på 243K $<T<$ 423K og med et absolutt trykk på null til 0,5 bar.

De mindre glasskulene ($\varnothing$0.38mm) ble også testet sammen med et høyporøst aluminiumskum. Formålet med metallskum i adsorptiv hydrogenlagring er å øke den effektive termiske ledningsevnen i tanken. Eksperimenter har vist at innføring av et aluminiumskum økte størrelsen av den effektive termiske ledningsevnen i en tank fylt med glassperler og luft med en faktor på 17 fra 0,22 W/m×K til 3.7W/m×K ved romtemperatur.

De første eksperimentene avslørte en kalibreringsfeil i Hot disk-programvaren som skaper en diskontinuitet i den effektive termiske ledningsevnen i området fra 273K$<T<$283K. Utenfor dette området gir den Hot Disk-måleoppsettet nøyaktige målinger av den effektive termiske ledningsevne til porøse materialer.

Hot Disk angir usikkerheten i hvert datapunkt til å være 5 %. I tillegg til dette kommer usikkerheten til den teoretiske modellen, på grunn av innføring av målte parametere slik som porøsitet. En usikkerhetsanalyse på ZBS-modellen ga en usikkerhet på omlag + -10 % for glassperler og + -5 % for MOF. Ved å summere usikkerheten i ZBS-modellen til usikkerheten gitt av forsøkene gir dette en total usikkerhet på henholdsvis 15 % for glassperler og 10 % for MOF.

Gjennom en minste-kvadrats-metode ble varmeledningsevnen til adsorbentene Cu-btc og Fe-btc bestemt. Temperaturavhengige funksjoner for begge MOF-materialene ble funnet ved sammenligning med ZBS-modellen. Denne modellen viste seg å gi gode estimater for den effektive varmeledningsevnen i porøse medier med mindre en 10 % relativt måleavvik.

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## Nomenclature

| | | | |
|---|---|---|---|
| $A$ | Area [m$^2$] | $l_s$ | Thickness of the fluid film adjacent to the contact surface of two particles [m] |
| $a$ | Ligament radius [m] | | |
| $a_T$ | Accommodation coefficient [1] | $l_{mfp}$ | Mean free path of molecular collisions [m] |
| $B$ | Shape-/deformation factor [1] | | |
| $b$ | Shape factor [1] | $M$ | Molecular weight [kg/kmol] |
| $C$ | Constant [1] | $m$ | Mass [kg] |
| $C_p$ | Heat capacity at constant pressure [J/K] | $N$ | ZBS Simplification parameter [1] |
| $C_{el}$ | Electronic volumetric specific heat [J/m$^3$K] | $N_A$ | Number of particles per unit area [1] |
| | | $N_L$ | Number of particles per unit length [1] |
| $\bar{c}$ | Mean molecular velocity [m/s] | $Nu_f$ | Radiation Nusselt number [1] |
| $c$ | Specific heat capacity [J/kgK] | $\boldsymbol{n}$ | Normal vector [1] |
| $D$ | Pore diameter [m] | $n$ | Number of contact points [1] |
| $D(\tau)$ | Function of TCT [1] | $P_0$ | Sensor power output [mW] |
| $d$ | Diameter [m] | $p$ | Pressure [Pa] |
| $\bar{d}_p$ | Sauter diameter [m] | $Q$ | Heat transfer [W] |
| $d_s$ | Equivalent spherical diameter [m] | $R$ | Particle radius [m] |
| $E$ | Radiation exitance [W] | $\tilde{R}$ | Universal gas constant =8314 [J/kmol·K] |
| $E_{fuel}$ | Lower heating value [kJ/kg] | $R_i$ | Initial Nickel resistance [ohm] |
| $E_s$ | Young's modulus [GPa] | $R(t)$ | Sensor resistance [ohm] |
| $E_{exp}$ | Expended fuel [kJ/kg] | $r$ | Radius [m] |
| $e$ | $= w/L$ | $S$ | Kaviany contact area parameter [1] |
| $F$ | Function of optical properties [1] | $S_F$ | Kaviany contact area parameter [1] |
| $f$ | Force exerted on particles [N] | $T$ | Temperature [K] |
| $g$ | $= a/L$ | $t$ | Time [s] |
| $h_c$ | Contact area heat transfer coefficient [W/m$^2$K] | $U$ | Simplification parameter [1] |
| | | $V$ | Volume [m$^3$] |
| $h_{rf}$ | Fluid radiation heat transfer coefficient [W/m$^2$K] | $W_i$ | Any physical quantity under consideration for volume-averaging [?] |
| $h_{rs}$ | Solid radiation heat transfer coefficient [W/m$^2$K] | $w$ | Node width [m] |
| $K$ | Solid-fluid conductivity ratio $= k_s/k_f$ [1] | $x$ | Direction in the Cartesian coordinate system [m] |
| $k$ | Thermal conductivity [W/m·K] | $x_0$ | Diameter of sectional area corresponding to one contact point [m] |
| $L$ | Length [m] | | |
| $l$ | Mean free path of gas molecules [m] | $x_r$ | System characteristic dimension [m] |
| $l_f$ | Thickness of a slab of solid giving the same heat resistance as a spherical particle [m] | $y$ | Direction in the Cartesian coordinate system [m] |
| | | $z$ | Direction in the Cartesian coordinate system [m] |

## Abbreviations

| | | | |
|---|---|---|---|
| DOE | US Department Of Energy | P&ID | Piping and Instrumentation Diagram |
| IES | International Energy Summit | REV | Representative Elementary Volume |
| LHV | Lover Heating Value | TPS | Transient Plane Source |
| MOF | Metal Organic Framework | TCR | Temperature Coefficient of Resistivity |
| NG | Natural Gas | TCT | Ratio of Total to Characteristic Time |
| NTNU | Norwegian University of Science and Technology | US | United States |
| | | ZBS | Zehner, Bauer and Schlünder |

**Greek letters**

| | |
|---|---|
| $\alpha$ | Thermal diffusivity [m$^2$/s] |
| $\alpha_i$ | Thermal diffusivity of layer material [mm$^2$/s] |
| $\beta$ | $= \Delta L/d_p$ [1] |
| $\gamma$ | $= l_s/d_p$ [1] |
| $\delta$ | Molecular diffusivity [m$^2$/s] |
| $\Delta_p$ | Probing depth [mm] |
| $\Delta T_i$ | Initial temperature difference [K] |
| $\Delta T(\tau)$ | Sample temperature difference [K] |
| $\Delta t_i$ | Initial time difference [s] |
| $\Delta X_i$ | Uncertainty of a parameter [1] |
| $\varepsilon$ | Porosity [1] |
| $\varepsilon_r$ | Emissivity [1] |
| $\zeta$ | Distribution parameter [1] |
| $\eta$ | Tortuosity factor [1] |
| $\theta$ | Characteristic time [s] |
| $\theta_0$ | Angle corresponding to the boundary of heat flow for one contact point [rad] |

| | |
|---|---|
| $\vartheta$ | Temperature coefficient of resistivity (TCR) [ohm/K] |
| $\lambda$ | Fluid to solid conductivity ratio $= k_f/k_s$ [1] |
| $\Lambda$ | Dimensionless solid conductivity $= k_s/4\sigma d_p \bar{T}^3$ [1] |
| $\mu_s$ | Poisson ratio [1] |
| $\xi$ | Insulating layer thickness [mm] |
| $\pi$ | Pi = 3.14159 [1] |
| $\rho$ | Density [kg/m$^3$] |
| $\sigma$ | Stephan Boltzmann constant = 5.67·10$^{-8}$ [W/m$^2$K$^4$] |
| $\tau$ | Total to characteristic time (TCT) |
| $\varphi$ | ZBS contact area parameter [1] |
| $\phi$ | $= l_v/d_p$ [1] |
| $\chi$ | Reflectivity [1] |
| $\psi$ | Absorptivity [1] |
| $\omega$ | Transmissivity [1] |

**Subscripts**

| | |
|---|---|
| 0 | Condition at 273K |
| a | Axial |
| alu | Aluminum |
| av | Average |
| b | Bed, bulk |
| bb | Black body |
| c | Core, crossectional, contact area |
| e | Effective |
| el | Electron |
| exp | Expended |
| f | Fluid |
| fs | Interface between fluid and solid |
| g | Gas |

| | |
|---|---|
| i | Denoting the ith particle size fraction |
| j | Denoting the phase (fluid or solid) |
| mfp | Mean free path |
| n | Denoting the nth section (A,B,C or D) |
| m | Zehner and Schlünder exponential constant |
| nc | Non-conducting |
| p | Particle |
| ph | Phonon |
| r | Radiation, radial |
| s | Solid, spherical |
| uc | Unit cell |
| v | Void |

**Superscripts**

| | |
|---|---|
| $\cdot$ | Quantity per unit volume |
| $^-$ | Average of quantity |
| $''$ | Quantity per unity area |
| $'''$ | Quantity per unit volume |
| * | Modified quantity |

**Other symbols**

| | |
|---|---|
| ⌀ | Diameter [mm] |

# Introduction

There will come a time when the fossil fuels that the world today so conveniently depends on will run out, and therefore we have to look for alternate energy sources and carriers for supporting future energy demand. The run out of fossil fuels will produce a gap that needs to be filled, and it has to be filled with sustainable renewable energy sources. Since hydrogen ($H_2$) can be made from electrolysis of water using renewable electricity coming from solar or wind power, hydrogen is a possible energy carrier for the future.

Recently, the European Commission conducted an extensive well-to-wheel analysis of various automotive fuels (IES, 2007). They consider the energy ratio $E_{exp}/E_{fuel}$, where $E_{fuel}$ is the energy content for combustion (lower heating value, LHV) and $E_{exp}$ is the energy expended to create the fuel. One of the most common, and short term beneficial production methods for $H_2$ is by steam reforming of natural gas, and this has and $E_{exp}/E_{fuel}$ ratio of 0.75 (IES, 2007). Hydrogen thus forms a basis for an energetically viable infrastructure.

Since hydrogen can be produced from electricity, hydrogen storage allows for a more efficient utilization of the grid. In particular, the use of grid-connected intermittent energy sources such as water or wind turbines can benefit from grid energy storage. Energy derived from these energy sources is variable by nature – the amount of electrical energy produced varies with time, day of the week, season, and random factors such as the weather. Hydrogen storage thus facilitates for environmentally friendly energy sources to be utilized outside whenever, working as a grid buffer.

Hydrogen also allows for the use of high-efficiency energy conversion devices, such as fuel cells, from which the products are only water and $O_2$. If $H_2$ made by renewable energy sources is combusted in a fuel cell, this gives a zero-emission energy chain which can be applicable for the transport sector in the future.

Compared with other fuels, $H_2$ has a lower heating value (LHV) of almost triple. However, there are a few challenges regarding transport and storage. Although hydrogen has a high energy density compared to other fuels, its volumetric density is only 0.084 kg/Nm$^3$, compared to the 0.65 kg/Nm$^3$ of Methane and 4.4 kg/Nm$^3$ of gasoline (Ullmann, 1989).
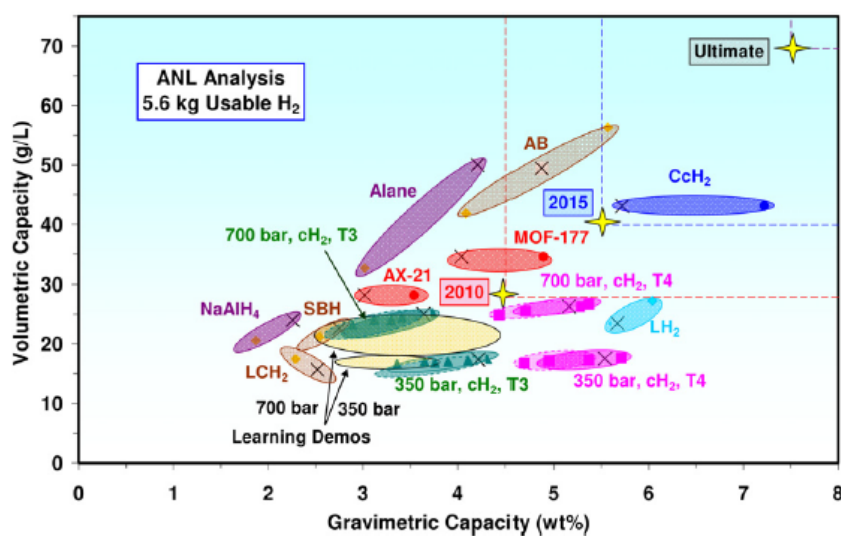


Fig. I-1 - Map of storage capacities of different hydrogen storage options (R.K. Ahluwalia, 2012).

1

Compression is the preferred method for hydrogen storage in automotive applications today. Fig. I-1 shows that the only storage method that meets the US Department of Energy (DOE) system targets of today is cryo-compression ($CcH_2$) (R.K. Ahluwalia, 2012). Hydrogen for use in cars is mainly compressed to 35…70 MPa. This calls for expensive storage materials. Other methods for hydrogen storage are liquefaction, cryo-compression, and chemical storage via metal hydrides or adsorption via physisorption. Onboard hydrogen storage for transportation applications is proven to be one of the most technically challenging barriers to the commercialization of hydrogen fueled light-duty vehicles. The technologies today do not meet the goals set for the future. The US Department of Energy has renewed the targets for light duty transportation. Some of the goals are given in Table I-1 below.

Table I-1 - Targets for hydrogen storage for light duty vehicles (US Department of energy, 2011).

| Storage parameter | Units | 2017 target | Ultimate target |
|---|---|---|---|
| System Gravimetric Capacity | kWh/kg | 1.8 | 2.5 |
| System Volumetric Capacity | kWh/L | 1.3 | 2.3 |
| Storage System Cost | $/kWh net | 12 | 8 |
| System fill time (5 kg) | min | 3.3 | 2.5 |
| | kg H2/min | 1.5 | 2.0 |

Metal-Organic Frameworks (MOF), with their high porosity and large surface area, serve excellently as sorption materials for hydrogen storage. NTNU, together with Max Planck Institute for Intelligent Systems (Stuttgart) and University Dresden, participate in a research and development-project for MOF materials. In order to meet future requirements by means of storage dimension dynamics, it is important to have knowledge of the thermophysical properties of the MOF materials.

One of the key challenges is the process of charging and discharging storage systems. In order to understand transient storage behavior, and to be able to improve the storage dynamic, elemental physical properties such as thermal conductivity and permeability must be characterized. Prior to this work, reports on permeability (transport capability) of MOF materials have been conducted. (Hubert, 2011); (Henriksen, 2012). This current work follows the earlier projects on thermal conductivity conducted by Jeremy Gauthier (2011) and Rasmussen and Eithun (2011). This work will provide new experimental data on the MOF materials Cu-btc (HKUST-1) and Fe-btc-xerogel as well as further research on heat transport enhancement devices such as open-cell aluminum foams. This work is organized in five main parts:

- Part 1 – Theoretical fundamentals for heat transport through porous media, with emphasis on predictive models and transport mechanisms.
- Part 2 – Description of the measurement setup, estimate of bed properties and uncertainty analysis.
- Part 3 – Results and analysis of the experiments made on MOF materials including a detailed uncertainty analysis.
- Part 4 – A new approach for determining the quality of aluminum foam.
- Part 5 – Conclusions and suggestions for further work.

# Part 1 - Heat transfer in porous media

## 1.1 The porous medium

A porous medium is a medium which consists of both a solid and a fluid phase. The fluid may be either gas, liquid or a combination. One may refer to porous media and mean one of two things:

- A solid structure with an interconnected void.
- A powder or a sample of particles which have a void in between them.

Both are porous. The first one can for example be a sponge-like material or porous rock. The only material described in this report which is a solid matrix is the metal-foam. The second type refers to granular porous media, like sand or powder. In this report the granular materials are glass beads or MOF (Metal Organic Framework). Both granular and solid-structure porous media are shown in Fig. 1.1. However, the MOF-particles themselves are porous, consisting of a solid structure containing extremely small pores. When hydrogen is adsorbed for storage the granular MOF-particles are packed tightly together in a packed bed (also referred to as fixed bed).
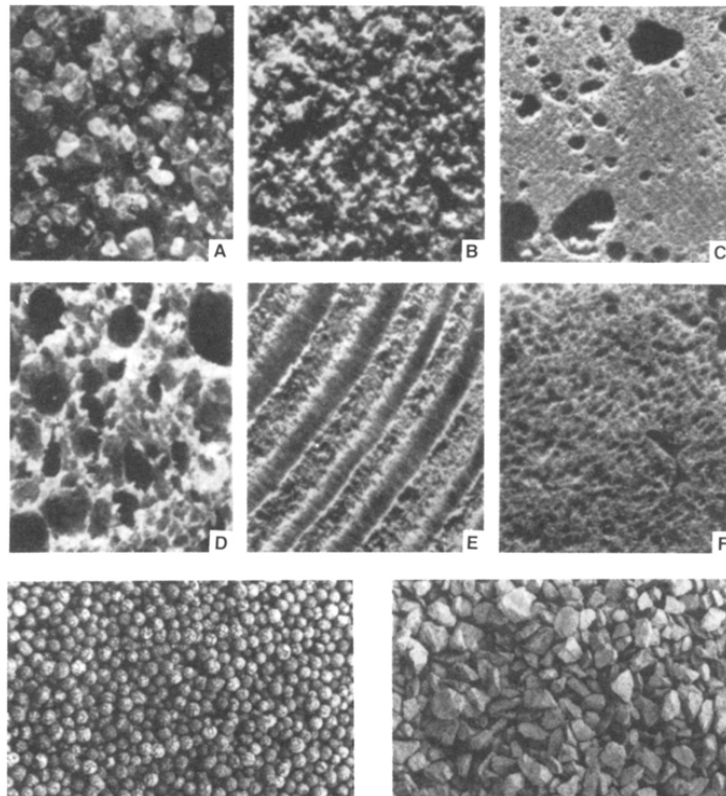


Fig. 1.1 - Top: Examples of natural porous materials: A) beach sand, B) sandstone, C) limestone, D) rye bread, E) wood, and F) human lung.

Bottom: Granular porous materials used in the construction industry, 0.5-cm-diameter Liapor spheres (left), and 1-cm-size crushed limestone (right) (Bejan 2006).

### 1.1.2 Porosity

The porosity is the volume fraction between the fluid and solid phase. It is described as the ratio between fluid volume and total volume, and is important to understand when conducting heat through a porous medium. For a packed bed, porosity is given as

$$\varepsilon = \frac{V_v}{V_b}. \tag{1.1}$$

Here, $V_v$ is the volume of the void/fluid and $V_b$ is the bulk volume, i.e. the total volume occupied by the material and the void in between. Defining the porosity this way assumes that (1-ε) is the fraction of the solid.

For granular porous materials, the porosity may vary greatly with packing. For example, for equally sized spheres, the lowest porosity possible is reported to be 0.259 (Kaviany, 1995). With random packing of equally shaped spheres however, the porosity varies along 0.37 – 0.43. Preliminary tests made in this work are done with glass beads of an almost perfect-sphere shape, and the porosity is therefore expected to lie within this range.
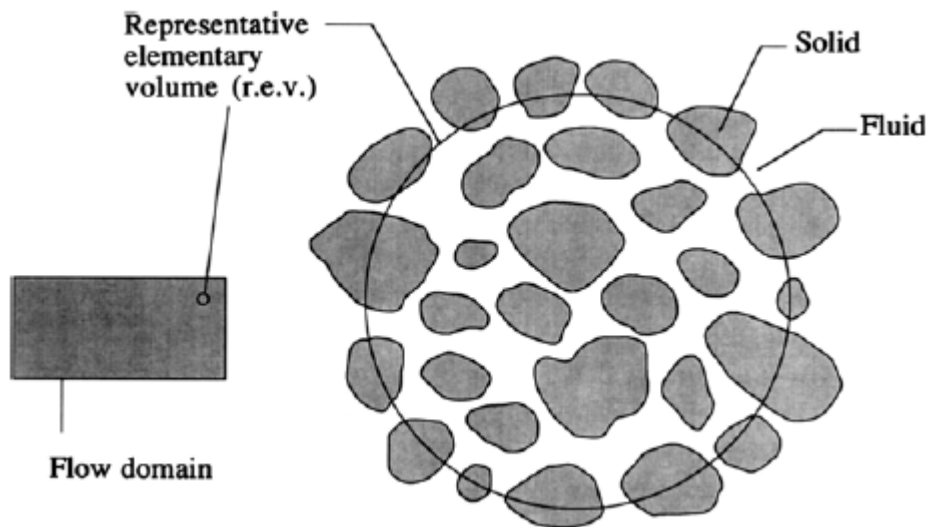


Fig. 1.2 - Porosity visualized (Bejan, 2006).

Fig. 1.2 displays a representative elementary volume (REV) of a porous medium. Since porous media is so complex in geometry, a common approach on heat transfer problems is volume averaging. This basically means to apply the basic energy and momentum equations on only a piece of the medium, a piece that within an acceptable statistical margin of error can represent the behavior of the entire medium (Tsotsas, 1987).

### 1.2 Heat transfer mechanisms

Heat transfer in porous media is carried out from different mechanisms. Fig 1.3 indicates the different heat transfer mechanisms in porous media. The effective thermal conductivity depends on solid and

fluid thermal conductivity, geometry of the solid medium, contact resistance between particles and the amount of heat transfer by radiation. Note that convective heat transfer will be assumed negligible throughout this work. Convection in porous media rarely occur on a notable level, because the fluid pore size between the solid structure is simply too small. All particles applied in this work have a particle diameter << 1 cm, and therefore convection will be negligible (Tavman, 1996).

1. Heat transfer through solid by heat conduction.

2. Heat transfer through the fluid by conduction.

3. Conductive heat transfer through the contact surfaces of adjacent particles.

4. Heat transfer between surfaces due to radiation.

5. Convective heat transfer (solid/gas/solid).
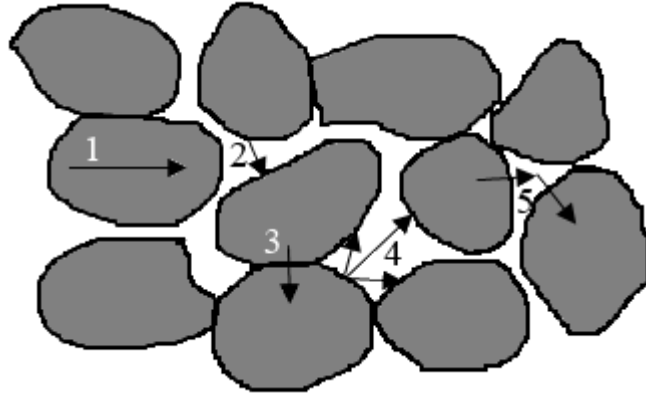
Fig. 1.3 - Heat transfer mechanisms in porous media.

As visualized in the above figure, all these processes may occur at the same time. The overall heat transported will then be a sum of the conductive heat transfer contribution both from the solid and the fluid. The radiation is expected to become increasingly significant as the temperature of the packed bed increases. In order to understand the process of heat transfer in porous media, which is a fairly complex issue, one must first begin to understand the general processes governing heat transfer.

### 1.2.2 Conduction

Conduction is spontaneous energy transfer from energetic particles of a medium to the adjacent less energetic ones (Cengel, 2006). These particles can be molecules, atoms, electrons or phonons. Conduction can occur in solids as well as fluids (gas or liquid). In fluids, the conduction occurs when particles collide and diffuses through the fluid during their random motion. In solids, it is due to the combination of lattice vibrations (phonons) of molecules and the energy transport by free electrons.

The magnitude of heat conduction is greater in solids than in fluids, because atoms are close together. Solids thereby allows for adjacent atoms to transfer energy to each other by lattice vibrations. In fluids, and especially gases, molecules are further away from each other, and this thus gives less conduction.

The rate of heat conduction depends on the geometry and the material of the medium, the temperature difference, as well as its thickness. This is governed by Fourier's law of heat conduction (Cengel, 2006), which yields

$$\dot{Q} = -kA_c \frac{dT}{dx}.$$ (1.2)

Here, *dT/dx* is the temperature gradient with respect to the x-direction, $A_c$ is the crossectional area, $\dot{Q}$ is the rate of conduction heat transfer, and $k$ is the thermal conductivity in W/(m·K).

The thermal conductivity is a thermophysical property, and the magnitude for different materials varies over a wide range. The thermal conductivity of a material depends on its state, chemical composition, and physical structure. It also varies with temperature and pressure. However, in most cases, thermal conductivity is much more dependent on temperature than on pressure. In that case, the pressure dependence can be neglected, and the thermal conductivity can be tabulated as a function of temperature only.
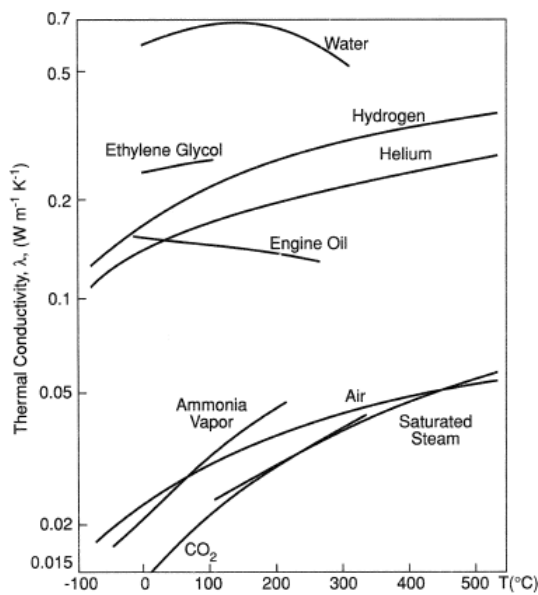


Fig. 1.4 - Thermal conductivity as a function of temperature for some fluid substances (Thermopedia.com).
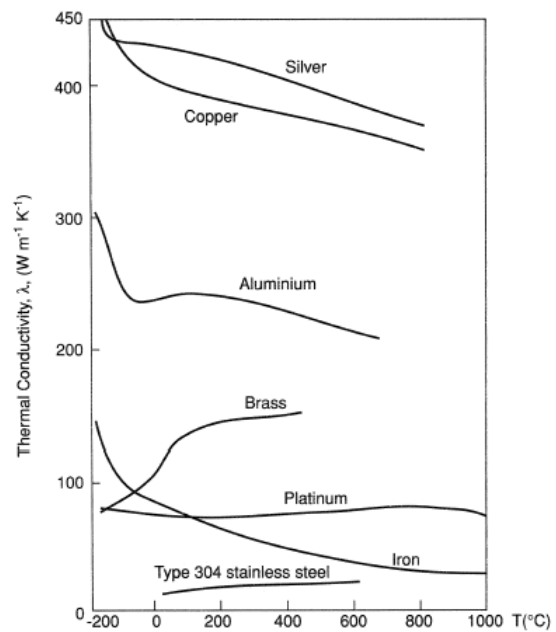
Fig. 1.5 - Thermal conductivity as a function of temperature for some solid substances (Thermopedia.com).

Fig. 1.4 and 1.5 shows the thermal conductivity as a function of temperature for various substances. As can be seen from the figure, fluids have a much lower thermal conductivity than solids.

This is the main challenge for hydrogen storage in porous materials today: Since a major part of a porous medium consists of a fluid, the thermal conductivity for the porous medium is low. Because of low thermal conductivity, porous media have difficulties transporting enough heat at a rate which allow for fast charge and discharge of a storage tank.

The physical meaning behind the solid thermal conductivity can be explained by kinetic theory. Kinetic theory gives the following expression (Incropera, 2007) for the thermal conductivity:

$$k = \frac{1}{3} C_{el} \bar{c} l_{mfp}, \tag{1.3}$$

where $C_{el}$ is the electron specific volumetric heat, $\bar{c}$ is mean molecular velocity, while $l_{mfp}$ is the mean free path between molecular collisions. As mentioned earlier, transport of thermal energy

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through a solid may be due to two effects: the migration of free electrons and lattice vibrational waves (phonons). For a given solid material, the thermal conductivity may therefore be expressed as a function of the electron conductivity $k_{el}$ (for conducting solids) and the phonon thermal conductivity $k_{ph}$ (for non-conducting solids), or $k = k_{el} + k_{ph}$.
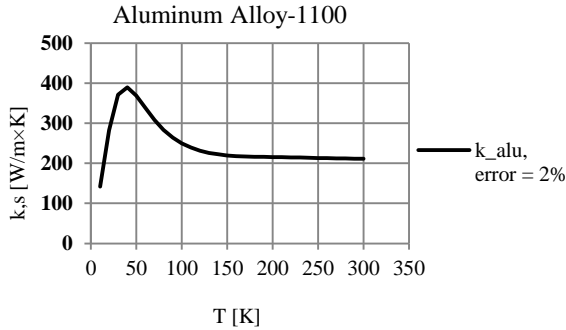


Fig. 1.6 - Thermal conductivity $k_s$ of Aluminum Alloy-1100 (NIST Chemistry Webbok).
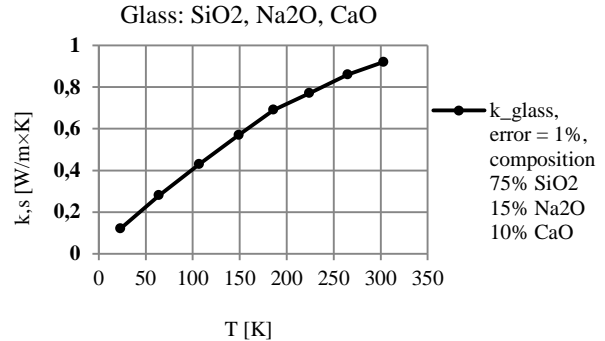
Fig. 1.7 - Solid thermal conductivity $k_s$ of glass (Handbook of glass data, 1987).

Figures 1.6 and 1.7 above displays plots of thermal conductivity versus temperature in the range of 4K<T<300K. Fig 1.6 is based on data from the NIST web-book. NIST apply an 8th degree polynomial relation in their calculations which has an uncertainty of 2%. The metal foam used in this work consists of Aluminum Alloy-1100, and as can be seen from Fig. 1.6, the solid conductivity has a grand peak at approximately 390 W/m·K and 40K before flattening out at around 215 W/m·K when 150K<T<300K. Fig. 1.7 shows the solid conductivity of a glass melt, whose composition is quite similar to the glass beads used for reference measurements in this work.

Heat conduction in gases and vapors mainly depend on the transfer of kinetic energy from the molecular movement. The kinetic gas theory states that the temperature of a gas element is proportional to the mean kinetic energy of its constituent molecules. This suggests that the thermal conductivity of a gas should depend on its temperature. According to a simple model of kinetic theory (traffic model), an approximated relation for gases can be (W. M. Rohsenow, 1961)

$$k = \frac{1}{3}\rho c_v \bar{c} l_{mfp}. \tag{1.4}$$

Here, $\rho$ is the gas density and $c_v$ is the specific heat capacity at constant volume. The mean molecular velocity $\bar{c}$ increases with increasing temperature and decreasing molecular weight. Hence, the thermal conductivity will also increase. The molecular weight of helium (He) is double the magnitude to that of hydrogen ($H_2$) and therefore the conductivity of hydrogen is higher. These trends are shown in Fig. 1.4. on the next page.

Air and N$_2$ at atmostpheric pressure

He and H$_2$ at atmostpheric pressure
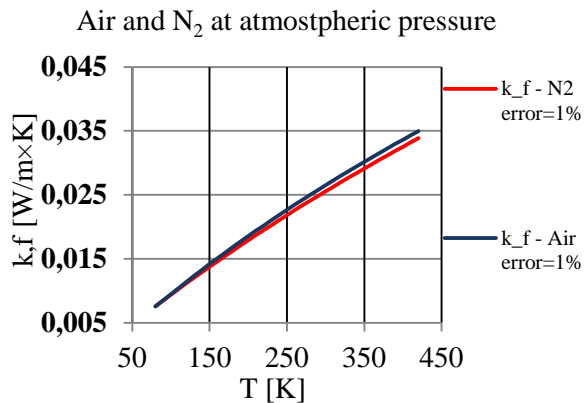


Fig. 1.8 - Conductivity of Air and N$_2$ as a function of temperature (VDI Heat Atlas, 2010).
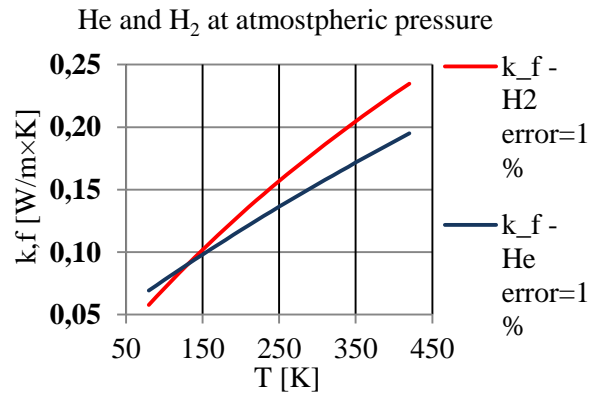
Fig. 1.9 - Conductivity of He and H$_2$ as a function of temperature (VDI Heat Atlas, 2010).

Fig. 1.8 and 1.9 show the fluid thermal conductivity of gases applied in this work. The plots are calculated from a 4$^{th}$ degree polynomial and have an uncertainty of 1%. From the figures it is clear that, at atmospheric pressure, the thermal conductivity of air and nitrogen at any given temperature between 80K and 420K are almost an order of magnitude less than helium and hydrogen thermal conductivity. Since the N$_2$ conductivity is almost equal to that of air, and He conductivity is almost equal to that of hydrogen, the measurements in this work were carried out with helium and nitrogen. Helium is non-flammable and a lot safer to handle than hydrogen (see the attached Risk Assessment Report).

### 1.2.3 Stefan Boltzmann law of radiation

Unlike conduction, where heat is transferred through a substance, energy transport can also take place without the presence of a physical medium. This process is known as thermal radiation and occurs when a substance emit electromagnetic waves. Electromagnetic waves are formed when accelerated charges or changing electric currents make an electric field in the medium (Cengel, 2006). Thermal radiation is emitted from a substance when energy in molecules, electrons and atoms is in transition. At a microscopic level, the measure of this process is temperature. Thus, thermal radiation will increase with increasing temperature. All substances are emitting electromagnetic waves; it can come from solid bodies as well as liquids and gases. All substances also possess the ability to absorb such energy.
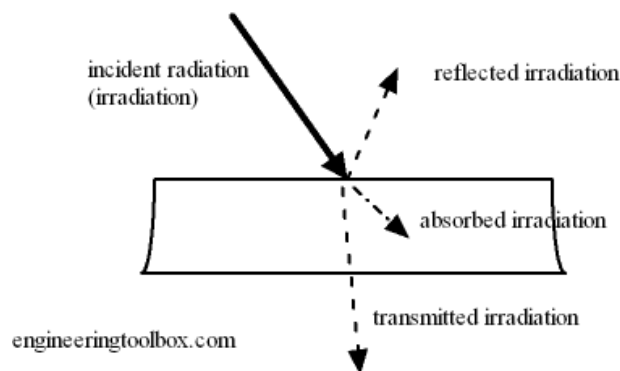


Fig. 1.10 - Incident radiation on a surface.

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As seen from Fig. 1.10, when radiation is incident on a body the surface will reflect part of it. The rest is absorbed within the body or even transmitted through it. When radiation impinges on a surface, the fraction that is reflected back is defined as the *reflectivity* $\chi$, the fraction that is absorbed is the *absorptivity* $\psi$, while the fraction transmitted is called the *transmissivity* $\omega$. This gives (Cengel, 2006):

$$\chi + \psi + \omega = 1. \tag{1.5}$$

A substance which has the ability to absorb a lot of radiation is called *opaque*. In opaque bodies, the energy that penetrates the body will be absorbed and stored within a very thin layer adjacent to the surface. For an opaque body, Eq. (1.5) then reduces to $\chi + \psi = 1$. In a *transparent* body, the material thickness required to substantially absorb radiation is large compared to the thickness of the body, and then most of the radiation will pass through without being absorbed. An ideal body, which absorbs all impinging radiation energy without reflection or transmission, is called *black body*. Hence, for a black body Eq. (1.5) reduces to $\psi = 1$.

A black body is defined as a perfect emitter and absorber of radiation (R. Siegel, 2002). The total emission of radiation per surface area and per unit time $E_{bb}''$ from a black body is related to the temperature $T$ of the surface through the Stephan Boltzmann law of radiation. The law yields

$$E_{bb}'' = \sigma T^4, \tag{1.6}$$

where $\sigma = 5.67 \cdot 10^{-8}$ [W/m$^2$K$^4$] is the Stephan Boltzmann constant.
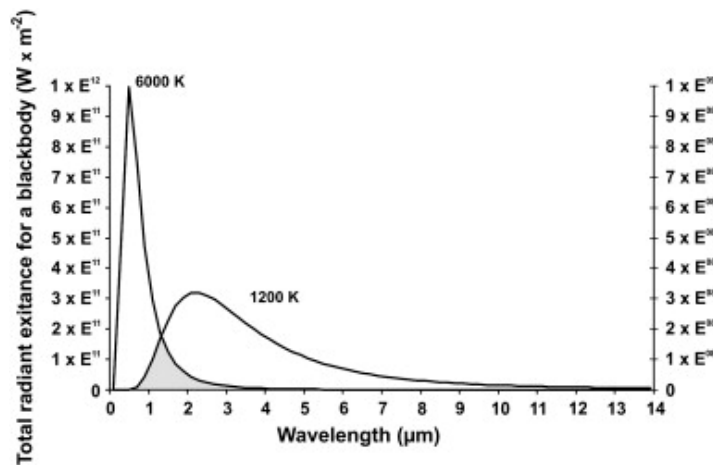


Fig. 1.11 - Black body exitance $E_{bb}''$ at various temperatures and wavelengths (L. Spampinato, 2011).

As can be seen from Fig. 1.11, the total radiant exitance (or emittance) from a surface will increase severely with temperature. Ordinary substances do not meet the specifications of a black body; they will emit a lower rate of radiation at the same given temperature. Thus, $E''$ being the exited radiation flux from an ordinary body per surface area and per unit time, the emissivity $\varepsilon_r$ of the surface will be

$$\varepsilon_r = \frac{E''}{\sigma T^4} = \frac{E''}{E_{bb}''}. \tag{1.7}$$

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Thus, for a black body, $\varepsilon_r = 1$. The emissivity of a material is defined as the ratio of the radiation emitted by the surface area at a given temperature to the radiation emitted by a black body of the same temperature. It will vary with temperature and wavelength and the direction of the emitted radiation. The emissivity of some common materials is given in Table 1.1 below.

Table 1.1 - Total emissivity of some common materials at given temperatures

| Material | Temperature [K] | Emissivity | Source |
|---|---|---|---|
| Aluminum Alloy-1100 | 366-700 | 0.05 | (Monarchserver.com) |
| Aluminum foil | 300 | 0.04 | (engineeringtoolbox.com) |
| Aluminum oxidized | 472 | 0.11 | (Monarchserver.com) |
| Aluminum unoxidized | 298-373 | 0.02-0.03 | (Monarchserver.com) |
| Glass | 293 | 0.94 | (Stephan Kabelac, 2010) |
| Glass, smooth | 273-473 | 0.95 | (Berkely Engineering Division) |
| Quartz glass | 300 | 0.93 | (engineeringtoolbox.com) |
| Steel oxidized | 300 | 0.79 | (engineeringtoolbox.com) |
| Steel polished | 300 | 0.07 | (engineeringtoolbox.com) |
| Styrofoam insulation | N/A | 0.6 | (Thermoworks.com) |

As seen from Table 1.1, the emissivity will increase when substances become more oxidized, and when temperature increases. The aluminum foam in this work consists of aluminum Alloy-1100 and clearly has a very low emissivity (0.05). Since the foam is cut and stored in ambient moist air in the lab, the edges will be somewhat oxidized, so an emissivity up until 0.1 could be closer to the real value. The glass beads one the other hand, is expected to have a very high emissivity, close to a black body (>0.9).

In order to understand the role that the effective thermal conductivity plays in porous media, one must take a closer look at the basic conduction heat transfer equations. The following section will address the basis for how the effective stagnant thermal conductivity can describe heat conduction in porous media.

## 1.3 Macroscopic transient heat conduction equations

As mentioned in section 1.1.2, heat transfer problems in porous media are most commonly approached macroscopically by averaging the microscopic heat transfer process over a representative elementary volume (REV). By assuming local thermal equilibrium within the fluid and solid phases one can describe the heat transfer process in two-face media by a single conduction equation. Then the problem comes down to construction of a correct correlation for describing the effective stagnant thermal conductivity of the whole mixture.

Assume a pack of solid particles surrounded by a fluid, as described in Fig. 1.2. The particles are considered to be spherical and of uniform size with particle diameter $d_p$. By assuming that $d_p$ is much larger than the typical molecule size, one can treat the fluid and solid both as macroscopically continuous, thus the macroscopic transient heat conduction equations are (Hsu, 1999)

$$\rho_f c_{pf} \frac{\partial T_f}{\partial t} = \nabla \cdot \left( k_f \nabla T_f \right), \qquad (1.8)$$

for the fluid and

$$\rho_s c_{ps} \frac{\partial T_s}{\partial t} = \nabla \cdot (k_s \nabla T_s), \tag{1.9}$$

for the solid, respectively. In the above equations, the subscripts $f$ and $s$ refer to the fluid and the solid phase, $\rho$ is the material density, $k$ is the thermal conductivity, and $c_p$ is the specific heat capacity at constant pressure. All properties are assumed constant. Furthermore, on the interface between the fluid and solid phases $A_{fs}$ the temperatures $T_f$ and $T_s$ will be equal. Hence,

$$\boldsymbol{n}_{fs} \cdot k_f \nabla T_f = \boldsymbol{n}_{fs} \cdot k_s \nabla T_s \text{ on } A_{fs}, \tag{1.10}$$

where $\boldsymbol{n}_{fs}$ is the normal vector from fluid to solid. Solving equations (1.8) - (1.10) in detail when the number of particles is large requires extensive numerical modeling; therefore it is more practical to apply the volume averaging-method. Now introducing the REV of volume $V$ and with $W_i$ denoting any physical quantity under consideration, the procedure of volume averaging can then be defined as

$$\overline{W}_j = \frac{1}{V_j} \iiint_{V_j} W_j dV. \tag{1.11}$$

Here, $V_j$ is the volume of the j-phase (thus either fluid or solid). The phase-averaged quantity $W_j$ can be considered continuous by assuming that the volume of the REV is much larger than the volume of a single particle yet much smaller than the entire flow domain volume. Defining $V=V_s+V_f$ then gives the opportunity to define the porosity as $\varepsilon=V_f/V$. Then, by averaging equations (1.8) and (1.9) over the REV and applying the divergence theorem, the volumetric phase averaged equations are (Hsu, 1999)

$$\rho_f c_{pf} \frac{\partial \varepsilon \overline{T}_f}{\partial t} = \overline{\nabla} \cdot \left[ k_f \overline{\nabla}(\varepsilon \overline{T}_f) \right] + \overline{\nabla} \cdot \left( k_f \overline{A}_{fs} \right) + Q_{fs}''', \tag{1.12}$$

and

$$\rho_s c_{ps} \frac{\partial [(1-\varepsilon)\overline{T}_s]}{\partial t} = \overline{\nabla} \cdot \{ k_s \overline{\nabla}[(1-\varepsilon)]\overline{T}_s \} - \overline{\nabla} \cdot \left( k_s \overline{A}_{fs} \right) - Q_{fs}''', \tag{1.13}$$

where $\overline{\nabla}$ is the gradient in the macroscopic coordinate system. In equations (1.12) and (1.13), the contribution due to thermal tortuosity is

$$\overline{\boldsymbol{A}}_{fs} = \frac{1}{V} \iint_{A_{fs}} T_f d\boldsymbol{s} = \frac{1}{V} \iint_{A_{fs}} T_s d\boldsymbol{s}, \tag{1.14}$$

and the interfacial heat transfer contribution $Q_{fs}'''$ is

$$Q_{fs}''' = \frac{1}{V} \iint_{A_{fs}} k_f \nabla T_f d\boldsymbol{s} = \frac{1}{V} \iint_{A_{fs}} k_s \nabla T_s d\boldsymbol{s} \tag{1.15}$$

with $d\boldsymbol{s}=\boldsymbol{n}_{fs} dA_{fs}$ as described in Eq. (1.10). The term on the left hand side of Eq. (1.12) and (1.13) represents the rate of thermal energy stored in the REV. On the right hand side, the first term represents the conductive heat transfer rate entering the fluid (1.12) and solid (1.13) phase through the REV boundary. The second term associates with the thermal tortuosity effect – an elongation in the thermal path travel due to the presence of solid particles. Note that magnitude of the tortuosity term in (1.12) differs from that in (1.13), due to the fact that the thermal conductivities of solid and fluid are

different. However, the magnitude of the interfacial heat transfer is the same. (This is obvious, since one can only assume that all heat transferred from the solid to the fluid is also gained in the fluid, and vice versa). Finally, the last term represents the heat transfer rate between the fluid and solid phases. The last to terms in both equations (1.12) and (1.13) have opposite signs because the source terms in (1.12) have to be sink terms in (1.13).

The assumption of having local thermal equilibrium has been proven not to be mathematically valid (Hsu, 1999), however, for any practical purposes it will suffice. Then, by assuming local thermal equilibrium, the local temperatures

$$\bar{T}_f = \bar{T}_s = \bar{\bar{T}}. \tag{1.16}$$

Inserting Eq. (1.16) into the phase averaged equations (1.12) and (1.13) then gives the transient heat conduction equation for stagnant porous media (Hsu, 1999)

$$\left(\rho_f c_{pf} + \rho_s c_{ps}\right)\frac{\partial \bar{T}}{\partial t} = \bar{\nabla} \cdot [k_e \bar{\nabla} \bar{T}], \tag{1.17}$$

where $k_e$ is the effective stagnant thermal conductivity of the mixture given by

$$k_e = \varepsilon k_f + (1 - \varepsilon)k_s + (1 - K)^2 \eta. \tag{1.18}$$

In (1.18), $K$ is the solid- to fluid thermal conductivity ratio, while $\eta$ is the thermal tortuosity parameter. The effective thermal conductivity then only depends on the interfacial geometry and the thermal properties of solid and fluid. Solutions to Eq. (1.17) has been extensively presented and debated in open literature. The problem then comes down to obtaining the correct value for the effective stagnant thermal conductivity.

## 1.4 Effective stagnant thermal conductivity of granular porous materials

Determining the effective stagnant thermal conductivity has been examined thoroughly for more than a century (Maxwell, 1873). Early experiments for measuring the effective thermal conductivity have been carried out in the late 60s (Kunii and Smith, 1960) (Krupiczka, 1967) along with theoretical predictive models. Most of these measurements were carried out in the range of $1<K<10^3$. Later, experiments with higher (Nozad et al, 1985) and lower (Prasad et al, 1989) values of $K$ were published. The predictive models for determining $k_e$ have been reviewed by Tsotsas (1987), and more recent by van Antwerpen et al. (2010). Over the years, several analytical composite-layer models have been proposed for $k_e$ (Kunii and Smith, 1960) (Zehner and Schlünder, 1970). Recently, Hsu et al. (1994) extended the Zehner-Schlünder model for spherical particles by introducing a particle touching parameter. Newer experiments on open-cell materials like metal foams were conducted by Boomsma and Poulikakos (2001).

According to the simpler models for effective stagnant thermal conductivity, $k_e$ depends on the geometry of the porous medium and the thermal conductivity of the fluid and solid phases. The overall conductivity can consequently be described as (VDI Heat Atlas, 2010)

$$k_e = k_e\left(\varepsilon, k_f, k_s\right) \tag{1.19}$$

Parameters listed above are so-called primary parameters, and most predictive models depend only on these three main parameters. However, as previously described, other parameters might also need to be taken into account, depending on the physical state of the medium (Tsotsas, 1987):

1. According to the kinetic gas theory, the thermal conductivity of unconfined, dilute gas is pressure-independent. However, with the presence of a solid medium the thermal conductivity of gas will decrease with decreasing pressure.
2. Chances of radiation heat transfer occurring increases with increasing temperature. This means that the effective thermal conductivity becomes a function also of optical properties (like emissivity $\varepsilon_r$), the relation between void and particles (which corresponds to the particle diameter $d_p$) of the media involved, as well as their temperature $T$.
3. Particles subjected to a force $f$ as a result of the pressure caused by their own weight pressing down on each other, will lead to an increase in heat transfer between particles since the contact area of the particles are enlarged (larger contact area means that the solid conductivity $k_s > k_f$ will transfer more heat between particles if $K > 1$). Deformation of particles depends mainly on the mechanical properties of the solid and the morphology of particle surface.
4. Heat transfer by free convection will occur in very large voids, and with sufficient solid-fluid temperature difference.

In experiments conducted in this work the pressure is maintained constant, it is therefore reasonable to state that one can neglect the pressure dependence when processing these data. As stated in section 1.2, convective heat transfer can be neglected. For this study, points 1 and 4 are therefore irrelevant. However, adding the secondary parameters to the list given in Eq. (1.19) gives the following functional dependence:

$$k_e = k_e \left( \begin{array}{c} \varepsilon, k_f, k_s, T, \varepsilon_r, d_p, \\ particle\ shape\ and\ size\ distribution, \\ particle\ mechanical\ and\ optical\ properties, \\ fluid\ thermodynamic\ and\ optical\ properties, \\ flattening\ of\ contact\ points \end{array} \right) \qquad (1.20)$$

### 1.4.1 Limiting relations

Predictive models need to describe the functional relationship in Eq. (1.19) or (1.20) in a general way, and in order to result in the most accurate solution they should take into account the primary parameters and as many of the secondary ones as possible. A good model should also be mathematically concise and be valid for a large range of both fluid and solid substances. It should also possess some flexibility by allowing for parameter-fitting regarding shape and size, and if possible be easy to apply. Furthermore, taking into account the primary parameters from Eq. (1.19), it should fulfill the following limiting physical relations (Shcumann, 1934):

1. $\varepsilon = 0$     $\Rightarrow$     $k_e = k_s$; the system consists of solid only.
2. $\varepsilon = 1$     $\Rightarrow$     $k_e = k_f$; the system consists of fluid only.
3. $k_s = k_f$     $\Rightarrow$     $k_e = k_s = k_f$; all phases have the same thermal conductivity.
4. $k_f \rightarrow \infty$     $\Rightarrow$     $k_e \rightarrow \infty$; particles can be bypassed for an infinitely good conducting

continuous phase.

5.  $k_s \rightarrow \infty$  $\Rightarrow$  $k_e \rightarrow \infty$; if particles touch each other.
6.  $k_f \rightarrow 0$  $\Rightarrow$  $k_e \rightarrow 0$; valid if particles touch each other, or has point contact.
7.  $k_s \rightarrow 0$  $\Rightarrow$  $k_e / k_f = \delta_e / \delta$; the reduced thermal conductivity of a mixture of non-conducting particles should be equal to the effective diffusion coefficient $\delta_e$ divided by the molecular diffusivity $\delta$.

Most of the above limitations are evident and do not require further explanation. However, regarding limiting relation 7, the conductivity of a substance containing a non-conducting dispersed phase is not actually *known*, though it is a definite function of its structure and of the conductivity of the continuous phase (Tsotsas, 1987).

### 1.4.2 Types of models

In order to keep track of all the models determining the effective thermal conductivity in porous media, one might feel the need to classify them on the basis of from which method they were obtained. Almost all models predicting the thermal conductivity of packed beds can then be divided into three different categories (Tsotsas, 1987):

Type I) - In type one models, the Laplace equation for heat conduction is solved analytically or numerically in order make an exact computation of the temperature field in and around the solid substance.

Type II) - Second type models are represented by thermal resistances for conduction in the solid and fluid phases. The limiting relations are also represented by the limiting theoretical correlations *parallel* and *series* arrangement, which are the simplest forms of the thermal resistance type models.

Type III) - Employing a unit cell as a basis for calculation, type three models sets the thermal conductivity in the unit cell equal to the conductivity of the whole mixture. This implies that the specific unit cell must be representative for an average of all the other unit cells in the mixture. If it is indeed so, these types of models give a very precise prediction of the effective thermal conductivity. In order to ensure easy calculation, either parallel heat flux lines (type IIIa) or parallel isotherms (type IIIb) are assumed.
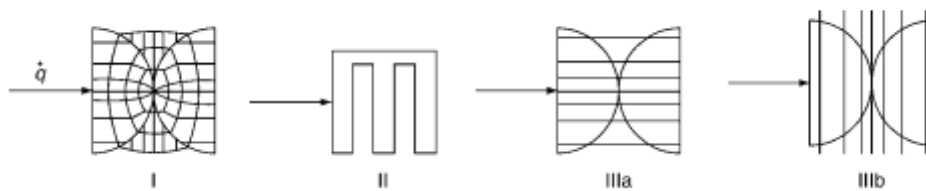


Fig. 1.12 - Types of models used to predict the thermal conductivity of packed beds. Type I: Exact analytical or numerical computation of the temperature field. Type II: Combination of resistances. Type IIIa: Unit cell with parallel heat flux lines. Type IIIb: Unit cell with parallel isotherms.

The above figure visualizes the three model types. For comparison purposes, Table 1.2 lists a selection of models along with their limiting relations and the secondary parameters they include. Regarding the limiting relation 7 (non-conducting solid particles), the value of $k_e / k_f$ is given. It is calculated with $\varepsilon = 0.4$ and $k_f = 0.25$ at 273K and atmospheric pressure. Values for limiting relation 7 for the Bauer and

Schlünder and IAEA-TECDOC models are from this work. Values for other models are taken from Tsotsaas (1987).

Table 1.2 - List of effective thermal conductivity models and their corresponding limiting relations.

| Author | Year | Type | Limiting relations | | | | | | | Secondary parameters | | | | | Source |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | Press. | Rad. | Form | Distr. | Cont. | |
| Maxwell | 1873 | Ia | Y | Y | Y | Y | N | Y | 0.308 | N | N | N | N | N | Tsotsaas |
| Kuuni and Smith | 1960 | IIIa | N | Y | Y | Y | Y | Y | 0.400 | N | Y | N | N | Y | Tsotsaas |
| Krupiczka | 1967 | Ia | N | N | Y | Y | Y | Y | 0.0 | N | N | N | N | N | Tsotsaas |
| Zehner and Schlünder | 1970 | IIIa | Y | Y | Y | Y | Y | Y | 0.225 | N | N | Y | Y | N | Tsotsaas |
| Bauer and Schlünder | 1978 | IIIa | Y | Y | Y | Y | Y | Y | 0.294 | Y | Y | Y | Y | Y | This work |
| IAEA-TECDOC-1163 | 2000 | IIIa | Y | Y | Y | Y | Y | Y | 0.250 | N | Y | Y | Y | Y | This work |

Y = Yes, N = No. Cont.=Contact area heat transfer (not to be mistaken for point contact heat transfer)

The model of Krupiczka has been shown to give good estimations in fluidized beds when $K<1$ (Prasad et al, 1989). The Zehner and Schlünder model tends to underestimate the effective thermal conductivity because it does not take into account the possibility of enlarged contact area between particles. This is however fixed in the upgraded Bauer-Schlünder model. This fix also accounts for radiation. This model is more commonly known as the Zehner-Bauer-Schlünder (hereby ZBS) model. Also, note that the ZBS model is not valid in the near-wall region, thus one need to ensure that the bulk is sufficiently large compared to the particle size when applying this model. Both the model proposed by the IAEA-TECDOC-1163 and the ZBS model have been proven to correlate well with experimental data (van Antwerpen et al, 2010).

As one can see from the Table 1.2, the type III models clearly provide a larger spectrum of applicability. The Models of type I and II will therefore be discussed briefly, while the type III models will be given a more detailed review.

## 1.5 Models based on the solution of the Laplace conduction equation (Type I)

The oldest and simplest analytical solution has been provided by Maxwell in 1873. This solution is

$$\frac{k_e}{k_f} = \frac{2\varepsilon + K(3 - 2\varepsilon)}{3 - \varepsilon + K\varepsilon}. \tag{1.21}$$

The Maxwell equation is valid for $\varepsilon \to 1$, because of the assumption that the solid particles are so far apart that they do not have contact with each other. In Eq. (1.21), $K$ denotes the solid to fluid conductivity ratio $k_s/k_f$. Since most granular porous media usually have porosities ranging from 0.3 to 0.5, this model may not be particularly applicable for this work.

Another model of type one is the Krupiczka (1967) correlation. He solved a set of two-dimensional heat conduction equations with no temperature-drop at the solid-fluid interface. He then applied these results in a spherical lattice and came up with the following model for stagnant thermal conductivity:

$$\frac{k_e}{k_f} = \lambda^{-a}, \tag{1.22}$$

with

$$a = 0.28 - 0.757 \log_{10}(\varepsilon) + 0.057 \log_{10} \lambda, \tag{1.23}$$

where $\lambda = k_f/k_s$ is the fluid to solid conductivity ratio. Prasad et al. (1989) reviewed this correlation for liquid fluid substances and found that it gave good results.

## 1.6 Thermal resistance models (Type II)

With the solid-fluid conductivity ratio $K=k_s/k_f$, the parallel and series arrangement resistance models yields:

$$\frac{k_e}{k_f} = \varepsilon + (1 - \varepsilon)K, \tag{1.24}$$

and

$$\frac{k_e}{k_f} = \frac{1}{\varepsilon + (1 - \varepsilon)/K}, \tag{1.25}$$

respectively. The maximum effective thermal conductivity for a two-phase system is given by a unidirectional heat flow through parallel layers of solid and fluid phases, while the minimum is given by a series arrangement (Dressler and Boegli, 1958). The two models from Eq. (1.24) and (1.25) act as the boundaries for the other thermal conductivity models.

## 1.7 Unit cell models (Type III)

Unit cell models seem to be the most applicable ones, and they also take in many of the secondary parameters from Eq. 1.18. The model of Kunii and Smith was obtained in 1960. They obtained the model by discretizing the solid and fluid phase into different modes acting in series and parallel.

### 1.7.1 Model of Kunii and Smith (1960)

Table 1.3 shows the limits for this model. Note that particle form and distribution is not taken into account. Hence, the model is only applicable for beds of mono-sized spherical particles.

Table 1.3 - Limiting relations and secondary parameters for Kunii and Smith's model.

| Author | Year | Type | Limiting relations | | | | | | | Secondary parameters | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | Press. | Rad. | Form | Distr. | Cont. |
| Kuuni and Smith | 1960 | IIIa | N | Y | Y | Y | Y | Y | 0.400 | N | Y | N | N | Y |
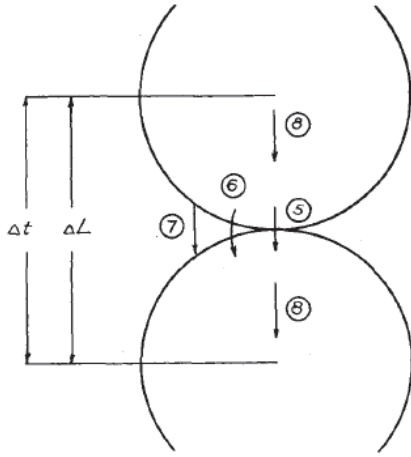
Fig. 1.13 - Kunii and Smith's heat transfer model for a packed bed of spherical particles.
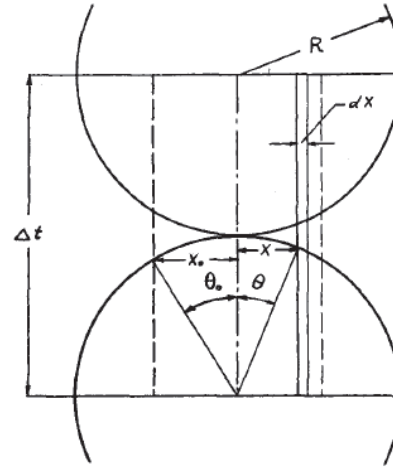
Fig. 1.14 - Model for heat transfer near particle contact points (Kunii and Smith, 1960). Note that they use $t$ for temperature instead of $T$ (this work).

In figures and above are Kunii and Smith's general model for heat transfer in a packed bed (Fig. 1.13) and their model for heat transfer near particle contact points (Fig. 1.14). They presume that the heat transfer occurs in the vertical direction by the following mechanisms:

1. Heat transfer through the fluid in the void space by conduction and by radiation between adjacent voids.
2. Heat transfer through solid:
   a. Conduction through contact area between adjacent particles.
   b. Conduction through the stagnant fluid near the contact surface.
   c. Radiation heat transfer between solid surfaces.
   d. Conduction through the solid phase.

According to figure 1.13, they then state that if the length between the center of the two spheres is $\Delta L$, then the effective thermal conductivity is given by $-k_e(\Delta T)/(\Delta L)=$ *[heat flux in void space]+[heat flux through solid]=[mechanism 1]+[mechanism 2]*. This yield

$$-k_e \frac{\Delta T}{\Delta L} = -k_f \varepsilon \frac{\Delta T}{\Delta L} + h_{rs} \cdot \varepsilon \cdot (-\Delta T) + \dot{Q}_s'', \tag{1.26}$$

where $h_{rs}$ is the thermal radiation heat transfer coefficient between solid surfaces and $\dot{Q}_s''$ is the heat flux rate through the solid per surface area. They then assume that the temperature drop in a particle equals the sum of the temperature drop in the solid phase and the temperature near the contact surface, or

$$\Delta T = \Delta T_s + \Delta T_{fs}. \tag{1.27}$$

The temperature drops $\Delta T_s$ in solid and $\Delta T_{fs}$ near the contact surface may be written in terms of

$$\Delta T_s = -\frac{\dot{Q}_s''}{(k_s/l_s)(1-\varepsilon)}, \tag{1.28}$$

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and

$$\Delta T_{fs} = -\frac{\dot{Q}_s''}{\left(h_c + h_{rs} + k_f/l_f\right)(1-\varepsilon)}. \tag{1.29}$$

In the above equations (1.28) and (1.29) the thickness of a slab of material which would give the same heat resistance as a spherically shaped particle is denoted $l_s$, while $l_f$ is the thickness of a slab of stationary fluid which would offer the same resistance as the filaments of fluid near the particle contact points. In Eq. (1.29), $h_c$ is the heat transfer coefficient representing the heat transfer through the contact area of adjacent particles. Combining Eq. (1.26) through (1.29) and then dividing by the fluid conductivity $k_f$ and inserting the quantities $\beta=\Delta L/d_p$, $\phi=l_f/d_p$ and $\gamma=l_s/d_p$ gives the expression

$$\frac{k_e}{k_f} = \varepsilon\left(1 + \beta\frac{h_{rf} \cdot d_p}{k_f}\right) + \frac{\beta(1-\varepsilon)}{\left[\dfrac{1}{(1/\phi) + (d_p/k_f)(h_c + h_{rs})} + \dfrac{\gamma}{K}\right]}, \tag{1.30}$$

where $h_{rf}$ and $h_{rs}$ is the radiation heat transfer coefficient in the void space (fluid) and solid, respectively. The numerical values of $\beta$, $\phi$, and $\gamma$ will be discussed further down. By neglecting the heat transfer through contact area $h_c$ (a valid assumption for every use except extremely low pressures (Kunii and Smith, 1960)), the above equation can also be written as

$$\frac{k_e}{k_f} = \varepsilon\left(1 + \beta\frac{(2-\varepsilon_r)}{\varepsilon_r + \dfrac{\varepsilon(1-\varepsilon_r)}{2(1-\varepsilon)}}Nu_f\right) + \frac{\beta(1-\varepsilon)}{\left[\dfrac{1}{(1/\phi) + Nu_f} + \dfrac{\gamma}{K}\right]}, \tag{1.31}$$

with the radiation Nusselt number (Vortmeyer, 1980)

$$Nu_f = \left[4\sigma T^3 \cdot \frac{\varepsilon_r}{(2-\varepsilon_r)}\right] \cdot \frac{d_p}{k_f} = h_{rs}\frac{d_p}{k_f} \tag{1.32}$$

By combining Eq. (1.30) through (1.32) one can obtain the relations for the radiation heat transfer coefficients

$$h_{rf} = \frac{4\sigma T^3}{\left[1 + \dfrac{\varepsilon}{2(1+\varepsilon)} \cdot \dfrac{(1-\varepsilon_r)}{\varepsilon_r}\right]}, \tag{1.33}$$

and

$$h_{rs} = 4\sigma T^3 \cdot \frac{\varepsilon_r}{(2-\varepsilon_r)}. \tag{1.34}$$

Now, one need to find the numerical values of the quantities $\beta$, $\phi$, and $\gamma$. Fig. 1.15 and 1.16 below visualizes Kunii and Smith's model for heat transfer directions through loose- and close-packed beds.

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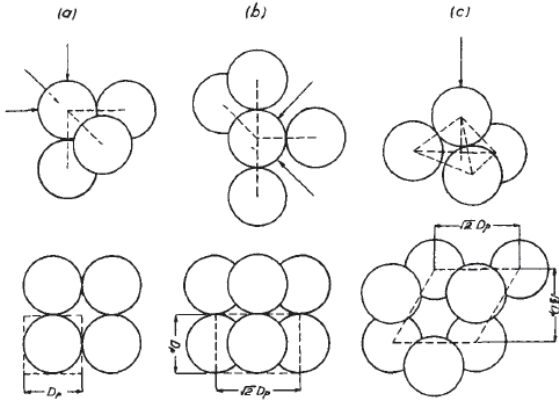
Fig. 1.15 - Heat transfer directions for loose packing of spheres (Kunii and Smith, 1960).
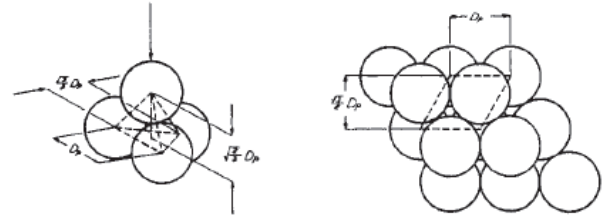
Fig. 1.16 - Heat transfer directions for close packing of spheres (Kunii and Smith, 1960).

The value of $\beta$ for close spherical packing as shown in Fig. 1.16 above is then

$$\beta = \frac{\Delta L}{d_p} = \frac{1}{d_p} \cdot \frac{1}{3}\left[\left(\frac{2}{9}\right)^{\frac{1}{2}} + 1 + \left(\frac{3}{2}\right)^{\frac{1}{2}}\right] d_p = 0.895. \tag{1.35}$$

For the loosest packing the value of $\beta$ should be unity, since the spheres are very far apart from one another. Hence, for loose packing, $\beta=1$. The value of $\gamma$ depends upon $l_s$, and Kunii and Smith assume that it is a length of a cylinder having the same size as a spherical particle. Hence,

$$\gamma = \frac{l_s}{d_p} = \frac{1}{d_p} \cdot \frac{\left[\left(\frac{\pi}{6}d_p{}^2\right)\right]}{\left[\left(\frac{\pi}{4}d_p{}^2\right)\right]} = \frac{2}{3}. \tag{1.36}$$

In order to obtain the $\phi$ value ($\phi=l_f/d_p$), they state that the fraction of the total heat flux through the fluid near one contact point is given by

$$\frac{1}{n} = \frac{\pi x_0^2}{\pi R^2} = \sin^2 \theta_0. \tag{1.37}$$

with the parameters explained in Fig. 1.14. With this in mind, the one can derive the following expression for the heat flux

$$Q'' = \int_0^{\theta_0} dQ'' = \pi R k_f \Delta T \left(-\frac{K}{K-1}\right)^2 \left\{\ln[K - (K-1)\cos\theta_0] - \frac{K-1}{K}(1 - \cos\theta_0)\right\}. \tag{1.38}$$

Equation (1.38) includes heat transfer through solid and filaments of fluid in series, thus the heat flux can also be described through the resistance of two steps:

$$Q'' = \frac{\pi (R\sin\theta_0)^2 \Delta T}{\dfrac{2R\left(\frac{2}{3}\right)}{k_s} + \dfrac{l_f}{k_f}}, \tag{1.39}$$

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where $2R(2/3)=l_s$. Since $l_f=d_p\,\phi$ and $d_p=2R$, combining equations (1.38) and (1.39) gives

$$\phi_{tight\ or\ loose} = \frac{1}{2} \cdot \frac{\left(\frac{K-1}{K}\right)^2 \sin^2 \theta_0}{\ln[K-(K-1)\cos\theta_0] - \left(\frac{K-1}{K}\right)(1-\cos\theta_0)} - \frac{2}{3K}. \tag{1.40}$$

The above equation, however only applies for one single contact point. They argue (based on Fig. 1.15) that the number of contact points can be assumed ass stated in Table 1.4 below.

Table 1.4 - Particle orientation (Kunii and Smith, 1960).

| Orientation (ref. Fig. 1.15) | a) | b) | c) |
|---|---|---|---|
| Contact point | 1 | 2 | 3 |
| Area corresponding to one particle | $2d_p^2$ | $2^{1/2}d_p^2$ | $3^{1/2}d_p^2$ |
| Number of directions | 3 | 2 | 4 |

The cases of the loosest and tightest packing available should then be considered. As shown in Fig. 1.14, the derivation of Eq. (140) is based upon case a). Therefore, the approximate average number of contact points $n_{loose} = (n_a + n_b + n_c)$ for a loose bed of spherical particles should be

$$n_{loose} = \frac{1}{3}\left[1 + \left(2 \cdot \frac{d_p^2}{\sqrt{2}d_p^2} \cdot \frac{2}{3}\right) + \left(3 \cdot \frac{d_p^2}{\sqrt{3}d_p^2} \cdot \frac{4}{3}\right)\right] \approx 1.5. \tag{1.41}$$

The equivalent number of contact points for tight packing is then obtained in the same manner (ref. Fig. 1.16):

$$n_{tight} = \left(6 \cdot \frac{d_p^2}{\sqrt{3}/2 \cdot d_p^2}\right) \approx 4\sqrt{3}. \tag{1.42}$$

The most dense packing available is $\varepsilon=0.26$ and the most loose is $\varepsilon=0.476$ (Kaviany, 1995). Kunii and Smith (1960) argue that a packed bed can be considered a composite of these two extremes. They then approximate $\phi$ as

$$\phi = \phi_{tight} + \left(\phi_{loose} + \phi_{tight}\right)\frac{\varepsilon - 0.26}{0.476 - 0.26}; 0.26 < \varepsilon < 0.476. \tag{1.43}$$

To sum up the effective thermal conductivity model by Kunii and Smith (1960), the equations and parameters needed are given in Table 1.5 below.

Table 1.5 - Kunii and Smith model summarized

| | |
|---|---|
| Effective thermal conductivity $k_e$ | Eq. (1.30) |
| Radiation heat transfer $h_{rf}$ and $h_{rs}$ | Eq. (1.33) and (1.34) |
| Point contact $\phi$ | Eq. (1.43) and (1.40) |
| Contact area (flattening) $h_c$ | N/A |
| | |
| Packing structure | Loose     Dense |

| | | |
|---|---|---|
| $\beta$ | 1 | 0.895 |
| $\gamma$ | 2/3 | 0.67 |
| $n$ | 1.5 | $4\sqrt{3}$ |

Note that the heat transfer $h_c$ due to flattening of particles (contact area) is neglected. The Kunii and Smith model should give good predictions of the effective thermal conductivity, but due to the neglecting of flattened contact area heat transfer, the values might be lower than the real case. Another popular type III model which takes this into consideration however, is the Zehner-Bauer-Schlünder model.

### 1.7.2 Model of Zehner, Bauer and Schlünder (1970-1978)

The model of Zehner and Schlünder (1970), which takes into consideration a unit cell of either perfectly spherically shaped or deformed particles, is widely used within literature and has been verified to give good estimations of the effective thermal conductivity for a wide range of solid-fluid conductivity ratios (Kandula, 2011). This model was later extended by improving the deformation factor for spheres (Hsu et al, 1994) and by adding heat transfer expressions related to contact area and radiation (Bauer and Schlünder, 1978). The combination of above mentioned models are more commonly known as the Zehner-Bauer-Schlünder (ZBS) model. As seen from Table 1.6 the model looks very promising; it takes in to account a lot of parameters, and is as previously mentioned valid for a large range of conductivity ratios.

Table 1.6 - Zehner/Bauer/Schlünder limiting relations and secondary parameters.

| Author | Year | Type | Limiting relations | | | | | | | Secondary parameters | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | Press. | Rad. | Form | Distr. | Cont. |
| Zenher/Bauer/Schlünder | 1970-1978 | IIIa | Y | Y | Y | Y | Y | Y | 0.294 | Y | Y | Y | Y | Y |
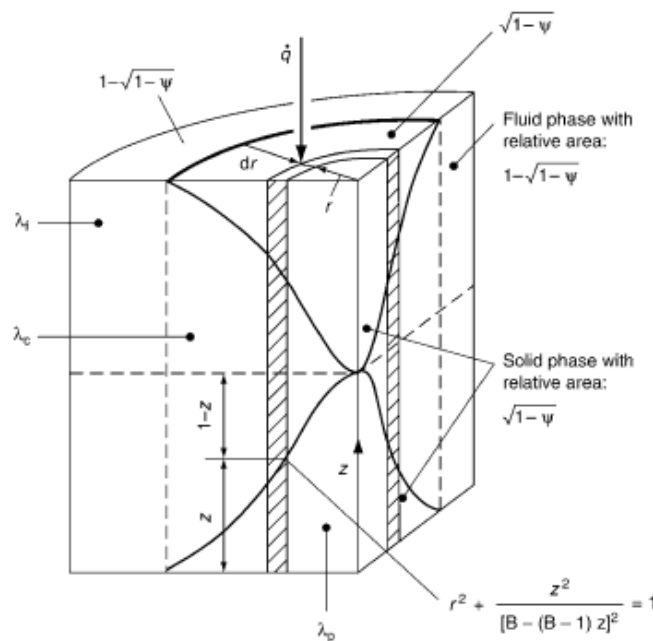


Fig. 1.17 - The unit cell from the ZBS model (VDI Heat Atlas, 2010).

Fig. 1.17 shows the unit cell as proposed by the ZBS model. Note that the notations from the figure differ from the notations used in this work. To avoid any misconception, the equivalent parameters are listed in Table 1.7 below.

Table 1.7 - Corresponding notations.

| Zehner/ Bauer /Schlünder | | This work |
|---|---|---|
| $\Psi$ | $\rightarrow$ | $\varepsilon$ |
| $\lambda_f$ | $\rightarrow$ | $k_f$ |
| $\lambda_p$ | $\rightarrow$ | $k_s$ |
| $\lambda_c$ | $\rightarrow$ | $k_c$ |
| $\dot{q}$ | $\rightarrow$ | $\dot{Q}_s''$ |

The main idea of this model is to repair the incorrect assumption of parallel heat flux lines by using fictitious non-spherical particles to simulate the thermal behavior of spherical ones. The main characteristic is thus the deformation factor $B$ which is modeled according to the equation (ref. Fig. 1.17) (Zehner and Schlünder, 1970)

$$r^2 = \frac{z^2}{[B-(B-1)z]^2},$$

(1.44)

With $r$ and $z$ as the radius and z-axis in the cylindrical coordinate system. For $B$=1, Eq. (1.44) describes the surface of a sphere. $B$<1 gives something like prolonged needles, and with $B$>1 one obtains cylinder-like bodies. The deformation factor gives the opportunity to change the width of the voids in the area around contact points. In this manner, the error caused by the assumption of parallel heat flux lines can be compensated for (to a certain extent). From Fig. 1.17, one can define the porosity of the unit cell as (Zehner and Schlünder, 1970)

$$\varepsilon = 1 - \frac{V_p}{V_{uc}}\sqrt{1-\varepsilon},$$

(1.45)

where $V_p$ is the volume of the deformed particle and $V_{uc}$ is the volume of the cylinder wrapped in the unit cell. Rearranging Eq. (1.45) gives

$$\varepsilon = 1 - \left(\frac{V_p}{V_{uc}}\right)^2.$$

(1.46)

It is then possible to compute $V_p/V_{uc}$ from Eq. (1.44) by a volume integral, thus giving the following relationship between porosity $\varepsilon$ and the deformation factor $B$:

$$\varepsilon = 1 - \left\{\frac{B}{(B-1)^3}[3 - 4B + B^2 + 2\ln B].\right\}^2.$$

(1.47)

$B$ can be explicitly computed from the porosity, and a good approximation of Eq. (1.46) is then given by (Zehner and Schlünder, 1970)

$$B = C\left(\frac{1-\varepsilon}{\varepsilon}\right)^m,$$

(1.48)

with $m=10/9$ and the shape-factor $C=1.25$. The parameters $C$ and and $m$ allows for fitting of particles other than spherical ones. They gave parameters for spheres, cylinders and broken particles from experimental data given in Table 1.8.

Table 1.8 - Shape factors

| Particle shape | C |
|---|---|
| Spheres | 1.25 |
| Broken particles | 1.4 |
| Cylinders | 2.5 |

It can be discussed whether Eq. (1.48) gives an accurate approximate for Eq. (1.47), and this was pointed out by Hsu et al. (1994). Based on a least square procedure the found that Eq. (1.48) with $C=1.364$ and $m=1.055$ resulted in a more accurate prediction for spherical particles.

That aside; with the deformation factor known, the conductivity of the core of the unit cell $k_c$ can be calculated from integrating over the parallel heat flux lines. By setting the conductivity of the whole mixture equal to that of the unit cell, one can thus obtain the formula for the ratio of the effective stagnant thermal conductivity to fluid conductivity (see Fig. 1.17)

$$\frac{k_e}{k_f} = 1 - \sqrt{1-\varepsilon} + \sqrt{1-\varepsilon}K_c \tag{1.49}$$

with $K_c = k_c/k_f$. The core-fluid conductivity ratio $K_c$ is then given by (Zehner and Schlünder, 1970)

$$K_c = \frac{2}{N}\left[\frac{B}{N^2} \cdot \frac{K-1}{K} \cdot \ln\left(\frac{K}{B}\right) - \frac{B+1}{2} - \frac{B-1}{N}\right], \tag{1.50}$$

where

$$N = \left(1 - \frac{B}{K}\right). \tag{1.51}$$

Equations (1.48) through (1.51) are together known as the Zehner and Schlünder model. It does not account for secondary parameters and is a function of only the primary parameters ($\varepsilon, k_s$ and $k_f$) and the deformation factor $B$. The given model was then extended, accounting for the secondary parameters presented in Eq. (1.20). The extension based on the figure of the unit cell (Fig. 1.17) is given as (Zehner and Schlünder, 1972); (Bauer and Schlünder, 1978):

$$\frac{k_e}{k_f} = \left(1 - \sqrt{1-\varepsilon}\right)\varepsilon\left[\frac{1}{\left(\varepsilon - 1 + \frac{1}{K_g}\right)} + K_r\right] + \sqrt{1-\varepsilon}[\varphi K + (1-\varphi)K_c], \tag{1.52}$$

with

$$K_c = \frac{2}{N} \left\{ \frac{B(K + K_r - 1)}{N^2 K_g K} \cdot \frac{K-1}{K} \cdot \ln \frac{K + K_r}{B \left[ K_g + (1 - K_g)(K + K_r) \right]} \right.$$

$$\left. + \frac{B+1}{2B} \left[ \frac{K_r}{K_g} - B \left( 1 + \frac{1 - K_g}{K_g} K_r \right) \right] - \frac{B-1}{NK_g} \right\},$$

(1.53)

$$N = \frac{1}{K_g} \left( 1 + \frac{K_r - BK_g}{K} \right) - B \left( \frac{1}{K_g} - 1 \right) \left( 1 + \frac{K_r}{K} \right),$$

(1.54)

and

$$B = C \left( \frac{1 - \varepsilon}{\varepsilon} \right)^{10/9} \cdot f(\zeta).$$

(1.55)

The contribution due to radiation is

$$K_r = \frac{k_r}{k_f} = \frac{4 \sigma T^3 d_p}{\left( \frac{2}{\varepsilon_r} - 1 \right)} \cdot \frac{1}{k_f},$$

(1.56)

while the gaseous conduction related to the Knudsen regime is given by:

$$K_g = \frac{k_g}{k_f} = \frac{1}{\left( 1 + l/d_p \right)}.$$

(1.57)

For beds packed with particles of various sizes, a mean particle size

$$\bar{d}_p = \left[ \sum_{i=1}^{n} \left( \frac{V_i}{V} \cdot \frac{1}{d_{pi}} \right) \right]^{-1},$$

(1.58)

should be used. In Eq. (1.58), $V_i/V$ is the volume of the $i$th particle size fraction $V_i$ referred to the total analyzed particle volume $V$. The letter $d_{pi}$ is the average particle diameter of fraction $i$. In the case of non-spherical particles, the diameter of a sphere of equal volume as the particle should be used (neglecting inner porosity), in place of $d_p$ and $d_{pj}$. The effective conductivity of thermal radiation $k_r$, or in dimensionless form $K_r$, is given by equation (1.56). This is a general Damköhler radiation term (Damköhler, 1937)

$$k_r = 4 \sigma F \bar{T}^3 x_r,$$

(1.59)

where $F$ is a function the optical properties of the surfaces in the mixture (in this case the emissivity $\varepsilon_r$ of the solid), $\sigma$ is the Stefan Boltzmann constant, $\bar{T}$ is the average bed temperature and $x_r$ is some characteristic dimension of the system (in this case the porosity $\varepsilon$ and particle diameter $d_p$). The temperature $\bar{T}$ is actually a result of an averaging assumption that $((T_1^4 - T_2^4)/(T_1 - T_2) \approx 4\bar{T}^3$, which is only valid as long as the steady state temperature drop $\Delta T$ across the unit cell is much smaller than the average bed temperature, i.e. $\Delta T \ll \bar{T}$.

The local version of Eq. (1.56) has been integrated over the unit cell, in order to account for the variation in distance between particles. Hence, this includes a correction for the thermal radiation exchange between adjacent particles. The section of the unit cell representing the void (fluid) is $1 - \sqrt{1 - \varepsilon}$. In order to give a true estimate of the radiation paths through the void, the particle diameter $d_p$ has been divided by the bed porosity $\varepsilon$ (Zehner and Schlünder, 1972).

Equation (1.57) denotes the pressure dependence. It relates the thermal conductivity of a gas-filled gap $k_g$ to the fluid conductivity $k_f$. In reduced form this gives $K_g = k_g/k_f$, hence, if the fluid is not a gas, $K_g \rightarrow 1$. In Eq. (1.57), $l/d_p$ is the Knudsen number, with $l$ being the modified mean free path of gas molecules. The gap size between particles is here represented by the particle diameter $d_p$. In order to account for the change in distance between particles, the local version Eq. (1.57) was integrated over the particle radius (Schlünder, 1981). The mean free path of the molecules $l$ is calculated from the following formula (Möllenkopf and Martin, 1982):

$$l = 2 \frac{2 - a_t}{a_t} \left( \frac{2\pi \tilde{R} T}{M_g} \right)^{1/2} \cdot \frac{k_f}{p\left(2C_{pf} - \tilde{R}/M_g\right)}, \tag{1.60}$$

where the thermal accommodation coefficient $a_t$, which is a measure of the effectiveness of energy transfer when gas molecules collide with the solid surface, is best given by (Song and Yovanovich, 1987)

$$a_t = exp\left[-0.57\left(\frac{T_s - T_0}{T_0}\right)\right]\left(\frac{M^*}{6.8 + M^*}\right) + \frac{2.4M}{(1 + M)^2}\left\{1 - exp\left[-0.57\left(\frac{T - T_0}{T_0}\right)\right]\right\}, \tag{1.61}$$

with

$$\begin{aligned} M^* &= \begin{cases} M_g \text{ for monoatomic gases} \\ 1.4M_g \text{ for diatomic gases} \end{cases} \\ M &= M_g/M_s \end{aligned} \tag{1.62}$$

In equations (1.60) through (1.62), $T_s$ is the temperature of the solid surface, $T_s = 273,15K$, $p$ is the pressure of the gas, $\tilde{R}$ is the universal gas constant, $Cp_g$ is the gas thermal heat capacity under constant pressure, while $M_g$ and $M_s$ is the molecular weight of gas and solid, respectively.

In order to describe the influence of particle shape, the deformation factor $B$ from Eq. (1.48) has been corrected with a distribution factor $f(\zeta)$ (see Eq. (1.55)). This distribution function is then

$$f(\zeta) = 1 + 3\zeta, \tag{1.63}$$

with the distribution parameter

$$\zeta = \left\{ \frac{\sum_{i=1}^{n}\left(\frac{V_i}{V} \cdot \frac{1}{d_{pi}^2}\right)}{\sum_{i=1}^{n}\left(\frac{V_i}{V} \cdot \frac{1}{d_{pi}}\right)^2} - 1 \right\}^{1/2}. \tag{1.64}$$

However, experimental investigations on beds packed with poly-dispersed media show that the thermal conductivity can be calculated accurately with a distribution function equal to unity (Tsotsas and Schlünder, 1991), i.e.

$$f(\zeta) = 1. \tag{1.65}$$

In Eq. (1.52), $\varphi$ is the flattening coefficient modeling the heat transfer through flattened contact areas between adjacent particles, and values for this are given in Table 1.9 below.

Table 1.9 - Flattening coefficients obtained by Zehner and Bauer (Schlünder, 1981).

| Particle shape | $\varphi$ | Material |
|---|---|---|
| Spheres | 0.0013 | Steel |
|  | 0.0077 | Ceramic |
|  | 0.0253 | Copper |
| Broken particles | 0.001 | |
| Cylinders | ? | |

Summing up the model of Zehner, Bauer and Schlünder, it takes into account particle distribution and shape, as well as heat transfer trough flattening of contact points. This shows promise regarding the experimental investigations of this work. This study will make use of both broken and cylindrically shaped particles as well as spheres (glass beads) and it is therefore expected that the ZBS model gives a very precise prediction of the effective thermal conductivity. A brief summary of the model is shown in Table 1.10 below.

Table 1.10 - ZBS model summarized

| | |
|---|---|
| Total effective thermal conductivity $k_e$ | Eq. (1.52) |
| Core thermal conductivity $K_c$ | Eq. (1.53) and (1.54) |
| Radiation heat transfer $K_r$ | Eq. (1.56) |
| Pressure dependence $K_g$ | Eq. (1.57), (1.60), (1.61) and (1.62) |
| Particle deformation factor $B$ | Eq. (1.55) |
| Particle diameter $d_p$ | Eq. (1.58) |
| Particle distribution $f(\zeta)$ | Eq. (1.65) |

| Particle shape and contact area (flattening) | C | $\varphi$ | Material |
|---|---|---|---|
| Spheres | 1.5 | 0.0013 | Steel |
|  |  | 0.0077 | Ceramic |
|  |  | 0.0253 | Copper |
| Broken | 1.4 | 0.001 | |
| Cylinders | 2.5 | | |

The ZBS model was, among other models, recently compared with a newer model (van Antwerpen et al, 2010) proposed by the IAEA-TECDOC-1163 (2000). This new model is based on the ZBS model and will be explained in the next section.

### 1.7.3 Model proposed by IAEA-TECDOC-1163 (2000)

The IAEA-TECDOC-1163 (2000) model utilizes the unit cell of the original Zehner and Schlünder (1970) model (Fig. 1.17; Eqs. (1.48)-(1.51)) and combines this with other models for contact area (Kaviany, 1995) and radiation (Breitbach and Barthels, 1980) heat transfer.

Table 1.11 - Limiting relations and secondary parameters for the IAEA-TECDOC-1163 model

| Author | Year | Type | Limiting relations | | | | | | | Secondary parameters | | | | |
|--------|------|------|---|---|---|---|---|---|---|---------|------|------|-------|-------|
|        |      |      | 1 | 2 | 3 | 4 | 5 | 6 | 7 | Press.  | Rad. | Form | Distr. | Cont. |
| IAEA-TECDOC-1163 | 2000 | IIIa | Y | Y | Y | Y | Y | Y | 0.250 | N | Y | Y | Y | Y |

Table 1.11 shows the parameter inputs of this model. Fulfilling every limiting relation and accounting for all secondary parameters except one shows that this might be a model applicable for various purposes. The dimensionless effective thermal conductivity ($k_e/k_f$) is given by the Zehner and Schlünder model from equations (1.48) through (1.51) with the following terms added (IAEA-TECDOC-1163, 2000):

The radiation heat transfer given by

$$K_r = \frac{k_r}{k_f} = \frac{4\sigma T^3 d_p}{k_f}\left\{\varepsilon \cdot \left[1 - \sqrt{1-\varepsilon}\right] + \frac{\sqrt{1-\varepsilon}}{\frac{2}{\varepsilon_r}-1}\cdot\frac{B+1}{B}\cdot\frac{1}{1+\frac{1}{(2/\varepsilon_r-1)\Lambda}}\right\}, \qquad (1.66)$$

where the dimensionless solid conductivity is $\Lambda = k_s/4\overline{T}^3 d_p$ and the heat transfer due to contact area flattening is

$$\frac{k_c}{k_f} = K\left[\frac{3(1-\mu_s^2)}{4E_s}fR\right]^{1/3}\cdot\frac{1}{0.531\cdot S}\left(\frac{N_A}{N_L}\right), \qquad (1.67)$$

with the external collinear force $f$ related to the external pressure through (Kaviany, 1995)

$$f = p\frac{S_F}{N_A}. \qquad (1.68)$$

Equations (1.67) and (1.68) were obtained on the basis of three different spherical arrangements (Table 1.12; Fig. 1.18), and are a function of the solid elastic properties Poisson ratio, $\mu_s$, and Young's modulus, $E_s$. $N_A$ and $N_L$ are the number of particles per unit area and length, respectively. $S_F$ is also given in Table 1.12.

Table 1.12 - Structural parameters for different packing of spheres (Kaviany, 1995).

| Parameter | Simple cubic | Body-centered cubic | Face-centered cubic |
|---|---|---|---|
| $\varepsilon$ | 0.476 | 0.32 | 0.26 |
| $N_L$ | $\dfrac{1}{2R}$ | $\dfrac{\sqrt{3}}{2R}$ | $\dfrac{\sqrt{3/8}}{R}$ |
| $N_A$ | $\dfrac{1}{4R^2}$ | $\dfrac{3}{16R^2}$ | $\dfrac{1}{2\sqrt{3}R^2}$ |
| $S$ | 1 | 0.25 | $\dfrac{1}{3}$ |
| $S_F$ | 1 | $\dfrac{\sqrt{3}}{4}$ | $\dfrac{1}{\sqrt{6}}$ |
| Number of contact points | 6 | 8 | 12 |
| Points used in the heat flow analysis | 2 | 8 | 6 |

a)                                  b)                                  c)



Simple cubic              Body-centered cubic              Face-centered cubic
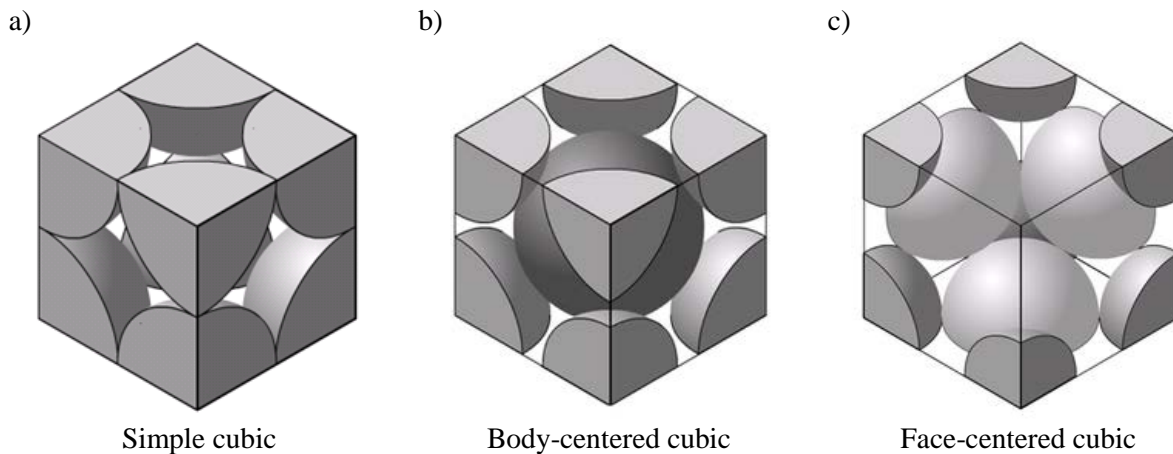
Fig. 1.18 - The three different packing arrangements applied for the derivation of the model.

The IAEA model should give good results when applied for spherical packing. If the contact area equation also manages to predict accurately with broken or cylindrical particles, it is expected that this model should perform well; it takes into account all secondary parameters from Eq. (1.20) except pressure dependence and distribution. For this study, pressure dependence is not really relevant, since all experiments are to be performed at constant pressure. The distribution function (1.65) can be set equal to unity, and as previously discussed in section 1.7.2, and the model will still perform well.

A summary of the IAEA-TECDOC-1163 model is given in Table 1.13.

Table 1.13 - IAEA-TECDOC-1163 model summarized

| | |
|---|---|
| Total effective thermal conductivity $k_e$ | Eq. (1.49) |
| Core thermal conductivity $K_c$ | Eq. (1.50) and (1.51) |
| Radiation heat transfer $K_r$ | Eq. (1.66) |
| Pressure dependence | N/A |
| Contact area heat transfer $k_c$ | Eq. (1.67)[A] and (1.68)[A] |
| Particle deformation factor $B$ | Eq. (1.48) |
| Particle diameter $d_p$ | Eq. (1.58) |
| | |
| Particle shape coefficients | C |
| Spheres | 1.25 |
| Broken | 1.4 |
| Cylinders | 2.5 |

[A]With structural parameters given in Table 1.12

All models may prove to give good results for different applications. The ZBS - and IAEA models are expected to perform well over a large range of fluid to conductivity ratios. The model by Kunii and Smith should also perform well, although it does not account for all secondary parameters. Non-radiation models such as those by Maxwell and Krupiczka may give good predictions when the temperature is sufficiently low and radiation heat transfer can be neglected. It is the goal of this work to generate as much experimental data as possible, in order to find a model that will accurately predict the effective thermal conductivity of various hydrogen adsorption substances filled with different gases. All previously described models are now compared together (Fig. 1.19) for a bed filled with spherical particles with a porosity $\varepsilon$ of 0.4 and air as the fluid medium.
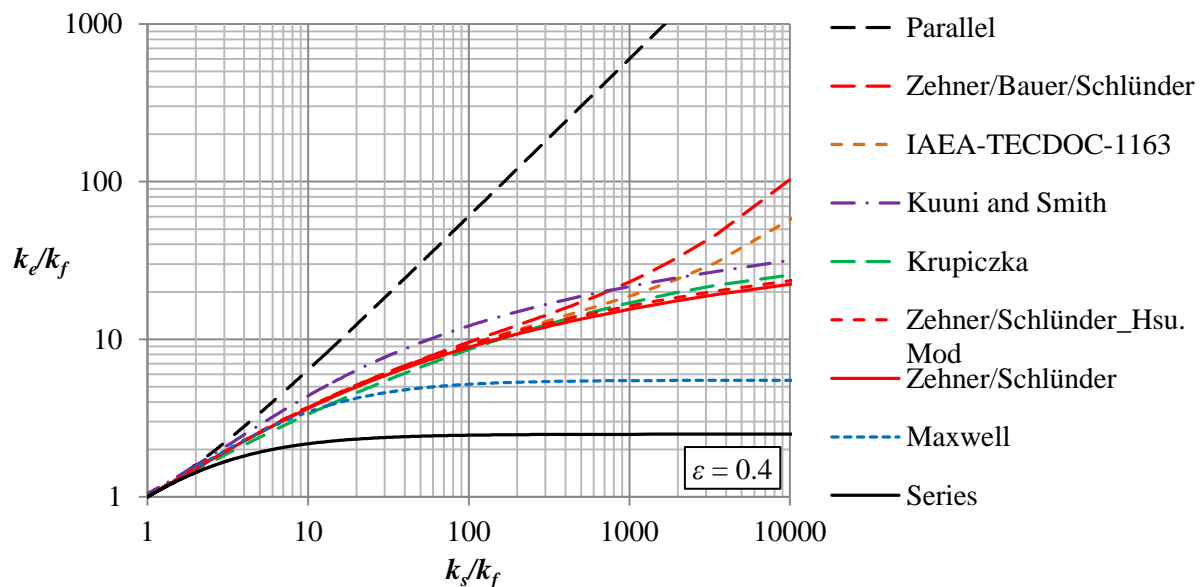


Fig. 1.19 - Comparison of models for a random loose packing of spherically shaped particles.

As Fig. 1.19 displays, all models go through the point 1.1, thus satisfying the limiting relation number three ($k_s=k_f=k_e$; Eq. (1.20)). The lower and upper bound is provided by the series and parallel

arrangement, as it should. If one considers the original Zehner and Schlünder model to be the most accurate, the model of Maxwell gives low values for $k_s/k_f > 10$. The Kunii and Smith model on the other hand, seems to slightly overpredict the thermal conductivity throughout the whole spectrum plotted. This may be due to their radiation term not working correctly. As can be seen from the figure (1.19) the radiation term of the IAEA-TECDOC-1163 and ZBS models starts to become apparent at approximately $k_s/k_f = 120$. This because a solid-fluid conductivity-ratio $K > 100$ only occurs at very high temperatures, and thus the radiation starts to come into effect.

For applications with solid-matrix porous media, however, the above mention models will not suffice. Relatively recent, some new models concerning continuously connected solid structure media (like metal foam) have been developed (Hsu et al, 1994); (Boomsma and Poulikakos, 2000). These models will be presented in the next section.

## 1.8 Effective stagnant thermal conductivity of solid matrix porous media

A solid matrix structure, like open cell metal foam, can make a significant contribution to the effective thermal conductivity of a packed bed, thus allowing for more efficient charging and discharging of a hydrogen-filled tank. Not many models exist for foam-like materials, and it remains to be seen if the models can give a good enough prediction for metal foam filled with granular porous media. If not, a new model shall be developed.

### 1.8.1 Hsu's phase-symmetry model (1994)

The phase-symmetry model by Hsu et al. (1994) is based on the method of Zehner and Schlünder (1970). However, in this approach both the solid and fluid phase is assumed continuously connected and having a phase-symmetry. For this reason, the Zehner and Schlünder equation for effective thermal conductivity (Eq. (1.49)) cannot be used because the expression is not symmetric with respect to the two phases. Hsu et al. (1994) therefore sketch a new unit cell given in Fig. 1.20 below.
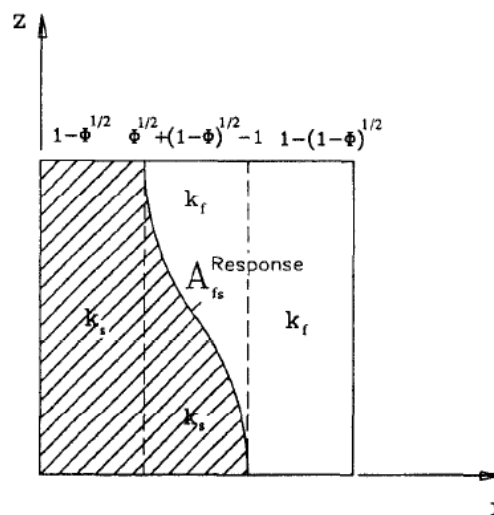


Fig. 1.20 - Unit cell for the phase-symmetric model (Hsu et al, 1994).

Note that they use $\Phi$ for porosity instead of $\varepsilon$ (this work). As displayed by the figure, Hsu et al. (1994) divides the unit cell into three layers: layer one consisting of pure solid, layer two consisting of a

symmetric solid-fluid composition, and layer three which is pure fluid. They then give a new function for the reduced effective thermal conductivity $k_e$ based on Eq. (1.49) and Fig. 1.20:

$$\frac{k_e}{k_f} = \left(1 - \sqrt{1-\varepsilon}\right) + \left(1 - \sqrt{\varepsilon}\right)K + \left(\sqrt{1-\varepsilon} + \sqrt{\varepsilon} - 1\right)K_{sf}, \tag{1.69}$$

where $K_{sf}$ is the dimensionless equivalent thermal conductivity of the composite layer, which can be expressed as

$$K_{sf} = \frac{k_{sf}}{k_f} = \frac{b(1-\lambda)}{(1-\lambda b)^2}\ln\left(\frac{1}{\lambda b}\right) - \frac{b-1}{1-\lambda b}, \tag{1.70}$$

with $\lambda = k_f/k_s$. The solid-fluid interface, as a function of the coordinate system parameters $z$ and $x$ and the deformation factor $b$, is given as

$$x + \frac{z}{b - (b-1)z} = 1. \tag{1.71}$$

By setting the volumetric fraction of the solid in the sandwiched layer equal to the integral over Eq. (1.70) the obtained the following relationship between $\varepsilon$ and $b$

$$\frac{\sqrt{\varepsilon} - \varepsilon}{\sqrt{1-\varepsilon} + \sqrt{\varepsilon} - 1} = \frac{b}{(1-b)}(b - 1 - \ln b). \tag{1.72}$$

They then found a good approximation of the new deformation factor $b$, which can be expressed as

$$b = C\left(\frac{1-\varepsilon}{\varepsilon}\right)^m \tag{1.73}$$

like before, but with $C=1$ and $m=0.9676$ giving the best fit with Eq. (1.72). Equation (1.73) satisfies the constraints of Eq. (1.72), $\varepsilon \to 0 \rightarrow b \to \infty$, and $\varepsilon \to 1 \rightarrow b \to 0$. Also, if $C=1$, then the constraint of $\varepsilon$ being 0.5 when $b=1$ (sphere) is satisfied as well.

### 1.8.2 Boomsma and Poulikakos's metal foam model (2000)

The model for effective stagnant thermal conductivity inside metal foam (Boomsma and Poulikakos, 2000) showed that the phase-symmetry model of Hsu et al. (1994) gave somewhat high predictions, while the predictions of Boomsma-Poulikakos model were quite accurate, compared with various measurements. However, they later released a corrigendum-paper (Boomsma and Poulikakos, 2011) where they altered their model.

Their model is based on modeling the foam as a tetrakaidecahedron with cylindrical ligaments and cubic nodes, as visualized in figures 1.21 through 1.23 (Boomsma and Poulikakos, 2000).
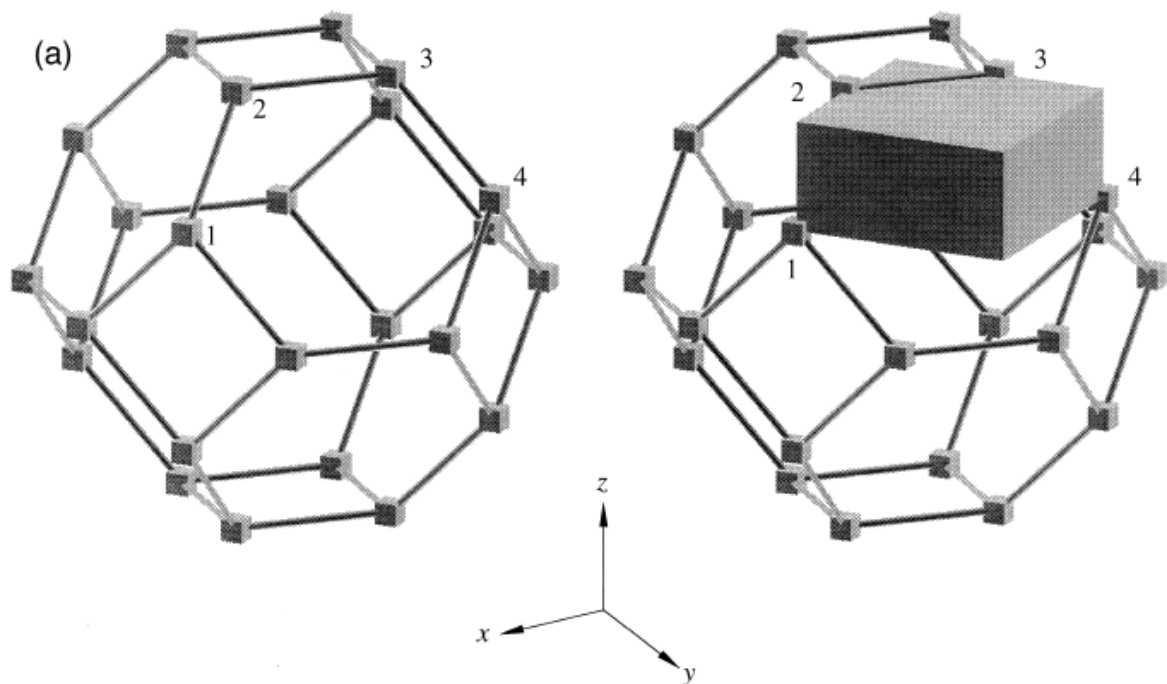
Fig. 1.21 - Their visualization of the foam structure. To the right: the unit cell between points 1-4 (Boomsma and Poulikakos, 2000)

Fig. 1.21 displays the foam structure, fig 1.23 displays the unit cell in relation to the image at the right side in Fig. 1.21, and Fig. 1.22 shows a photo of their metal foam. In Fig. 1.21, the Cartesian coordinate system is shown in order to explain the technical modeling of the foam shown in Fig. 1.23. In Fig 1.23, they denote the width of the node to $r$. However, this work will denote the node with $w$ instead, so that it is not mistaken for radius. Furthermore, the length of each ligament $L$ is denoted with subscripts $A, B, C$ and $D$ for each subsection of the unit cell (Fig 1.23). The radius of the ligaments is $a$. The unit cell consists of six squares and eight hexagons. This shape is the most likely shape that the foam will attain from the foam manufacturing process (Thompson, 1995), and selecting this as an approximate for the foam structure thus gives a good approximate on how the foam will look like as a whole.



32
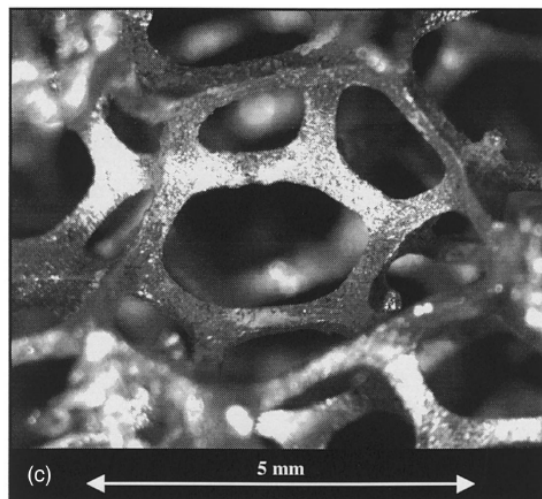
Fig. 1.22 - Picture of metal foam (Boomsma and Poulikakos, 2000).
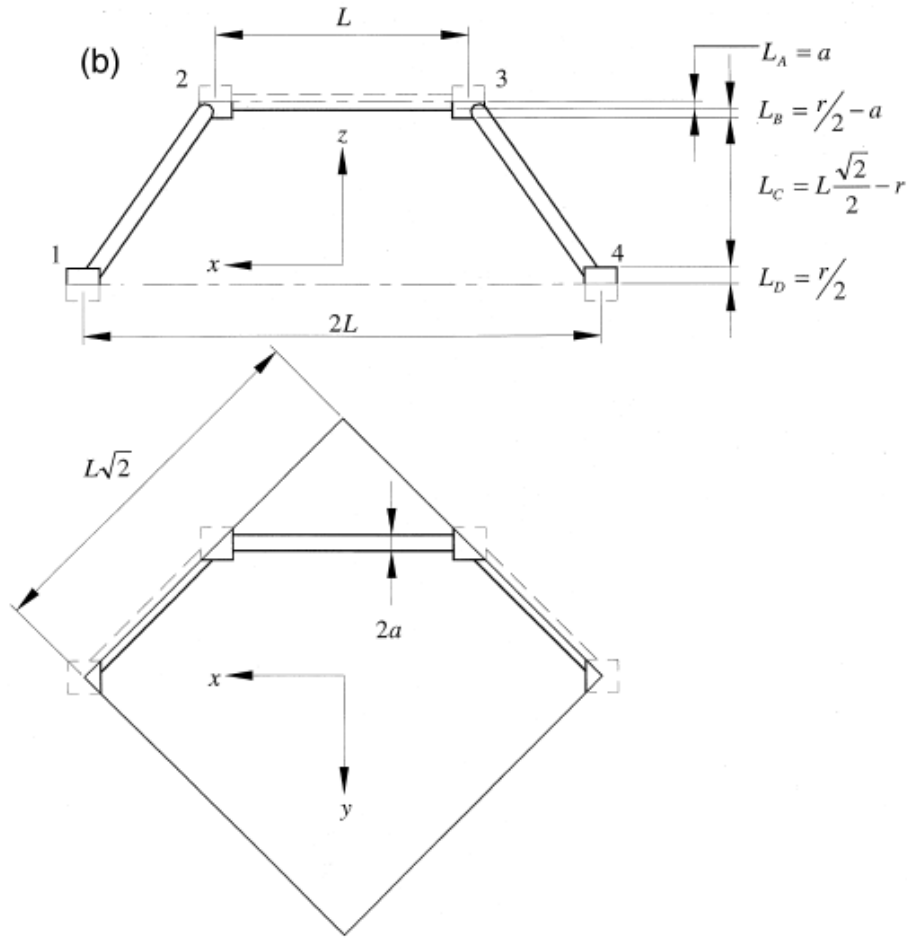


Fig. 1.23 - The metal foam modeled in a Cartesian coordinate system (Boomsma and Poulikakos, 2000).

Boomsma and Poulikakos (2000) choose one sixteenth of the tetrakaidecahedron as their unit cell, because this feature holds all geometric information on the structure. From Fig. 1.23, the length of the rectangular unit cell in the z-direction is

$$L_{uc} = L\frac{\sqrt{2}}{2}, \qquad (1.74)$$

The two other variables, $a$ and $w$, divide the cell into four different sections from top to bottom (z-direction). The first section, $A$, have a height of $a$. This is because the other half of this ligament belongs to the unit cell above. Further down, in the $B$-section, the height of this section is $w/2 - a$. Proceeding to section $D$, the height is the half of one node, $w/2$, because half the node belong to the unit cell below. Finally, the height for section $C$ is the difference between the total unit cell height (Eq. 1.74) and sum of all the other sections. Hence,

$$L_C = L_{uc} - (L_A + L_B + L_D), \qquad (1.75)$$

and

$$L_C = L\frac{\sqrt{2}}{2} - \left[a + \left(\frac{r}{2} - a\right) + \frac{r}{2}\right] = L\frac{\sqrt{2}}{2} - r. \tag{1.76}$$

Having defined these heights for the different subsections one can simply multiply the area of each section in the x-y-plane (see Fig. 1.22, bottom) by their respective heights in order to find the volume $V$ of each section :

$$V_A = 2aL^2. \tag{1.77}$$

$$V_B = (w - 2a)L^2. \tag{1.78}$$

$$V_C = 2\left(\frac{1}{2}L\sqrt{2} - w\right)L^2. \tag{1.79}$$

$$V_D = rL^2. \tag{1.80}$$

They then introduce a set of non-dimensional parameters. With the dimensionless parameters $e=w/L$ and $g=a/L$, they then calculate the volume of the unit cell occupied by solid as (Boomsma and Poulikakos, 2000); (Boomsma and Poulikakos, 2011)

$$V_{A,s} = \left[e^2 + \frac{1}{2}g\pi(1-e)\right]gL^3. \tag{1.81}$$

$$V_{B,s} = \left(\frac{1}{2}e - g\right)e^2L^3. \tag{1.82}$$

$$V_{C,s} = \left(1 - e\sqrt{2}\right)\pi g^2 L^3. \tag{1.83}$$

$$V_{D,s} = \frac{1}{4}e^3L^3. \tag{1.84}$$

By substituting the dimensionless parameters $e$ and $g$ also into equations (1.77) through (1.80), one can define the porosity by

$$\varepsilon = 1 - \frac{V_s}{V} = 1 - \left(\sum_{n=A}^{n=D} V_{n,s} \Bigg/ \sum_{n=A}^{n=D} V_n\right) = \tag{1.85}$$

$$1 - \frac{\sqrt{2}}{2}\left[ge^2 + \frac{1}{2}\pi g^2(1-e) + e^2\left(\frac{1}{2}e - g\right) + \pi g^2\left(1 - e\sqrt{2}\right) + \frac{1}{4}e^3\right]$$

Solving Eq. (1.85) in terms of $g$ gives (Boomsma and Poulikakos, 2011)

$$g = \sqrt{\frac{\sqrt{2}\left[2 - (3\sqrt{2}e^3/4) - 2e\right]}{\pi\left(3 - 2e\sqrt{2} - e\right)}}. \tag{1.86}$$

In order to determine the effective conductivity of the unit cell, the thermal conductivity is then averaged over each section as

$$k_n = \frac{V_{n,s}k_s + (V_n - V_{n,s})k_f}{V_n} \qquad (1.87)$$

where $n$ denotes the sections *A, B, C* or *D*, respectively. Based on the Fourier law of heat conduction (Eq. 1.2) in the z-direction, through a series of four sections, the effective thermal conductivity of the unit cell yields

$$k_e = \frac{L_A + L_B + L_C + L_D}{(L_A/k_A) + (L_B/k_B) + (L_C/k_C) + (L_D/k_D)}. \qquad (1.88)$$

One can then obtain the effective thermal conductivity by substituting for section lengths $L_n$, each of the thermal conductivities $k_n$, and the positive solution for $g$, and then fitting the parameter $e$ on the basis of experimental data. However, this expression will be rather long and untidy. Therefore, it seems more appropriate to introduce the simplifying notations (Boomsma and Poulikakos, 2000); (Boomsma and Poulikakos, 2011)

$$U_A = \frac{4g}{2e^2 + \pi g(1-e)k_s + [4 - 2e^2 - \pi g(1-e)]k_f}. \qquad (1.89)$$

$$U_B = \frac{(e - 2g)^2}{e^2(e - 2g)k_s + [2e - 4g - e^2(e - 2g)]k_f}. \qquad (1.90)$$

$$U_C = \frac{\sqrt{2} - 2e}{\sqrt{2}\pi g^2 k_s + (2 - \sqrt{2}\pi g^2)k_f}. \qquad (1.91)$$

$$U_D = \frac{2e}{e^2 k_s + (4 - e^2)k_f}. \qquad (1.92)$$

Then, the solution for the effective thermal conductivity yields

$$k_e = \frac{\sqrt{2}}{2(U_A + U_B + U_C + U_D)}. \qquad (1.93)$$

The parameter $e$, which is actually equal to the radius of a node divided by the radius of a ligament, now works as a fitting parameter. This means that it can be applied for other types of foams having the same structure (i.e. smaller voids; same structure), by fitting the curve to the experimental data. Plotting this model versus the phase-symmetry model of Hsu et al. (1994) and the ZBS-model gives the predictions showed in Fig. 1.24 on the next page.
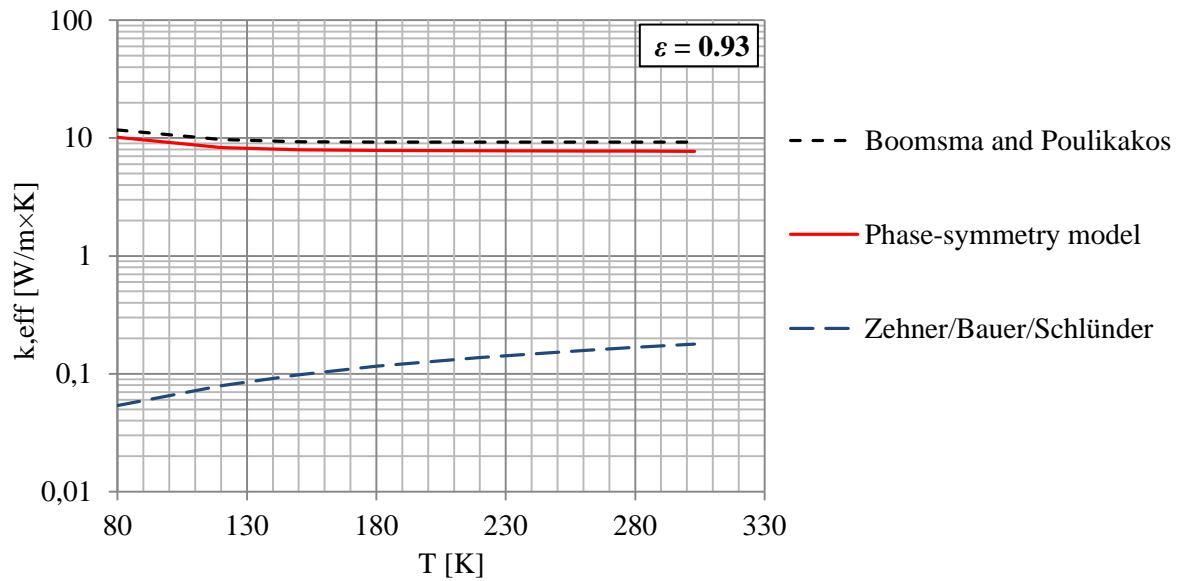
Fig. 1.24 - Comparison between solid matrix models and the ZBS model

In Fig. 1.24, the ZBS model is applied on spherical glass beads with air as fluid. The Boomsma-Poulikakos and phase-symmetry models are applied to aluminum foam filled with the same air and glass beads as applied with the ZBS model. The foam (aluminum Alloy 1100) has a porosity of 0.93. As seen from Fig. 1.24, both foam models suggest that a high-porosity aluminum foam should increase the magnitude of the effective thermal conductivity of a bed with glass beads with about a 100 times. This is a severe improvement and should be worth the reduction in permeability that the foam causes. This increase comes from the thermal conductivity of aluminum which is in the magnitude of 200 W/m·K (NIST Chemistry Webbok) in room temperature compared to approximately 1W/m·K of the glass beads (Handbook of glass data, 1987).

# Part 2 - Experiments

The experiments aim to determine the effective stagnant thermal conductivity of MOF material filled with gases such as nitrogen ($N_2$) and helium (He) as a function of temperature. Measurements were performed in the range of 243K<$T$<423K, and 1bar<p<1.5bar (absolute). In order to ensure the repeatability of measurements, each data point is measured three times, and each experiment (filling) was also repeated at least three times.

Effective stagnant thermal conductivity experiments were first conducted on glass beads, which worked as a reference material, with air and $N_2$ as the fluid phase. Second, experiments on the MOF materials Cu-btc (HKUST-1) and Fe-btc-xerogel were performed with both $N_2$ and He. Then, the glass beads were measured together with a high-porosity aluminum foam in order to examine the effect of the foam. The thermal conductivity of helium is quite similar to that of hydrogen, and thus the results with He will gives a good read on what to expect when hydrogen is applied. The experiment setup used is a Hot Disk TPS 2500s.

## 2.1 Transient Plane Source method (TPS)

Measurements of thermal conductivity by the means of the TPS-method have previously been documented elsewhere (M. Gustavsson, 1994). This method is based on the use of a transiently heated plane sensor, which consists of an electrically conducting pattern in the shape of a double spiral, and this has been etched out of a thin nickel foul. This spiral is sandwiched between two thin samples of insulating material (Kapton/Mica). During a measurement, the sensor is placed carefully between two sample pieces, as Fig. 2.1 depicts.
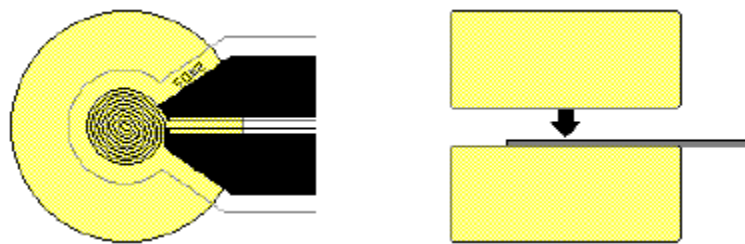


Fig. 2.1 - Sensor position between sample pieces

The solution of the thermal conductivity equation is based on the assumption that the Hot Disk sensor is located in an infinite medium, which means that the transient recording must be interrupted as soon as any influence from the outside boundaries of the two sample pieces is recorded by the sensor. Hence, the thermal penetration depth must not reach the outside boundaries of the sample pieces during transient recording. An estimation of how far this thermal wave has proceeded through the sample is called the probing depth. The probing depth is thus very important, and it is defined as (M. Gustavsson, 1994)

$$\Delta_p = 2\sqrt{\alpha t}, \tag{2.1}$$

where $\alpha = k/(\rho \cdot C_p)$ is the thermal diffusivity of the sample and $t$ is the measuring time of the experiment. A consequence of this equation is that the distance from any point of the sensor to any point on the surface of the two sample pieces must exceed $\Delta_p$ if the total measuring time is $t$.

The theory of the TPS-method is developed under the assumption that the Hot Disk sensor consists of a certain number of concentric ring heat sources located in an infinitely large sample. A constant electric power is supplied to the sensor, creating an increase in temperature which is related to the sensor resistance $R(t)$ through the relation (M. Gustavsson, 1994)

$$R(t) = R_0\{1 + \vartheta[\Delta T_i + \Delta T_{av}(\tau)]\}. \tag{2.2}$$

In Eq. (2.2), $R_i$ is the initial nickel resistance at the beginning of a recording, $\vartheta$ is the temperature coefficient of resistivity (TCR) of the nickel foil, and $\Delta T_i$ is the constant temperature difference that develops momentarily over the thin insulating layers covering the two sides of the sensor material. $\Delta T_{av}(\tau)$ is the temperature increase of the sample of the other side of the insulating layer. From Eq. (2.2), the temperature increase recorded by the sensor can be described by

$$\Delta T_i + \Delta T_{av}(\tau) = \frac{1}{\vartheta}\left[\frac{R(t)}{R_i} - 1\right]. \tag{2.3}$$

The instant temperature difference $\Delta T_i$ becomes constant after a very short time ($\Delta t_i$). Then, $\Delta t_i$ can be estimated as (M. Gustavsson, 1994)

$$\Delta t_i = \frac{\xi^2}{\alpha_i}, \tag{2.4}$$

where $\xi$ is the thickness of the insulating layer and $\alpha_i$ is the thermal diffusivity of the layer material. The spatial average can be obtained through the equation (M. Gustavsson, 1994)

$$\Delta T_{av}(\tau) = \frac{P_0}{\pi^{3/2}(r \cdot k)}D(\tau). \tag{2.5}$$

In Eq. (2.5), $P_0$ is the power output from the sensor, $r$ is the radius of the disk, $k$ is the thermal conductivity of the sample and $D(\tau)$ is a dimensionless time dependent function defined

$$D(\tau) = \sqrt{\frac{t}{\theta}}; \ \theta = \frac{r^2}{k}. \tag{2.6}$$

In this equation (2.6), $t$ is the time measured from the start of the transient recording, and $\theta$ is the characteristic time. The ratio total to characteristic time (TCT) need to be between 0.3 and 0.9 in order for the thermal diffusivity to be estimated correctly. Furthermore, the total temperature increase should be between 2K and 5K (M. Gustavsson, 1994). It is important that all measurements are kept within these constraints as well as the probing depth boundaries. In order to ensure that all measurement values for the effective thermal conductivity are kept within the given uncertainty range of 5% (M. Gustavsson, 1994), the above constraints must be satisfied. The thermal conductivity can be obtained by fitting the experimental data to the linear curve in Eq. (2.5).

## 2.2 Instrumentation

The Hot Disk TPS 2500S is supplied by the Swedish company Hot-Disk AB. It utilizes a small electrical power (<2W) to heat up a special shaped resistance, which is used as a temperature sensor simultaneously. The transient temperature rise of the sensor is recorded and analyzed by the TPS2500S. As result the material thermal diffusivity, specific heat capacity and thermal conductivity are determined. Different material types e.g. solid materials or powders can be measured. In this particular case different sorts of powders are used for the measurements.

Porous media is poured in to a sample holder, and a Hot Disk sensor is placed in between the samples.
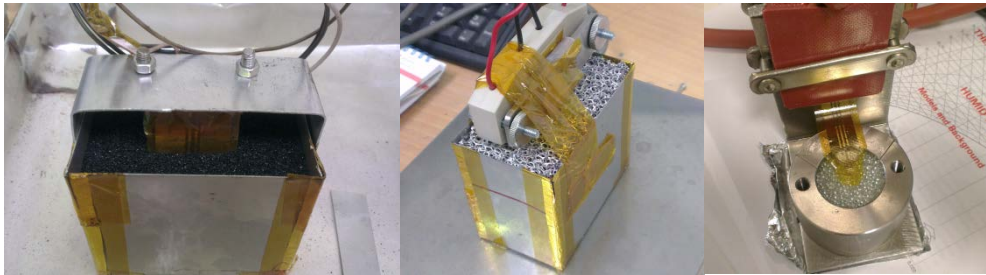


Fig. 2.2 - Sample holders

Different sample holders are used for different types of porous media. Graphic examples are given in Fig. 2.2. If room-temperature measurements with air are conducted, measurements can start as soon as the wires are connected to the sensor.



Fig. 2.3 - Vacuum cell

Most measurements however, are conducted at a range of -30 °C<T<150 °C with pressurized gas (He/N2, at 0.1 ...0.4 bar relative). In order to do this, the sample holder, or sometimes the porous media itself, is lowered into the vacuum / pressure cell. This pressure cell is provided by Hot Disk AB as well and can withstand pressures of 1 bar relative and 200°C (see attachment B, in the Risk Assessment Report). Wires are then connected to the vacuum / pressure cell itself (from inside and outside), as shown in Figure 2.3. The lid is then sealed with a silicone gasket and if necessary with silicon vacuum grease and lowered into a thermal bath. The oil bath can stabilize at any given temperature between -35 and +200 degrees Celsius. After connecting the vacuum suction line with pressure sensors and the gas supply (Figure 2.4 or 2.5, next page) a final leakage test is necessary to guaranty safe and reliable measurements. Once thermal bath has stabilized, measurements can start.

Fig. 2.4 - P&ID flow chart of the thermal conductivity experimental setup.

As shown in Fig. 2.4 and Fig. 2.5, compressed gas (N2 or He at approximately 200 bar) from a gas bottle is supplied after opening the shut-off valve (1.00). A maximal allowed pressure is set with the pressure regulators (max 1.4 bar (absolute)) (1.01) and (1.02). Experiments are preformed usually at 1.2...1.4 bar (absolute). Next follows a needle valve (1.03) for extra security, and a venting valve (1.04). The venting valve is there in order to ensure that pressure can be released if it is too high, or for venting before gas change. By turning the pressure regulator (1.02) up, or turning it down and venting through (1.04) one can regulate the pressure. By opening up the sample circuit valve (1.05) the system is supplied with gas.

The porous medium is located inside the pressure cell (1.08), which again is lowered in the liquid bath (1.09). Before one starts any measurements the porous medium must be evacuated. This is because the porous powder contains humid air which can interfere with the measurement. Evacuation is monitored through the vacuum pressure indicators (1.06) and (1.07). In order to evacuate, the sample circuit valve (1.05) is closed, the vacuum pump (1.10) is started and its valve is opened. When vacuum reaches $10^{-2}$ torr (0.13 bar), the valve on the vacuum pump (1.10) is closed and the sample circuit valve (1.05) is opened. Gas is then purged into the system. The Hot Disk Digital Controller TPS2500S (2.01) governs the measurements together with a timetable set up on the computer (2.02).

To ensure that all humidity is gone, the porous medium must also be activated. In order to heat it, the liquid bath controller (1.11) is then set to 150 degrees Celsius. The evacuation process is then repeated five times over.

A "Procedure for running experiments" summarizes all important steps for carrying out safe measurements and is added in the Risk Assessment Report at the very end of the document.
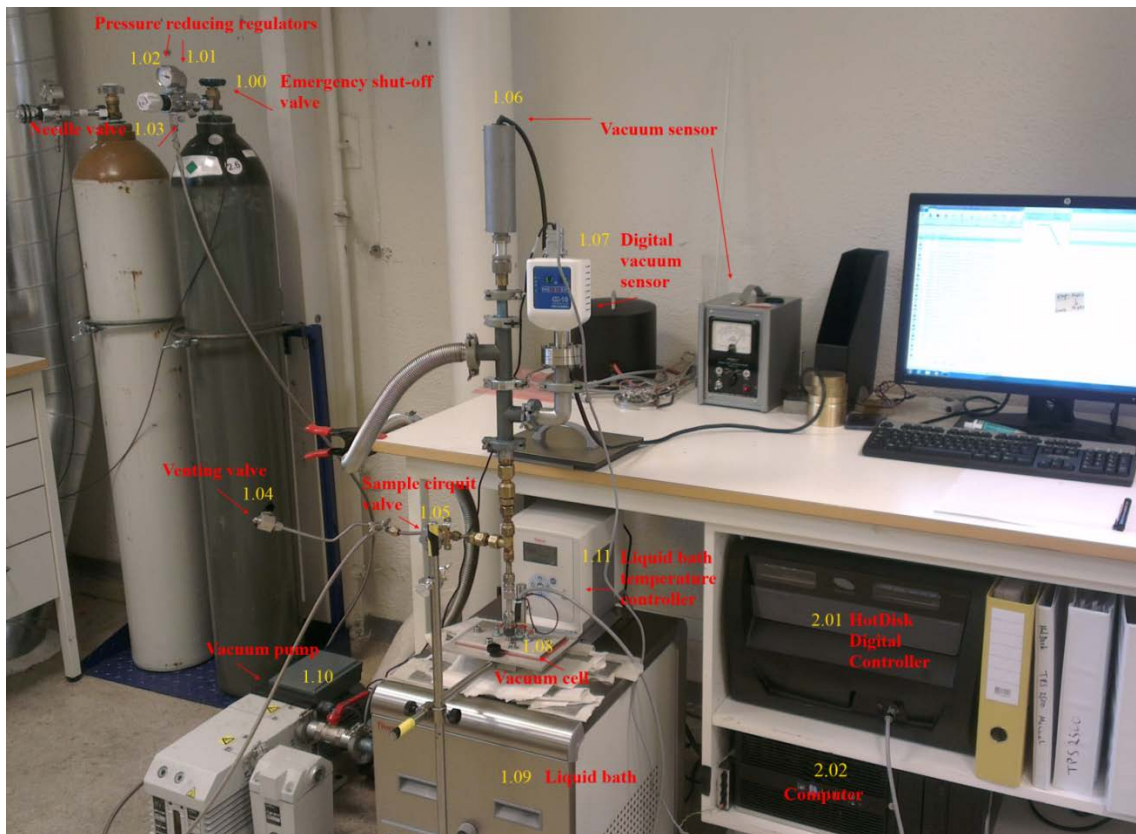


Fig. 2.5 - The Hot-Disk experimental setup.

Table 2.1 - Sensor/Actuator list for the experimental setup.

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| Lfd. Nr. | P&ID Nr. | measurement position | | measure-/actuating variable | | | device specification | | | signal | comments |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | medium | position | sensor / device | S/A | EMSR | device type | measure-/ control value | Unit | range | |
| | | | | | | | (z.B. producer, name.) | | | | |
| 1 | 1.00 | compressed N2/He | gas bottle | shut-off valve | A | | Yara Praxair | 200bar | bar | | |
| 2 | 1.01 | compressed N2/He | gas bottle | Pressure reducing regulator | A/S | PIC | Yara Praxair | 200bar | bar | p_max=315 bar | |
| 3 | 1.02 | compressed N2/He | gas bottle | Pressure reducing regulator | A/S | PIC | Yara Praxair | operating @ < 0.5 bar | bar | p_max=5 bar | |
| 4 | 1.03 | compressed N2/He | gas bottle | Needle valve | A | | Yara Praxair | operating @ 0.2/0.4 bar | bar | p_max= 60 bar | |
| 5 | 1.04 | compressed N2/He | between gas bottle/sample circuit | Venting valve | A | | Swagelok SS 43GS 6mm | operating @ 0.2/0.4 bar | bar | 0.5bar | |
| 6 | 1.05 | compressed or evacuated N2/He | between venting valve/sample holder | Sample circuit valve | A | | Danfoss | operating @ 0.2/0.4 bar | bar | 0.5bar | |
| 7 | 1.06 | compressed or evacuated N2/He | above sample holder | Vacuum pressure indicator | S | PIR | SPEEDIVAC | 0.005…1 torr | torr | 0.005…1 torr | 1 torr =0.013 bar |
| 8 | 1.07 | compressed or evacuated N2/He | above sample holder | Vacuum pressure indicator | S | PIR | TELEVAC CC-10 | $10^{-9}...10^{-3}$ torr $10^{-3}...10^{-1}$ torr $10^{-1}...10^{1}$ torr $10^{1}...10^{3}$ torr | torr | $10^{-9}...10^{3}$ torr | can log to pc |
| 9 | 1.08 | compressed or evacuated N2/He | in liquid bath | Vacuum pressure cell (sample inside) | - | | HotDisk Vacuum cell 11502 | operating @ 0.2/0.4 bar | bar | Tmax =200$^o$C p_max=2.5 bar | |
| 10 | 1.09 | Oil | floor | Liquid bath | - | | AC200 | | | | |
| 11 | 1.10 | compressed or evacuated N2/He | floor next to liquid bath | Vacuum pump | A | | Oerlikon AF-16-25 | p=$10^{3}...5\cdot10^{-3}$ | torr | p=$10^{3}...5\cdot10^{-3}$ torr | <=$10^{-4}$ mbar (given from producer) |
| 12 | 1.11 | | on top of liquid bath | liquid bath temperature controller | A/S | TIC | Thermo Scientific AC 200 | T=243…423 | K | T=238…473K | |
| 13 | 2.01 | | in desk next to liquid bath | Hotdisk digital measurement controller | A/S | DRC | HotDisk Th. Const. Analyser TPS 2500S | | | | Bridge: Keithley 2400, Off balance: Keithley 2700 (6.5 digits resolution) |
| 14 | 2.02 | | in desk next to liquid bath | Computer | A/S | DRA | HotDisk Th. Const. Ana. Version 7.0.16 | Thermal conductivity k | W/mK | 0.005-500 W/mK | Repeatability +-0.5%, Thermal conductivity uncertainty=+-5% |

## 2.3 Investigated materials and their relevant properties

As mentioned before, different reference materials are tested, as well as the Cu-btc and Fe-btc-particles. The small glass beads are almost the same size as the Fe-btc, and can therefore give a good assumption for the magnitude of the effective thermal conductivity when applying Fe-btc.
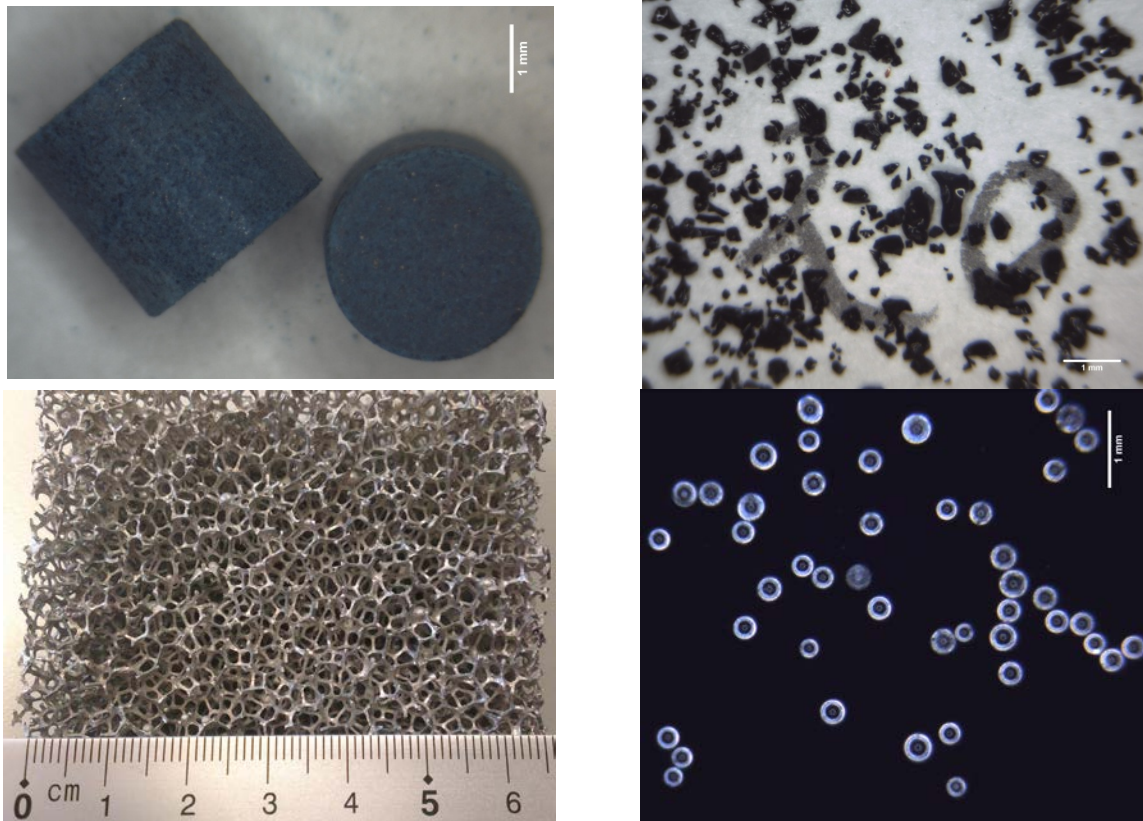


Fig. 2.6 - From top left to bottom right: Cu-btc pellets, Fe-btc, aluminum foam, glass beads.

### 2.3.2 Density

The density of glass beads was obtained by dividing measured volume by measured mass. The respective Cu-btc and Fe-btc densities were obtained by bathing a significant amount of them in ethanol, measuring the volume before and after. The densities given are in dry (activated) condition. This means that the porosity inside the particle is also taken into account. The density of each material is given in Table 2.2 below:

Table 2.2 - Density.

| Material | Particle density $\rho_p$ [kg/m$^3$] | Source |
|---|---|---|
| Glass | 2602 | (Henriksen, 2012) |
| Fe-btc | 1085.4 | (Schlemminger, 2013) |
| Cu-btc | 946.9 | (Schlemminger, 2013) |
| Foam | 2700 | (NIST Chemistry Webbok) |

The bulk densities were obtained by dividing the measured mass $m_b$ by the bulk volume $V_b$:

$$\rho_b = \frac{m_b}{V_b}.$$ 

(2.7)

When conducting experiments with MOF, there is a chance that the particles contain moisture. In order to determine the correct porosity, the mass and volume of the bed was measured before and after each experiment.

### 2.3.3 Porosity

Rearranging Eq. (1.1, part 1), the porosity of a bed of granular porous media can be defined also as

$$\varepsilon = 1 - \frac{\rho_b}{\rho_p}$$ 

(2.8)

Here, $\rho_p$ is the density of the solid particles and $\rho_b$ is the total bulk density. The porosity is determined individually for each bed. The bulk density is obtained by weighing the powder before it is put into the cylinder, and then measuring the height $H_b$ inside the sample holder. The porosity of the aluminum foam is obtained from the relation

$$\varepsilon_{foam} = 1 - \frac{V_{foam}}{V_b} = 1 - \frac{(m/\rho)_{foam}}{V_b}.$$ 

(2.9)

### 2.3.4 Particle diameter

The glass beads and the Fe-btc particles are too small to measure by hand. In addition, the Fe-btc particles are of arbitrary shape, and it is therefore best to determine the diameter from a mass distribution. The sieving analysis was done in accordance with the ISO-2591-1 and ISO-9276-1 standards for sieving and representation of sieving results. Distribution is given in Fig. 2.6 and 2.7.
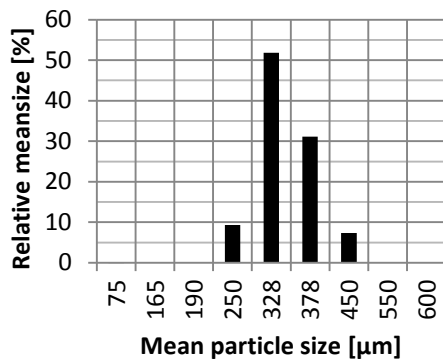


Fig. 2.7 - Relative undersize – glass beads        Fig. 2.8 - Cumulative undersize – glass beads
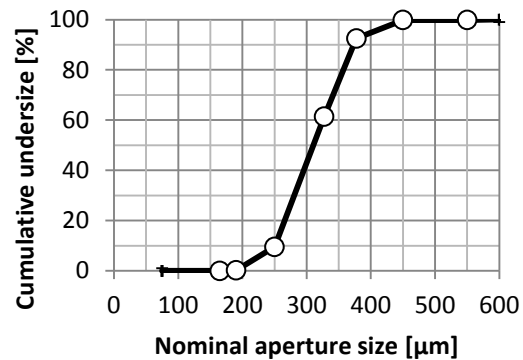
At first sieves were weighed individually before putting them together and pouring powders in the top sieve. The sieves were then shaken using a mechanical shaker. The glass beads were shaken for 20 minutes. Because the Fe-btc is so light and small, the particles have great difficulty coming through

the sieves. Sieving time were therefore 3×20 minutes, pounding them lightly with the palm in between. Afterwards, the sieves were weighed once again, to determine the mass of solids. The process is then repeated five times over. The sieving results are presented in full in Appendix A.

Distributions obtained are cumulative mass distribution, and particle diameter is computed modifying Eq. (1.50, Part 1). Since the particle density $\rho_{pi}$ for the $i$th fraction is the same as the total particle density $\rho_p$, becomes

$$\bar{d}_p = \left[\sum_{i=1}^{n}\left(\frac{V_i}{V}\cdot\frac{1}{d_{pi}}\right)\right] = \left[\sum_{i=1}^{n}\left(\frac{V_i/\rho_p}{V/\rho_p}\cdot\frac{1}{d_{pj}}\right)\right] = \left[\sum_{i=1}^{n}\left(\frac{V_i/\rho_{pi}}{V/\rho}\cdot\frac{1}{d_{pi}}\right)\right] = \left[\sum_{i=1}^{n}\left(\frac{m_i}{m}\cdot\frac{1}{d_{pi}}\right)\right]. \qquad (2.10)$$

An average particle diameter for the large glass beads were obtained by hand measurement (Henriksen, 2012).

The Cu-btc particles are of cylindrical shape, and a different particle diameter must be applied. Loreline Hubert (2011), whose report also covered Cu-btc, estimated the length $L_p$ and diameter $d_p$ to be respectively 3.04 mm and 3.09 mm. The equivalent spherical particle diameter $d_s$ is given by (Kaviany, 1995)

$$d_s = \sqrt{\frac{A_p}{\pi}}, \qquad (2.11)$$

where $A_p$ is the surface area of the particle. The particle diameters used in measurements are then given in Table 2.3 below.

Table 2.3 - Particle diameter of various materials.

| Material | Distribution diameter $d_p$ [mm] | Spherical diameter $d_s$ [mm] | Source |
|---|---|---|---|
| Glass beads – small | 0.338 | | This work |
| Glass beads - large | 1.395 | | (Henriksen, 2012) |
| Fe-btc, fillings 1-2 | 0.456 | | This work |
| Fe-btc, fillings 3-5 | 0.440 | | This work |
| Cu-btc | | 3.764 | (Hubert, 2011) |
| Foam | N/A | | |

## 2.4 Data reduction

In order to determine the effective thermal conductivity correctly, the solid conductivity $k_s$ and the fluid conductivity $k_f$ as a function of temperature need to be known. The fluid conductivity of air, He and $N_2$ can be expressed as (VDI Heat Atlas, 2010)

$$k_f = C_1 + C_2 T + C_3 T^2 + C_4 T^3 + C_5 T^4, \qquad (2.12)$$

with the constants given in Table 2.4.

Table 2.4 - Constants for $k_f$ 4$^{th}$ degree polynomial.

| Medium | $10^3 \cdot C_1$ | $10^3 \cdot C_2$ | $10^6 \cdot C_3$ | $10^9 \cdot C_4$ | $10^{12} \cdot C_5$ | Eq. range | Eq. error |
|---|---|---|---|---|---|---|---|
| Air | -0.908 | 0.112 | -0.084333 | 0.056964 | -0.015631 | 223K<$T$<773K | 1% |
| $N_2$ | -0.133 | 0.101 | -0.060650 | 0.033610 | -0.007100 | 223K<$T$<773K | 1% |
| He | 34.00 | 0.457 | -0.214890 | 0.100710 | -0.019140 | 223K<$T$<773K | 1% |

The solid conductivity of glass beads and the aluminum foam also need to be known. The MOF conductivities are found by fitting theoretical models to the experimental data. However, in order to know which of the theoretical models to apply, all the properties for the reference material must be known. Conductivity of Aluminum Alloy 1100 is given as (NIST Chemistry Webbok) by the relation

$$\log_{10} k_{s,alu} = C_1 + C_2 \log_{10} T + C_3 \log_{10} T^2 + C_4 \log_{10} T^3 + C_5 \log_{10} T^4$$

$$+ C_6 \log_{10} T^5 + C_7 \log_{10} T^6 + C_8 \log_{10} T^7 + C_9 \log_{10} T^8$$

(2.13)

Table 2.5 - Constants for $k_{s,alu}$ 8$^{th}$ degree polynomial.

| | |
|---|---|
| $C_1$ | 23.39172 |
| $C_2$ | -148.5733 |
| $C_3$ | 422.1917 |
| $C_4$ | -653.6664 |
| $C_5$ | 607.0402 |
| $C_6$ | -346.152 |
| $C_7$ | 118.4276 |
| $C_8$ | -22.2781 |
| $C_9$ | 1.770187 |
| Range | 4K<$T$<300K |
| Eq. error | 2% |

Constants for Eq. (2.13) is given in Table 2.5. As seen from the Table, the range is not wide enough. Experiments are done in the range of 243<$T$<423K. Therefore the rest of the range is covered by an extrapolation equation. Fig. 2.8 displays this function graphically. The equation is given as

$$k_{s,alu} = 8 \cdot 10^{-5} \cdot T^2 - 0.0803 \cdot T + 228.55.$$

(2.14)
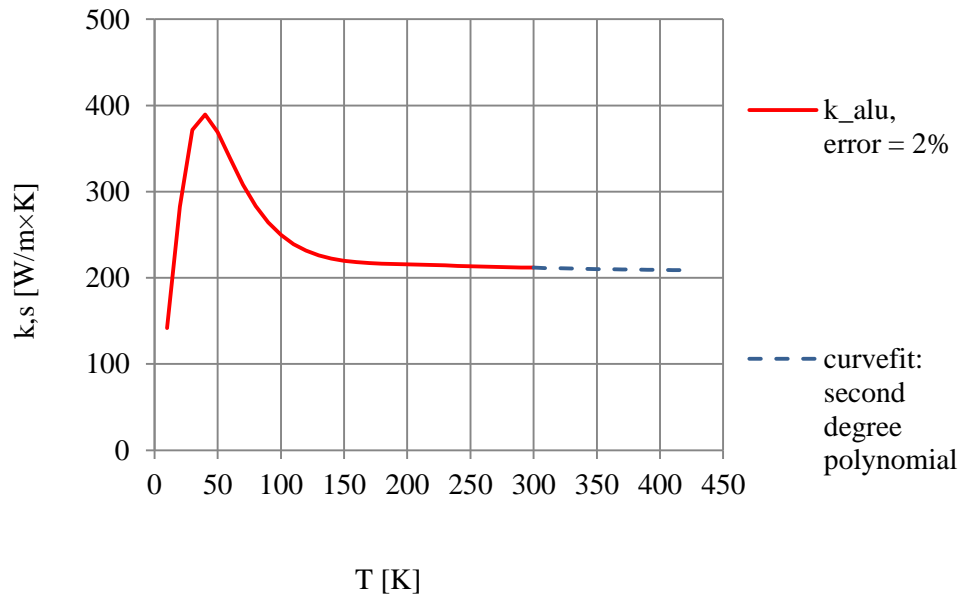
Fig. 2.9 - Solid conductivity of aluminum alloy 1100.

The solid conductivity of glass beads is also an extrapolation, based on data from the Handbook of Glass Data (1987) and an algorithm developed by glassproperties.com. The computation of the thermal conductivity with the exact composition of the glass beads is given in Appendix B. The equation, with $C$=1.2, is expressed as

$$k_{s,glass} = C \cdot (-4.8502 \cdot 10^{-8} \cdot T^2 + 4.4634 \cdot 10^{-3} \cdot T + 1.5825 \cdot 10^{-2}). \qquad (2.15)$$
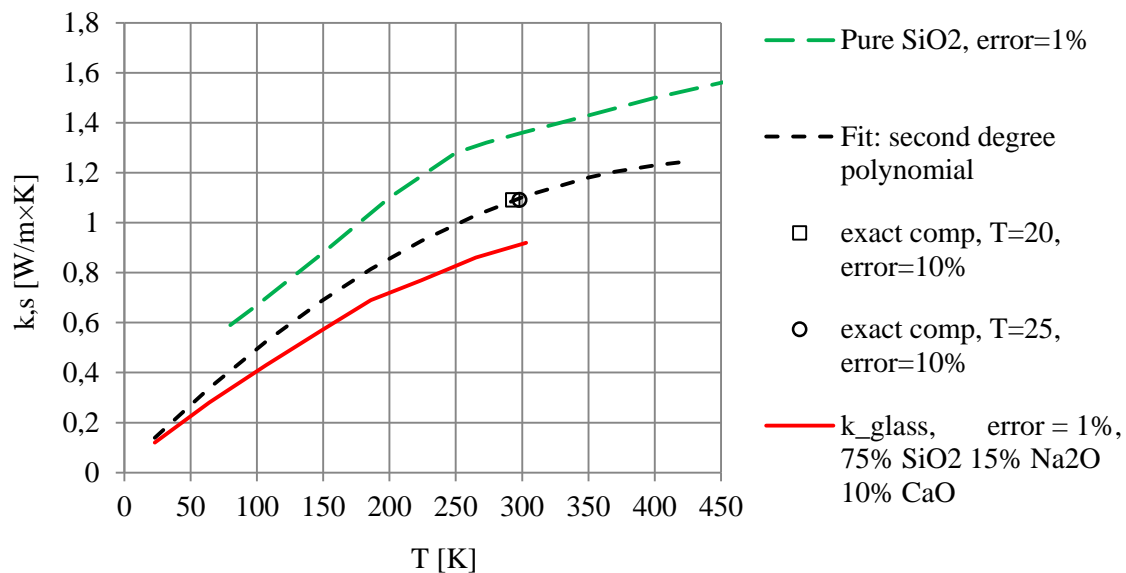


Fig. 2.10 - Solid conductivity of glass beads as a function of temperature (Handbook of glass data, 1987); (Glassproperties.com).

The exact composition of the glass beads is (KarlRoth.com):

- $SiO_2$ 72.5 %
- $Na_2O$ 13.0 %
- $K_2O$ 0.2 %
- CaO 9.6 %
- MgO 4.22 %
- $Al_2O_3$ 0.58 %
- $Fe_2O_3$ 0.11 %.

As seen in Fig. 2.9 above, the thermal conductivity of the curve-fit equation follows the same trend as the thermal conductivity of a glass-melt (red line; (Handbook of glass data, 1987)) with almost the exact same composition as glass beads. The extrapolated equation (Eq. 2.15) is actually the second order polynomial for the near-exact composition (red line) in Fig. 2.10 multiplied by the constant $C=1.2$. This constant was inserted into the equation in order to fit the curve to the thermal conductivity magnitude for the exact composition (which is $k=1.086$, at room temperature, i.e. 293K<T<298K). The thermal conductivity for pure $SiO_2$ (Ed. James, 2001) also follows this same trend.

## 2.5 Uncertainty analysis

The producer of the Hot Disk system gives the uncertainties on the effective thermal conductivity rendered in Table 2.6.

Table 2.6 - Given uncertainties (M. Gustavsson, 1994).

|  | Effective thermal conductivity $k_e$ |
| --- | --- |
| Uncertainty | 5% |
| Reproducibility | 2% |

The effective stagnant thermal conductivity that comes out of the software is given with 5% uncertainty. But each data point is also given with some parameters not affected by the Hot Disk program. These include:

- The porosity of the bed $\varepsilon$, which is a function of the bed mass $m_b$ and bed volume $V_b$.
- Temperature at a given data point, $T$.
- Pressure at a given data point, $p$.

The uncertainty of a variable

$$R = R(X_1, X_2, X_3, \cdots, X_N), \qquad (2.16)$$

depending on the calculated or measured values $(X_1, \cdots, X_N)$, can be expressed as (Moffat, 1988):

$$\delta R = \left\{ \sum_{i=1}^{N} \left( \frac{\delta R}{\delta X_i} \delta X_i \right)^2 \right\}^{\frac{1}{2}}.$$  (2.17)

Rewriting Eq. (2.8), and the porosity is expressed as

$$\varepsilon = 1 - \frac{m_b/V_b}{\rho_s} = 1 - \frac{m_b/h_b l_b w_b}{\rho_s},$$  (2.18)

where $h_b$, $l_b$ and $w_b$ is the height, length and width of the bed, respectively. Then

$$\varepsilon = \varepsilon(m_b, H_b, l_b, w_b, \rho_s).$$  (2.19)

Rewriting Eq. (2.17) in terms of $\varepsilon$ then gives the uncertainty equation for the porosity

$$\delta\varepsilon = \left\{ \left( \frac{\delta\varepsilon}{\delta m_b} \Delta m_b \right)^2 + \left( \frac{\delta\varepsilon}{\delta H_b} \Delta H_b \right)^2 + \left( \frac{\delta\varepsilon}{\delta l_b} \Delta l_b \right)^2 + \left( \frac{\delta\varepsilon}{\delta w_b} \Delta w_b \right)^2 + \left( \frac{\delta\varepsilon}{\delta \rho_s} \Delta \rho_s \right)^2 \right\}^{\frac{1}{2}},$$  (2.20)

or in terms of relative uncertainty

$$\frac{\Delta\varepsilon}{\varepsilon} = \left\{ \left( \frac{\Delta m_b}{m_b} \right)^2 + \left( \frac{\Delta H_b}{H_b} \right)^2 + \left( \frac{\Delta l_b}{l_b} \right)^2 + \left( \frac{\Delta w_b}{w_b} \right)^2 + \left( \frac{\Delta \rho_s}{\rho_s} \right)^2 \right\}^{\frac{1}{2}}.$$  (2.21)

The temperature is controlled by the liquid bath controller, which has an uncertainty of $\Delta T = \pm 0.1$K. The pressure is controlled by the pressure regulator. All Experiments are done at constant pressure. However, the pressure may vary as much as $\Delta p = \pm 0.2$ bar.

Since the experiments on MOF are to be fitted with the theory, an uncertainty analysis on the parameters affecting the theoretical models was conducted. The parameters making an impact on the theoretical models are:

- Porosity, $\varepsilon$.
- Temperature, $T$.
- Pressure, $p$.
- Solid conductivity, $k_s$
- Fluid conductivity, $k_f$

The solid and fluid conductivities are mainly a function of temperature. The temperatures calculated from the liquid bath controller are very accurate.

The conductivity of glass of solids varies greatly with pressure when the pressure is low, however, conductivity of solids will level out and become constant in the vicinity of 1000 hPa (=1 bar) (E. S. Huetter, 2008). As all measurements were carried out in ambient-like pressures, the relative error of solid conductivity of MOF is assumed constant at 2%. The curve-fit for glass beads is on the other hand based on experimental data with 1% uncertainty as well as theoretical correlations with 10%

uncertainty. Therefore, the relative conductivity error of the thermal conductivity of glass beads is assumed to be 15%.

The conductivity of fluids as a function of temperature has a given (Table 2.4) relative error of 1%. The fluid conductivity variation due to pressure is examined in Table 2.7 (NIST Chemistry Webbok).

Table 2.7 -  Comparison of fluid thermal conductivity at various pressures

|  | Temp. | Thermal conductivity | | Increase ($p_n \rightarrow p_{n+1}$) | |
|---|---|---|---|---|---|
|  |  | $N_2$ | He | $N_2$ | He |
| $p_1$=1 bar | 240 | 0.02168 | 0.13372 |  |  |
|  | 420 | 0.03341 | 0.19692 |  |  |
| $p_2$=1.2 bar | 240 | 0.02168 | 0.13374 | 0.032 % | 0.015 % |
|  | 420 | 0.03342 | 0.19694 | 0.015 % | 0.010 % |
| $p_3$=1.4 bar | 240 | 0.02169 | 0.13375 | 0.032 % | 0.007 % |
|  | 420 | 0.03342 | 0.19695 | 0.015 % | 0.005 % |

Table 2.7 shows that it is acceptable to assume that the relative error on fluid conductivity is <0.05%.

Uncertainties will be given as a lower and upper band for the theoretical models. The experimental result uncertainties are visualized as error bares around the experimental data points. Note that the uncertainty of a data point most likely is > 5%, due to the effect of the parameters that the computer software does not take into account (porosity variation, temperature variation, pressure variation).

The density of glass beads was estimated manually on a set of larger glass beads (⌀4.9mm) (Henriksen, 2012) to b 2602 kg/m$^3$. However, the producer states that the density is approximately 2500 kg/m$^3$ (KarlRoth.com). This gives a discrepancy of approximately 5%. A 5% discrepancy from the given value indicates that the actual density may even be as low as 2400 kg/m$^3$. Therefore the total density uncertainty will be assumed to be 10%.

The constant relative uncertainty of each variable is given in Table 2.8

Table 2.8 - Uncertainties.

| Parameter | Uncertainty [$\Delta X_i$] | Rel. uncertainty [$\Delta X_i/X_i$] |
|---|---|---|
| $\varepsilon$ |  | Eq. (2.10) |
| $m_b$ | 0.1g |  |
| $H_b$ | 1 mm |  |
| $l_b$ | 1 mm |  |
| $w_b$ | 1 mm |  |
| $\rho_s$ |  | 10 % |
| $T$ | 0.1 K |  |
| $p$ | 0.2 bar |  |
| $k_s$ |  | MOF: 2% |
|  |  | Glass: 15% |
| $k_f$ |  | 0.05% |

# Part 3 - Results and analysis

Experiments were performed on large (∅1.395 mm) and smaller (∅0.38 mm) glass beads as well as Fe-btc and Cu-btc MOF. Last, some experiments were performed on aluminum foam filled with the small glass beads and $N_2$ or air as fluid. All graphs presented in this section show the measured effective stagnant thermal conductivity (y-axis, W/m·K) plotted versus temperature (x-axis, K) plotted versus theoretical models. Experiments with $N_2$ or He are performed at a constant absolute pressure of 1.2 bar, if another pressure is not specified. A selection results will be presented and discussed in this chapter. All results for the glass beads, Cu-btc, Fe-btc and aluminum foam are shown in Appendix C, D, E, and G, respectively. These Appendices also contain the data from which the plots are produced.

## 3.1 Reference material

In order to find a theoretical model which can be used to determine the MOF solid conductivity ($k_s$), experiments were first conducted on glass beads of various sizes. Some of the results are displayed in Fig. 3.1 - 3.3. The rest of the glass beads results are given in Appendix C. Preliminary results for the effective thermal conductivity of glass beads with air as fluid are shown in Fig. 3.1 with properties given in Table 3.1 below.

Table 3.1 - Properties of glass beads experiments.

| material | m_bulk m,b [mg] | H_bulk H [m] | D_bulk D [m] | Volume V,b [m3] | Density ρ,bulk [kg/m3] | Porosity ε [1] | Uncertainty dε/ε |
|---|---|---|---|---|---|---|---|
| glass_1.395mm | 13261 | 0,0174 | 0,025 | 8,5E-06 | 1556 | 0,40 | 12,24 % |
| glass_1.395mm | 13614 | 0,0186 | 0,025 | 9,1E-06 | 1493 | 0,43 | 12,06 % |
| glass_1.395mm | 12742 | 0,0172 | 0,025 | 8,5E-06 | 1506 | 0,42 | 12,26 % |
| glass_0.38mm | 12992 | 0,0168 | 0,025 | 8,2E-06 | 1575 | 0,39 | 12,33 % |
| glass_0.38mm | 13923 | 0,0181 | 0,025 | 8,9E-06 | 1566 | 0,40 | 12,12 % |
| glass_0.38mm | 14684 | 0,0189 | 0,025 | 9,3E-06 | 1584 | 0,39 | 12,02 % |
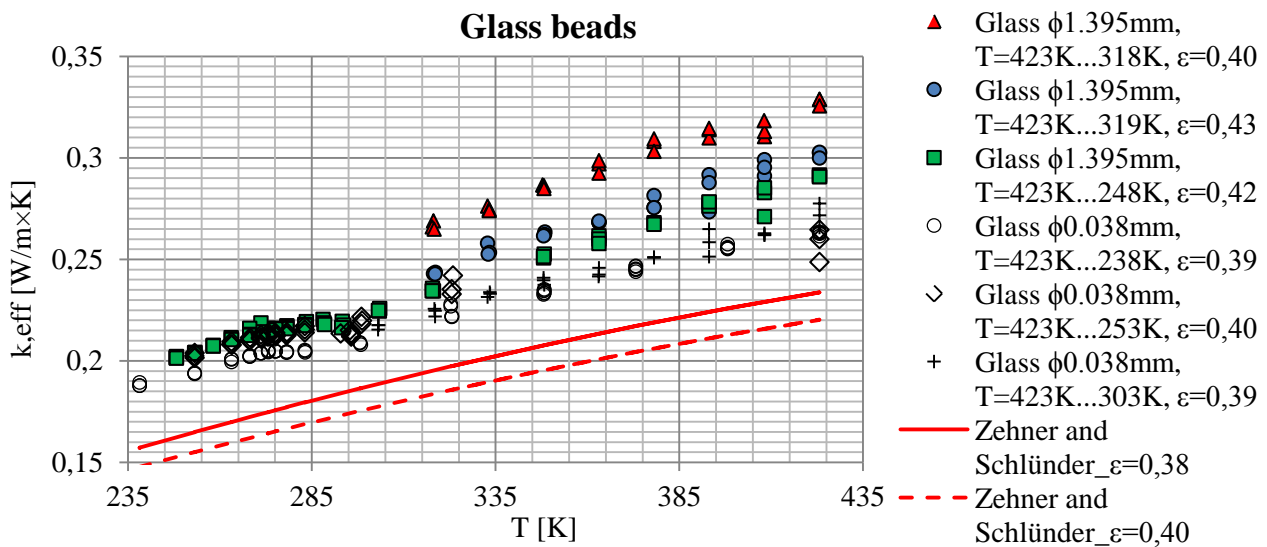


Fig. 3.1 - Effective thermal conductivity of six different fillings of glass beads with air as fluid.

The figure (3.1) shows that the effective thermal conductivity has an ascending curve from low to high temperature. The conductivity of the larger glass beads (colored, filled markers) has a higher magnitude than the small glass beads (black, non-filled markers). The large glass beads have as well a difference of approximately 11% from the lower (green squares) measurement series to the lower (red triangles) measurement series. This could be due to a change in porosity. As seen from the two lines for the Zehner and Schlünder model (Fig. 3.1; red stapled and red solid line), changing the porosity from 0.38 to 0.4 (5%), results in a reduction of the effective conductivity of a little less than 5%. Summing up the porosity uncertainties and the TPS measurement device uncertainties then gives an uncertainty of approximately ±10% which can explain the variation between experiments performed on the same material.

Furthermore, the results do not match the theoretical models at a given porosity. Each measurement point has an uncertainty of 5%, but repeated measurements show that the effective thermal conductivity consequently is approximately 15% higher in magnitude than suggested by the theoretical models. However, the porosity errors (displayed in Table 3.1) are quite high, and this could explain some of this behavior. As an example, take the top red triangles from Fig 3.1 (glass $\varnothing$1.395mm, $\varepsilon$=0.4): With the porosity error of 12.24%, this series could actually have porosity as low as 0.35. As stated in the uncertainty analysis in part two, the porosity error affects both measurements and the theoretical model. The porosity error, which is mainly due to the particle density error (10%) may be responsible for some of this behavior. Though, the most likely scenario is that the humidity (20% relative humidity for all measurements) in the air causes an increase in the effective thermal conductivity due to the formation of water droplets at the vicinity of contact points between particles, thus increasing the effective conductivity of the bed (as shown in Fig. 1.4, the thermal conductivity of water is severely higher than that of air).

Last, Fig 1.3 show a trend of discontinuity in the magnitude of the effective thermal conductivity in the range of 265<$T$<295K. At these temperatures, the effective thermal conductivity of the bed seems to flatten out. This discontinuity could be due to icing, and the issue may remove itself when experiments with evacuated material flushed with nitrogen gas are performed.

The figures 3.2 and 3.3 show the effective thermal conductivity of glass beads filled with $N_2$ gas. A lower and upper band for the ZBS-model is added in these figures. This is the upper and lower relative uncertainty bounds of this model due to the parameters discussed in the uncertainty analysis (sect. 2.5). The error bars represent the 5% error for each data point given by Hot Disk.

Figure 3.2 shows the effective stagnant thermal conductivity of a packed bed filled with large glass beads ($\varnothing$1.395mm) with $N_2$ gas at 1.2 bar absolute pressure. The figure clearly shows a resemblance with the theoretical models: The conductivity of the experimental results increases with increasing temperature, following the ascending trend of the theoretical models. The results follow the high uncertainty bound of the ZBS-model. The model of Kunii and Smith and the Maxwell model predict high and low, respectively. The model from IAEA-TECDOC-1163 predict slightly lower than the ZBS-model. All these trends also show in the comparison from part 1 (Fig. 1.19). The figures 3.2 and 3.3 show that the ZBS model gives the best prediction for the effective thermal conductivity. However, due to the large uncertainties of the porosity and solid conductivity, the ZBS-model has a relative uncertainty of about ±10%. The 5% uncertainty for each measurement point also needs to be taken into account. Generally, a relative uncertainty over 10% is not acceptable, and therefore it is somewhat problematic that the uncertainties of the solid conductivity and porosity are so large. For

future applications, the choice of glass beads as a reference material should therefore be reconsidered. For effective thermal conductivity experiments, the most important properties of a reference material are density (for calculation of the porosity) and solid thermal conductivity (as input parameter in the theoretical models). The thermophysical properties of a reference material should be well-known.
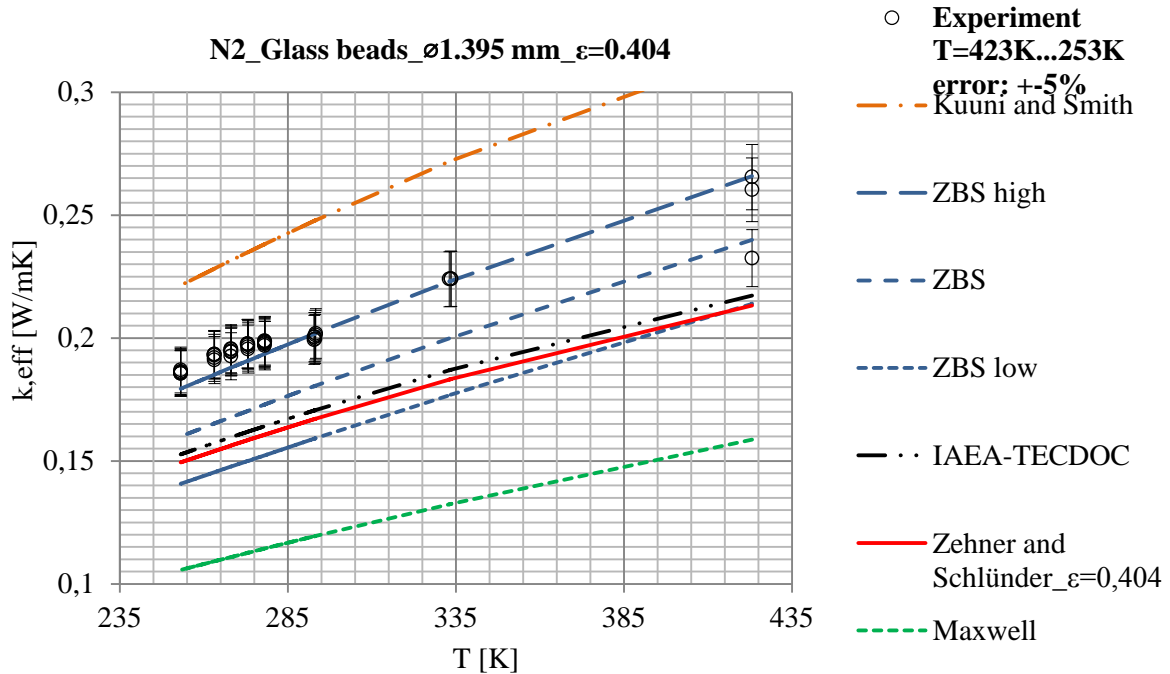


Fig. 3.2 - Effective thermal conductivity of large glass beads and $N_2$ plotted versus temperature.
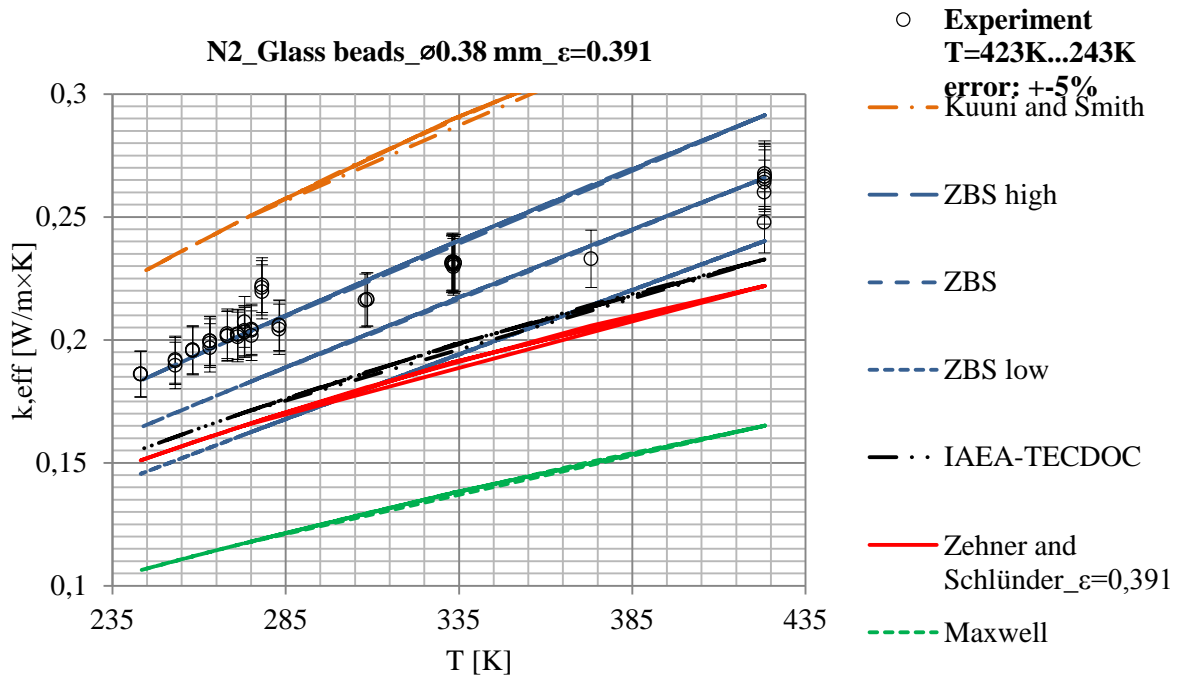


Fig. 3.3 - The effective thermal conductivity of small glass beads with $N_2$ gas.

Fig. 3.3 shows the effective thermal conductivity of a bulk filled with small glass beads (∅0.38 mm) and nitrogen gas. These measurements were done at temperatures ranging from T_high= 243K down to T_low= 243K. Then, the measurements were repeated from T_low and up to T_high again. The repeatability is very good; the figure shows that all data points are almost identical. All results keep within the ZBS relative uncertainty bounds, except for the values at approximately 277 K. What caused this is uncertain. It could be some disturbance in the measurement environment, or a computational or calibration-related error in the Hot Disk software. The latter issue is addressed further down on this page.

By comparing the small glass beads experiments conducted with $N_2$ gas (Fig. 3.3) with the ones conducted with air (Fig. 3.1), one can see that the magnitude of the thermal conductivity seems to be about the same compared to their respective Zehner and Schlünder curves. This indicates that it might not be any humidity-effect on the thermal conductivity, but perhaps rather a miscalculation of the particle density (which can lead to a large miscalculation of the porosity).

A major concern is that the discontinuity of the effective thermal conductivity shown in Fig. 3.2 through 3.4 around $T=273K$ is still present, even though the experiments were conducted with pure $N_2$ as fluid. This behavior is clearly not supported by the theoretical models, which give ascending, almost linear, curves for thermal conductivity from $T=243K$ to $T=423K$. Since the bulk material may contain moisture from the setup mantling process, a possible explanation is that this was due to the formation of ice around $T=273K$. However, this is not the case, since the air has been evacuated prior to experiment startup. Another possible scenario is that the system had a leakage. After performing several measurements, the conclusion was that this was indeed the case. The silicon grease used for sealing the pressure cell was seeping through and into the pressure cell during measurements. A photo taken after an experiment is displayed below (Fig. 3.2).
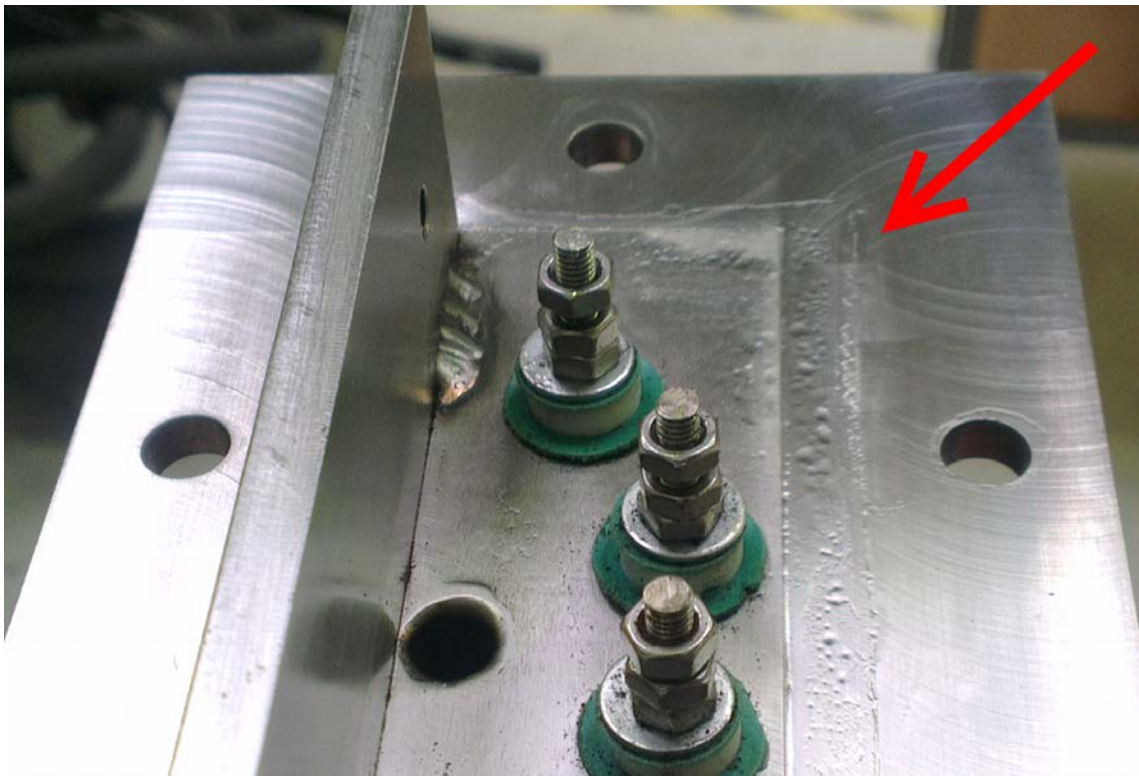


Fig. 3.4 - Silicon vapor seeping inside the pressure/vacuum cell.

The photo (Fig. 3.4) clearly shows that silicon vapor has been seeping inside the container through the sealing. If this affects the measurements around $T$=273K, it would most likely also affect the measurements at other temperatures as well, so this being the cause of the problem is unlikely.

Another proposal is that the Hot Disk TPS software is unable to compute the thermal conductivity correctly in the area around $T$=273K, due to some calibration error. After checking with the producer (mail attached in Appendix F), the conclusion was that a calibration error is most likely to have caused this behavior. Since there was no time to address this concern at the time of this discovery, the results in the range of 263K<$T$<283K were not taken into account during the fitting of MOF solid conductivities.

Summarizing the glass beads experiments, it is clear that the theoretical model that is best fit to use for determining the solid conductivity of the MOF materials is the ZBS model. The Hot Disk setup have a very good repeatability, and measures the effective thermal conductivity of a granular porous bed of glass beads in the temperature range of 243K<$T$<263K and 283K<$T$<423K within 15% accuracy. However, in order to confirm the validity of this experimental setup within 10% accuracy, more experiments with a different reference material should be carried out.

## 3.2 MOF results

The MOF materials Cu-btc and Fe-btc were measured in the range of 243<$T$<423K. Some of the results from these experiments are given in visual form further down. All of the plots, as well as the produced data, are given in Appendix D (Cu-btc) and Appendix E (Fe-btc). The results for the effective thermal conductivity $k_s$ were fitted to the ZBS model through the temperature-dependent relation

$$k_s = k_{s0} \left(\frac{T}{T_0}\right)^n,$$ 

(3.1)

where $T_0$=273K, $k_{s0}$ is the solid thermal conductivity at $T_0$, and $n$ is an exponential coefficient. The ZBS model was chosen on the basis of it providing the most accurate predictions, and as well because it is a versatile model which takes into account all of the secondary parameters given in Eq. 1.20. The fit for the solid thermal conductivity can thus be applied for prediction of larger storage systems as well. The optimal values for $k_{s0}$ and $n$ were found setting the absolute of the sum of all effective thermal conductivity differences ($\Delta k_e$) between the ZBS model and the measured values to a minimum. This gives

$$\left.\begin{matrix} k_{s0} \\ n \end{matrix}\right|_{optimal} = MIN\left\{\left|\sum_{i=1}^{N}\Delta k_e\right|\right\} = MIN\left\{\sqrt{\sum_{i=1}^{N}\left(k_{e,ZBS} - k_{e,measured}\right)^2}\right\}.$$ 

(3.2)

The solution to equations (3.1) and (3.2) were obtained using Solver in Excel. These values are displayed in Table 3.2.

Table 3.2 - Optimized constants

|  | Cu-btc | Fe-btc |
|---|---|---|
| $k_{s0}$ | 0.33 | 0.28 |
| $n$ | 0.11 | 0.26 |
| $\lvert\sum\Delta k_e\rvert/\sum k_{ZBS}$ | 5.01% | 5.44% |

Table 3.2 also shows that using Eq. (3.1) to approximate the solid thermal conductivity gives a deviance from the ZBS model of approximately 5 %.

Fig 3.5 and 3.6 shows the measured effective thermal conductivity of two fillings of Cu-btc MOF filled with helium and nitrogen, respectively. In these figures, the error bars represents the 5% error that comes with the measurements. The measurements are performed at a constant pressure of 1.2 bar, except the Cu-btc experiment with He, which is at 1.4 bar. The difference between the measurements at 1.2 bar and 1.4 bar is much lower than the uncertainty of the measurement itself (see Appendix D), and it should therefore not be a problem comparing $N_2$ and He experiments performed at 1.2 bar and 1.4 bar, respectively. All theoretical models in Fig. 3.5 through Fig. 3.8 are based on the solid conductivity obtained from the fitting (Eq. (3.1) and (3.2); Table 3.2).
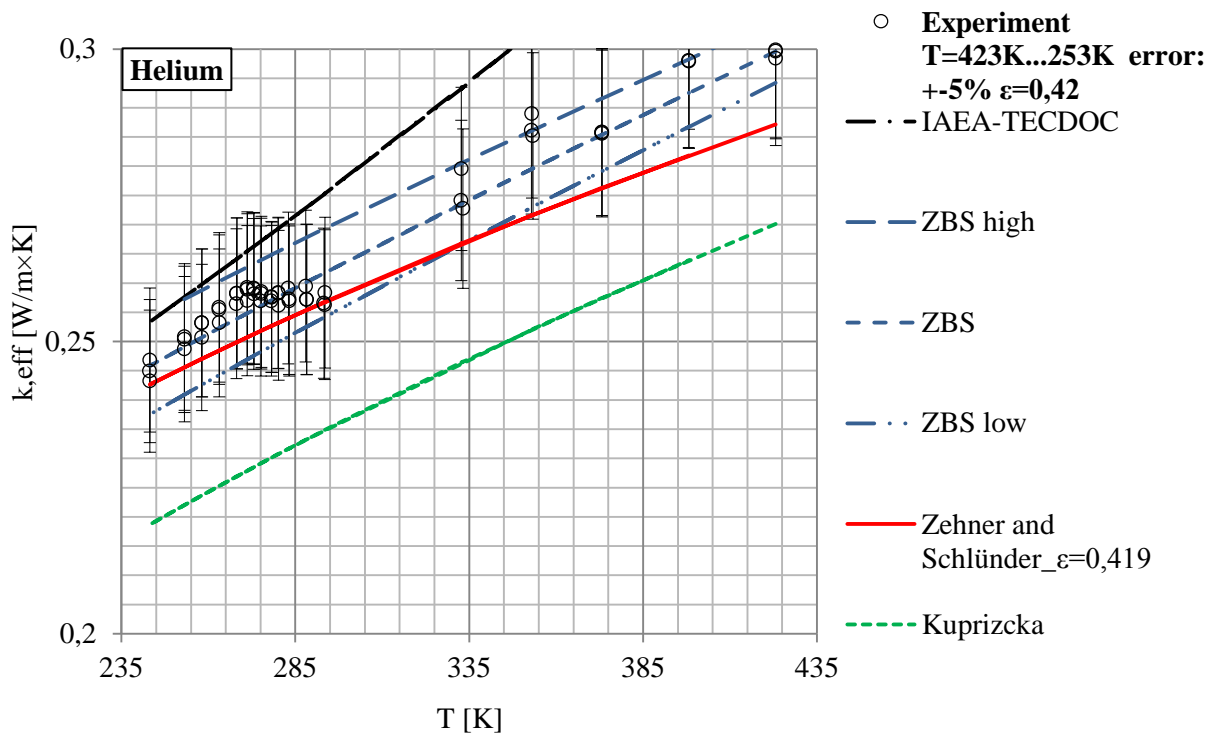


Fig. 3.5 - Effective thermal conductivity of Cu-btc with helium (He) gas at 1.4 bar absolute pressure.

In figures 3.5 and 3.6, all the measured values of the effective thermal conductivity are within the relative uncertainty bounds of the ZBS model. The figures show a discrepancy between the effective thermal conductivity of the bulk filled with helium and the one filled with nitrogen. This is to be expected, since the thermal conductivity of helium is so much larger than nitrogen (ref. Fig. 1.4).
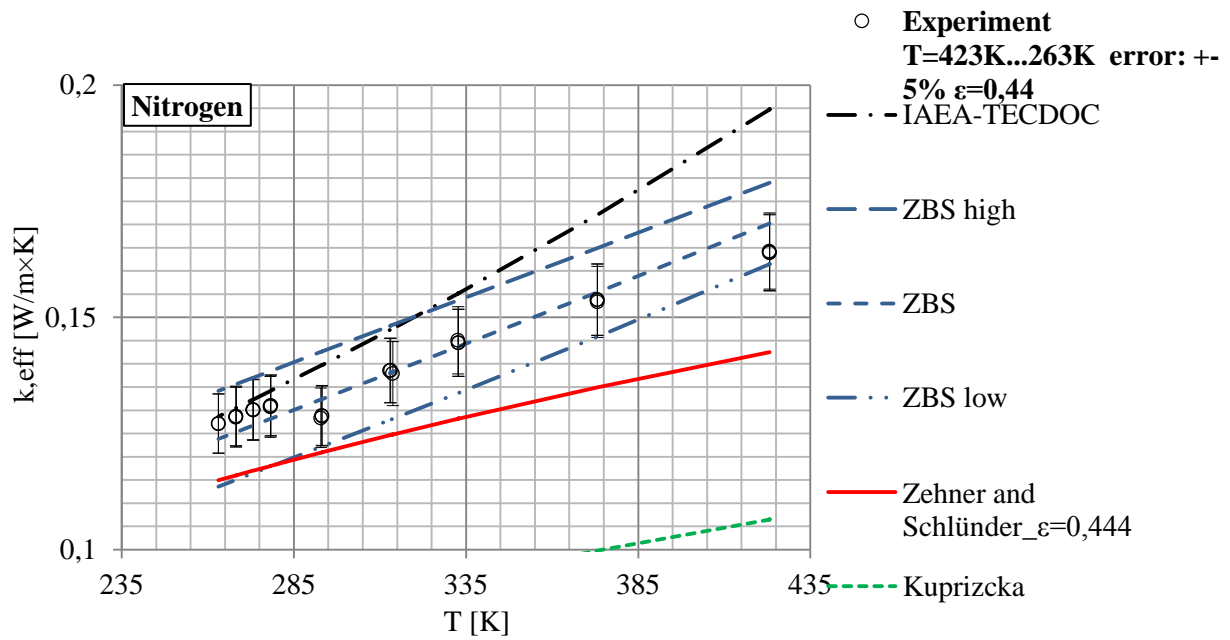
Fig. 3.6 - Effective thermal conductivity of Cu-btc with nitrogen ($N_2$) gas at 1.2 bar absolute pressure.

Fig. 3.5 and 3.6 also show that the experimental data correspond very well with the slope of the ZBS-model curve. This indicates that the temperature dependent function for the solid conductivity has a correct relation between the solid thermal conductivity $k_{so}$ at 273K and the temperature T.

Furthermore, the discontinuity of the effective thermal conductivity around $T$=273K is still there. This continuity showing up in all measurements indicates that it is in fact due to a computational error from the TPS Hot Disk software. The results from the Fe-btc also show this same behavior.

Figures 3.7 and 3.8 show the results for the effective thermal conductivity of Fe-btc with helium and nitrogen gas, respectively.
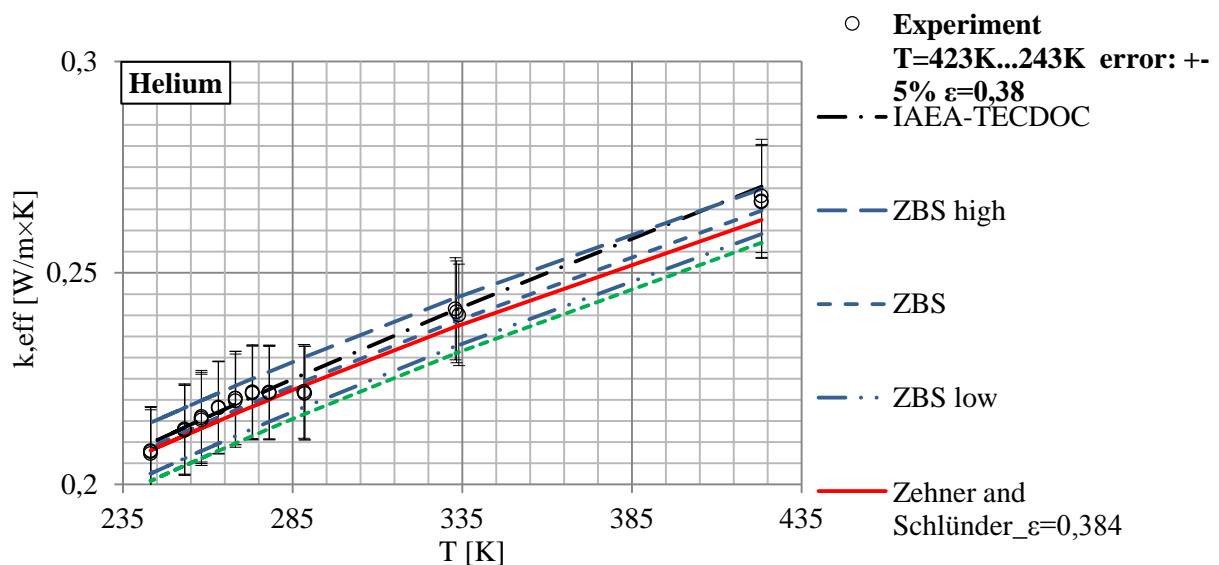


Fig. 3.7 - Thermal conductivity of Fe-btc with helium (He) gas.
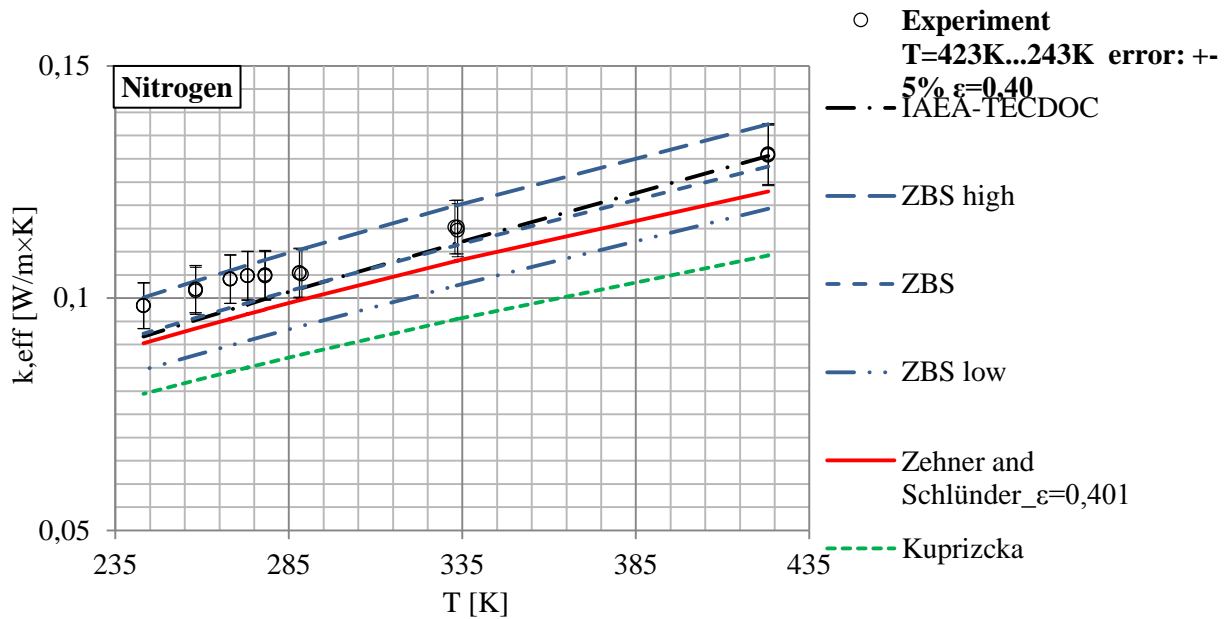
Fig. 3.8 - Thermal conductivity of Fe-btc with nitrogen (N₂) gas.

As for the Cu-btc, the measured effective thermal conductivities of Fe-btc shown in Figures 3.7 and 3.8 are also within the relative uncertainty bounds of the ZBS model. The slope of the experimental data fits very well with the ZBS model for Fe-btc as well.

However, comparing the predictions for the effective thermal conductivity of Cu-btc and N₂ (Fig. 3.6) with the predictions of Fe-btc with N₂ in (Fig. 3.8) shows that Cu-btc predictions has a greater discrepancy between the ZBS and the IAEA-TECDOC model as the temperature increases. The trend is also shown when comparing the predictions with helium gas. The fact that the gas does not affect this tendency suggests that it is due to the difference in material structure. Also, the fact that the discrepancy increases with temperature suggests that it is due to the radiation term of each of the models being different. Looking at the equations for the respective models (part 1, section 1.7.2 and 1.7.3) gives a perfectly good explanation:

Both models require the use of the deformation factor $B$ from Eq. (1.55). However, the constant $C$ in the deformation factor term is 2.5 for cylindrical particles (Cu-btc), 1.4 for broken particles (Fe-btc) and 1.25 for spherical particles. Investigating the works on the IAEA-TECDOC radiation term (Eq. (1.66)) from the original author reveals that the term was derived under the assumption that the particles are spherically shaped (Breitbach and Barthels, 1980). Hence, they only allow for $C$ to be equal to 1.25. With the deformation factor being larger for Cu-btc particles, the last term inside the bracket of the IAEA-TECDOC radiation term will result in a radiation term of greater magnitude than that of the ZBS model (Eq. 1.56). The particle diameter of Cu-btc is approximately 3.76/0.43≈8 times greater than that of Fe-btc. Since the IAEA-TECDOC radiation term is multiplied with the particle diameter $d_p$ and $T^3$ this effect will increase with temperature. This shows that this model is actually not applicable for the purpose of this work. The ZBS model however, provide accurate predictions for both broken and cylindrically shaped particles.

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A comparison of the predictions for the effective thermal conductivity from the ZBS model at 285K and 385K, for both Cu-btc (Fig. 3.5 and 3.6) and Fe-btc (Fig. 3.7 and 3.8), is shown in Table 3.3.

Table 3.3 - The increase of the effective thermal conductivity when switching from $N_2$ to He.

|  | Cu-btc | | | Fe-btc | | |
|---|---|---|---|---|---|---|
|  | N2 | He | Increase | N2 | He | Increase |
| $k_{ZBS}$ at 285K | 0,13 | 0,26 | 100 % | 0,102 | 0,22025 | 116 % |
| $k_{ZBS}$ at 385K | 0,16 | 0,29 | 81 % | 0,132 | 0,2504 | 90 % |

As shown in the above table, a bulk of Fe-btc receives a higher increase in thermal conductivity than a bulk of Cu-btc when the gas is switched. This is the result of their respective solid conductivities having a different temperature variation as well as a different magnitude. The solid conductivity of Cu-btc is already much higher than that of Fe-btc, and this is why the effect of changing to a more conductive gas will not provide the same effect for the Cu-btc. The temperature variation of the solid thermal conductivity of both MOF materials is shown in Fig. 3.9 below.
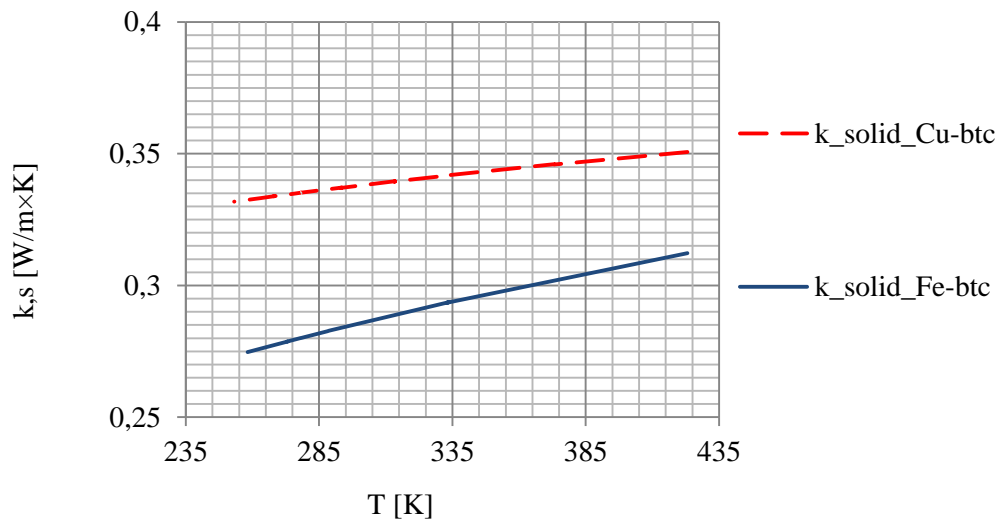


Fig. 3.9 - The variation of solid thermal conductivity of Cu-btc and Fe-btc as a function of temperature (Based on Eq. (3.1) and (3.2) with values from Table 3.2).

In Fig 3.9, the increase of solid thermal conductivity of Fe-btc is almost linear, while the Cu-btc thermal conductivity has a slight descending increase over the temperature range. The solid thermal conductivities of both Cu-btc and Fe-btc are fitted values from the ZBS model. The conductivity of Cu-btc is higher than Fe-btc. This is to be expected, since Cu-btc is copper-based and thus generally should have better heat transfer capabilities than an iron-based substance. However, confirming that the obtained relations give the true values for the thermal conductivity requires more experimental data from various gases.

The magnitude of the effective thermal conductivity of a packed bed of MOF material is not very large. Even though changing gas from nitrogen to helium provides approximately a 100% increase, the aluminum foam supposedly increases the effective thermal conductivity by two orders of magnitude.

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At the time of writing this, there was not enough time available to test the performance of the aluminum foam together with a MOF material. However, experiments were made on foam filled with glass beads and nitrogen gas.

## 3.3 Foam effect

Experiments on an aluminum open-cell foam were conducted with nitrogen and glass beads. The results are given in full in Appendix G. The effective thermal conductivity of a packed bed with glass beads is in the magnitude of 0.2. According to the predicted values of the Phase-symmetry model (1994) and the model of Boomsma and Poulikakos (2000) showed in Fig 1.24, the magnitude of the effective thermal conductivity of the bed should increase approximately 50 times when applying a high-porosity aluminum foam. The experiments conducted by this work, plotted in Fig. 3.10, shows that this is not the case.
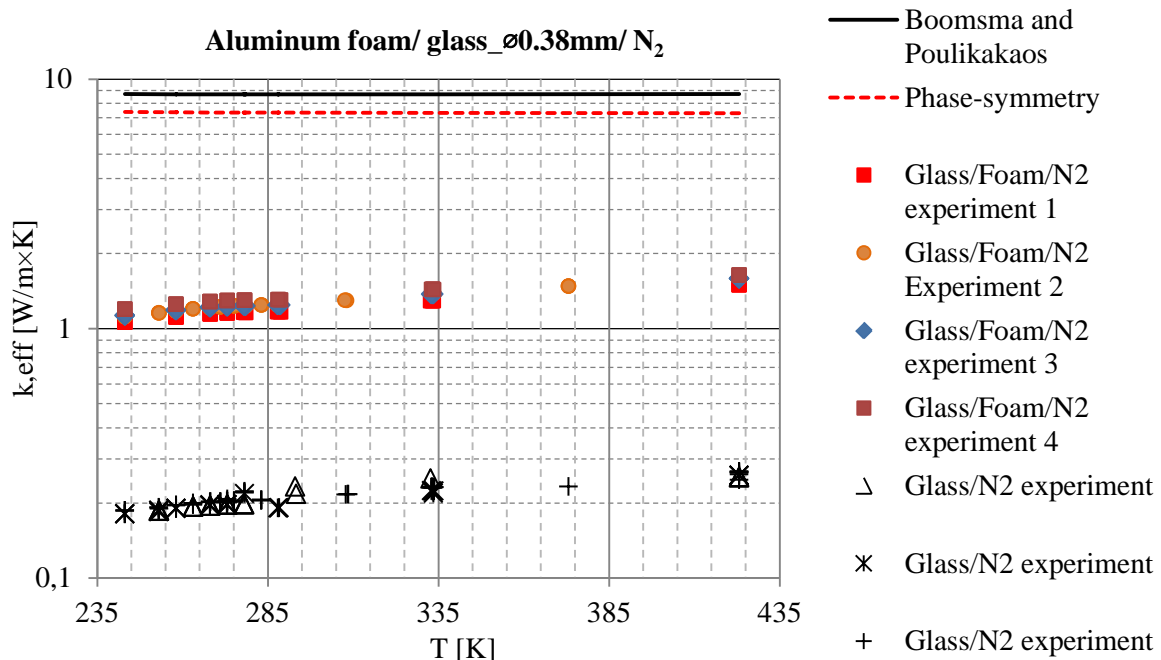


Fig. 3.10 - Top: Predictions of the effective thermal conductivity of glass/foam/$N_2$.
Middle: Measured effective thermal conductivity of glass/foam/$N_2$.
Bottom: Measured effective thermal conductivity of glass/$N_2$.

In Fig. 3.10, the middle placed colored markers show the results for the effective thermal conductivity of a bulk containing a high-porosity aluminum foam filled with small glass beads (⌀0.38mm) and nitrogen gas. The bottom results (black, non-filled markers) show the effective thermal conductivity of glass beads with nitrogen only. At the top the model of Boomsma and Poulikakos (2000) (with shape-parameter e=0.18) predict a thermal conductivity of approximately constant magnitude of 9 W/m·K. The Phase-symmetry-model of Hsu et al. (1994) predict a little lower, the effective thermal conductivity being constant at approximately 7 W/m·K. The conductivity of aluminum is descending from 235 to 435 K, contrary to other substances such as the glass beads and nitrogen. The theoretical models show that the descending conductivity of aluminum counteracts the ascending conductivities of the other substances, creating almost linear predictions for the effective thermal conductivity.

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Fig. 3.10 clearly shows that both the experiments with and without foam have the same ascending tendency over the temperature range, suggesting that in reality the foam does not affect the thermal conductivity to the same extent as predicted in theory.

Regarding the experimental results, Fig. 3.10 shows that the use of this particular aluminum foam only increases the effective thermal conductivity about 6 times, compared to approximately 50 times for the theoretical models. The magnitude of the effective thermal conductivity of the foam with small glass beads and nitrogen gas ranges from 0.18 to 0.25 W/m·K over the temperature range. Previous experiments performed on aluminum-based foam (AlSi$_7$Mg, $k_s \approx 160$W/m·K) also show this same magnitude for the effective thermal conductivity (Inger-Anne Rasmussen, 2011).

What is now clear is that the theoretical models are not able to predict the effective thermal conductivity correctly for these particular foams. This could be the result of two different scenarios:

1.  The theoretical models dos not give an accurate prediction for foam-like structures.
2.  The measurements are wrongly executed.

According to other authors, the effective thermal conductivity of pure-aluminum foam ($\varepsilon$=0.937) filled with air should be in the magnitude of 4.5 W/m·K (Valmidi, 1999). This indicates that both of the above scenarios are likely to be true. On this basis the decision was made, to modify the phase-symmetry model of Hsu et al. (1994) and compare this to a new set of foam experiments. This model will be presented in part 4.

## Part 4 - Modified phase-symmetry model for aluminum foam

In this part, a modification to the phase-symmetry model of Hsu et al. (1994) described in section 1.8.1 will be presented. The original model only assumes phase symmetry and a continuous solid and fluid phase, and does not take into account the extra distance that the heat must travel due to the structure of the foam. In the present approach, this effect will be included by the introduction of a dimensionless geometrical tortuosity factor.

A new set of experiments on the same aluminum foam ($\varepsilon$=0.93) with $\varnothing$0.38mm glass beads and air as fluid were performed. These experiments were carried out with an external force exerted on the foam in order to ensure good contact between the foam and the Hot Disk sensor. In addition, the surfaces of the foam parts in contact with the sensor were finished using a wide belt finishing machine.

### 4.1 Model development

The unit cell from the original phase-symmetry model is pictured in Fig. 1.20. The unit cell of the new modified model is depicted below.
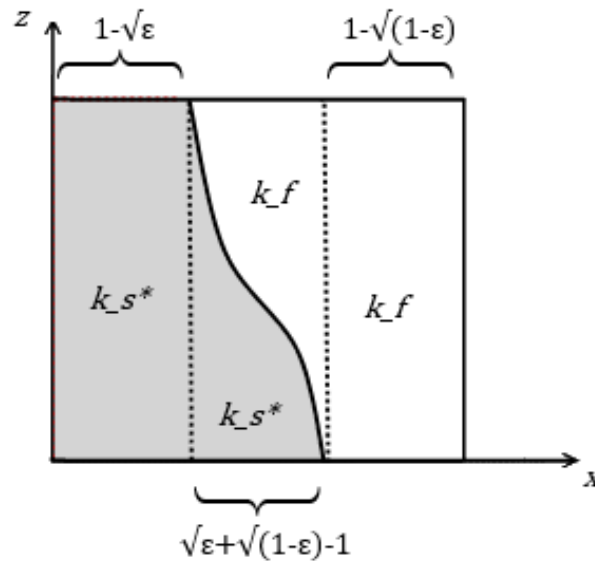


Fig. 4.1 - Unit cell for the modified phase-symmetry model.

Fig 4.1 shows the unit cell for the modified phase symmetry model. In the figure, $k_s^*$ is the solid conductivity to be modified. The phase symmetry model can then be expressed as follows (Hsu et al, 1994):

$$k_e = (1 - \sqrt{1-\varepsilon})k_f + (1 - \sqrt{\varepsilon})k_s^* + (\sqrt{1-\varepsilon} + \sqrt{\varepsilon} - 1)k_{sf}, \qquad (4.1)$$

where $k_{sf}$ is the equivalent thermal conductivity of the composite layer, which can be expressed as

$$k_{sf} = k_f \left[ \frac{b(1-\lambda)}{(1-\lambda b)^2} \ln\left(\frac{1}{\lambda b}\right) - \frac{b-1}{1-\lambda b} \right], \tag{4.2}$$

with $\lambda = k_f/k_s^*$. The shape factor $b$ is the same as before (Eq. 1.73).

The path of heat transfer in the solid phase is then modified by a dimensionless tortuosity factor $\eta$. The relation between the modified solid conductivity and the regular solid conductivity can be described by the relation

$$k_s^* = \eta \cdot k_s, \tag{4.3}$$

where

$$\eta = \frac{pore\ diameter\ [m]}{acual\ heat\ transfer\ path\ [m]}. \tag{4.4}$$

In order to find the value of the tortuosity factor, one must take a look at the actual structure of the foam. The Hot Disk sensor measures the heat transfer in the axial and radial direction. As demonstrated in Fig 4.2 below, the 2-dimensional pore structure in the radial and axial direction has the shape of a hexagon and pentagon, respectively.
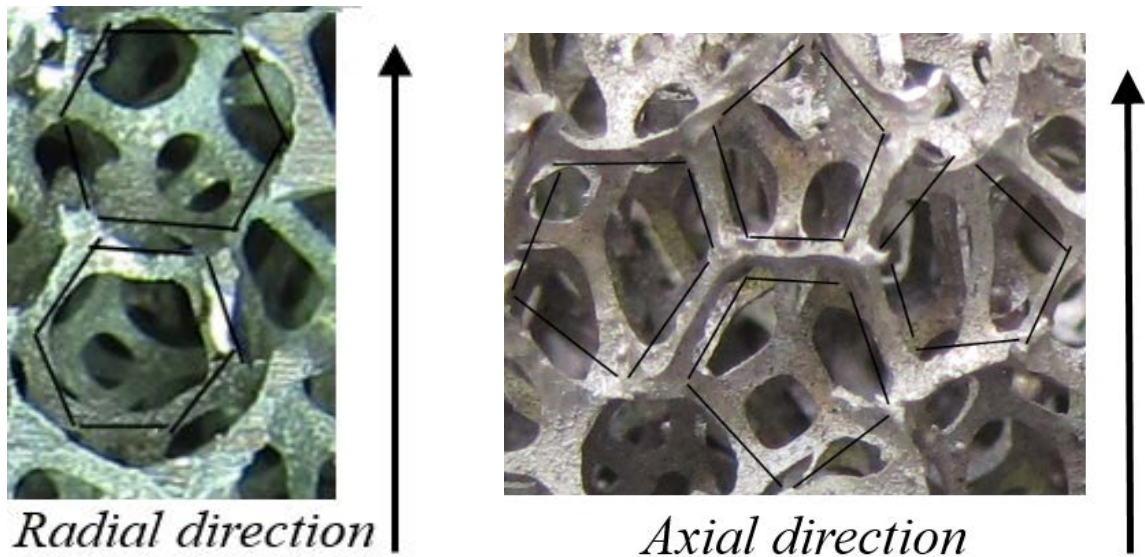


Fig. 4.2 - The pore structure visualized.

Hence, the ratio of the pore diameter to the actual heat transfer path in 2 dimensions can be approximated as shown in Fig. 4.3 below. The original phase symmetry model does not take into account the path of heat transfer, it only accounts for the ratio between the respective phases.
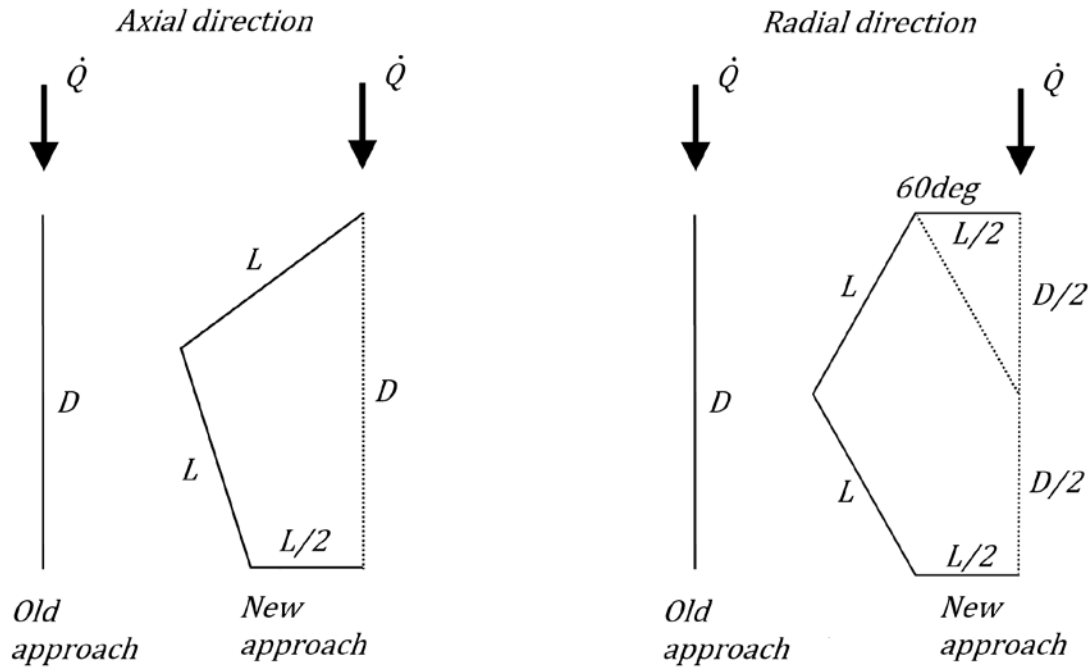
Fig. 4.3 - Actual heat transfer path and the heat transfer path of the original phase-symmetry model.

In Fig. 4.3, $D$ is the pore diameter, and $L$ is the length of the side of the pentagon and hexagon. The influence of the pore size diameter can be neglected due to the fact that the ratio of the pore diameter to the actual heat transfer path will be se same regardless of a change in the pore diameter. The heat transfer path in the axial direction will be half of a pentagon circumference, while the path in the radial direction will be the circumference of half a hexagon. Thus, the tortuosity factor in the axial direction can be expressed as

$$\eta_{high} = \frac{D}{2.5L} = \frac{\frac{L}{2}(\sqrt{5}+1)}{2.5L} = \frac{1}{5}(\sqrt{5}+1) \approx 0.647, \tag{4.5}$$

while the tortuosity factor in the radial direction is

$$\eta_{low} = \frac{D}{3L} = \frac{1}{3}\tan(60°) \approx 0.557. \tag{4.6}$$

These values for $\eta$ will provide a lower and upper band for the new phase symmetry model. Note that this approach involves the use of the ZBS model on the glass beads and the air, in order to gain an effective thermal conductivity for the *phase* that the foam is filled with. An overview of this approach is given in Table 4.1 below.

Table 4.1 - New foam approach overview.

| Model applied | Effective thermal conductivity | Input parameters | | |
|---|---|---|---|---|
| | | porosity | fluid | solid |
| Phase-symmetry | $k_e$ | $\varepsilon_{foam}$ | $k_{ZBS}$ | $k_s* = \eta k_s$ |
| Zehner/Bauer/Schlünder | $k_{ZBS}$ | $\varepsilon_{glass}$ | $k_{air}$ | $k_{glass}$ |

A comparison with the original phase symmetry model and the model of Boomsma and Poulikakaos (2000) (e=0.18) is given in Fig. 4.4 below.
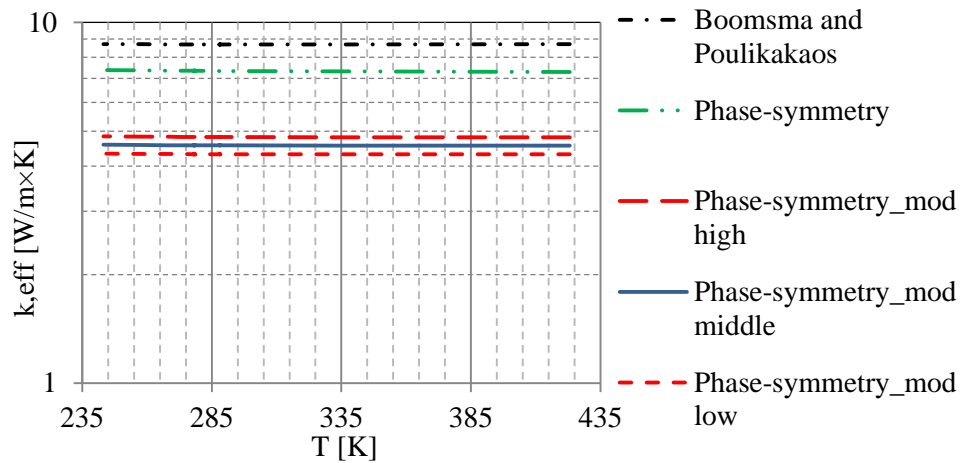


Fig. 4.4 - The modified phase symmetry model compared with the original.

Fig. 4.4 shows the prediction for the effective thermal conductivity of various theoretical models plotted versus temperature at 1.2 bar absolute pressure. The bed consists of aluminum foam ($\varepsilon$=0.93) with glass beads and air as fluid. The figure shows that the modified phase-symmetry model predicts the effective thermal conductivity to be in the magnitude of 4.7 W/m·K. This is very close to the experiments of Valmidi (1999), which had a thermal conductivity of 4.5 W/m·K ($\varepsilon$=0.937, 1 bar, air).

## 4.2 Comparison with experimental results

In a late stage of this study, experiments were performed on foam with small glass beads and air as fluid. The experiments were performed at 298 K and ambient pressure. Pictures of the setup are provided in Fig. 4.5.
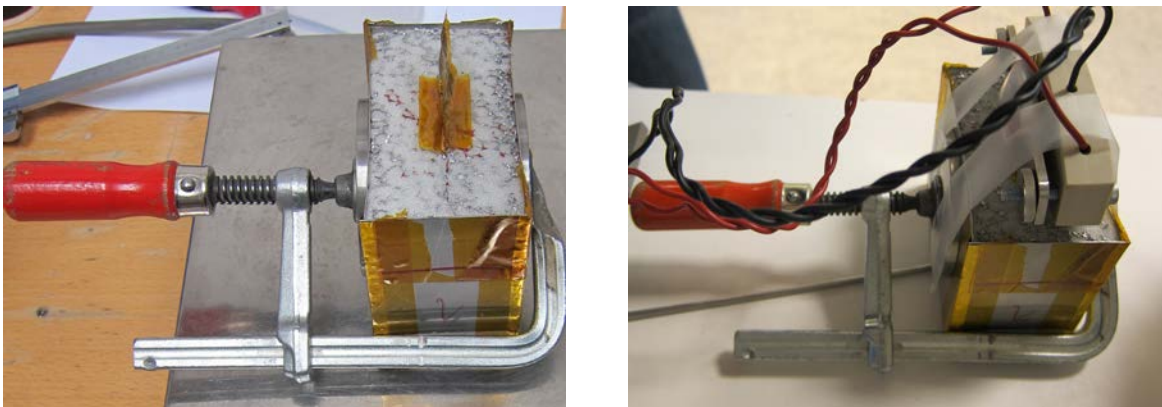


Fig. 4.5 - New measurement setup

The new experiments were conducted due to a suspicion that the previous experiments gave low values for the effective thermal conductivity, as shown in Fig. 3.10. Fig. 4.5 gives a visualization of the new experimental setup, and shows that the foam is subjected to compression in the axial direction, in order to reach an acceptable contact surface between the foam and the sensor.

The Hot Disk software enables both isotropic and anisotropic measurements. The anisotropic measurement module provides values for the effective thermal conductivity in both the radial and axial directions. The effective thermal conductivity of an anisotropic measurement is then given as (M. Gustavsson, 1994)

$$k_e = \sqrt{k_a \cdot k_r}, \tag{4.7}$$

where $k_a$ and $k_r$ is the measured thermal conductivity of the bed in the axial and radial direction, respectively. Contrary to the isotropic method, the anisotropic measurement method requires the input of the bed volumetric specific heat capacity $\rho c_p$ [MJ/m³K]. This was estimated to be 1.35 MJ/(m³K).

The experiments were performed once, and then the pressure was increased. This process was then repeated two times over. The experimental data are given in Table 4.2. Some of these data are plotted in Fig. 4.6 and 4.7.
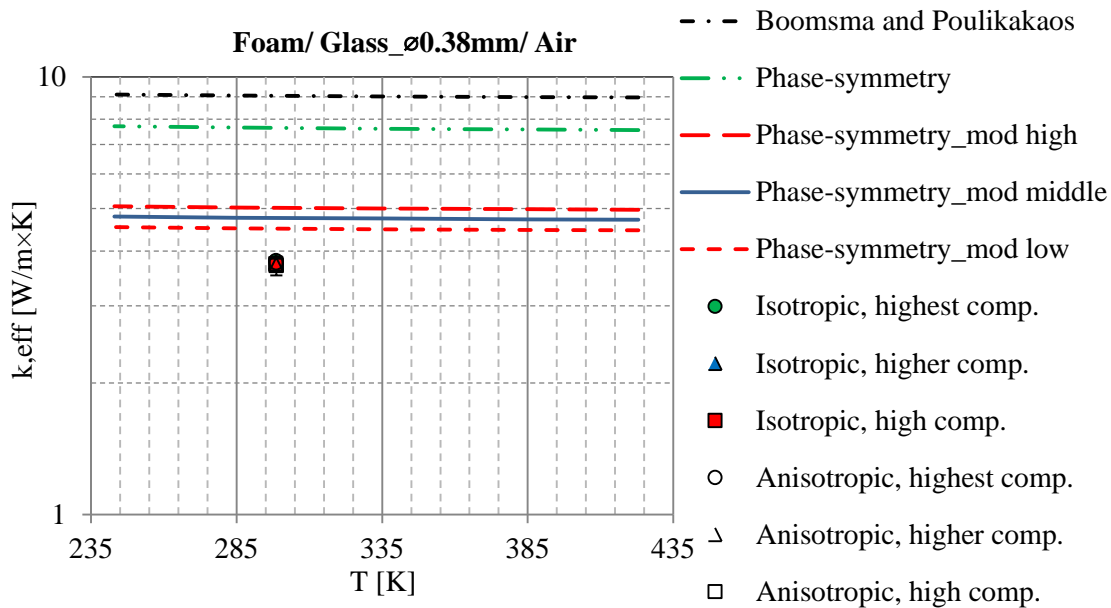


Fig. 4.6 - New foam measurements performed at 298K plotted versus the theoretical models.

Fig 4.6 shows the theoretical models plotted over the whole temperature range, and the experimental results from both isotropic and anisotropic measurements performed at 298K. A more close-up plot is given in Fig. 4.7. The magnitudes of the exerted pressure on the foam were not known, and therefore the exerted force is denoted as from high to the highest compression available. Fig. 4.6 show however little difference between the measurements, and according to Table 4.2 the variation from the lowest to the highest measured effective thermal conductivity is as little as 3%. The experimental data is approximately 20% lower than the middle bound of the new modified phase-symmetry model (blue, solid line). The reason for this will be discussed further down.

Table 4.2 - Experimental data from the new foam measurements.

**Aluminum foam/Glass_⌀0.38mm/ Air**

| ε_tot | 0,36309 | R | 8314 | e | 0,174667 |
| ε_spec | 0,39 | Cp_gas | 1009 | η_high | 0,647214 |
| ε_foam | 0,931 | dp | 0,00038 | η_low | 0,57735 |

| T | Cp_mes. | k_measured | | | k_f | k_s_glass | k_ZBS | k_s_alu | k_phase-sym_mod | | | Exp. | Compression. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Effective | Axial | Radial | | | | | High bound | Low bound | Middle | method | |
| [K] | [MJ/m³K] | [W/m·K] | [W/m·K] | [W/m·K] | [W/m·K] | [W/m·K] | [W/m·K] | [W/m·K] | [W/m·K] | [W/m·K] | [W/m·K] | | |
| 298,65 | 1,348217 | 3,70654 | 3,162517 | 4,344148 | 0,0263 | 1,099465 | 0,189279 | 211,7847 | 5,024172 | 4,504187 | 4,764185 | Aniso. | High |
| 298,65 | 1,348217 | 3,728154 | 3,241302 | 4,288132 | 0,0263 | 1,099465 | 0,189279 | 211,7847 | 5,024172 | 4,504187 | 4,764185 | Aniso. | Higher |
| 298,65 | 1,348217 | 3,753695 | 3,361703 | 4,191396 | 0,0263 | 1,099465 | 0,189279 | 211,7847 | 5,024172 | 4,504187 | 4,764185 | Aniso. | Highest |
| 298,55 | 1,134521 | 3,733641 | - | - | 0,0263 | 1,099277 | 0,189265 | 211,7847 | 5,024154 | 4,50417 | 4,764168 | Iso. | High |
| 298,55 | 1,160301 | 3,766763 | - | - | 0,0263 | 1,099277 | 0,189265 | 211,7847 | 5,024154 | 4,50417 | 4,764168 | Iso. | Higher |
| 298,55 | 1,155828 | 3,78877 | - | - | 0,0263 | 1,099277 | 0,189265 | 211,7847 | 5,024154 | 4,50417 | 4,764168 | Iso. | Highest |
| 298,55 | 1,162978 | 3,798265 | - | - | 0,0263 | 1,099277 | 0,189265 | 211,7847 | 5,024154 | 4,50417 | 4,764168 | Iso. | Highest |
| 298,55 | 1,145366 | 3,792659 | - | - | 0,0263 | 1,099277 | 0,189265 | 211,7847 | 5,024154 | 4,50417 | 4,764168 | Iso. | Highest |
| 298,55 | 1,15525 | 3,790111 | - | - | 0,0263 | 1,099277 | 0,189265 | 211,7847 | 5,024154 | 4,50417 | 4,764168 | Iso. | Highest |
| 298,55 | 1,162441 | 3,788542 | - | - | 0,0263 | 1,099277 | 0,189265 | 211,7847 | 5,024154 | 4,50417 | 4,764168 | Iso. | Highest |
| 298,55 | 1,142308 | 3,779273 | - | - | 0,0263 | 1,099277 | 0,189265 | 211,7847 | 5,024154 | 4,50417 | 4,764168 | Iso. | Highest |
| 298,55 | 1,149854 | 3,802789 | - | - | 0,0263 | 1,099277 | 0,189265 | 211,7847 | 5,024154 | 4,50417 | 4,764168 | Iso. | Highest |
| 298,55 | 1,153614 | 3,793865 | - | - | 0,0263 | 1,099277 | 0,189265 | 211,7847 | 5,024154 | 4,50417 | 4,764168 | Iso. | Highest |
| 298,55 | 1,163294 | 3,790825 | - | - | 0,0263 | 1,099277 | 0,189265 | 211,7847 | 5,024154 | 4,50417 | 4,764168 | Iso. | Highest |
| 298,55 | 1,159858 | 3,80246 | - | - | 0,0263 | 1,099277 | 0,189265 | 211,7847 | 5,024154 | 4,50417 | 4,764168 | Iso. | Highest |
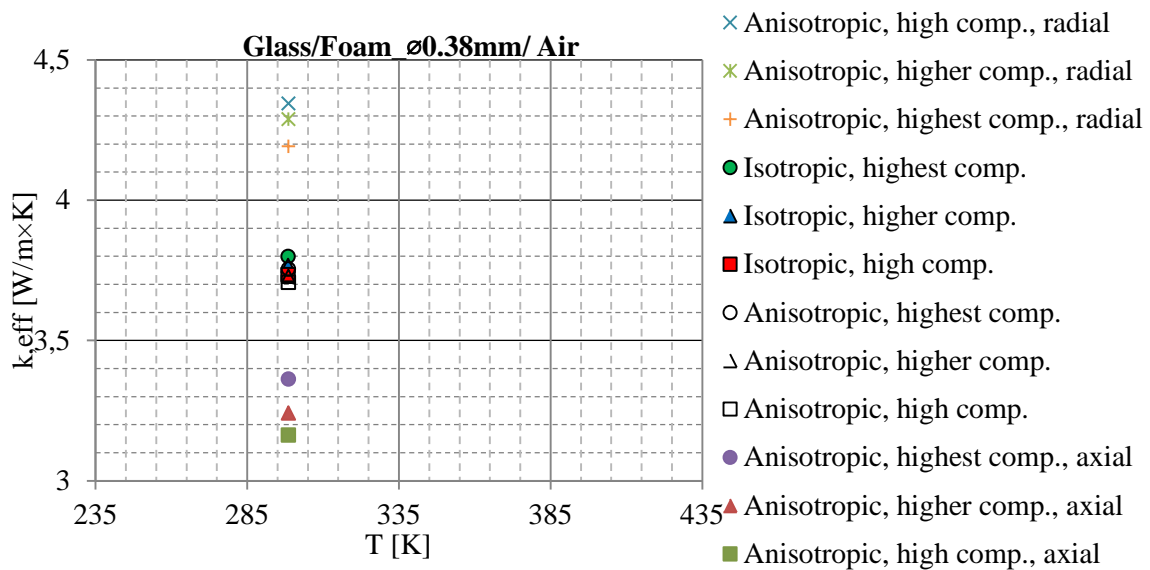
Fig. 4.7 - Anisotropic and isotropic effective thermal conductivity measurements of aluminum foam at 298K and ambient pressure. The foam porosity is $\varepsilon$=0.93.

Fig 4.7 shows the same experimental data as Fig. 4.6, and in addition it shows the axial and radial components of the anisotropic measurements. The Fig. 4.7 shows, that if the compression is increased (from high$\rightarrow$highest) the thermal conductivity increases. This is to be expected, since the thermal contact between the sensor and the foam will be increased, and thus increasing the quality of the measurement and thereby also the conductivity. Beside this, two conclusions can be drawn from this plot:

1.  The radial component of the thermal conductivity (Fig. 4.7, Top three markers) does not increase when compression is increased. Due to the improvement in contact area between the sensor and the foam, the thermal conductivity should therefore increase when applying higher compression. However, this was not the case. A possible explanation is that the compression that is exerted on the foam in the axial direction will *increase* the heat transfer path in the radial direction, and thus decreasing the thermal conductivity in the radial direction. In the axial direction it will be the other way around.

2.  The magnitude of the axial thermal conductivity component should, according to the new modified phase-symmetry model, be higher than the radial thermal conductivity. An example is given in Table 4.3.

Table 4.3 - Radial and axial thermal conductivity.

| Compression | k_Experiment [W/m·K] | | k_Phase-sym_mod [W/m·K] | |
|---|---|---|---|---|
| | Axial | Radial | High | Low |
| High | 3,163 | 4,344 | 5,024 | 4,504 |
| Higher | 3,241 | 4,288 | 5,024 | 4,504 |
| Highest | 3,362 | 4,191 | 5,024 | 4,504 |

The values for the thermal conductivity given in Table 4.3 suggest that the axial component should be in the magnitude of approximately 5 W/m·K. This implies that either the pentagon is not a satisfactory way to model the pore structure in the axial direction, or the contact area between the sensor and the aluminum foam needs to be improved even further. Either way, this is an issue that needs to be examined through further experimental investigations. The reduction in axial conductivity could also be due to bubble-like structures inside the foam, as shown in Fig. 4.8 below.
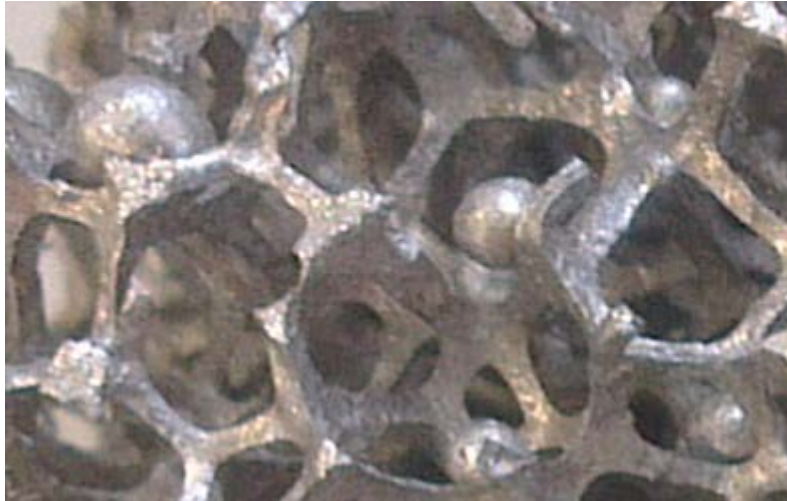


Fig. 4.8 - A close-up of the aluminum foam applied in the experiments.

Fig. 4.8 also shows signs of damage on the ligaments. Some of them may be broken, and thus extending the path of heat transfer by conduction through the foam. The foam consists of pure aluminum, whose solid conductivity is very high in ambient conditions (211.79 W/m·K at 298K; (NIST Chemistry Webbok)). The bubbles in Fig. 4.8 may have been formed during the foam production process, and although the bubbles consist of pure aluminum, they do not contribute to heat transfer in either direction. The bubbles are in fact only taking up space inside the bed, and this can be modeled theoretically with the introduction of a non-conducting foam porosity $\varepsilon_{nc}$ which can be related to the conducting foam porosity $\varepsilon_{cond}$ through the relation

$$\varepsilon_{foam} = \varepsilon_{nc} + \varepsilon_{cond}. \tag{4.8}$$

This introduction implies that $\varepsilon_{foam} < \varepsilon_{nc}$. At the moment of this discovery there was not enough time to address this issue, however it is an issue that needs to be investigated further.

Summing up the experiments conducted on aluminum foam, the conclusion is that with the current measurement techniques one can expect an increase in thermal conductivity at 298 K and ambient pressure from 0.22 W/m·K to approximately 3.7 W/m·K with the use of a high porosity ($\varepsilon$=0.93) aluminum foam. The magnitude increases by a factor of 17. However, the new theoretical model which was developed suggests that the thermal conductivity should be in the magnitude of 4.7 W/m·K. The discrepancy between theory and experimental results therefore needs to be addressed in future works.

# Part 5 - Conclusions

## 5.1 Conclusion

The goal of this work was to determine the effective stagnant thermal conductivity of the MOF materials Cu-btc and Fe-btc, and as well examine the effect of introducing heat transfer enhancement structures such as aluminum foam or wires. Furthermore, it was important to confirm the validity of the Hot Disk TPS measurement setup.

First, experiments were performed on glass beads ($\varnothing$0.38mm and $\varnothing$1.395mm), which served as a reference material, in order to understand the setup and find a suitable correlation for determining the solid conductivity of the MOF. The model of Zehner/Bauer/Schlünder (ZBS) was chosen for this task because of its best fit with the glass beads experimental data.

In addition to the 5% uncertainty on each measured data point come the uncertainties of the ZBS model, due to the input of measured parameters such as porosity. The uncertainty analysis revealed that the experimental data fitted within the uncertainty bounds of the ZBS model, which were approximately ±10%. Adding the uncertainty of the ZBS model to the uncertainty of the experiments gives a total uncertainty of 15% from the glass beads experiments.

Solid conductivity of MOF materials Fe-btc and Cu-btc were then successfully determined. Temperature dependent functions for the solid conductivity were obtained through a least square procedure with the ZBS model. The obtained solid conductivity can be expressed by the function

$$k_s = k_{s0} \left( \frac{T}{T_0} \right)^n, \tag{5.1}$$

with the values given in Table 5.1 below. This function is valid in the range of 240K$< T <$ 420 K.

Table 5.1 - Optimized constants

|  | Cu-btc | Fe-btc |
|---|---|---|
| $k_{s0}$ | 0.33 | 0.28 |
| $n$ | 0.11 | 0.26 |
| $\lvert\sum\Delta k_e\rvert/\sum k_{ZBS}$ | 5.01% | 5.44% |

Also, a modified phase-symmetry model for determination of the effective stagnant thermal conductivity for high-porosity metal foams was developed. The model was obtained by modifying the model of Hsu et al. (1994) with the introduction of a dimensionless tortuosity factor accounting for extended heat transfer path through the solid structure. The effective stagnant thermal conductivity is given as

$$k_e = (1 - \sqrt{1 - \varepsilon})k_f + (1 - \sqrt{\varepsilon})k_s^* + (\sqrt{1 - \varepsilon} + \sqrt{\varepsilon} - 1)k_{sf}, \tag{5.2}$$

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with

$$k_{sf} = k_f \left[ \frac{b(1-\lambda)}{(1-\lambda b)^2} \ln\left(\frac{1}{\lambda b}\right) - \frac{b-1}{1-\lambda b} \right], \tag{5.3}$$

where

$$b = \left(\frac{1-\varepsilon}{\varepsilon}\right)^{0.9676}, \tag{5.4}$$

$$\lambda = k_f/k_s^*, \tag{5.5}$$

and

$$k_s^* = \eta \cdot k_s. \tag{5.6}$$

The tortuosity factor $\eta$ is given to be

$$\eta = \begin{cases} 0.647; axial\ direction \\ 0.557; radial\ direction \end{cases} \tag{5.7}$$

The $\eta$-value in the axial and radial direction result in a high and low band for the effective thermal conductivity. The middle bound of this model is expressed as

$$k_e = \sqrt{k_{e,a} \cdot k_{e,r}}, \tag{5.8}$$

where $k_{e,a}$ and $k_{e,r}$ is the effective thermal conductivity of the bed in the axial and radial direction, respectively. This modified phase-symmetry model is applicable for high porosity metal foams, and gives values for the effective thermal conductivity in the magnitude of 4.5 W/m·K at 298 K and ambient pressure.

Experimental investigations on aluminum foam filled with glass beads and air (298K, 1 bar) show that the foam increases the effective thermal conductivity of the bed from 0.22 W/m·K to 3.7W/m·K. According to the newly developed model, as well as other authors (Valmidi, 1999), this value should be ~4.5 W/m·K for this type of foam. This therefore needs to be investigated further.

Overall, the Hot Disk measurement setup provides accurate measurements of the effective thermal conductivity of porous materials. However, due to a calibration error, the setup show a discontinuity in the effective thermal conductivity in the range of 273<$T$<283K. Moreover, during measurements of heat distribution structures, it was discovered that the contact between the structure and the sensor needs to be as high as possible.

## 5.2 Further work

Conducting measurements with glass beads as a reference material proved somewhat problematic, because no data existed on the thermophysical properties of the exact composition of the glass melt applied. In order to ensure a total uncertainty below 10%, a pure substance whose thermophysical properties are well-known should be applied in future investigations.

Furthermore, the following improvements should be made of the measurement setup:

- The reason for the silicon seepage into the vacuum/pressure cell should be investigated. A new sealing or another type of vacuum grease should be applied.
- The discontinuity in the effective thermal conductivity in the range of 263K<T<283K needs to be investigated further.
- A cryo-probe should be installed and calibrated, in order to perform experiments on MOF and aluminum foam in temperatures below 240K – the actual working range of a cryo-adsorption hydrogen storage.

Also, further experiments, both isotropic and anisotropic, should be performed on various metal foams. The measurement techniques used in this work gave values for the foam effective thermal conductivity up to ~3.7 W/m·K. The theory suggests that it should be in the magnitude of 4.7 W/m·K. Anisotropic measurements show that the axial thermal conductivity component is only ~3 W/m·K, while the model suggests that this value should be in the vicinity of 5 W/m·K. In order to find the reason for this discrepancy, new foam experiments with emphasis on improving the thermal contact between the Hot Disk sensor and the sample should be performed. Moreover, a larger sensor can be applied to reduce the structural influences of the foam. However, this may also require that the sample size needs to be increased.

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## Appendix A - Particle diameter

Particle diameter of small glass beads are given in Fig- A-1. This particle diameter was used for all measurements with the small glass beads. This also applies for the larger glass beads, whose sieving results are displayed in Fig. A-2 The particle diameter for Fe-bc in fillings one and two is shown in Fig A-3, and the one in fillings three, four and five, is shown in Fig. A-4.

| Methode of sieving | | dry | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Size and shape of sieves | | round | 200 mm | | | | | | | | | | |
| Sieving medium : | | Woven wire cloth | | | | | | | | | | | |
| Sieves maked: | | DIN/ISO 3310-1 | 5 times | | | | | | | | | | |
| Duration of sieving | | 20 min +, five times | | | | | | | | | | | |
| Amplitude | | 0,6-0,8 | | | | | | | | | | | |
| Mass m_0 [g] | total | 620,3 | | Balance accuracy | | +- | 0,05 | [g] | | | Relative error | | |
| | mesh diameter | | | particle size sieves | | mean size | m_0 | m_1 | m_solid | dm/m | | m_sample | ∑m_sample/m | mi/m/dpi |
| | [µm] | | [µm] | | [µm] | [µm] | [g] | [g] | [g] | [%] | | [%] | |
| 1 | 0 | | 150 | >>d> | 0 | 75 | 1628,6 | 1628,4 | -0,2 | -25,00 % | | -0,03 | -0,03 | -4,299 |
| 2 | 150 | | 180 | >>d> | 150 | 165 | 1764,3 | 1764,5 | 0,2 | 25,00 % | | 0,03 | 0,00 | 1,954089 |
| 3 | 180 | | 200 | >>d> | 180 | 190 | 1886,2 | 1887,3 | 1,1 | 4,55 % | | 0,18 | 0,18 | 9,333345 |
| 4 | 200 | | 300 | >>d> | 200 | 250 | 1787,3 | 1845,3 | 58 | 0,09 % | | 9,35 | 9,53 | 374,0126 |
| 5 | 300 | | 355 | >>d> | 300 | 327,5 | 1870,6 | 2192,3 | 321,7 | 0,02 % | | 51,85 | 61,39 | 1583,573 |
| 6 | 355 | | 400 | >>d> | 355 | 377,5 | 2021,9 | 2215 | 193,1 | 0,03 % | | 31,12 | 92,52 | 824,6384 |
| 7 | 400 | | 500 | >>d> | 400 | 450 | 1906,8 | 1952,3 | 45,5 | 0,11 % | | 7,33 | 99,85 | 163,0036 |
| 8 | 500 | | 600 | >>d> | 500 | 550 | 1932,4 | 1932,4 | 0 | 0,00 % | | 0,00 | 99,85 | 0 |
| 9 | 600 | | | >>d> | 600 | 600 | 2009,5 | 2010,6 | 1,1 | 4,55 % | | 0,18 | 100,03 | 2,955559 |
| Original mass | [g] | 620,3 | | | | | | | | | ∑mi/m/dpi | | 2955,171 |
| Toal of fraction masses | [g] | 620,5 | +-0,45 | | | | | sauter diameter ds [m] = | | [∑mi/m/dpi]⁻¹ | | 0,000338 |
| Accumulated error | [g] | 0,073 % | | | | | | | | | | | |



Fig. A-1 Particle diameter of small glass beads.

| | sieving analysis 08.05.2013___ glass beads ROTH-A555.1___ 1.25<dp<1.65 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| m tot | 407,2 | | | | | | | | |
| siev | | | | | mean | m_o_siev | m_1_siev | mbed | mi/m/dpi |
| 1 | 0 | >> | 1000 | 500 | 381,3 | 381,3 | 0 | 0 |
| 2 | 1000 | >> | 1250 | 1125 | 433,2 | 433,3 | 0,1 | 0,218292949 |
| 3 | 1250 | >> | 1400 | 1325 | 387,6 | 415,6 | 28 | 51,89605961 |
| 4 | 1400 | >> | 1400 | 1400 | 479,4 | 858,4 | 379 | 664,8189728 |
| | | | | | | ∑mi/m/dpi | | 716,9333253 |
| | | | | | | particle diameter | (∑mi/m/dpi)^(-1) | 0,0013948 m |

Fig. A-2 Particle diameter of the larger glass beads.

| Material | Fe_btc | sieving 2 |
|---|---|---|
| Methode of sieving | dry | |
| Size and shape of testsievs | round | 200 mm |
| Sieving medium : | Woven wire cloth | |
| Sieves maked: | DIN/ISO 3310-1 | |
| Duration of sieving | 3 x 20 min | |
| Amplitude | 0,1-1 | |

| Mass m_0 [g] | 171,9 | Balance accuracy | +- | 0,05 | [g] |
|---|---|---|---|---|---|

| | | mesh diameter [µm] | particle size [µm] | | particle size [µm] | mean size [µm] | m_0_siev [g] | m_1_siev [g] | m_sample [g] | Relative error dm/m [%] | m_sample | ∑m_sampl [%] | mi/m/dpi |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 0 | 100 | >>d> | 0 | 50 | 381,3 | 381,3 | 0 | 0 % | 0 | 0,0 | 0 |
| 3 | 2 | 100 | 150 | >>d> | 100 | 125 | 348,4 | 348,6 | 0,2 | 25 % | 0 | 0,1 | 9,307737 |
| 3 | 3 | 150 | 200 | >>d> | 150 | 175 | 352,6 | 353,1 | 0,5 | 10 % | 0 | 0,4 | 16,62096 |
| 3 | 4 | 200 | 300 | >>d> | 200 | 250 | 356,9 | 360,5 | 3,6 | 1 % | 2 | 2,5 | 83,76963 |
| 3 | 5 | 300 | 400 | >>d> | 300 | 350 | 374,2 | 409 | 34,8 | 0 % | 20 | 22,7 | 578,4094 |
| 5 | 6 | 400 | 600 | >>d> | 400 | 500 | 369,6 | 484,7 | 115,1 | 0 % | 67 | 89,7 | 1339,151 |
| 4 | 7 | 600 | 630 | >>d> | 600 | 615 | 402 | 414,8 | 12,8 | 0 % | 7 | 97,1 | 121,0763 |
| 4 | 8 | 630 | 710 | >>d> | 630 | 670 | 407,3 | 412,1 | 4,8 | 1 % | 3 | 99,9 | 41,67643 |
| 4 | 9 | 710 | 1250 | >>d> | 710 | 980 | 402,7 | 402,8 | 0,1 | 50 % | 0 | 100,0 | 0,593606 |
| | 10 | 1250 | 1400 | >>d> | 1250 | 1325 | 387,5 | 387,5 | 0 | 0 % | 0 | 100,0 | 0 |
| | 11 | 1400 | | >>d> | 1400 | 1400 | 479,4 | 479,4 | 0 | 0 % | 0 | 100,0 | 0 |

| Original mass | [g] | 171,9 | | | | | | ∑mi/m/dpi | 2190,6 |
|---|---|---|---|---|---|---|---|---|---|
| Toal of fraction masses | [g] | 171,9 | +-0,45 | NB! Write original mass | | | | | |
| Accumulated error | [g] | 0,262 % | | | | sauter diameter ds [m] = | | [∑mi/m/dp]$^{-1}$ | 0,000456 |

diameter Fe_btc series 1 and 2



Fig. A-3 Particle diameter of Fe-btc used for measurement series 1 and 2.

| container | mean size | m_sample | mi/m/dpi | |
|---|---|---|---|---|
| 3.. | 125 | 0,3 | 13,913043 | |
| 3… | 175 | 0,6 | 19,875776 | |
| 3… | 250 | 4,5 | 104,34783 | |
| 3… | 350 | 39,4 | 652,58799 | |
| 3… | 500 | 127,7 | 1480,5797 | |
| m | | 172,5 | 2271,3043 | |
| | | [∑mi/m/dpi]$^{-1}$ | 0,000440 | diameter fe_btc series 3, 4 and 5 |
| 5… | 500 | 71,5 | | amount of 5 |

Fig. A-4 Particle diameter of Fe-btc used for measurement series 3 through 5.

**Appendix B - Exact composition of glass beads**

Source: (Glassproperties.com). The source claim that this applies for room temperature. This indicates that the data is valid between 293K and 298K.

## Calculation of the thermal (phonon) conductivity of glass at room temperature

*M. K. Choudhary, R. M. Potter: "Heat Transfer in Glass-Forming Melts"; Chapter 9 in: "Properties of Glass-Formation Melts" edited by D. L. Pye, A. Montenaro, I. Joseph; CRC Press, Boca Raton, Florida, May 2005, ISBN: 1-57444-662-2*

Excel file designed by Alexander Fluegel, November 2007, info@glassproperties.com, http://glassproperties.com/thermal-conductivity/

| Glass composition in wt% | | Thermal conductivity in W / (m · K) | Glass composition in mol% | |
|---|---|---|---|---|
| $SiO_2$ | 72,290 | 1,086 | $SiO_2$ | 70,886 |
| $B_2O_3$ | 0,000 | | $B_2O_3$ | 0,000 |
| $Al_2O_3$ | 0,580 | The error of the prediction | $Al_2O_3$ | 0,335 |
| $Li_2O$ | 0,000 | is about 10% of the | $Li_2O$ | 0,000 |
| $Na_2O$ | 13,000 | predicted value. | $Na_2O$ | 12,358 |
| $K_2O$ | 0,200 | | $K_2O$ | 0,125 |
| $Cs_2O$ | 0,000 | | $Cs_2O$ | 0,000 |
| MgO | 4,220 | | MgO | 6,169 |
| CaO | 9,600 | | CaO | 10,086 |
| SrO | 0,000 | | SrO | 0,000 |
| BaO | 0,000 | | BaO | 0,000 |
| PbO | 0,000 | | PbO | 0,000 |
| $Fe_2O_3$ | 0,110 | | $Fe_2O_3$ | 0,041 |
| $TiO_2$ | 0,000 | | $TiO_2$ | 0,000 |
| MnO | 0,000 | | MnO | 0,000 |
| ZnO | 0,000 | | ZnO | 0,000 |
| CoO | 0,000 | | CoO | 0,000 |
| NiO | 0,000 | | NiO | 0,000 |
| CuO | 0,000 | | CuO | 0,000 |
| $Bi_2O_3$ | 0,000 | | $Bi_2O_3$ | 0,000 |
| $Sb_2O_3$ | 0,000 | | $Sb_2O_3$ | 0,000 |
| F | 0,000 | | F | 0,000 |

## Appendix C - Glass beads results

Glass beads with $N_2$, filling 1, measurement error =5%

| | | |
|---|---|---|
| ε_bulk | 0,404064 | |
| ρ_bulk | 1550,03 | kg/m$^3$ |
| d_p | 0,001395 | m |

| T [K] | k [W/m·K] | Cp_gas [J/kgK] | k_f [W/m·K] | k_s [W/m·K] | K=k_s/k_f | k_ZBS [W/m·K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,164 | 0,232 | 1046,360 | 0,034 | 1,243 | 36,497 | 0,240 | 11 % |
| 423,146 | 0,260 | 1046,358 | 0,034 | 1,243 | 36,498 | 0,240 | 11 % |
| 423,137 | 0,265 | 1046,357 | 0,034 | 1,243 | 36,498 | 0,240 | 11 % |
| 333,071 | 0,224 | 1040,026 | 0,028 | 1,157 | 41,430 | 0,200 | 12 % |
| 333,605 | 0,224 | 1040,045 | 0,028 | 1,158 | 41,402 | 0,200 | 12 % |
| 333,515 | 0,224 | 1040,042 | 0,028 | 1,158 | 41,407 | 0,200 | 12 % |
| 292,879 | 0,199 | 1039,143 | 0,025 | 1,088 | 43,472 | 0,180 | 12 % |
| 293,104 | 0,200 | 1039,146 | 0,025 | 1,089 | 43,461 | 0,180 | 12 % |
| 293,165 | 0,200 | 1039,146 | 0,025 | 1,089 | 43,458 | 0,181 | 12 % |
| 278,138 | 0,198 | 1039,056 | 0,024 | 1,058 | 44,199 | 0,173 | 12 % |
| 278,110 | 0,198 | 1039,056 | 0,024 | 1,058 | 44,200 | 0,173 | 12 % |
| 278,126 | 0,198 | 1039,056 | 0,024 | 1,058 | 44,199 | 0,173 | 12 % |
| 273,178 | 0,196 | 1039,049 | 0,024 | 1,048 | 44,441 | 0,170 | 12 % |
| 273,164 | 0,197 | 1039,049 | 0,024 | 1,048 | 44,442 | 0,170 | 12 % |
| 272,998 | 0,198 | 1039,049 | 0,024 | 1,047 | 44,450 | 0,170 | 12 % |
| 268,147 | 0,194 | 1039,053 | 0,023 | 1,037 | 44,686 | 0,168 | 12 % |
| 268,153 | 0,195 | 1039,053 | 0,023 | 1,037 | 44,685 | 0,168 | 12 % |
| 268,114 | 0,196 | 1039,053 | 0,023 | 1,037 | 44,687 | 0,168 | 12 % |
| 263,137 | 0,192 | 1039,065 | 0,023 | 1,025 | 44,928 | 0,165 | 12 % |
| 263,112 | 0,193 | 1039,065 | 0,023 | 1,025 | 44,929 | 0,165 | 12 % |
| 263,135 | 0,193 | 1039,065 | 0,023 | 1,025 | 44,928 | 0,165 | 12 % |
| 253,140 | 0,186 | 1039,114 | 0,022 | 1,002 | 45,408 | 0,160 | 12 % |
| 253,129 | 0,186 | 1039,114 | 0,022 | 1,002 | 45,408 | 0,160 | 12 % |
| 253,146 | 0,187 | 1039,114 | 0,022 | 1,002 | 45,408 | 0,160 | 12 % |
| 253,157 | 0,186 | 1039,114 | 0,022 | 1,002 | 45,407 | 0,160 | 12 % |
| 253,138 | 0,186 | 1039,114 | 0,022 | 1,002 | 45,408 | 0,160 | 12 % |
| 253,159 | 0,186 | 1039,114 | 0,022 | 1,002 | 45,407 | 0,160 | 12 % |
| 263,178 | 0,191 | 1039,065 | 0,023 | 1,025 | 44,926 | 0,165 | 12 % |
| 263,141 | 0,193 | 1039,065 | 0,023 | 1,025 | 44,928 | 0,165 | 12 % |
| 263,148 | 0,193 | 1039,065 | 0,023 | 1,025 | 44,927 | 0,165 | 12 % |
| 268,111 | 0,193 | 1039,053 | 0,023 | 1,037 | 44,687 | 0,168 | 12 % |
| 268,162 | 0,195 | 1039,053 | 0,023 | 1,037 | 44,685 | 0,168 | 12 % |
| 268,151 | 0,194 | 1039,053 | 0,023 | 1,037 | 44,685 | 0,168 | 12 % |
| 273,208 | 0,196 | 1039,049 | 0,024 | 1,048 | 44,440 | 0,170 | 12 % |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 273,148 | 0,197 | 1039,049 | 0,024 | 1,048 | 44,443 | 0,170 | 12 % |
| 273,120 | 0,198 | 1039,049 | 0,024 | 1,048 | 44,444 | 0,170 | 12 % |
| 278,159 | 0,197 | 1039,056 | 0,024 | 1,059 | 44,198 | 0,173 | 12 % |
| 278,195 | 0,199 | 1039,056 | 0,024 | 1,059 | 44,196 | 0,173 | 12 % |
| 278,170 | 0,199 | 1039,056 | 0,024 | 1,059 | 44,197 | 0,173 | 12 % |
| 292,883 | 0,200 | 1039,143 | 0,025 | 1,088 | 43,472 | 0,180 | 12 % |
| 293,225 | 0,201 | 1039,147 | 0,025 | 1,089 | 43,455 | 0,181 | 12 % |
| 293,340 | 0,202 | 1039,148 | 0,025 | 1,089 | 43,449 | 0,181 | 12 % |
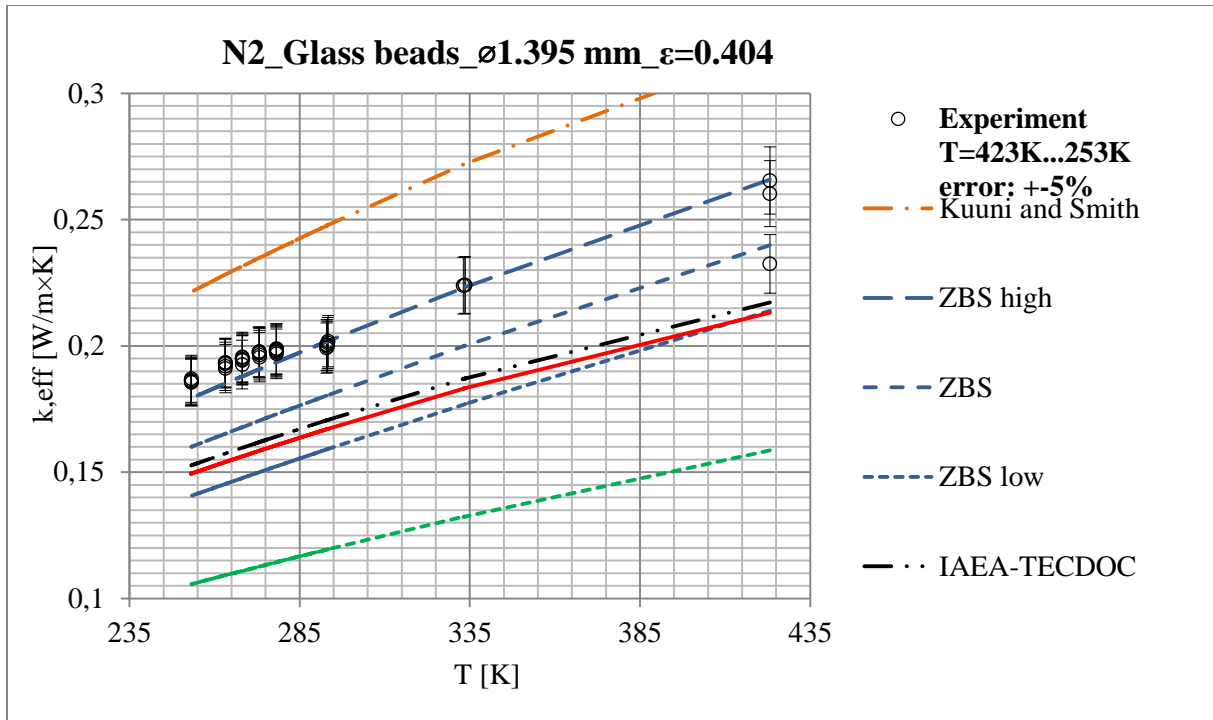
Glass beads with $N_2$, filling 1, measurement error =5%

ε_bulk  0,425965

ρ_bulk  1493,06  kg/m$^3$

d_p  0,00038  m

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,170 | 0,253 | 1046,360 | 0,034 | 1,243 | 36,496 | 0,246 | 10 % |
| 423,160 | 0,254 | 1046,359 | 0,034 | 1,243 | 36,497 | 0,246 | 10 % |
| 423,144 | 0,254 | 1046,358 | 0,034 | 1,243 | 36,498 | 0,246 | 10 % |
| 332,757 | 0,250 | 1040,015 | 0,028 | 1,157 | 41,446 | 0,198 | 11 % |
| 293,060 | 0,232 | 1039,145 | 0,025 | 1,089 | 43,463 | 0,176 | 11 % |
| 293,364 | 0,217 | 1039,148 | 0,025 | 1,089 | 43,448 | 0,177 | 11 % |
| 278,126 | 0,197 | 1039,056 | 0,024 | 1,058 | 44,199 | 0,168 | 12 % |
| 278,097 | 0,197 | 1039,056 | 0,024 | 1,058 | 44,201 | 0,168 | 12 % |
| 278,121 | 0,197 | 1039,056 | 0,024 | 1,058 | 44,200 | 0,168 | 12 % |
| 273,144 | 0,197 | 1039,049 | 0,024 | 1,048 | 44,443 | 0,166 | 12 % |
| 273,191 | 0,197 | 1039,049 | 0,024 | 1,048 | 44,440 | 0,166 | 12 % |
| 273,174 | 0,197 | 1039,049 | 0,024 | 1,048 | 44,441 | 0,166 | 12 % |
| 268,150 | 0,195 | 1039,053 | 0,023 | 1,037 | 44,685 | 0,163 | 12 % |
| 268,201 | 0,195 | 1039,052 | 0,023 | 1,037 | 44,683 | 0,163 | 12 % |
| 268,214 | 0,195 | 1039,052 | 0,023 | 1,037 | 44,682 | 0,163 | 12 % |
| 263,196 | 0,193 | 1039,065 | 0,023 | 1,026 | 44,925 | 0,160 | 12 % |
| 263,157 | 0,193 | 1039,065 | 0,023 | 1,025 | 44,927 | 0,160 | 12 % |
| 263,138 | 0,193 | 1039,065 | 0,023 | 1,025 | 44,928 | 0,160 | 12 % |
| 253,168 | 0,186 | 1039,114 | 0,022 | 1,002 | 45,407 | 0,155 | 12 % |
| 253,147 | 0,186 | 1039,114 | 0,022 | 1,002 | 45,408 | 0,155 | 12 % |
| 253,156 | 0,186 | 1039,114 | 0,022 | 1,002 | 45,407 | 0,155 | 12 % |

**N2_Glass beads_⌀0.38 mm_ε=0.426**
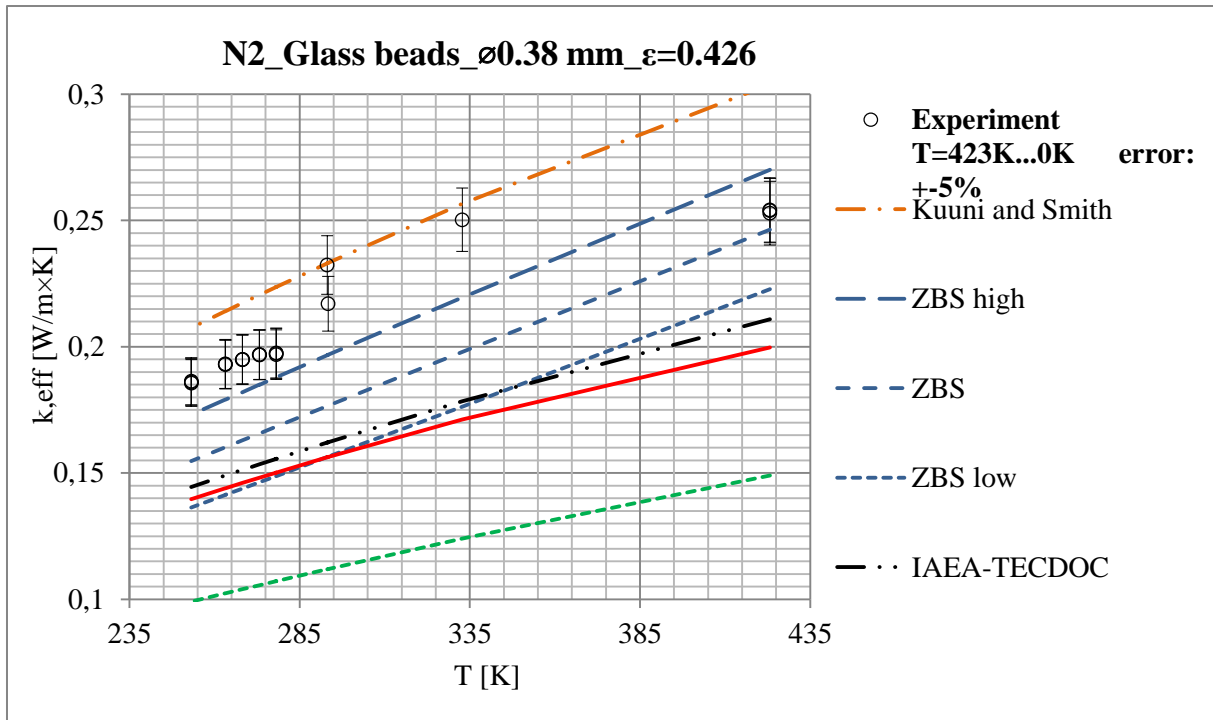
Glass beads with $N_2$, filling 2, measurement error =5%

| | | |
|---|---|---|
| $\varepsilon$_bulk | 0,396707583 | |
| $\rho$_bulk | 1569,16 | kg/m$^3$ |
| d_p | 0,00038 | m |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 333,599 | 0,219 | 1040,045 | 0,028 | 1,158 | 41,402 | 0,213 | 11 % |
| 287,958 | 0,191 | 1039,102 | 0,025 | 1,079 | 43,716 | 0,188 | 11 % |
| 288,328 | 0,191 | 1039,105 | 0,025 | 1,079 | 43,697 | 0,188 | 11 % |
| 288,286 | 0,191 | 1039,105 | 0,025 | 1,079 | 43,700 | 0,188 | 11 % |
| 423,160 | 0,258 | 1046,359 | 0,034 | 1,243 | 36,497 | 0,262 | 10 % |
| 423,143 | 0,258 | 1046,358 | 0,034 | 1,243 | 36,498 | 0,262 | 10 % |
| 423,144 | 0,258 | 1046,358 | 0,034 | 1,243 | 36,498 | 0,262 | 10 % |
| 333,732 | 0,223 | 1040,049 | 0,028 | 1,158 | 41,395 | 0,213 | 11 % |
| 333,049 | 0,224 | 1040,025 | 0,028 | 1,157 | 41,431 | 0,213 | 11 % |
| 333,367 | 0,223 | 1040,036 | 0,028 | 1,158 | 41,414 | 0,213 | 11 % |
| 278,200 | 0,218 | 1039,056 | 0,024 | 1,059 | 44,196 | 0,182 | 11 % |
| 273,123 | 0,197 | 1039,049 | 0,024 | 1,048 | 44,444 | 0,179 | 11 % |
| 273,125 | 0,197 | 1039,049 | 0,024 | 1,048 | 44,444 | 0,179 | 11 % |
| 273,155 | 0,197 | 1039,049 | 0,024 | 1,048 | 44,442 | 0,179 | 11 % |
| 268,102 | 0,196 | 1039,053 | 0,023 | 1,037 | 44,688 | 0,176 | 11 % |
| 268,145 | 0,196 | 1039,053 | 0,023 | 1,037 | 44,686 | 0,176 | 11 % |
| 268,123 | 0,195 | 1039,053 | 0,023 | 1,037 | 44,687 | 0,176 | 11 % |
| 258,133 | 0,190 | 1039,086 | 0,022 | 1,014 | 45,169 | 0,170 | 11 % |
| 258,146 | 0,190 | 1039,086 | 0,022 | 1,014 | 45,168 | 0,170 | 11 % |
| 258,132 | 0,190 | 1039,086 | 0,022 | 1,014 | 45,169 | 0,170 | 11 % |
| 243,142 | 0,181 | 1039,188 | 0,021 | 0,977 | 45,884 | 0,162 | 12 % |
| 243,168 | 0,181 | 1039,188 | 0,021 | 0,977 | 45,883 | 0,162 | 12 % |
| 243,147 | 0,181 | 1039,188 | 0,021 | 0,977 | 45,884 | 0,162 | 12 % |
| 253,134 | 0,184 | 1039,114 | 0,022 | 1,002 | 45,408 | 0,168 | 12 % |
| 253,149 | 0,186 | 1039,114 | 0,022 | 1,002 | 45,407 | 0,168 | 12 % |
| 253,146 | 0,187 | 1039,114 | 0,022 | 1,002 | 45,408 | 0,168 | 12 % |

N2_Glass beads_⌀0.38 mm_ε=0.397
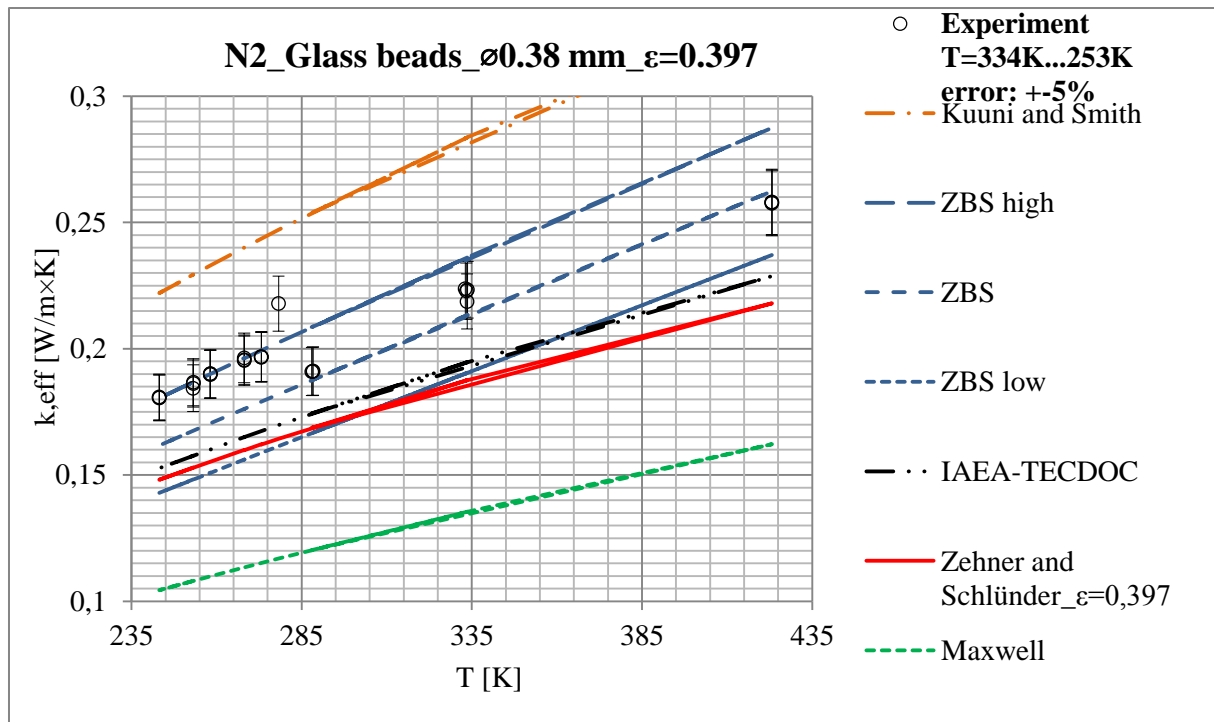
Glass beads with N$_2$, filling 3, measurement error =5%

ε_bulk   0,390662

ρ_bulk   1584,89   kg/m$^3$

d_p      0,00038   m

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,135 | 0,248 | 1046,357 | 0,034 | 1,243 | 36,498 | 0,266 | 10 % |
| 333,210 | 0,232 | 1040,031 | 0,028 | 1,157 | 41,422 | 0,216 | 11 % |
| 333,758 | 0,231 | 1040,050 | 0,028 | 1,158 | 41,394 | 0,217 | 11 % |
| 332,998 | 0,231 | 1040,023 | 0,028 | 1,157 | 41,433 | 0,216 | 11 % |
| 278,224 | 0,222 | 1039,056 | 0,024 | 1,059 | 44,195 | 0,185 | 11 % |
| 273,145 | 0,207 | 1039,049 | 0,024 | 1,048 | 44,443 | 0,182 | 11 % |
| 423,117 | 0,260 | 1046,355 | 0,034 | 1,243 | 36,500 | 0,266 | 10 % |
| 423,159 | 0,268 | 1046,359 | 0,034 | 1,243 | 36,497 | 0,266 | 10 % |
| 423,128 | 0,264 | 1046,356 | 0,034 | 1,243 | 36,499 | 0,266 | 10 % |
| 333,832 | 0,231 | 1040,053 | 0,028 | 1,158 | 41,390 | 0,217 | 11 % |
| 333,234 | 0,231 | 1040,032 | 0,028 | 1,158 | 41,421 | 0,216 | 11 % |
| 333,561 | 0,230 | 1040,043 | 0,028 | 1,158 | 41,404 | 0,217 | 11 % |
| 278,200 | 0,220 | 1039,056 | 0,024 | 1,059 | 44,196 | 0,185 | 11 % |
| 273,168 | 0,203 | 1039,049 | 0,024 | 1,048 | 44,442 | 0,182 | 11 % |
| 273,078 | 0,204 | 1039,049 | 0,024 | 1,048 | 44,446 | 0,182 | 11 % |
| 273,194 | 0,203 | 1039,049 | 0,024 | 1,048 | 44,440 | 0,182 | 11 % |
| 268,171 | 0,203 | 1039,053 | 0,023 | 1,037 | 44,684 | 0,179 | 11 % |
| 268,205 | 0,202 | 1039,052 | 0,023 | 1,037 | 44,683 | 0,179 | 11 % |
| 268,143 | 0,201 | 1039,053 | 0,023 | 1,037 | 44,686 | 0,179 | 11 % |
| 258,207 | 0,196 | 1039,086 | 0,022 | 1,014 | 45,165 | 0,173 | 11 % |
| 258,155 | 0,196 | 1039,086 | 0,022 | 1,014 | 45,168 | 0,173 | 11 % |
| 258,190 | 0,196 | 1039,086 | 0,022 | 1,014 | 45,166 | 0,173 | 11 % |
| 243,165 | 0,186 | 1039,188 | 0,021 | 0,977 | 45,883 | 0,164 | 12 % |
| 243,147 | 0,186 | 1039,188 | 0,021 | 0,977 | 45,884 | 0,164 | 12 % |
| 243,162 | 0,186 | 1039,188 | 0,021 | 0,977 | 45,883 | 0,164 | 12 % |
| 253,153 | 0,190 | 1039,114 | 0,022 | 1,002 | 45,407 | 0,170 | 11 % |
| 253,133 | 0,191 | 1039,114 | 0,022 | 1,002 | 45,408 | 0,170 | 11 % |
| 253,140 | 0,192 | 1039,114 | 0,022 | 1,002 | 45,408 | 0,170 | 11 % |
| 263,145 | 0,197 | 1039,065 | 0,023 | 1,025 | 44,927 | 0,176 | 11 % |
| 263,163 | 0,199 | 1039,065 | 0,023 | 1,025 | 44,927 | 0,176 | 11 % |
| 263,137 | 0,200 | 1039,065 | 0,023 | 1,025 | 44,928 | 0,176 | 11 % |
| 263,158 | 0,200 | 1039,065 | 0,023 | 1,025 | 44,927 | 0,176 | 11 % |
| 271,142 | 0,201 | 1039,049 | 0,023 | 1,043 | 44,540 | 0,181 | 11 % |
| 271,214 | 0,202 | 1039,049 | 0,023 | 1,044 | 44,537 | 0,181 | 11 % |
| 271,125 | 0,202 | 1039,049 | 0,023 | 1,043 | 44,541 | 0,181 | 11 % |
| 275,134 | 0,202 | 1039,051 | 0,024 | 1,052 | 44,346 | 0,183 | 11 % |
| 275,141 | 0,204 | 1039,051 | 0,024 | 1,052 | 44,345 | 0,183 | 11 % |
| 275,143 | 0,204 | 1039,051 | 0,024 | 1,052 | 44,345 | 0,183 | 11 % |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 283,125 | 0,204 | 1039,074 | 0,024 | 1,069 | 43,954 | 0,188 | 11 % |
| 283,194 | 0,206 | 1039,074 | 0,024 | 1,069 | 43,951 | 0,188 | 11 % |
| 283,122 | 0,206 | 1039,074 | 0,024 | 1,069 | 43,954 | 0,188 | 11 % |
| 307,927 | 0,216 | 1039,354 | 0,026 | 1,116 | 42,718 | 0,202 | 11 % |
| 308,487 | 0,216 | 1039,364 | 0,026 | 1,117 | 42,690 | 0,202 | 11 % |
| 308,588 | 0,216 | 1039,366 | 0,026 | 1,118 | 42,684 | 0,202 | 11 % |
| 373,131 | 0,233 | 1042,050 | 0,031 | 1,207 | 39,299 | 0,239 | 10 % |
| 423,152 | 0,266 | 1046,359 | 0,034 | 1,243 | 36,498 | 0,266 | 10 % |
| 423,197 | 0,265 | 1046,363 | 0,034 | 1,243 | 36,495 | 0,266 | 10 % |
| 423,162 | 0,266 | 1046,360 | 0,034 | 1,243 | 36,497 | 0,266 | 10 % |
| 332,987 | 0,232 | 1040,023 | 0,028 | 1,157 | 41,434 | 0,216 | 11 % |
| 333,021 | 0,231 | 1040,024 | 0,028 | 1,157 | 41,432 | 0,216 | 11 % |
| 333,396 | 0,230 | 1040,037 | 0,028 | 1,158 | 41,413 | 0,216 | 11 % |
| 278,106 | 0,221 | 1039,056 | 0,024 | 1,058 | 44,200 | 0,185 | 11 % |
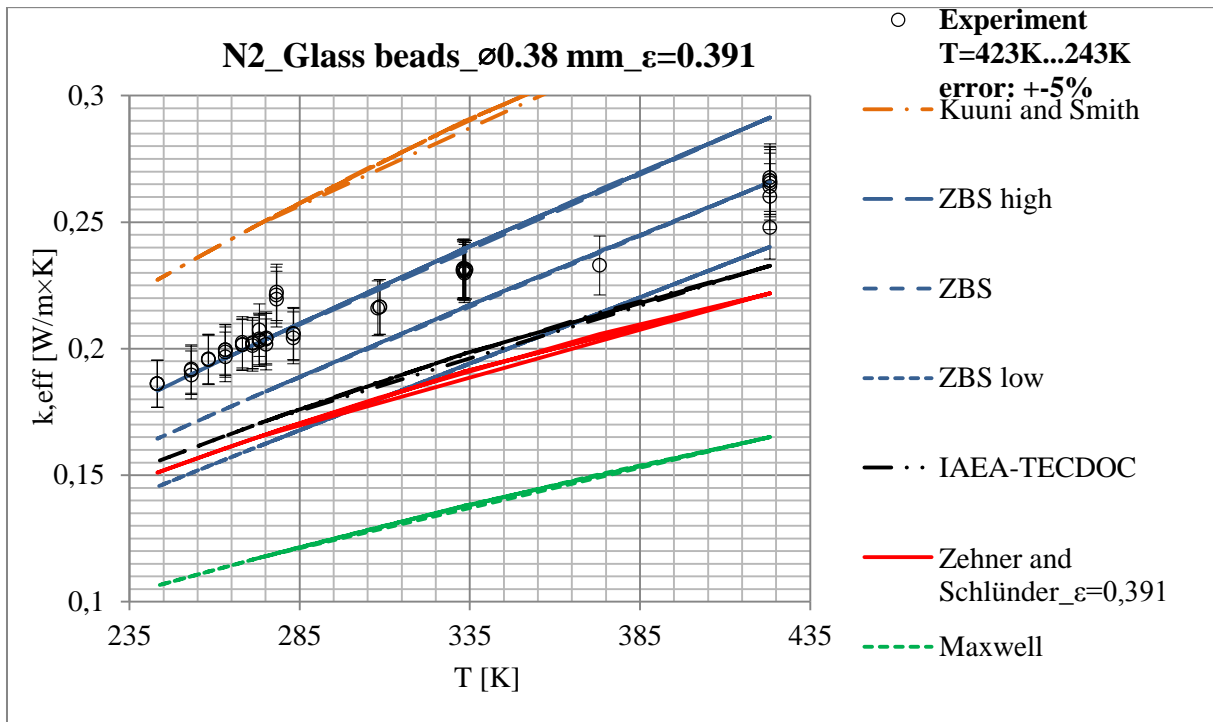
## Appendix D - Cu-btc results

Cu-btc with He, filling 1, measurement error =5%

| | | | | |
|---|---|---|---|
| ε_bulk | 0,415206 | |
| ρ_bulk | 553,16 | kg/m³ |
| d_p | 0,00376 | m |
| p | 1,2 | bar |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,141 | 0,296 | 5196,250 | 0,196 | 0,351 | 1,790 | 0,300 | 2 % |
| 423,131 | 0,296 | 5196,250 | 0,196 | 0,351 | 1,790 | 0,300 | 2 % |
| 423,136 | 0,296 | 5196,250 | 0,196 | 0,351 | 1,790 | 0,300 | 2 % |
| 373,125 | 0,283 | 5196,250 | 0,179 | 0,346 | 1,928 | 0,286 | 2 % |
| 373,142 | 0,282 | 5196,250 | 0,179 | 0,346 | 1,928 | 0,286 | 2 % |
| 373,166 | 0,282 | 5196,250 | 0,179 | 0,346 | 1,928 | 0,286 | 2 % |
| 332,898 | 0,270 | 5196,250 | 0,166 | 0,342 | 2,061 | 0,274 | 3 % |
| 333,047 | 0,270 | 5196,250 | 0,166 | 0,342 | 2,061 | 0,274 | 3 % |
| 332,708 | 0,270 | 5196,250 | 0,166 | 0,342 | 2,062 | 0,274 | 3 % |
| 313,066 | 0,262 | 5196,250 | 0,159 | 0,339 | 2,136 | 0,268 | 3 % |
| 313,562 | 0,261 | 5196,250 | 0,159 | 0,340 | 2,134 | 0,268 | 3 % |
| 312,679 | 0,262 | 5196,250 | 0,159 | 0,339 | 2,138 | 0,268 | 3 % |
| 293,328 | 0,253 | 5196,250 | 0,152 | 0,337 | 2,218 | 0,262 | 3 % |
| 293,045 | 0,254 | 5196,250 | 0,152 | 0,337 | 2,220 | 0,262 | 3 % |
| 293,596 | 0,253 | 5196,250 | 0,152 | 0,337 | 2,217 | 0,262 | 3 % |
| 278,228 | 0,255 | 5196,250 | 0,147 | 0,335 | 2,287 | 0,258 | 3 % |
| 278,131 | 0,255 | 5196,250 | 0,147 | 0,335 | 2,287 | 0,258 | 3 % |
| 278,225 | 0,255 | 5196,250 | 0,147 | 0,335 | 2,287 | 0,258 | 3 % |
| 273,176 | 0,255 | 5196,250 | 0,145 | 0,335 | 2,311 | 0,256 | 3 % |
| 273,125 | 0,256 | 5196,250 | 0,145 | 0,335 | 2,311 | 0,256 | 3 % |
| 273,196 | 0,255 | 5196,250 | 0,145 | 0,335 | 2,311 | 0,256 | 3 % |
| 268,134 | 0,254 | 5196,250 | 0,143 | 0,334 | 2,336 | 0,255 | 3 % |
| 268,079 | 0,255 | 5196,250 | 0,143 | 0,334 | 2,336 | 0,255 | 3 % |
| 268,159 | 0,254 | 5196,250 | 0,143 | 0,334 | 2,336 | 0,255 | 3 % |
| 263,128 | 0,253 | 5196,250 | 0,141 | 0,333 | 2,361 | 0,253 | 3 % |
| 263,147 | 0,253 | 5196,250 | 0,141 | 0,333 | 2,361 | 0,253 | 3 % |
| 263,146 | 0,252 | 5196,250 | 0,141 | 0,333 | 2,361 | 0,253 | 3 % |
| 253,128 | 0,246 | 5196,250 | 0,137 | 0,332 | 2,414 | 0,250 | 3 % |
| 253,153 | 0,246 | 5196,250 | 0,137 | 0,332 | 2,413 | 0,250 | 3 % |
| 253,143 | 0,246 | 5196,250 | 0,137 | 0,332 | 2,413 | 0,250 | 3 % |

**Cu-btc_filling 1_He**

Legend:
- ○ **Experiment T=423K...253K error: +-5% ε=0.42**
- — · · — IAEA-TECDOC
- — — ZBS high
- – – – ZBS
- — · — ZBS low
- ——— Zehner and Schlünder_ε=0,415
- - - - - Kuprizcka

x-axis: T [K]
y-axis: k,eff [W/m×K]

Cu-btc with N2, filling 1, measurement error =5%

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| ε_bulk | 0,415206 | | | | | | |
| ρ_bulk | 553,16 | kg/m$^3$ | | | | | |
| d_p | 0,00376 | m | | | | | |
| p | 1,2 | bar | | | | | |
| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
| 423,183 | 0,159 | 1046,362 | 0,034 | 0,351 | 10,295 | 0,176 | 5 % |
| 423,128 | 0,158 | 1046,356 | 0,034 | 0,351 | 10,296 | 0,176 | 5 % |
| 423,147 | 0,158 | 1046,358 | 0,034 | 0,351 | 10,296 | 0,176 | 5 % |
| 373,126 | 0,148 | 1042,050 | 0,031 | 0,346 | 11,263 | 0,162 | 6 % |
| 373,136 | 0,147 | 1042,051 | 0,031 | 0,346 | 11,263 | 0,162 | 6 % |
| 373,167 | 0,147 | 1042,053 | 0,031 | 0,346 | 11,263 | 0,162 | 6 % |
| 332,755 | 0,138 | 1040,015 | 0,028 | 0,342 | 12,243 | 0,151 | 7 % |
| 332,849 | 0,138 | 1040,018 | 0,028 | 0,342 | 12,241 | 0,151 | 7 % |
| 333,022 | 0,138 | 1040,024 | 0,028 | 0,342 | 12,236 | 0,151 | 7 % |
| 313,556 | 0,132 | 1039,468 | 0,027 | 0,340 | 12,793 | 0,145 | 7 % |
| 313,694 | 0,132 | 1039,471 | 0,027 | 0,340 | 12,789 | 0,145 | 7 % |
| 313,107 | 0,132 | 1039,458 | 0,027 | 0,339 | 12,807 | 0,145 | 7 % |
| 278,170 | 0,125 | 1039,056 | 0,024 | 0,335 | 13,995 | 0,135 | 8 % |
| 278,186 | 0,125 | 1039,056 | 0,024 | 0,335 | 13,994 | 0,135 | 8 % |
| 278,108 | 0,125 | 1039,056 | 0,024 | 0,335 | 13,997 | 0,135 | 8 % |
| 273,265 | 0,124 | 1039,049 | 0,024 | 0,335 | 14,185 | 0,134 | 8 % |
| 273,211 | 0,125 | 1039,049 | 0,024 | 0,335 | 14,187 | 0,134 | 8 % |
| 273,210 | 0,125 | 1039,049 | 0,024 | 0,335 | 14,187 | 0,134 | 8 % |
| 268,169 | 0,123 | 1039,053 | 0,023 | 0,334 | 14,389 | 0,133 | 8 % |
| 268,171 | 0,123 | 1039,053 | 0,023 | 0,334 | 14,389 | 0,133 | 8 % |
| 268,141 | 0,124 | 1039,053 | 0,023 | 0,334 | 14,390 | 0,133 | 8 % |
| 263,146 | 0,121 | 1039,065 | 0,023 | 0,333 | 14,598 | 0,131 | 8 % |
| 263,162 | 0,122 | 1039,065 | 0,023 | 0,333 | 14,597 | 0,131 | 8 % |

Cu-btc with N2, filling 2, measurement error =5%

| | |
|---|---|
| ε_bulk | 0,444422 |
| ρ_bulk | 525,52 kg/m³ |
| d_p | 0,00376 m |
| p | 1,2 bar |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,141 | 0,164 | 1046,357 | 0,034 | 0,351 | 10,296 | 0,170 | 5 % |
| 423,173 | 0,164 | 1046,361 | 0,034 | 0,351 | 10,295 | 0,170 | 5 % |
| 423,147 | 0,164 | 1046,358 | 0,034 | 0,351 | 10,296 | 0,170 | 5 % |
| 373,165 | 0,154 | 1042,053 | 0,031 | 0,346 | 11,263 | 0,155 | 6 % |
| 373,138 | 0,154 | 1042,051 | 0,031 | 0,346 | 11,263 | 0,155 | 6 % |
| 373,150 | 0,153 | 1042,052 | 0,031 | 0,346 | 11,263 | 0,155 | 6 % |
| 332,703 | 0,145 | 1040,013 | 0,028 | 0,342 | 12,245 | 0,144 | 7 % |
| 332,815 | 0,145 | 1040,017 | 0,028 | 0,342 | 12,242 | 0,144 | 7 % |
| 332,743 | 0,145 | 1040,014 | 0,028 | 0,342 | 12,244 | 0,144 | 7 % |
| 313,091 | 0,139 | 1039,457 | 0,027 | 0,339 | 12,807 | 0,138 | 7 % |
| 312,888 | 0,139 | 1039,453 | 0,026 | 0,339 | 12,813 | 0,138 | 7 % |
| 313,736 | 0,138 | 1039,472 | 0,027 | 0,340 | 12,788 | 0,138 | 7 % |
| 293,124 | 0,129 | 1039,146 | 0,025 | 0,337 | 13,453 | 0,132 | 8 % |
| 293,200 | 0,129 | 1039,147 | 0,025 | 0,337 | 13,451 | 0,132 | 8 % |
| 292,866 | 0,128 | 1039,143 | 0,025 | 0,337 | 13,462 | 0,132 | 8 % |
| 278,347 | 0,131 | 1039,056 | 0,024 | 0,335 | 13,988 | 0,128 | 8 % |
| 278,227 | 0,131 | 1039,056 | 0,024 | 0,335 | 13,992 | 0,128 | 8 % |
| 278,191 | 0,131 | 1039,056 | 0,024 | 0,335 | 13,994 | 0,128 | 8 % |
| 273,215 | 0,130 | 1039,049 | 0,024 | 0,335 | 14,187 | 0,127 | 8 % |
| 273,182 | 0,130 | 1039,049 | 0,024 | 0,335 | 14,188 | 0,127 | 8 % |
| 273,132 | 0,130 | 1039,049 | 0,024 | 0,335 | 14,190 | 0,127 | 8 % |
| 268,177 | 0,129 | 1039,052 | 0,023 | 0,334 | 14,389 | 0,125 | 8 % |
| 268,240 | 0,129 | 1039,052 | 0,023 | 0,334 | 14,386 | 0,125 | 8 % |
| 268,189 | 0,129 | 1039,052 | 0,023 | 0,334 | 14,388 | 0,125 | 8 % |
| 263,116 | 0,127 | 1039,065 | 0,023 | 0,333 | 14,599 | 0,124 | 8 % |
| 263,111 | 0,127 | 1039,065 | 0,023 | 0,333 | 14,599 | 0,124 | 8 % |

Cu-btc with He, filling 2, measurement error =5%

| | | |
|---|---|---|
| ε_bulk | 0,444422 | |
| ρ_bulk | 525,52 | kg/m$^3$ |
| d_p | 0,00376 | m |
| p | 1,2 | bar |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,177 | 0,301 | 5196,250 | 0,196 | 0,351 | 1,790 | 0,297 | 2 % |
| 423,165 | 0,300 | 5196,250 | 0,196 | 0,351 | 1,790 | 0,297 | 2 % |
| 423,149 | 0,300 | 5196,250 | 0,196 | 0,351 | 1,790 | 0,297 | 2 % |
| 332,812 | 0,277 | 5196,250 | 0,166 | 0,342 | 2,061 | 0,270 | 3 % |
| 332,772 | 0,276 | 5196,250 | 0,166 | 0,342 | 2,062 | 0,270 | 3 % |
| 333,692 | 0,277 | 5196,250 | 0,166 | 0,342 | 2,058 | 0,270 | 3 % |
| 293,269 | 0,263 | 5196,250 | 0,152 | 0,337 | 2,219 | 0,258 | 3 % |
| 292,899 | 0,263 | 5196,250 | 0,152 | 0,337 | 2,220 | 0,258 | 3 % |
| 292,865 | 0,263 | 5196,250 | 0,152 | 0,337 | 2,220 | 0,257 | 3 % |
| 278,440 | 0,266 | 5196,250 | 0,147 | 0,335 | 2,286 | 0,253 | 3 % |
| 278,168 | 0,267 | 5196,250 | 0,147 | 0,335 | 2,287 | 0,253 | 3 % |
| 278,046 | 0,266 | 5196,250 | 0,147 | 0,335 | 2,288 | 0,253 | 3 % |
| 273,202 | 0,267 | 5196,250 | 0,145 | 0,335 | 2,311 | 0,251 | 3 % |
| 273,142 | 0,267 | 5196,250 | 0,145 | 0,335 | 2,311 | 0,251 | 3 % |
| 273,103 | 0,267 | 5196,250 | 0,145 | 0,335 | 2,311 | 0,251 | 3 % |
| 268,204 | 0,266 | 5196,250 | 0,143 | 0,334 | 2,335 | 0,250 | 3 % |
| 268,148 | 0,266 | 5196,250 | 0,143 | 0,334 | 2,336 | 0,250 | 3 % |
| 268,187 | 0,266 | 5196,250 | 0,143 | 0,334 | 2,335 | 0,250 | 3 % |
| 263,165 | 0,267 | 5196,250 | 0,141 | 0,333 | 2,361 | 0,248 | 3 % |
| 263,102 | 0,265 | 5196,250 | 0,141 | 0,333 | 2,361 | 0,248 | 3 % |
| 263,223 | 0,267 | 5196,250 | 0,141 | 0,333 | 2,360 | 0,248 | 3 % |
| 253,121 | 0,261 | 5196,250 | 0,137 | 0,332 | 2,414 | 0,245 | 3 % |
| 253,115 | 0,262 | 5196,250 | 0,137 | 0,332 | 2,414 | 0,245 | 3 % |
| 253,174 | 0,261 | 5196,250 | 0,137 | 0,332 | 2,413 | 0,245 | 3 % |

| Cu-btc with He, filling 3, measurement error =5% | | | | | | |
|---|---|---|---|---|---|---|

| ε_bulk | 0,419108 | |
|---|---|---|
| ρ_bulk | 549,47 | kg/m³ |
| d_p | 0,00376 | m |
| p | 1,4 | bar |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,144 | 0,300 | 5196,250 | 0,196 | 0,351 | 1,790 | 0,300 | 2 % |
| 423,148 | 0,298 | 5196,250 | 0,196 | 0,351 | 1,790 | 0,300 | 2 % |
| 423,150 | 0,300 | 5196,250 | 0,196 | 0,351 | 1,790 | 0,300 | 2 % |
| 373,186 | 0,286 | 5196,250 | 0,179 | 0,346 | 1,928 | 0,285 | 2 % |
| 373,159 | 0,286 | 5196,250 | 0,179 | 0,346 | 1,928 | 0,285 | 2 % |
| 373,160 | 0,286 | 5196,250 | 0,179 | 0,346 | 1,928 | 0,285 | 2 % |
| 332,767 | 0,280 | 5196,250 | 0,166 | 0,342 | 2,062 | 0,274 | 3 % |
| 332,713 | 0,274 | 5196,250 | 0,166 | 0,342 | 2,062 | 0,274 | 3 % |
| 333,232 | 0,273 | 5196,250 | 0,166 | 0,342 | 2,060 | 0,274 | 3 % |
| 293,551 | 0,258 | 5196,250 | 0,152 | 0,337 | 2,217 | 0,262 | 3 % |
| 293,463 | 0,256 | 5196,250 | 0,152 | 0,337 | 2,218 | 0,262 | 3 % |
| 293,230 | 0,257 | 5196,250 | 0,152 | 0,337 | 2,219 | 0,262 | 3 % |
| 282,991 | 0,259 | 5196,250 | 0,148 | 0,336 | 2,265 | 0,259 | 3 % |
| 283,255 | 0,257 | 5196,250 | 0,148 | 0,336 | 2,263 | 0,259 | 3 % |
| 283,218 | 0,257 | 5196,250 | 0,148 | 0,336 | 2,264 | 0,259 | 3 % |
| 278,139 | 0,257 | 5196,250 | 0,147 | 0,335 | 2,287 | 0,257 | 3 % |
| 278,140 | 0,258 | 5196,250 | 0,147 | 0,335 | 2,287 | 0,257 | 3 % |
| 278,221 | 0,258 | 5196,250 | 0,147 | 0,335 | 2,287 | 0,257 | 3 % |
| 273,094 | 0,258 | 5196,250 | 0,145 | 0,335 | 2,311 | 0,255 | 3 % |
| 273,093 | 0,259 | 5196,250 | 0,145 | 0,335 | 2,311 | 0,255 | 3 % |
| 273,065 | 0,259 | 5196,250 | 0,145 | 0,335 | 2,312 | 0,255 | 3 % |
| 268,070 | 0,256 | 5196,250 | 0,143 | 0,334 | 2,336 | 0,254 | 3 % |
| 268,196 | 0,258 | 5196,250 | 0,143 | 0,334 | 2,335 | 0,254 | 3 % |
| 268,196 | 0,258 | 5196,250 | 0,143 | 0,334 | 2,335 | 0,254 | 3 % |
| 263,159 | 0,253 | 5196,250 | 0,141 | 0,333 | 2,361 | 0,252 | 3 % |
| 263,120 | 0,255 | 5196,250 | 0,141 | 0,333 | 2,361 | 0,252 | 3 % |
| 263,139 | 0,256 | 5196,250 | 0,141 | 0,333 | 2,361 | 0,252 | 3 % |
| 253,144 | 0,249 | 5196,250 | 0,137 | 0,332 | 2,413 | 0,249 | 3 % |
| 253,155 | 0,251 | 5196,250 | 0,137 | 0,332 | 2,413 | 0,249 | 3 % |
| 253,119 | 0,250 | 5196,250 | 0,137 | 0,332 | 2,414 | 0,249 | 3 % |
| 243,182 | 0,243 | 5196,250 | 0,134 | 0,330 | 2,469 | 0,246 | 3 % |
| 243,180 | 0,247 | 5196,250 | 0,134 | 0,330 | 2,469 | 0,246 | 3 % |
| 243,160 | 0,245 | 5196,250 | 0,134 | 0,330 | 2,469 | 0,246 | 3 % |
| 258,117 | 0,251 | 5196,250 | 0,139 | 0,332 | 2,387 | 0,251 | 3 % |
| 258,108 | 0,253 | 5196,250 | 0,139 | 0,332 | 2,387 | 0,251 | 3 % |
| 258,168 | 0,253 | 5196,250 | 0,139 | 0,332 | 2,387 | 0,251 | 3 % |
| 271,181 | 0,257 | 5196,250 | 0,144 | 0,334 | 2,321 | 0,255 | 3 % |
| 271,212 | 0,259 | 5196,250 | 0,144 | 0,334 | 2,321 | 0,255 | 3 % |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 271,198 | 0,259 | 5196,250 | 0,144 | 0,334 | 2,321 | 0,255 | 3 % |
| 275,108 | 0,257 | 5196,250 | 0,145 | 0,335 | 2,302 | 0,256 | 3 % |
| 275,229 | 0,258 | 5196,250 | 0,145 | 0,335 | 2,301 | 0,256 | 3 % |
| 275,213 | 0,258 | 5196,250 | 0,145 | 0,335 | 2,301 | 0,256 | 3 % |
| 280,204 | 0,256 | 5196,250 | 0,147 | 0,335 | 2,278 | 0,258 | 3 % |
| 280,113 | 0,258 | 5196,250 | 0,147 | 0,335 | 2,278 | 0,258 | 3 % |
| 280,119 | 0,258 | 5196,250 | 0,147 | 0,335 | 2,278 | 0,258 | 3 % |
| 288,016 | 0,259 | 5196,250 | 0,150 | 0,336 | 2,242 | 0,260 | 3 % |
| 288,273 | 0,257 | 5196,250 | 0,150 | 0,336 | 2,241 | 0,260 | 3 % |
| 288,262 | 0,257 | 5196,250 | 0,150 | 0,336 | 2,241 | 0,260 | 3 % |
| 353,042 | 0,289 | 5196,250 | 0,173 | 0,344 | 1,992 | 0,280 | 2 % |
| 353,322 | 0,285 | 5196,250 | 0,173 | 0,344 | 1,991 | 0,280 | 2 % |
| 352,927 | 0,286 | 5196,250 | 0,173 | 0,344 | 1,992 | 0,280 | 2 % |
| 398,164 | 0,301 | 5196,250 | 0,188 | 0,348 | 1,856 | 0,293 | 2 % |
| 398,136 | 0,298 | 5196,250 | 0,188 | 0,348 | 1,856 | 0,293 | 2 % |
| 398,177 | 0,298 | 5196,250 | 0,188 | 0,348 | 1,856 | 0,293 | 2 % |

Cu-btc with N2, filling 3, measurement error =5%

| | | |
|---|---|---|
| ε_bulk | 0,419108 | |
| ρ_bulk | 549,47 | kg/m³ |
| d_p | 0,00376 | m |
| p | 1,4 | bar |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,150 | 0,164 | 1046,358 | 0,034 | 0,351 | 10,296 | 0,176 | 5 % |
| 423,180 | 0,166 | 1046,362 | 0,034 | 0,351 | 10,295 | 0,176 | 5 % |
| 423,141 | 0,166 | 1046,357 | 0,034 | 0,351 | 10,296 | 0,176 | 5 % |
| 373,142 | 0,152 | 1042,051 | 0,031 | 0,346 | 11,263 | 0,161 | 6 % |
| 373,134 | 0,155 | 1042,051 | 0,031 | 0,346 | 11,263 | 0,161 | 6 % |
| 373,123 | 0,155 | 1042,050 | 0,031 | 0,346 | 11,263 | 0,161 | 6 % |
| 332,783 | 0,141 | 1040,016 | 0,028 | 0,342 | 12,243 | 0,150 | 7 % |
| 333,645 | 0,143 | 1040,046 | 0,028 | 0,342 | 12,219 | 0,150 | 7 % |
| 332,853 | 0,145 | 1040,018 | 0,028 | 0,342 | 12,241 | 0,150 | 7 % |
| 293,073 | 0,129 | 1039,145 | 0,025 | 0,337 | 13,455 | 0,139 | 7 % |
| 293,658 | 0,131 | 1039,151 | 0,025 | 0,337 | 13,435 | 0,139 | 7 % |
| 292,921 | 0,132 | 1039,144 | 0,025 | 0,337 | 13,460 | 0,139 | 7 % |
| 283,612 | 0,127 | 1039,076 | 0,024 | 0,336 | 13,791 | 0,136 | 8 % |
| 283,142 | 0,130 | 1039,074 | 0,024 | 0,336 | 13,809 | 0,136 | 8 % |
| 283,630 | 0,130 | 1039,076 | 0,024 | 0,336 | 13,791 | 0,136 | 8 % |
| 278,220 | 0,126 | 1039,056 | 0,024 | 0,335 | 13,993 | 0,134 | 8 % |
| 278,155 | 0,129 | 1039,056 | 0,024 | 0,335 | 13,995 | 0,134 | 8 % |
| 278,144 | 0,130 | 1039,056 | 0,024 | 0,335 | 13,996 | 0,134 | 8 % |
| 273,179 | 0,126 | 1039,049 | 0,024 | 0,335 | 14,188 | 0,133 | 8 % |
| 273,166 | 0,129 | 1039,049 | 0,024 | 0,335 | 14,188 | 0,133 | 8 % |
| 273,205 | 0,129 | 1039,049 | 0,024 | 0,335 | 14,187 | 0,133 | 8 % |
| 268,108 | 0,124 | 1039,053 | 0,023 | 0,334 | 14,391 | 0,132 | 8 % |
| 268,102 | 0,128 | 1039,053 | 0,023 | 0,334 | 14,392 | 0,132 | 8 % |
| 268,201 | 0,128 | 1039,052 | 0,023 | 0,334 | 14,388 | 0,132 | 8 % |
| 258,167 | 0,120 | 1039,086 | 0,022 | 0,332 | 14,812 | 0,129 | 8 % |
| 258,125 | 0,124 | 1039,086 | 0,022 | 0,332 | 14,814 | 0,129 | 8 % |
| 258,172 | 0,124 | 1039,086 | 0,022 | 0,332 | 14,812 | 0,129 | 8 % |
| 253,123 | 0,117 | 1039,114 | 0,022 | 0,332 | 15,038 | 0,127 | 8 % |
| 253,150 | 0,121 | 1039,114 | 0,022 | 0,332 | 15,037 | 0,127 | 8 % |
| 253,143 | 0,122 | 1039,114 | 0,022 | 0,332 | 15,037 | 0,127 | 8 % |
| 243,163 | 0,115 | 1039,188 | 0,021 | 0,330 | 15,510 | 0,124 | 8 % |
| 243,162 | 0,117 | 1039,188 | 0,021 | 0,330 | 15,510 | 0,124 | 8 % |
| 243,161 | 0,117 | 1039,188 | 0,021 | 0,330 | 15,510 | 0,124 | 8 % |
| 258,134 | 0,119 | 1039,086 | 0,022 | 0,332 | 14,814 | 0,129 | 8 % |
| 258,140 | 0,123 | 1039,086 | 0,022 | 0,332 | 14,813 | 0,129 | 8 % |
| 258,130 | 0,124 | 1039,086 | 0,022 | 0,332 | 14,814 | 0,129 | 8 % |
| 271,171 | 0,123 | 1039,049 | 0,023 | 0,334 | 14,268 | 0,132 | 8 % |
| 271,166 | 0,128 | 1039,049 | 0,023 | 0,334 | 14,268 | 0,132 | 8 % |

| 271,148 | 0,129 | 1039,049 | 0,023 | 0,334 | 14,269 | 0,132 | 8 % |
|---|---|---|---|---|---|---|---|
| 275,118 | 0,124 | 1039,051 | 0,024 | 0,335 | 14,112 | 0,134 | 8 % |
| 275,195 | 0,128 | 1039,051 | 0,024 | 0,335 | 14,109 | 0,134 | 8 % |
| 275,138 | 0,129 | 1039,051 | 0,024 | 0,335 | 14,111 | 0,134 | 8 % |
| 280,181 | 0,125 | 1039,062 | 0,024 | 0,335 | 13,919 | 0,135 | 8 % |
| 280,155 | 0,129 | 1039,062 | 0,024 | 0,335 | 13,920 | 0,135 | 8 % |
| 280,206 | 0,130 | 1039,062 | 0,024 | 0,335 | 13,918 | 0,135 | 8 % |
| 288,677 | 0,127 | 1039,107 | 0,025 | 0,337 | 13,609 | 0,137 | 7 % |
| 288,226 | 0,130 | 1039,104 | 0,025 | 0,336 | 13,625 | 0,137 | 7 % |
| 288,124 | 0,130 | 1039,103 | 0,025 | 0,336 | 13,628 | 0,137 | 7 % |
| 353,128 | 0,148 | 1040,885 | 0,029 | 0,344 | 11,722 | 0,155 | 6 % |
| 353,141 | 0,150 | 1040,886 | 0,029 | 0,344 | 11,722 | 0,155 | 6 % |
| 353,307 | 0,150 | 1040,894 | 0,029 | 0,344 | 11,718 | 0,156 | 6 % |

| | Cu-btc with He, filling 4, measurement error =5% | | | | | | |
|---|---|---|---|---|---|---|---|

ε_bulk 0,451856
ρ_bulk 518,49 kg/m³
d_p 0,00376 m
p 1,2 bar

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,163 | 0,306 | 5196,250 | 0,196 | 0,351 | 1,790 | 0,297 | 2 % |
| 423,139 | 0,300 | 5196,250 | 0,196 | 0,351 | 1,790 | 0,297 | 2 % |
| 423,152 | 0,300 | 5196,250 | 0,196 | 0,351 | 1,790 | 0,297 | 2 % |
| 372,955 | 0,293 | 5196,250 | 0,179 | 0,346 | 1,928 | 0,281 | 2 % |
| 373,409 | 0,296 | 5196,250 | 0,180 | 0,346 | 1,927 | 0,282 | 2 % |
| 372,883 | 0,290 | 5196,250 | 0,179 | 0,346 | 1,929 | 0,281 | 2 % |
| 332,836 | 0,280 | 5196,250 | 0,166 | 0,342 | 2,061 | 0,269 | 3 % |
| 332,782 | 0,278 | 5196,250 | 0,166 | 0,342 | 2,062 | 0,269 | 3 % |
| 333,305 | 0,275 | 5196,250 | 0,166 | 0,342 | 2,060 | 0,269 | 3 % |
| 292,842 | 0,260 | 5196,250 | 0,152 | 0,337 | 2,220 | 0,256 | 3 % |
| 293,146 | 0,259 | 5196,250 | 0,152 | 0,337 | 2,219 | 0,256 | 3 % |
| 292,846 | 0,260 | 5196,250 | 0,152 | 0,337 | 2,220 | 0,256 | 3 % |
| 283,533 | 0,260 | 5196,250 | 0,148 | 0,336 | 2,262 | 0,253 | 3 % |
| 283,106 | 0,258 | 5196,250 | 0,148 | 0,336 | 2,264 | 0,253 | 3 % |
| 283,198 | 0,257 | 5196,250 | 0,148 | 0,336 | 2,264 | 0,253 | 3 % |
| 278,211 | 0,257 | 5196,250 | 0,147 | 0,335 | 2,287 | 0,252 | 3 % |
| 278,262 | 0,257 | 5196,250 | 0,147 | 0,335 | 2,287 | 0,252 | 3 % |
| 278,238 | 0,257 | 5196,250 | 0,147 | 0,335 | 2,287 | 0,252 | 3 % |
| 273,124 | 0,258 | 5196,250 | 0,145 | 0,335 | 2,311 | 0,250 | 3 % |
| 273,152 | 0,258 | 5196,250 | 0,145 | 0,335 | 2,311 | 0,250 | 3 % |
| 273,093 | 0,258 | 5196,250 | 0,145 | 0,335 | 2,311 | 0,250 | 3 % |
| 268,092 | 0,256 | 5196,250 | 0,143 | 0,334 | 2,336 | 0,248 | 3 % |
| 268,214 | 0,257 | 5196,250 | 0,143 | 0,334 | 2,335 | 0,248 | 3 % |
| 268,146 | 0,257 | 5196,250 | 0,143 | 0,334 | 2,336 | 0,248 | 3 % |
| 263,161 | 0,253 | 5196,250 | 0,141 | 0,333 | 2,361 | 0,247 | 3 % |
| 263,134 | 0,254 | 5196,250 | 0,141 | 0,333 | 2,361 | 0,247 | 3 % |
| 263,128 | 0,254 | 5196,250 | 0,141 | 0,333 | 2,361 | 0,247 | 3 % |
| 253,137 | 0,249 | 5196,250 | 0,137 | 0,332 | 2,413 | 0,243 | 4 % |
| 253,179 | 0,250 | 5196,250 | 0,137 | 0,332 | 2,413 | 0,243 | 4 % |
| 253,129 | 0,250 | 5196,250 | 0,137 | 0,332 | 2,413 | 0,243 | 4 % |
| 243,141 | 0,241 | 5196,250 | 0,134 | 0,330 | 2,469 | 0,240 | 4 % |
| 243,183 | 0,245 | 5196,250 | 0,134 | 0,330 | 2,469 | 0,240 | 4 % |
| 243,173 | 0,246 | 5196,250 | 0,134 | 0,330 | 2,469 | 0,240 | 4 % |
| 258,179 | 0,250 | 5196,250 | 0,139 | 0,332 | 2,387 | 0,245 | 3 % |
| 258,142 | 0,251 | 5196,250 | 0,139 | 0,332 | 2,387 | 0,245 | 3 % |
| 258,148 | 0,251 | 5196,250 | 0,139 | 0,332 | 2,387 | 0,245 | 3 % |
| 271,149 | 0,256 | 5196,250 | 0,144 | 0,334 | 2,321 | 0,249 | 3 % |
| 271,139 | 0,257 | 5196,250 | 0,144 | 0,334 | 2,321 | 0,249 | 3 % |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 271,148 | 0,257 | 5196,250 | 0,144 | 0,334 | 2,321 | 0,249 | 3 % |
| 275,195 | 0,256 | 5196,250 | 0,145 | 0,335 | 2,301 | 0,251 | 3 % |
| 275,200 | 0,257 | 5196,250 | 0,145 | 0,335 | 2,301 | 0,251 | 3 % |
| 275,190 | 0,257 | 5196,250 | 0,145 | 0,335 | 2,301 | 0,251 | 3 % |
| 280,106 | 0,258 | 5196,250 | 0,147 | 0,335 | 2,278 | 0,252 | 3 % |
| 280,109 | 0,257 | 5196,250 | 0,147 | 0,335 | 2,278 | 0,252 | 3 % |
| 280,117 | 0,257 | 5196,250 | 0,147 | 0,335 | 2,278 | 0,252 | 3 % |
| 288,716 | 0,260 | 5196,250 | 0,150 | 0,337 | 2,239 | 0,255 | 3 % |
| 288,290 | 0,257 | 5196,250 | 0,150 | 0,336 | 2,241 | 0,255 | 3 % |
| 288,277 | 0,257 | 5196,250 | 0,150 | 0,336 | 2,241 | 0,255 | 3 % |
| 352,989 | 0,289 | 5196,250 | 0,173 | 0,344 | 1,992 | 0,275 | 2 % |
| 353,276 | 0,283 | 5196,250 | 0,173 | 0,344 | 1,991 | 0,275 | 2 % |
| 353,004 | 0,283 | 5196,250 | 0,173 | 0,344 | 1,992 | 0,275 | 2 % |
| 398,130 | 0,297 | 5196,250 | 0,188 | 0,348 | 1,856 | 0,289 | 2 % |
| 398,172 | 0,295 | 5196,250 | 0,188 | 0,348 | 1,856 | 0,289 | 2 % |
| 398,164 | 0,295 | 5196,250 | 0,188 | 0,348 | 1,856 | 0,289 | 2 % |

Cu-btc with N2, filling 4, measurement error =5%

| | | | | |
|---|---|---|---|
| ε_bulk | 0,451856 | |
| ρ_bulk | 518,49 | kg/m$^3$ |
| d_p | 0,00376 | m |
| p | 1,2 | bar |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,152 | 0,161 | 1046,359 | 0,034 | 0,351 | 10,296 | 0,169 | 5 % |
| 423,147 | 0,164 | 1046,358 | 0,034 | 0,351 | 10,296 | 0,169 | 5 % |
| 423,162 | 0,163 | 1046,360 | 0,034 | 0,351 | 10,295 | 0,169 | 5 % |
| 373,163 | 0,148 | 1042,053 | 0,031 | 0,346 | 11,263 | 0,154 | 6 % |
| 373,153 | 0,150 | 1042,052 | 0,031 | 0,346 | 11,263 | 0,154 | 6 % |
| 373,131 | 0,150 | 1042,050 | 0,031 | 0,346 | 11,263 | 0,154 | 6 % |
| 333,468 | 0,136 | 1040,040 | 0,028 | 0,342 | 12,224 | 0,142 | 7 % |
| 333,604 | 0,138 | 1040,045 | 0,028 | 0,342 | 12,220 | 0,142 | 7 % |
| 332,773 | 0,139 | 1040,015 | 0,028 | 0,342 | 12,243 | 0,142 | 7 % |
| 293,142 | 0,123 | 1039,146 | 0,025 | 0,337 | 13,453 | 0,131 | 8 % |
| 293,117 | 0,125 | 1039,146 | 0,025 | 0,337 | 13,453 | 0,131 | 8 % |
| 293,205 | 0,125 | 1039,147 | 0,025 | 0,337 | 13,450 | 0,131 | 8 % |
| 283,595 | 0,121 | 1039,076 | 0,024 | 0,336 | 13,792 | 0,128 | 8 % |
| 283,282 | 0,124 | 1039,074 | 0,024 | 0,336 | 13,803 | 0,128 | 8 % |
| 283,245 | 0,124 | 1039,074 | 0,024 | 0,336 | 13,805 | 0,128 | 8 % |
| 278,228 | 0,119 | 1039,056 | 0,024 | 0,335 | 13,992 | 0,126 | 8 % |
| 278,188 | 0,123 | 1039,056 | 0,024 | 0,335 | 13,994 | 0,126 | 8 % |
| 278,232 | 0,123 | 1039,056 | 0,024 | 0,335 | 13,992 | 0,126 | 8 % |
| 273,029 | 0,119 | 1039,049 | 0,024 | 0,334 | 14,194 | 0,125 | 8 % |
| 273,203 | 0,123 | 1039,049 | 0,024 | 0,335 | 14,187 | 0,125 | 8 % |
| 273,182 | 0,123 | 1039,049 | 0,024 | 0,335 | 14,188 | 0,125 | 8 % |
| 268,173 | 0,118 | 1039,053 | 0,023 | 0,334 | 14,389 | 0,124 | 8 % |
| 268,129 | 0,121 | 1039,053 | 0,023 | 0,334 | 14,391 | 0,124 | 8 % |
| 268,112 | 0,122 | 1039,053 | 0,023 | 0,334 | 14,391 | 0,124 | 8 % |
| 263,155 | 0,116 | 1039,065 | 0,023 | 0,333 | 14,597 | 0,122 | 8 % |
| 263,152 | 0,120 | 1039,065 | 0,023 | 0,333 | 14,597 | 0,122 | 8 % |
| 263,176 | 0,121 | 1039,065 | 0,023 | 0,333 | 14,596 | 0,122 | 8 % |
| 253,157 | 0,113 | 1039,114 | 0,022 | 0,332 | 15,036 | 0,119 | 9 % |
| 253,120 | 0,116 | 1039,114 | 0,022 | 0,332 | 15,038 | 0,119 | 9 % |
| 253,188 | 0,117 | 1039,114 | 0,022 | 0,332 | 15,035 | 0,119 | 9 % |

Cu-btc_filling 4_N2
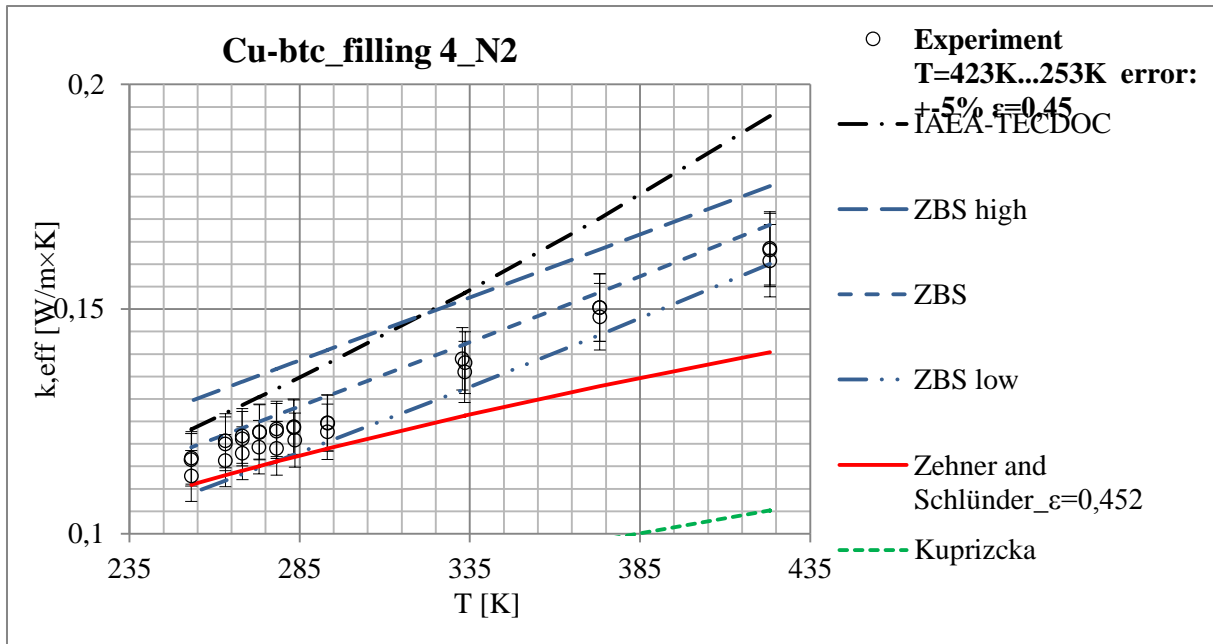
**Appendix E - Fe-btc results**

| Fe-btc with N2, filling 1, measurement error =5% | | | | | | | |
|---|---|---|---|---|---|---|---|

| $\varepsilon$_bulk | 0,423 | | | | | | |
|---|---|---|---|---|---|---|---|
| $\rho$_bulk | 730,15 | kg/m$^3$ | | | | | |
| d_p | 0,00046 | m | | | | | |
| p | 1,2 | bar | | | | | |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,147 | 0,126 | 1046,358 | 0,034 | 0,312 | 9,168 | 0,123 | 7 % |
| 423,167 | 0,128 | 1046,360 | 0,034 | 0,312 | 9,168 | 0,123 | 7 % |
| 423,150 | 0,128 | 1046,358 | 0,034 | 0,312 | 9,168 | 0,123 | 7 % |
| 333,270 | 0,114 | 1040,033 | 0,028 | 0,294 | 10,503 | 0,107 | 8 % |
| 332,712 | 0,114 | 1040,013 | 0,028 | 0,293 | 10,514 | 0,107 | 8 % |
| 332,884 | 0,113 | 1040,019 | 0,028 | 0,293 | 10,510 | 0,107 | 8 % |
| 288,635 | 0,104 | 1039,107 | 0,025 | 0,283 | 11,437 | 0,098 | 8 % |
| 288,162 | 0,104 | 1039,104 | 0,025 | 0,283 | 11,448 | 0,098 | 8 % |
| 288,130 | 0,104 | 1039,103 | 0,025 | 0,283 | 11,449 | 0,098 | 8 % |
| 278,191 | 0,104 | 1039,056 | 0,024 | 0,280 | 11,694 | 0,096 | 9 % |
| 278,069 | 0,104 | 1039,056 | 0,024 | 0,280 | 11,697 | 0,096 | 9 % |
| 278,147 | 0,104 | 1039,056 | 0,024 | 0,280 | 11,695 | 0,096 | 9 % |
| 273,175 | 0,104 | 1039,049 | 0,024 | 0,279 | 11,824 | 0,095 | 9 % |
| 273,216 | 0,104 | 1039,049 | 0,024 | 0,279 | 11,823 | 0,095 | 9 % |
| 273,184 | 0,104 | 1039,049 | 0,024 | 0,279 | 11,824 | 0,095 | 9 % |
| 268,107 | 0,103 | 1039,053 | 0,023 | 0,277 | 11,959 | 0,093 | 9 % |
| 268,189 | 0,103 | 1039,052 | 0,023 | 0,277 | 11,957 | 0,093 | 9 % |
| 268,165 | 0,103 | 1039,053 | 0,023 | 0,277 | 11,958 | 0,093 | 9 % |
| 258,113 | 0,101 | 1039,086 | 0,022 | 0,275 | 12,240 | 0,091 | 9 % |
| 258,132 | 0,101 | 1039,086 | 0,022 | 0,275 | 12,240 | 0,091 | 9 % |
| 258,216 | 0,101 | 1039,086 | 0,022 | 0,275 | 12,237 | 0,091 | 9 % |

Fe-btc with He, filling 2, measurement error =5%

| | | |
|---|---|---|
| ε_bulk | 0,401 | |
| ρ_bulk | 668,32 | kg/m$^3$ |
| d_p | 0,00046 | m |
| p | 1,2 | bar |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,147 | 0,260 | 5196,250 | 0,196 | 0,312 | 1,594 | 0,263 | 2 % |
| 423,147 | 0,261 | 5196,250 | 0,196 | 0,312 | 1,594 | 0,263 | 2 % |
| 423,151 | 0,263 | 5196,250 | 0,196 | 0,312 | 1,594 | 0,263 | 2 % |
| 333,330 | 0,233 | 5196,250 | 0,166 | 0,294 | 1,769 | 0,237 | 3 % |
| 332,754 | 0,233 | 5196,250 | 0,166 | 0,293 | 1,770 | 0,236 | 3 % |
| 333,830 | 0,233 | 5196,250 | 0,166 | 0,294 | 1,768 | 0,237 | 3 % |
| 287,847 | 0,218 | 5196,250 | 0,150 | 0,283 | 1,884 | 0,222 | 3 % |
| 288,035 | 0,217 | 5196,250 | 0,150 | 0,283 | 1,883 | 0,222 | 3 % |
| 288,702 | 0,216 | 5196,250 | 0,150 | 0,283 | 1,881 | 0,222 | 3 % |
| 278,669 | 0,216 | 5196,250 | 0,147 | 0,280 | 1,910 | 0,219 | 3 % |
| 278,529 | 0,216 | 5196,250 | 0,147 | 0,280 | 1,910 | 0,219 | 3 % |
| 278,554 | 0,216 | 5196,250 | 0,147 | 0,280 | 1,910 | 0,219 | 3 % |
| 273,077 | 0,216 | 5196,250 | 0,145 | 0,279 | 1,926 | 0,217 | 3 % |
| 273,345 | 0,216 | 5196,250 | 0,145 | 0,279 | 1,925 | 0,217 | 3 % |
| 273,106 | 0,216 | 5196,250 | 0,145 | 0,279 | 1,926 | 0,217 | 3 % |
| 268,228 | 0,214 | 5196,250 | 0,143 | 0,277 | 1,941 | 0,215 | 3 % |
| 268,042 | 0,215 | 5196,250 | 0,143 | 0,277 | 1,941 | 0,215 | 3 % |
| 268,167 | 0,215 | 5196,250 | 0,143 | 0,277 | 1,941 | 0,215 | 3 % |
| 258,181 | 0,211 | 5196,250 | 0,139 | 0,275 | 1,972 | 0,212 | 3 % |
| 258,167 | 0,210 | 5196,250 | 0,139 | 0,275 | 1,972 | 0,212 | 3 % |
| 258,151 | 0,210 | 5196,250 | 0,139 | 0,275 | 1,972 | 0,212 | 3 % |

Fe-btc_filling 2_He

Fe-btc with N2, filling 2, measurement error =5%

| ε_bulk | 0,401 | |
|--------|-------|------------|
| ρ_bulk | 668,32 | kg/m$^3$ |
| d_p | 0,00046 | m |
| p | 1,2 | bar |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|-------|-----------|----------------|-------------|-------------|-----------|---------------|-------------|
| 423,144 | 0,129 | 1046,358 | 0,034 | 0,312 | 9,168 | 0,128 | 6 % |
| 423,134 | 0,129 | 1046,357 | 0,034 | 0,312 | 9,168 | 0,128 | 6 % |
| 423,173 | 0,129 | 1046,361 | 0,034 | 0,312 | 9,168 | 0,129 | 6 % |
| 333,773 | 0,113 | 1040,051 | 0,028 | 0,294 | 10,494 | 0,112 | 7 % |
| 333,064 | 0,114 | 1040,026 | 0,028 | 0,293 | 10,507 | 0,111 | 7 % |
| 333,207 | 0,113 | 1040,031 | 0,028 | 0,294 | 10,504 | 0,111 | 7 % |
| 288,762 | 0,103 | 1039,108 | 0,025 | 0,283 | 11,434 | 0,102 | 7 % |
| 288,114 | 0,104 | 1039,103 | 0,025 | 0,283 | 11,450 | 0,102 | 7 % |
| 288,074 | 0,104 | 1039,103 | 0,025 | 0,283 | 11,450 | 0,102 | 7 % |
| 278,187 | 0,107 | 1039,056 | 0,024 | 0,280 | 11,694 | 0,100 | 7 % |
| 278,144 | 0,105 | 1039,056 | 0,024 | 0,280 | 11,695 | 0,100 | 7 % |
| 278,346 | 0,104 | 1039,056 | 0,024 | 0,280 | 11,690 | 0,100 | 7 % |
| 272,990 | 0,109 | 1039,049 | 0,024 | 0,279 | 11,829 | 0,099 | 7 % |
| 273,208 | 0,105 | 1039,049 | 0,024 | 0,279 | 11,823 | 0,099 | 7 % |
| 273,125 | 0,105 | 1039,049 | 0,024 | 0,279 | 11,825 | 0,099 | 7 % |
| 268,051 | 0,104 | 1039,053 | 0,023 | 0,277 | 11,961 | 0,098 | 7 % |
| 268,141 | 0,104 | 1039,053 | 0,023 | 0,277 | 11,958 | 0,098 | 7 % |
| 268,189 | 0,104 | 1039,052 | 0,023 | 0,277 | 11,957 | 0,098 | 7 % |
| 258,177 | 0,101 | 1039,086 | 0,022 | 0,275 | 12,238 | 0,096 | 7 % |
| 261,279 | 0,101 | 1039,072 | 0,023 | 0,276 | 12,149 | 0,096 | 7 % |
| 258,075 | 0,101 | 1039,086 | 0,022 | 0,275 | 12,241 | 0,096 | 7 % |

Fe-btc_filling 2_N2

k,eff [W/m×K]

T [K]

Experiment
T=423K...258K error:
±5% ε=0,40
IAEA-TECDOC
ZBS high
ZBS
ZBS low
Zehner and
Schlünder_ε=0,401
Kuprizcka

Fe-btc with He, filling 3, measurement error =5%

| ε_bulk | 0,386 | |
|---|---|---|
| ρ_bulk | 679,72 | kg/m$^3$ |
| d_p | 0,00044 | m |
| p | 1,2 | bar |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,148 | 0,230 | 5196,250 | 0,196 | 0,312 | 1,594 | 0,265 | 2 % |
| 423,174 | 0,228 | 5196,250 | 0,196 | 0,312 | 1,594 | 0,265 | 2 % |
| 423,167 | 0,227 | 5196,250 | 0,196 | 0,312 | 1,594 | 0,265 | 2 % |
| 332,975 | 0,223 | 5196,250 | 0,166 | 0,293 | 1,770 | 0,238 | 2 % |
| 333,496 | 0,223 | 5196,250 | 0,166 | 0,294 | 1,769 | 0,239 | 2 % |
| 332,872 | 0,223 | 5196,250 | 0,166 | 0,293 | 1,770 | 0,238 | 2 % |
| 288,135 | 0,218 | 5196,250 | 0,150 | 0,283 | 1,883 | 0,224 | 3 % |
| 288,204 | 0,218 | 5196,250 | 0,150 | 0,283 | 1,883 | 0,224 | 3 % |
| 288,152 | 0,218 | 5196,250 | 0,150 | 0,283 | 1,883 | 0,224 | 3 % |
| 278,129 | 0,219 | 5196,250 | 0,147 | 0,280 | 1,911 | 0,221 | 3 % |
| 278,125 | 0,220 | 5196,250 | 0,147 | 0,280 | 1,911 | 0,221 | 3 % |
| 278,166 | 0,220 | 5196,250 | 0,147 | 0,280 | 1,911 | 0,221 | 3 % |
| 273,145 | 0,220 | 5196,250 | 0,145 | 0,279 | 1,926 | 0,219 | 3 % |
| 273,287 | 0,220 | 5196,250 | 0,145 | 0,279 | 1,926 | 0,219 | 3 % |
| 273,138 | 0,220 | 5196,250 | 0,145 | 0,279 | 1,926 | 0,219 | 3 % |
| 268,137 | 0,219 | 5196,250 | 0,143 | 0,277 | 1,941 | 0,217 | 3 % |
| 268,135 | 0,219 | 5196,250 | 0,143 | 0,277 | 1,941 | 0,217 | 3 % |
| 268,114 | 0,219 | 5196,250 | 0,143 | 0,277 | 1,941 | 0,217 | 3 % |
| 258,119 | 0,215 | 5196,250 | 0,139 | 0,275 | 1,972 | 0,214 | 3 % |
| 258,123 | 0,215 | 5196,250 | 0,139 | 0,275 | 1,972 | 0,214 | 3 % |
| 258,152 | 0,215 | 5196,250 | 0,139 | 0,275 | 1,972 | 0,214 | 3 % |
| 243,162 | 0,208 | 5196,250 | 0,134 | 0,270 | 2,021 | 0,209 | 3 % |
| 243,143 | 0,209 | 5196,250 | 0,134 | 0,270 | 2,022 | 0,209 | 3 % |
| 243,171 | 0,209 | 5196,250 | 0,134 | 0,270 | 2,021 | 0,209 | 3 % |
| 253,124 | 0,213 | 5196,250 | 0,137 | 0,273 | 1,988 | 0,212 | 3 % |
| 253,143 | 0,213 | 5196,250 | 0,137 | 0,273 | 1,988 | 0,212 | 3 % |
| 253,145 | 0,213 | 5196,250 | 0,137 | 0,273 | 1,988 | 0,212 | 3 % |
| 263,156 | 0,218 | 5196,250 | 0,141 | 0,276 | 1,956 | 0,216 | 3 % |
| 263,146 | 0,218 | 5196,250 | 0,141 | 0,276 | 1,956 | 0,216 | 3 % |
| 263,140 | 0,218 | 5196,250 | 0,141 | 0,276 | 1,956 | 0,216 | 3 % |
| 271,140 | 0,220 | 5196,250 | 0,144 | 0,278 | 1,932 | 0,218 | 3 % |
| 271,132 | 0,220 | 5196,250 | 0,144 | 0,278 | 1,932 | 0,218 | 3 % |
| 271,170 | 0,220 | 5196,250 | 0,144 | 0,278 | 1,932 | 0,218 | 3 % |
| 275,155 | 0,220 | 5196,250 | 0,145 | 0,279 | 1,920 | 0,220 | 3 % |
| 275,175 | 0,220 | 5196,250 | 0,145 | 0,279 | 1,920 | 0,220 | 3 % |
| 275,167 | 0,220 | 5196,250 | 0,145 | 0,279 | 1,920 | 0,220 | 3 % |
| 283,152 | 0,219 | 5196,250 | 0,148 | 0,281 | 1,897 | 0,222 | 3 % |
| 283,139 | 0,219 | 5196,250 | 0,148 | 0,281 | 1,897 | 0,222 | 3 % |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 283,147 | 0,219 | 5196,250 | 0,148 | 0,281 | 1,897 | 0,222 | 3 % |
| 307,817 | 0,221 | 5196,250 | 0,157 | 0,288 | 1,831 | 0,230 | 3 % |
| 308,204 | 0,220 | 5196,250 | 0,157 | 0,288 | 1,830 | 0,231 | 3 % |
| 308,520 | 0,220 | 5196,250 | 0,157 | 0,288 | 1,829 | 0,231 | 3 % |
| 373,137 | 0,229 | 5196,250 | 0,179 | 0,302 | 1,684 | 0,250 | 2 % |
| 373,145 | 0,229 | 5196,250 | 0,179 | 0,302 | 1,684 | 0,250 | 2 % |
| 373,160 | 0,230 | 5196,250 | 0,179 | 0,302 | 1,684 | 0,250 | 2 % |

| | | | Fe-btc with N2, filling 3 measurement error =5% | | | | |
|---|---|---|---|---|---|---|---|

| ε_bulk | 0,386 | |
|---|---|---|
| ρ_bulk | 679,72 | kg/m$^3$ |
| d_p | 0,00044 | m |
| p | 1,2 | bar |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,135 | 0,135 | 1046,357 | 0,034 | 0,312 | 9,168 | 0,132 | 6 % |
| 423,164 | 0,135 | 1046,360 | 0,034 | 0,312 | 9,168 | 0,132 | 6 % |
| 423,149 | 0,135 | 1046,358 | 0,034 | 0,312 | 9,168 | 0,132 | 6 % |
| 333,348 | 0,119 | 1040,036 | 0,028 | 0,294 | 10,502 | 0,115 | 7 % |
| 332,779 | 0,120 | 1040,016 | 0,028 | 0,293 | 10,512 | 0,115 | 7 % |
| 333,785 | 0,119 | 1040,051 | 0,028 | 0,294 | 10,494 | 0,115 | 7 % |
| 288,469 | 0,109 | 1039,106 | 0,025 | 0,283 | 11,441 | 0,105 | 7 % |
| 288,680 | 0,109 | 1039,107 | 0,025 | 0,283 | 11,436 | 0,106 | 7 % |
| 287,981 | 0,109 | 1039,102 | 0,025 | 0,283 | 11,453 | 0,105 | 7 % |
| 278,286 | 0,109 | 1039,056 | 0,024 | 0,280 | 11,692 | 0,103 | 7 % |
| 278,157 | 0,109 | 1039,056 | 0,024 | 0,280 | 11,695 | 0,103 | 7 % |
| 278,305 | 0,109 | 1039,056 | 0,024 | 0,280 | 11,691 | 0,103 | 7 % |
| 273,188 | 0,109 | 1039,049 | 0,024 | 0,279 | 11,823 | 0,102 | 7 % |
| 273,381 | 0,109 | 1039,049 | 0,024 | 0,279 | 11,818 | 0,102 | 7 % |
| 273,161 | 0,109 | 1039,049 | 0,024 | 0,279 | 11,824 | 0,102 | 7 % |
| 268,115 | 0,108 | 1039,053 | 0,023 | 0,277 | 11,959 | 0,101 | 7 % |
| 268,149 | 0,108 | 1039,053 | 0,023 | 0,277 | 11,958 | 0,101 | 7 % |
| 268,181 | 0,108 | 1039,052 | 0,023 | 0,277 | 11,957 | 0,101 | 7 % |
| 258,175 | 0,105 | 1039,086 | 0,022 | 0,275 | 12,238 | 0,099 | 7 % |
| 258,119 | 0,106 | 1039,086 | 0,022 | 0,275 | 12,240 | 0,099 | 7 % |
| 258,132 | 0,106 | 1039,086 | 0,022 | 0,275 | 12,240 | 0,099 | 7 % |
| 243,148 | 0,102 | 1039,188 | 0,021 | 0,270 | 12,700 | 0,095 | 7 % |
| 243,139 | 0,102 | 1039,188 | 0,021 | 0,270 | 12,700 | 0,095 | 7 % |
| 243,145 | 0,102 | 1039,188 | 0,021 | 0,270 | 12,700 | 0,095 | 7 % |
| 253,109 | 0,103 | 1039,114 | 0,022 | 0,273 | 12,388 | 0,098 | 7 % |
| 253,146 | 0,104 | 1039,114 | 0,022 | 0,273 | 12,387 | 0,098 | 7 % |
| 253,163 | 0,104 | 1039,114 | 0,022 | 0,273 | 12,387 | 0,098 | 7 % |
| 263,140 | 0,106 | 1039,065 | 0,023 | 0,276 | 12,097 | 0,100 | 7 % |
| 263,127 | 0,107 | 1039,065 | 0,023 | 0,276 | 12,097 | 0,100 | 7 % |
| 263,143 | 0,107 | 1039,065 | 0,023 | 0,276 | 12,096 | 0,100 | 7 % |
| 271,177 | 0,108 | 1039,049 | 0,023 | 0,278 | 11,877 | 0,102 | 7 % |
| 271,154 | 0,108 | 1039,049 | 0,023 | 0,278 | 11,877 | 0,102 | 7 % |
| 271,072 | 0,108 | 1039,049 | 0,023 | 0,278 | 11,879 | 0,102 | 7 % |
| 275,191 | 0,108 | 1039,051 | 0,024 | 0,279 | 11,771 | 0,103 | 7 % |
| 275,160 | 0,109 | 1039,051 | 0,024 | 0,279 | 11,772 | 0,103 | 7 % |
| 275,161 | 0,109 | 1039,051 | 0,024 | 0,279 | 11,772 | 0,103 | 7 % |
| 283,141 | 0,108 | 1039,074 | 0,024 | 0,281 | 11,570 | 0,104 | 7 % |
| 283,186 | 0,109 | 1039,074 | 0,024 | 0,281 | 11,569 | 0,104 | 7 % |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 283,253 | 0,109 | 1039,074 | 0,024 | 0,281 | 11,567 | 0,104 | 7 % |
| 308,833 | 0,112 | 1039,371 | 0,026 | 0,288 | 10,985 | 0,110 | 7 % |
| 308,648 | 0,113 | 1039,367 | 0,026 | 0,288 | 10,988 | 0,110 | 7 % |
| 308,830 | 0,113 | 1039,371 | 0,026 | 0,288 | 10,985 | 0,110 | 7 % |
| 373,179 | 0,117 | 1042,054 | 0,031 | 0,302 | 9,840 | 0,123 | 6 % |
| 373,133 | 0,124 | 1042,051 | 0,031 | 0,302 | 9,840 | 0,123 | 6 % |

| | | | | |
|---|---|---|---|---|
| | Fe-btc with He, filling 4, measurement error =5% | | | |

| | | |
|---|---|---|
| $\varepsilon$_bulk | 0,384 | |
| $\rho$_bulk | 666,68 | kg/m$^3$ |
| d_p | 0,00044 | m |
| p | 1,4 | bar |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,151 | 0,268 | 5196,250 | 0,196 | 0,312 | 1,594 | 0,265 | 2 % |
| 423,156 | 0,267 | 5196,250 | 0,196 | 0,312 | 1,594 | 0,265 | 2 % |
| 423,159 | 0,267 | 5196,250 | 0,196 | 0,312 | 1,594 | 0,265 | 2 % |
| 332,958 | 0,242 | 5196,250 | 0,166 | 0,293 | 1,770 | 0,239 | 2 % |
| 333,985 | 0,240 | 5196,250 | 0,166 | 0,294 | 1,767 | 0,239 | 2 % |
| 333,292 | 0,241 | 5196,250 | 0,166 | 0,294 | 1,769 | 0,239 | 2 % |
| 288,589 | 0,222 | 5196,250 | 0,150 | 0,283 | 1,882 | 0,224 | 3 % |
| 288,546 | 0,222 | 5196,250 | 0,150 | 0,283 | 1,882 | 0,224 | 3 % |
| 288,312 | 0,222 | 5196,250 | 0,150 | 0,283 | 1,882 | 0,224 | 3 % |
| 278,136 | 0,222 | 5196,250 | 0,147 | 0,280 | 1,911 | 0,221 | 3 % |
| 278,112 | 0,222 | 5196,250 | 0,147 | 0,280 | 1,911 | 0,221 | 3 % |
| 278,114 | 0,222 | 5196,250 | 0,147 | 0,280 | 1,911 | 0,221 | 3 % |
| 273,167 | 0,222 | 5196,250 | 0,145 | 0,279 | 1,926 | 0,219 | 3 % |
| 273,154 | 0,222 | 5196,250 | 0,145 | 0,279 | 1,926 | 0,219 | 3 % |
| 273,134 | 0,222 | 5196,250 | 0,145 | 0,279 | 1,926 | 0,219 | 3 % |
| 268,144 | 0,220 | 5196,250 | 0,143 | 0,277 | 1,941 | 0,218 | 3 % |
| 268,156 | 0,220 | 5196,250 | 0,143 | 0,277 | 1,941 | 0,218 | 3 % |
| 268,176 | 0,220 | 5196,250 | 0,143 | 0,277 | 1,941 | 0,218 | 3 % |
| 258,113 | 0,216 | 5196,250 | 0,139 | 0,275 | 1,972 | 0,214 | 3 % |
| 258,128 | 0,216 | 5196,250 | 0,139 | 0,275 | 1,972 | 0,214 | 3 % |
| 258,127 | 0,215 | 5196,250 | 0,139 | 0,275 | 1,972 | 0,214 | 3 % |
| 243,172 | 0,207 | 5196,250 | 0,134 | 0,270 | 2,021 | 0,209 | 3 % |
| 243,168 | 0,208 | 5196,250 | 0,134 | 0,270 | 2,021 | 0,209 | 3 % |
| 243,206 | 0,208 | 5196,250 | 0,134 | 0,270 | 2,021 | 0,209 | 3 % |
| 253,184 | 0,213 | 5196,250 | 0,137 | 0,273 | 1,988 | 0,212 | 3 % |
| 253,170 | 0,213 | 5196,250 | 0,137 | 0,273 | 1,988 | 0,212 | 3 % |
| 253,128 | 0,213 | 5196,250 | 0,137 | 0,273 | 1,988 | 0,212 | 3 % |
| 263,152 | 0,218 | 5196,250 | 0,141 | 0,276 | 1,956 | 0,216 | 3 % |
| 263,142 | 0,218 | 5196,250 | 0,141 | 0,276 | 1,956 | 0,216 | 3 % |
| 263,126 | 0,218 | 5196,250 | 0,141 | 0,276 | 1,956 | 0,216 | 3 % |

Fe-btc_filling 4_He

Fe-btc with N2, filling 4, measurement error =5%

| ε_bulk | 0,384 | |
|---|---|---|
| ρ_bulk | 666,68 | kg/m$^3$ |
| d_p | 0,00044 | m |
| p | 1,2 | bar |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,161 | 0,138 | 1046,360 | 0,034 | 0,312 | 9,168 | 0,132 | 7 % |
| 423,181 | 0,137 | 1046,362 | 0,034 | 0,312 | 9,168 | 0,132 | 7 % |
| 423,151 | 0,137 | 1046,358 | 0,034 | 0,312 | 9,168 | 0,132 | 7 % |
| 333,063 | 0,123 | 1040,026 | 0,028 | 0,293 | 10,507 | 0,115 | 8 % |
| 333,299 | 0,122 | 1040,034 | 0,028 | 0,294 | 10,503 | 0,115 | 8 % |
| 333,700 | 0,122 | 1040,048 | 0,028 | 0,294 | 10,495 | 0,115 | 8 % |
| 288,441 | 0,112 | 1039,106 | 0,025 | 0,283 | 11,442 | 0,106 | 8 % |
| 288,203 | 0,111 | 1039,104 | 0,025 | 0,283 | 11,447 | 0,106 | 8 % |
| 288,310 | 0,111 | 1039,105 | 0,025 | 0,283 | 11,445 | 0,106 | 8 % |
| 288,596 | 0,111 | 1039,107 | 0,025 | 0,283 | 11,438 | 0,106 | 8 % |
| 278,134 | 0,111 | 1039,056 | 0,024 | 0,280 | 11,696 | 0,104 | 8 % |
| 278,145 | 0,111 | 1039,056 | 0,024 | 0,280 | 11,695 | 0,104 | 8 % |
| 278,100 | 0,111 | 1039,056 | 0,024 | 0,280 | 11,696 | 0,104 | 8 % |
| 273,149 | 0,111 | 1039,049 | 0,024 | 0,279 | 11,824 | 0,103 | 8 % |
| 273,152 | 0,112 | 1039,049 | 0,024 | 0,279 | 11,824 | 0,103 | 8 % |
| 273,131 | 0,111 | 1039,049 | 0,024 | 0,279 | 11,825 | 0,103 | 8 % |
| 268,147 | 0,111 | 1039,053 | 0,023 | 0,277 | 11,958 | 0,101 | 8 % |
| 268,159 | 0,110 | 1039,053 | 0,023 | 0,277 | 11,958 | 0,101 | 8 % |
| 268,157 | 0,110 | 1039,053 | 0,023 | 0,277 | 11,958 | 0,101 | 8 % |
| 268,174 | 0,110 | 1039,053 | 0,023 | 0,277 | 11,957 | 0,101 | 8 % |
| 268,164 | 0,110 | 1039,053 | 0,023 | 0,277 | 11,958 | 0,101 | 8 % |
| 268,161 | 0,110 | 1039,053 | 0,023 | 0,277 | 11,958 | 0,101 | 8 % |
| 243,157 | 0,103 | 1039,188 | 0,021 | 0,270 | 12,699 | 0,096 | 8 % |
| 243,164 | 0,104 | 1039,188 | 0,021 | 0,270 | 12,699 | 0,096 | 8 % |
| 243,172 | 0,104 | 1039,188 | 0,021 | 0,270 | 12,699 | 0,096 | 8 % |
| 253,148 | 0,106 | 1039,114 | 0,022 | 0,273 | 12,387 | 0,098 | 8 % |
| 253,133 | 0,106 | 1039,114 | 0,022 | 0,273 | 12,388 | 0,098 | 8 % |
| 253,191 | 0,106 | 1039,114 | 0,022 | 0,273 | 12,386 | 0,098 | 8 % |
| 263,145 | 0,108 | 1039,065 | 0,023 | 0,276 | 12,096 | 0,100 | 8 % |
| 263,131 | 0,109 | 1039,065 | 0,023 | 0,276 | 12,097 | 0,100 | 8 % |
| 263,170 | 0,109 | 1039,065 | 0,023 | 0,276 | 12,096 | 0,100 | 8 % |
| 271,145 | 0,110 | 1039,049 | 0,023 | 0,278 | 11,877 | 0,102 | 8 % |
| 271,187 | 0,111 | 1039,049 | 0,023 | 0,278 | 11,876 | 0,102 | 8 % |
| 271,125 | 0,111 | 1039,049 | 0,023 | 0,278 | 11,878 | 0,102 | 8 % |
| 275,163 | 0,110 | 1039,051 | 0,024 | 0,279 | 11,772 | 0,103 | 8 % |
| 275,159 | 0,111 | 1039,051 | 0,024 | 0,279 | 11,772 | 0,103 | 8 % |
| 275,140 | 0,111 | 1039,051 | 0,024 | 0,279 | 11,772 | 0,103 | 8 % |
| 283,143 | 0,111 | 1039,074 | 0,024 | 0,281 | 11,570 | 0,105 | 8 % |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 283,140 | 0,111 | 1039,074 | 0,024 | 0,281 | 11,570 | 0,105 | 8 % |
| 308,947 | 0,115 | 1039,373 | 0,026 | 0,288 | 10,982 | 0,110 | 8 % |
| 308,585 | 0,115 | 1039,366 | 0,026 | 0,288 | 10,990 | 0,110 | 8 % |
| 308,442 | 0,115 | 1039,363 | 0,026 | 0,288 | 10,993 | 0,110 | 8 % |
| 373,176 | 0,132 | 1042,053 | 0,031 | 0,302 | 9,840 | 0,123 | 7 % |
| 373,176 | 0,135 | 1042,053 | 0,031 | 0,302 | 9,840 | 0,123 | 7 % |
| 373,137 | 0,129 | 1042,051 | 0,031 | 0,302 | 9,840 | 0,123 | 7 % |

| | Fe-btc with He, filling 5, measurement error =5% | | | | | | |
|---|---|---|---|---|---|---|---|

| ε_bulk | 0,401 | | |
|---|---|---|---|
| ρ_bulk | 650,52 | kg/m³ | |
| d_p | 0,00044 | m | |
| p | 1,4 | bar | |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,169 | 0,264 | 5196,250 | 0,196 | 0,312 | 1,594 | 0,263 | 2 % |
| 423,127 | 0,262 | 5196,250 | 0,196 | 0,312 | 1,594 | 0,263 | 2 % |
| 423,128 | 0,262 | 5196,250 | 0,196 | 0,312 | 1,594 | 0,263 | 2 % |
| 333,493 | 0,236 | 5196,250 | 0,166 | 0,294 | 1,769 | 0,237 | 2 % |
| 332,969 | 0,237 | 5196,250 | 0,166 | 0,293 | 1,770 | 0,236 | 2 % |
| 333,521 | 0,236 | 5196,250 | 0,166 | 0,294 | 1,768 | 0,237 | 2 % |
| 288,122 | 0,218 | 5196,250 | 0,150 | 0,283 | 1,883 | 0,222 | 3 % |
| 288,739 | 0,217 | 5196,250 | 0,150 | 0,283 | 1,881 | 0,222 | 3 % |
| 288,323 | 0,218 | 5196,250 | 0,150 | 0,283 | 1,882 | 0,222 | 3 % |
| 278,218 | 0,218 | 5196,250 | 0,147 | 0,280 | 1,911 | 0,219 | 3 % |
| 278,195 | 0,218 | 5196,250 | 0,147 | 0,280 | 1,911 | 0,219 | 3 % |
| 278,198 | 0,219 | 5196,250 | 0,147 | 0,280 | 1,911 | 0,219 | 3 % |
| 273,206 | 0,218 | 5196,250 | 0,145 | 0,279 | 1,926 | 0,217 | 3 % |
| 273,139 | 0,218 | 5196,250 | 0,145 | 0,279 | 1,926 | 0,217 | 3 % |
| 273,114 | 0,218 | 5196,250 | 0,145 | 0,279 | 1,926 | 0,217 | 3 % |
| 268,135 | 0,216 | 5196,250 | 0,143 | 0,277 | 1,941 | 0,215 | 3 % |
| 268,170 | 0,217 | 5196,250 | 0,143 | 0,277 | 1,941 | 0,215 | 3 % |
| 268,161 | 0,216 | 5196,250 | 0,143 | 0,277 | 1,941 | 0,215 | 3 % |
| 258,165 | 0,212 | 5196,250 | 0,139 | 0,275 | 1,972 | 0,212 | 3 % |
| 258,112 | 0,212 | 5196,250 | 0,139 | 0,275 | 1,972 | 0,212 | 3 % |
| 258,134 | 0,211 | 5196,250 | 0,139 | 0,275 | 1,972 | 0,212 | 3 % |
| 243,154 | 0,204 | 5196,250 | 0,134 | 0,270 | 2,022 | 0,206 | 3 % |
| 243,144 | 0,203 | 5196,250 | 0,134 | 0,270 | 2,022 | 0,206 | 3 % |
| 243,140 | 0,204 | 5196,250 | 0,134 | 0,270 | 2,022 | 0,206 | 3 % |
| 253,118 | 0,208 | 5196,250 | 0,137 | 0,273 | 1,988 | 0,210 | 3 % |
| 253,150 | 0,209 | 5196,250 | 0,137 | 0,273 | 1,988 | 0,210 | 3 % |
| 253,104 | 0,208 | 5196,250 | 0,137 | 0,273 | 1,988 | 0,210 | 3 % |
| 263,113 | 0,215 | 5196,250 | 0,141 | 0,276 | 1,956 | 0,214 | 3 % |
| 263,137 | 0,214 | 5196,250 | 0,141 | 0,276 | 1,956 | 0,214 | 3 % |
| 263,148 | 0,215 | 5196,250 | 0,141 | 0,276 | 1,956 | 0,214 | 3 % |
| 271,175 | 0,217 | 5196,250 | 0,144 | 0,278 | 1,932 | 0,216 | 3 % |
| 271,157 | 0,217 | 5196,250 | 0,144 | 0,278 | 1,932 | 0,216 | 3 % |
| 271,141 | 0,217 | 5196,250 | 0,144 | 0,278 | 1,932 | 0,216 | 3 % |
| 275,151 | 0,218 | 5196,250 | 0,145 | 0,279 | 1,920 | 0,218 | 3 % |
| 275,151 | 0,218 | 5196,250 | 0,145 | 0,279 | 1,920 | 0,218 | 3 % |
| 275,176 | 0,218 | 5196,250 | 0,145 | 0,279 | 1,920 | 0,218 | 3 % |
| 283,224 | 0,218 | 5196,250 | 0,148 | 0,281 | 1,897 | 0,220 | 3 % |
| 283,182 | 0,218 | 5196,250 | 0,148 | 0,281 | 1,897 | 0,220 | 3 % |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 283,106 | 0,219 | 5196,250 | 0,148 | 0,281 | 1,897 | 0,220 | 3 % |
| 308,680 | 0,225 | 5196,250 | 0,157 | 0,288 | 1,828 | 0,229 | 3 % |
| 308,528 | 0,225 | 5196,250 | 0,157 | 0,288 | 1,829 | 0,229 | 3 % |
| 308,585 | 0,224 | 5196,250 | 0,157 | 0,288 | 1,829 | 0,229 | 3 % |
| 373,146 | 0,248 | 5196,250 | 0,179 | 0,302 | 1,684 | 0,249 | 2 % |
| 373,169 | 0,248 | 5196,250 | 0,179 | 0,302 | 1,684 | 0,249 | 2 % |
| 373,133 | 0,249 | 5196,250 | 0,179 | 0,302 | 1,684 | 0,249 | 2 % |

| | Fe-btc with N2, filling 5, measurement error =5% | | | | | | |
|---|---|---|---|---|---|---|---|

| $\varepsilon\_bulk$ | 0,401 | | | | | | |
|---|---|---|---|---|---|---|---|
| $\rho\_bulk$ | 650,52 | kg/m$^3$ | | | | | |
| $d\_p$ | 0,00044 | m | | | | | |
| p | 1,2 | bar | | | | | |

| T [K] | k [W/m×K] | Cp_gas [J/kgK] | k_f [W/m×K] | k_s [W/m×K] | K=k_s/k_f | k_ZBS [W/m×K] | Δk_ZBS/kZBS |
|---|---|---|---|---|---|---|---|
| 423,154 | 0,131 | 1046,359 | 0,034 | 0,312 | 9,168 | 0,128 | 7 % |
| 423,136 | 0,131 | 1046,357 | 0,034 | 0,312 | 9,168 | 0,128 | 7 % |
| 423,139 | 0,131 | 1046,357 | 0,034 | 0,312 | 9,168 | 0,128 | 7 % |
| 332,886 | 0,115 | 1040,019 | 0,028 | 0,293 | 10,510 | 0,111 | 8 % |
| 333,601 | 0,115 | 1040,045 | 0,028 | 0,294 | 10,497 | 0,111 | 8 % |
| 333,664 | 0,115 | 1040,047 | 0,028 | 0,294 | 10,496 | 0,111 | 8 % |
| 288,735 | 0,105 | 1039,108 | 0,025 | 0,283 | 11,435 | 0,102 | 8 % |
| 288,098 | 0,106 | 1039,103 | 0,025 | 0,283 | 11,450 | 0,102 | 8 % |
| 288,022 | 0,105 | 1039,103 | 0,025 | 0,283 | 11,452 | 0,102 | 8 % |
| 278,174 | 0,105 | 1039,056 | 0,024 | 0,280 | 11,695 | 0,100 | 8 % |
| 278,171 | 0,105 | 1039,056 | 0,024 | 0,280 | 11,695 | 0,100 | 8 % |
| 278,156 | 0,105 | 1039,056 | 0,024 | 0,280 | 11,695 | 0,100 | 8 % |
| 273,122 | 0,105 | 1039,049 | 0,024 | 0,279 | 11,825 | 0,099 | 8 % |
| 273,091 | 0,105 | 1039,049 | 0,024 | 0,279 | 11,826 | 0,099 | 8 % |
| 273,145 | 0,105 | 1039,049 | 0,024 | 0,279 | 11,825 | 0,099 | 8 % |
| 268,154 | 0,104 | 1039,053 | 0,023 | 0,277 | 11,958 | 0,098 | 8 % |
| 268,121 | 0,104 | 1039,053 | 0,023 | 0,277 | 11,959 | 0,098 | 8 % |
| 268,142 | 0,104 | 1039,053 | 0,023 | 0,277 | 11,958 | 0,098 | 8 % |
| 258,163 | 0,102 | 1039,086 | 0,022 | 0,275 | 12,239 | 0,096 | 8 % |
| 258,139 | 0,102 | 1039,086 | 0,022 | 0,275 | 12,240 | 0,096 | 8 % |
| 258,134 | 0,102 | 1039,086 | 0,022 | 0,275 | 12,240 | 0,096 | 8 % |
| 243,165 | 0,098 | 1039,188 | 0,021 | 0,270 | 12,699 | 0,092 | 8 % |
| 243,152 | 0,098 | 1039,188 | 0,021 | 0,270 | 12,700 | 0,092 | 8 % |
| 243,131 | 0,098 | 1039,188 | 0,021 | 0,270 | 12,700 | 0,092 | 8 % |

Fe-btc_filling 5_N2
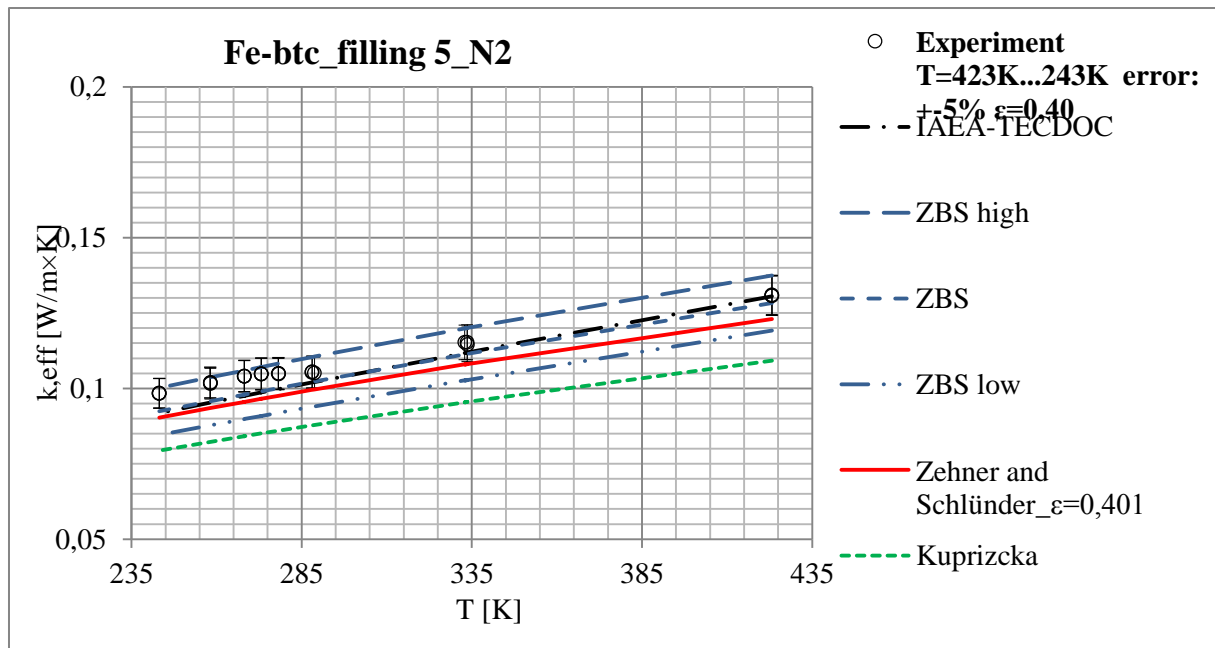
**Appendix F - Mail from Hot Disk**

---

**From:** Daniel Cederkrantz [mailto:daniel.cederkrantz@hotdisk.se]
**Sent:** 30. mai 2013 16:59
**To:** Christian Schlemminger
**Subject:** Re: Questions to cryo probe and measurment results

Hello again Christian,

I've been looking at your data, and I see two possible reasons for the discontinuity around 0 C.

1) The silicone oil that seeps into the chamber. However, I find this unlikely unless water joins the silicone oil in finding its way into the chamber. On a side-note here, I've been discussing the silicone oil problem with the manufacturer and they are now considering the problem. As a quick-fix they suggest arranging some kind of simple gasket or lid which prevents the silicone oil leaving the bath.

2) The TCR-values around 0 C could be less than perfect. I plotted the TCR-values (which you find here: C:\HotDiskTPS_7.1\data\Config\TCR-values.tcr) and there is a hint of a "bump" in the data around 0 C. I will investigate this phenomenon, and correct the TCR-data if needed, but I suspect this will take some time. However, you have the equipment necessary to gather TCR-data. If you decide to calibrate TCR on your own, just edit the TCR-file with you new TCR-values. You can even add new temperatures if you want to minimize the interpolation of TCR-values. It is also this file you need to edit when you add the TCR-values for temperatures below -80 C.

Testing of maximum cable-temperature remains to be performed.

Daniel

## Appendix G - Foam results

| Glass/Foam/N2 experiment 1 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| $\varepsilon\_tot$ | 0,396808 | | R | 8314 | | e | 0,174667 | |
| $\varepsilon\_spec$ | 0,425047 | | Cp_gas | 1039 | | $\eta\_high$ | 0,647214 | |
| $\varepsilon\_foam$ | 0,933564 | | dp | 0,00038 | | $\eta\_low$ | 0,57735 | |
| | | | | | | $\eta\_middle$ | 0,612282 | |

| T | k_e | k_f | k_s_glass | k_ZBS | k_s_alu | k_phase-sym_mod | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | High bound | Low Bound | Middle |
| [K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] |
| 423,14 | 1,495707 | 0,034064 | 1,243263 | 0,213317 | 208,8957 | 4,804016 | 4,310467 | 4,557241 |
| 423,14 | 1,497131 | 0,034064 | 1,243263 | 0,213317 | 208,8957 | 4,804016 | 4,310467 | 4,557241 |
| 423,137 | 1,495335 | 0,034063 | 1,243261 | 0,213316 | 208,8957 | 4,804016 | 4,310466 | 4,557241 |
| 333,602 | 1,290802 | 0,027971 | 1,158053 | 0,181862 | 210,665 | 4,808484 | 4,310821 | 4,559653 |
| 333,235 | 1,289158 | 0,027945 | 1,157511 | 0,181719 | 210,6749 | 4,808544 | 4,310858 | 4,559701 |
| 332,739 | 1,290415 | 0,02791 | 1,156777 | 0,181524 | 210,6883 | 4,808625 | 4,310908 | 4,559767 |
| 288,865 | 1,163587 | 0,024742 | 1,080516 | 0,16348 | 211,8803 | 4,815015 | 4,314521 | 4,564768 |
| 287,947 | 1,166929 | 0,024675 | 1,078681 | 0,163084 | 211,8989 | 4,81499 | 4,314452 | 4,564721 |
| 288,225 | 1,165652 | 0,024695 | 1,079238 | 0,163204 | 211,8932 | 4,814994 | 4,31447 | 4,564732 |
| 277,914 | 1,161333 | 0,023931 | 1,057986 | 0,158712 | 212,1877 | 4,816529 | 4,315319 | 4,565924 |
| 278,606 | 1,158105 | 0,023983 | 1,059451 | 0,159016 | 212,1633 | 4,81633 | 4,315176 | 4,565753 |
| 278,299 | 1,159503 | 0,02396 | 1,058802 | 0,158881 | 212,1741 | 4,816417 | 4,315238 | 4,565828 |
| 273,148 | 1,153445 | 0,023575 | 1,047745 | 0,156603 | 212,3694 | 4,818202 | 4,316566 | 4,567384 |
| 273,164 | 1,152823 | 0,023576 | 1,04778 | 0,156611 | 212,3687 | 4,818195 | 4,316561 | 4,567378 |
| 273,144 | 1,153376 | 0,023575 | 1,047737 | 0,156602 | 212,3696 | 4,818203 | 4,316568 | 4,567385 |
| 268,236 | 1,142562 | 0,023207 | 1,036914 | 0,15441 | 212,5785 | 4,820379 | 4,318254 | 4,569316 |
| 268,138 | 1,142635 | 0,0232 | 1,036695 | 0,154366 | 212,5829 | 4,820426 | 4,318291 | 4,569358 |
| 268,167 | 1,141879 | 0,023202 | 1,03676 | 0,154379 | 212,5816 | 4,820412 | 4,31828 | 4,569346 |
| 258,15 | 1,112312 | 0,022445 | 1,013793 | 0,149838 | 213,0531 | 4,825763 | 4,322527 | 4,574145 |
| 258,17 | 1,110462 | 0,022447 | 1,01384 | 0,149847 | 213,0521 | 4,825751 | 4,322518 | 4,574135 |
| 258,158 | 1,110635 | 0,022446 | 1,013812 | 0,149842 | 213,0527 | 4,825758 | 4,322523 | 4,574141 |
| 243,17 | 1,058731 | 0,021299 | 0,977268 | 0,142882 | 213,7914 | 4,834303 | 4,329339 | 4,581821 |
| 243,163 | 1,059883 | 0,021299 | 0,97725 | 0,142879 | 213,7918 | 4,834307 | 4,329342 | 4,581825 |
| 243,123 | 1,060475 | 0,021296 | 0,977149 | 0,14286 | 213,7937 | 4,834329 | 4,329359 | 4,581844 |

| Glass/Foam/N2 experiment 2 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| ε_tot | 0,378012 | | R | 8314 | | e | 0,174667 | |
| ε_spec | 0,404496 | | Cp_gas | 1039 | | η_high | 0,647214 | |
| ε_foam | 0,934526 | | dp | 0,00038 | | η_low | 0,57735 | |
| | | | | | | η_middle | 0,612282 | |

| T | k_e | k_f | k_s_glass | k_ZBS | k_s_alu | k_phase-sym_mod | | |
| | | | | | | High bound | Low Bound | Middle |
| [K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] |
|---|---|---|---|---|---|---|---|---|
| 423,136 | 1,601409 | 0,034063 | 1,243261 | 0,225745 | 208,8957 | 4,749694 | 4,263399 | 4,506553 |
| 423,16 | 1,598644 | 0,034065 | 1,243271 | 0,225752 | 208,8954 | 4,749696 | 4,263401 | 4,506555 |
| 423,158 | 1,597921 | 0,034065 | 1,24327 | 0,225752 | 208,8954 | 4,749696 | 4,263401 | 4,506555 |
| 333,798 | 1,383352 | 0,027985 | 1,158341 | 0,192981 | 210,6597 | 4,752145 | 4,261812 | 4,506984 |
| 333,551 | 1,3814 | 0,027967 | 1,157978 | 0,192879 | 210,6664 | 4,752178 | 4,261829 | 4,507009 |
| 333,108 | 1,381736 | 0,027936 | 1,157324 | 0,192697 | 210,6783 | 4,752238 | 4,261862 | 4,507055 |
| 287,871 | 1,242345 | 0,024669 | 1,078528 | 0,173108 | 211,9005 | 4,757265 | 4,264085 | 4,51068 |
| 287,846 | 1,240873 | 0,024667 | 1,078478 | 0,173096 | 211,9011 | 4,757264 | 4,264083 | 4,510678 |
| 288,444 | 1,236423 | 0,024711 | 1,079676 | 0,173368 | 211,8887 | 4,757293 | 4,26414 | 4,510721 |
| 278,571 | 1,227102 | 0,02398 | 1,059377 | 0,168836 | 212,1645 | 4,758301 | 4,264516 | 4,511413 |
| 278,094 | 1,229075 | 0,023944 | 1,058368 | 0,168615 | 212,1813 | 4,758421 | 4,264597 | 4,511513 |
| 278,205 | 1,228762 | 0,023953 | 1,058603 | 0,168666 | 212,1774 | 4,758392 | 4,264577 | 4,51149 |
| 273,149 | 1,22474 | 0,023575 | 1,047748 | 0,166307 | 212,3694 | 4,75996 | 4,265703 | 4,512836 |
| 273,149 | 1,223047 | 0,023575 | 1,047748 | 0,166307 | 212,3694 | 4,75996 | 4,265703 | 4,512836 |
| 273,127 | 1,223404 | 0,023574 | 1,0477 | 0,166297 | 212,3702 | 4,759968 | 4,265709 | 4,512843 |
| 268,117 | 1,210505 | 0,023198 | 1,036648 | 0,163935 | 212,5838 | 4,761996 | 4,267245 | 4,514625 |
| 268,091 | 1,208131 | 0,023196 | 1,03659 | 0,163922 | 212,585 | 4,762008 | 4,267254 | 4,514636 |
| 268,125 | 1,208094 | 0,023199 | 1,036666 | 0,163939 | 212,5834 | 4,761993 | 4,267242 | 4,514622 |
| 258,119 | 1,172856 | 0,022443 | 1,01372 | 0,159149 | 213,0546 | 4,766926 | 4,271089 | 4,519012 |
| 258,167 | 1,17279 | 0,022446 | 1,013833 | 0,159172 | 213,0523 | 4,7669 | 4,271069 | 4,518989 |
| 258,115 | 1,173265 | 0,022442 | 1,013711 | 0,159147 | 213,0548 | 4,766928 | 4,271091 | 4,519014 |
| 243,161 | 1,115929 | 0,021299 | 0,977245 | 0,151807 | 213,7919 | 4,774806 | 4,277269 | 4,526042 |
| 243,166 | 1,113643 | 0,021299 | 0,977257 | 0,151809 | 213,7916 | 4,774803 | 4,277267 | 4,526039 |
| 243,171 | 1,11467 | 0,021299 | 0,97727 | 0,151812 | 213,7914 | 4,774801 | 4,277265 | 4,526037 |
| 253,13 | 1,156673 | 0,022063 | 1,001844 | 0,156724 | 213,3008 | 4,769588 | 4,273183 | 4,52139 |
| 253,148 | 1,154958 | 0,022064 | 1,001887 | 0,156733 | 213,2999 | 4,769578 | 4,273176 | 4,521381 |
| 253,154 | 1,155314 | 0,022065 | 1,001902 | 0,156736 | 213,2996 | 4,769575 | 4,273173 | 4,521378 |
| 263,146 | 1,199547 | 0,022823 | 1,025394 | 0,161567 | 212,8124 | 4,764342 | 4,269064 | 4,516707 |
| 263,091 | 1,198774 | 0,022819 | 1,025268 | 0,161541 | 212,815 | 4,764369 | 4,269085 | 4,516731 |
| 263,136 | 1,198776 | 0,022823 | 1,025371 | 0,161562 | 212,8128 | 4,764347 | 4,269067 | 4,516712 |
| 271,162 | 1,221215 | 0,023427 | 1,0434 | 0,165373 | 212,4516 | 4,760715 | 4,266269 | 4,513496 |
| 271,132 | 1,221394 | 0,023424 | 1,043334 | 0,165359 | 212,4529 | 4,760727 | 4,266278 | 4,513507 |
| 271,155 | 1,220988 | 0,023426 | 1,043385 | 0,16537 | 212,4519 | 4,760718 | 4,266271 | 4,513499 |
| 275,105 | 1,233312 | 0,023722 | 1,051983 | 0,167223 | 212,2919 | 4,759289 | 4,26521 | 4,512254 |
| 275,211 | 1,232007 | 0,023729 | 1,052211 | 0,167272 | 212,2878 | 4,759255 | 4,265185 | 4,512225 |
| 275,205 | 1,231066 | 0,023729 | 1,052198 | 0,167269 | 212,2881 | 4,759257 | 4,265187 | 4,512226 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 283,114 | 1,24205 | 0,024317 | 1,068858 | 0,170933 | 212,0198 | 4,757467 | 4,264014 | 4,510745 |
| 283,276 | 1,241926 | 0,024329 | 1,069192 | 0,171007 | 212,0152 | 4,757448 | 4,264006 | 4,510732 |
| 283,09 | 1,240256 | 0,024316 | 1,068809 | 0,170922 | 212,0205 | 4,75747 | 4,264015 | 4,510747 |
| 307,98 | 1,29967 | 0,026138 | 1,116496 | 0,182059 | 211,4073 | 4,75638 | 4,26433 | 4,51036 |
| 308,2 | 1,296259 | 0,026154 | 1,116885 | 0,182155 | 211,4005 | 4,756338 | 4,264303 | 4,510325 |
| 307,629 | 1,298313 | 0,026113 | 1,115874 | 0,181906 | 211,4182 | 4,756449 | 4,264373 | 4,510416 |
| 373,138 | 1,479611 | 0,030718 | 1,207184 | 0,208381 | 209,7256 | 4,748741 | 4,26055 | 4,504652 |
| 373,16 | 1,47833 | 0,03072 | 1,207206 | 0,208389 | 209,7251 | 4,74874 | 4,26055 | 4,504651 |
| 373,143 | 1,477543 | 0,030718 | 1,207189 | 0,208383 | 209,7255 | 4,748741 | 4,26055 | 4,504652 |

| | | | | Glass/Foam/N2 experiment 3 | | | | |
|---|---|---|---|---|---|---|---|---|

| ε_tot | 0,368833 | | R | 8314 | | e | 0,174667 | |
| ε_spec | 0,394674 | | Cp_gas | 1039 | | η_high | 0,647214 | |
| ε_foam | 0,934526 | | dp | 0,00038 | | η_low | 0,57735 | |
| | | | | | | η_middle | 0,612282 | |

| T | k_e | k_f | k_s_glass | k_ZBS | k_s_alu | k_phase-sym_mod | | |
| | | | | | | High bound | Low Bound | Middle |
| [K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] |
|---|---|---|---|---|---|---|---|---|
| 423,157 | 1,586984 | 0,034065 | 1,24327 | 0,213322 | 208,8954 | 4,804017 | 4,310468 | 4,557249 |
| 423,121 | 1,587249 | 0,034062 | 1,243254 | 0,213311 | 208,8959 | 4,804015 | 4,310465 | 4,557246 |
| 423,176 | 1,587353 | 0,034066 | 1,243278 | 0,213328 | 208,8952 | 4,804018 | 4,31047 | 4,55725 |
| 333,276 | 1,374061 | 0,027948 | 1,157572 | 0,181735 | 210,6738 | 4,808537 | 4,310854 | 4,559701 |
| 333,537 | 1,372245 | 0,027966 | 1,157957 | 0,181837 | 210,6667 | 4,808495 | 4,310828 | 4,559667 |
| 332,913 | 1,375093 | 0,027922 | 1,157035 | 0,181593 | 210,6836 | 4,808597 | 4,310891 | 4,559749 |
| 288,494 | 1,242644 | 0,024715 | 1,079775 | 0,16332 | 211,8877 | 4,815001 | 4,31449 | 4,56475 |
| 288,746 | 1,240326 | 0,024733 | 1,080279 | 0,163428 | 211,8827 | 4,81501 | 4,31451 | 4,564765 |
| 287,906 | 1,243214 | 0,024672 | 1,078599 | 0,163066 | 211,8998 | 4,81499 | 4,31445 | 4,564724 |
| 278,569 | 1,232047 | 0,02398 | 1,059373 | 0,159 | 212,1646 | 4,81634 | 4,315184 | 4,565766 |
| 278,087 | 1,234315 | 0,023944 | 1,058353 | 0,158788 | 212,1815 | 4,816478 | 4,315282 | 4,565885 |
| 278,092 | 1,234265 | 0,023944 | 1,058364 | 0,15879 | 212,1813 | 4,816477 | 4,315281 | 4,565884 |
| 273,192 | 1,228913 | 0,023579 | 1,047841 | 0,156623 | 212,3676 | 4,818184 | 4,316553 | 4,567373 |
| 273,203 | 1,228172 | 0,023579 | 1,047865 | 0,156628 | 212,3672 | 4,81818 | 4,31655 | 4,567369 |
| 273,102 | 1,228605 | 0,023572 | 1,047645 | 0,156583 | 212,3713 | 4,81822 | 4,31658 | 4,567405 |
| 268,119 | 1,21562 | 0,023198 | 1,036653 | 0,154357 | 212,5837 | 4,820435 | 4,318298 | 4,569371 |
| 268,1 | 1,217149 | 0,023197 | 1,03661 | 0,154349 | 212,5846 | 4,820444 | 4,318305 | 4,569379 |
| 268,187 | 1,215822 | 0,023203 | 1,036805 | 0,154388 | 212,5807 | 4,820402 | 4,318273 | 4,569342 |
| 258,085 | 1,184314 | 0,02244 | 1,01364 | 0,149809 | 213,0563 | 4,8258 | 4,322557 | 4,574183 |
| 258,152 | 1,184025 | 0,022445 | 1,013798 | 0,149839 | 213,053 | 4,825762 | 4,322526 | 4,574148 |
| 258,123 | 1,183569 | 0,022443 | 1,013729 | 0,149826 | 213,0544 | 4,825778 | 4,322539 | 4,574163 |
| 243,141 | 1,127817 | 0,021297 | 0,977194 | 0,142868 | 213,7928 | 4,834319 | 4,329351 | 4,581839 |
| 243,144 | 1,128659 | 0,021297 | 0,977202 | 0,14287 | 213,7927 | 4,834318 | 4,32935 | 4,581838 |
| 243,143 | 1,128118 | 0,021297 | 0,977199 | 0,142869 | 213,7927 | 4,834318 | 4,32935 | 4,581838 |
| 253,155 | 1,162237 | 0,022065 | 1,001904 | 0,147541 | 213,2996 | 4,828642 | 4,324829 | 4,57674 |
| 253,175 | 1,162855 | 0,022066 | 1,001952 | 0,14755 | 213,2986 | 4,828631 | 4,32482 | 4,576729 |
| 253,138 | 1,163668 | 0,022064 | 1,001863 | 0,147533 | 213,3004 | 4,828652 | 4,324837 | 4,576749 |

Glass/Foam/N2 experiment 4

| ε_tot | 0,389464 | R | 8314 | e | 0,174667 |
|---|---|---|---|---|---|
| ε_fspec | 0,416333 | Cp_gas | 1039 | η_high | 0,647214 |
| ε_foam | 0,935462 | dp | 0,00038 | η_low | 0,57735 |
| | | | | η_middle | 0,612282 |

| T | k_e | k_f | k_s_glass | k_ZBS | k_s_alu | k_phase-sym_mod | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | High bound | Low Bound | Middle |
| [K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] | [W/m×K] |
| 423,15 | 1,636716 | 0,034064 | 1,243267 | 0,218492 | 208,8955 | 4,675974 | 4,196761 | 4,436374 |
| 423,154 | 1,637426 | 0,034065 | 1,243269 | 0,218493 | 208,8955 | 4,675975 | 4,196762 | 4,436374 |
| 423,152 | 1,635095 | 0,034064 | 1,243268 | 0,218492 | 208,8955 | 4,675975 | 4,196761 | 4,436374 |
| 333,428 | 1,438211 | 0,027959 | 1,157796 | 0,186385 | 210,6697 | 4,678838 | 4,195621 | 4,437234 |
| 333,06 | 1,435379 | 0,027933 | 1,157253 | 0,186237 | 210,6796 | 4,678889 | 4,195649 | 4,437274 |
| 333,723 | 1,431661 | 0,027979 | 1,158231 | 0,186502 | 210,6617 | 4,678797 | 4,195598 | 4,437203 |
| 288,239 | 1,307131 | 0,024696 | 1,079266 | 0,167396 | 211,8929 | 4,68418 | 4,198196 | 4,441193 |
| 288,816 | 1,302173 | 0,024739 | 1,080418 | 0,167651 | 211,8813 | 4,684211 | 4,198253 | 4,441237 |
| 288,023 | 1,305473 | 0,02468 | 1,078833 | 0,167301 | 211,8973 | 4,684172 | 4,198177 | 4,441179 |
| 278,223 | 1,299067 | 0,023954 | 1,058641 | 0,162935 | 212,1767 | 4,685354 | 4,198728 | 4,442046 |
| 278,133 | 1,298212 | 0,023947 | 1,058451 | 0,162894 | 212,1799 | 4,685378 | 4,198744 | 4,442065 |
| 278,31 | 1,299013 | 0,023961 | 1,058825 | 0,162974 | 212,1737 | 4,685332 | 4,198713 | 4,442027 |
| 273,138 | 1,294658 | 0,023575 | 1,047724 | 0,160635 | 212,3698 | 4,686952 | 4,199887 | 4,443424 |
| 273,164 | 1,292856 | 0,023576 | 1,04778 | 0,160646 | 212,3687 | 4,686942 | 4,19988 | 4,443416 |
| 273,172 | 1,293754 | 0,023577 | 1,047798 | 0,16065 | 212,3684 | 4,686939 | 4,199878 | 4,443413 |
| 268,097 | 1,283389 | 0,023196 | 1,036603 | 0,158331 | 212,5847 | 4,689008 | 4,201455 | 4,445236 |
| 268,124 | 1,281708 | 0,023199 | 1,036664 | 0,158343 | 212,5835 | 4,688996 | 4,201446 | 4,445225 |
| 268,149 | 1,281034 | 0,0232 | 1,03672 | 0,158355 | 212,5824 | 4,688985 | 4,201437 | 4,445215 |
| 258,177 | 1,252357 | 0,022447 | 1,013856 | 0,153729 | 213,0518 | 4,693918 | 4,205303 | 4,449615 |
| 258,137 | 1,250687 | 0,022444 | 1,013762 | 0,153711 | 213,0537 | 4,693939 | 4,20532 | 4,449634 |
| 258,114 | 1,250016 | 0,022442 | 1,013708 | 0,1537 | 213,0549 | 4,693951 | 4,20533 | 4,449644 |
| 243,143 | 1,195376 | 0,021297 | 0,977199 | 0,146583 | 213,7927 | 4,701867 | 4,211568 | 4,456721 |
| 243,15 | 1,194642 | 0,021298 | 0,977217 | 0,146586 | 213,7924 | 4,701863 | 4,211565 | 4,456718 |
| 243,15 | 1,194623 | 0,021298 | 0,977217 | 0,146586 | 213,7924 | 4,701863 | 4,211565 | 4,456718 |

glass/foam_⌀0.38mm_N2
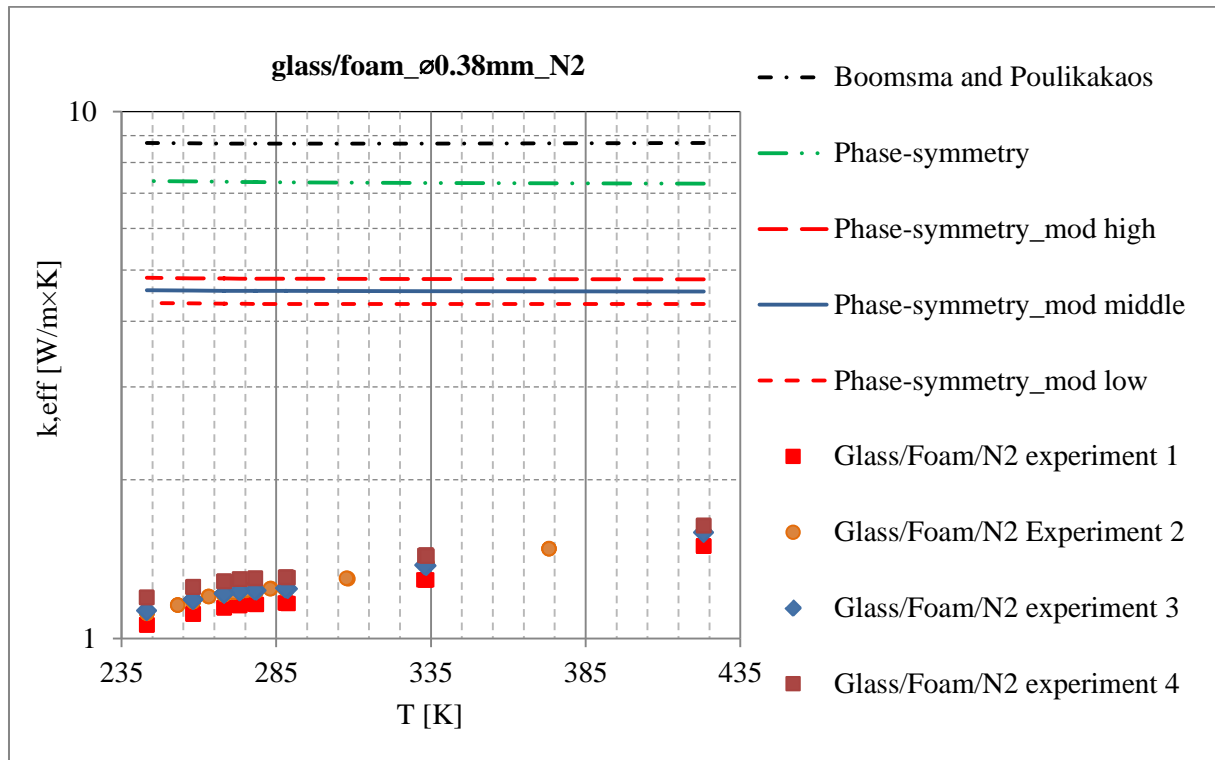
**Appendix H - Risk Assessment Report**

# Risk Assessment Report

Termisk konduktivitetsrigg: måling av termisk konduktivitet i porøse medier.

## [Thermal conductivity rig: measurements of thermal conductivity in porous media]

| Prosjekttittel | Conductivity rig: thermal conductivity measurements in porous media |
|---|---|
| Prosjektleder | Erling Næss/Christian Schlemminger |
| Enhet | NTNU |
| HMS-koordinator | Erik Langørgen |
| Linjeleder | Olav Bolland |
| Plassering | VATlab |
| Romnummer | Finlab |
| Riggansvarlig | Jan Georg Henriksen/Christian Schlemminger |
| Risikovurdering utført av | Erik Langørgen/Christian Schlemminger/Jan Georg Henriksen |
| | |

# TABLE OF CONTENTS

# 1    INTRODUCTION

The experiment is about measuring the thermal conductivity in a packed bed of porous and non porous media, in order to calculate the respective conductivity's of the sample media.

The rig is located in the FinLab in floor 1B, inside the VATlab.

# 2    ORGANISATION

| Rolle | NTNU | Sintef |
|---|---|---|
| Lab Ansvarlig: | Morten Grønli | Harald Mæhlum |
| Linjeleder: | Olav Bolland | Mona Mølnvik |
| HMS ansvarlig:<br>HMS koordinator<br>HMS koordinator | Olav Bolland<br>Erik Langørgen<br>Bård Brandåstrø | Mona Mølnvik<br>Harald Mæhlum |
| Romansvarlig: | Jan Georg Henriksen | |
| Prosjektleder: | Erling Næss/Christian Schlemminger | |
| Ansvarlig riggoperatører: | Jan Georg Henriksen | |

# 3    RISK MANAGEMENT IN THE PROJECT

| Hovedaktiviteter risikostyring | Nødvendige tiltak, dokumentasjon | DATE |
|---|---|---|
| Prosjekt initiering | Prosjekt initiering mal | 15.01.2013 |
| Veiledningsmøte<br>Guidance Meeting | Skjema for Veiledningsmøte med pre-risikovurdering | n.a |
| Innledende risikovurdering<br>Initial Assessment | Fareidentifikasjon – HAZID<br>Skjema grovanalyse | 20.02.2013 |
| Vurdering av teknisk sikkerhet<br>Evaluation of technical security | Prosess-HAZOP<br>Tekniske dokumentasjoner | 15.05.2013 |
| Vurdering av operasjonell sikkerhet<br>Evaluation of operational safety | Prosedyre-HAZOP<br>Opplæringsplan for operatører | 15.05.2013 |
| Sluttvurdering, kvalitetssikring<br>Final assessment, quality assurance | Uavhengig kontroll<br>Utstedelse av apparaturkort<br>Utstedelse av forsøk pågår kort | 15.05.2013 |

# 4    DRAWINGS, PHOTOS, DESCRIPTIONS OF TEST SETUP

**Test setup description/location of equipment**

The thermal conductivity measurement rig is located in the FinLab in the VATLab. The HotDisk TPS 2500S is supplied by the Swedish company Hot Disc AB. It utilizes a small electrical power (<2W) to heat up a special shaped resistance, which is used as a temperature sensor simultaneously. The transient temperature rise of the sensor is recorded and analyzed by the TPS2500S. As result the material thermal diffusivity, specific heat capacity and thermal conductivity are determined. Different material

types e.g. solid materials or powders can be measured. In the particular case different sorts of powders are used for the measurements.
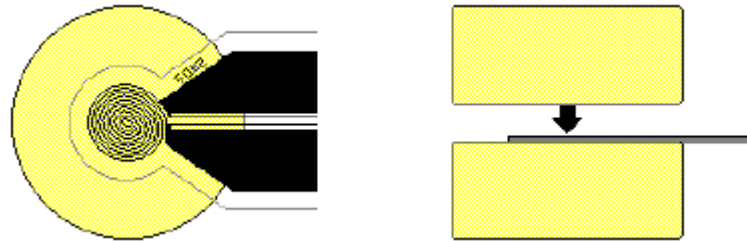


Figure 1 Sensor position between sample pieces

. Porous media is poured in to a sample holder, and a HotDisk sensor is placed in between the samples, as shown in Figure. 1.
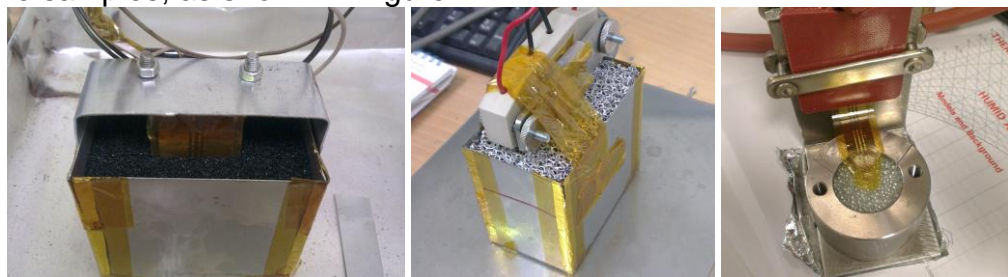


Figure 2 Sample holders

Different sample holders are used for different types of porous media. Graphic examples are given in Fig. 2. If room-temperature measurements with air are conducted, measurements can start as soon as the wires are connected to the sensor.



Figure 3 Vacuum cell

Most measurements however, are conducted at a range of -30 °C<T<150 °C with pressurized gas (He/N2, at 0.1 ...0.4 bar relative). In order to do this, the sample holder, or sometimes the porous media itself, is lowered into the vacuum / pressure cell. This pressure cell is provided by HotDisk AB as well and can withstand pressures of 1 bar relative and 200°C, see attachment B. Wires are then connected to the vacuum / pressure cell itself (from inside and outside), as shown in Figure 3. The lid is then sealed with a silicone gasket and if necessary with silicon vacuum grease and lowered into a thermal bath. The oil bath can stabilize at any given temperature between -35 and +200 degrees Celsius. After connecting the vacuum suction line with pressure sensors and the gas supply (Figure 4 or 5, next page) a

final leakage test is necessary to guaranty safe and reliable measurements. Once thermal bath has stabilized, measurements can start.
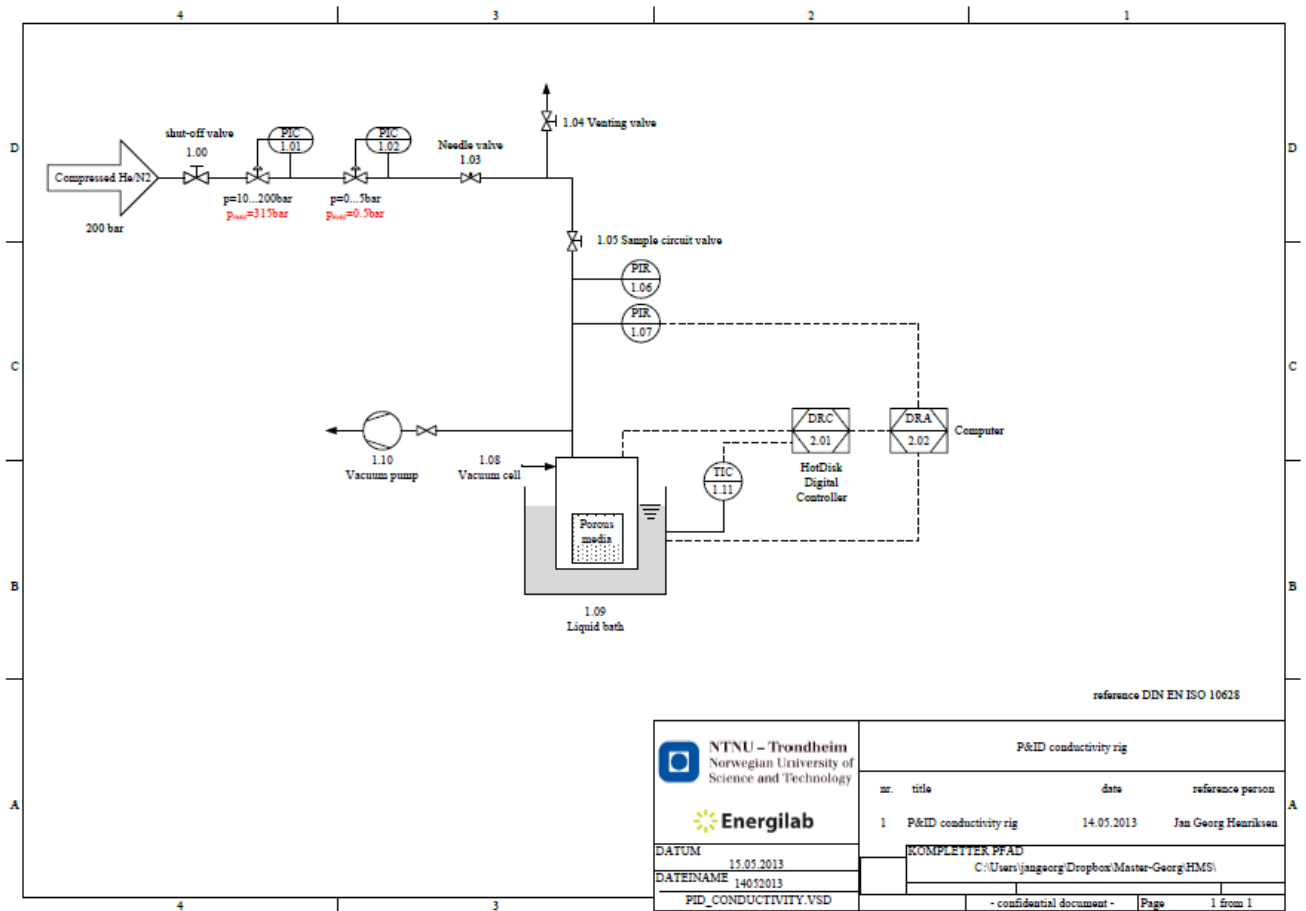


Figure 4 flow schematic conductivity rig (larger version attached at the very end of the report)
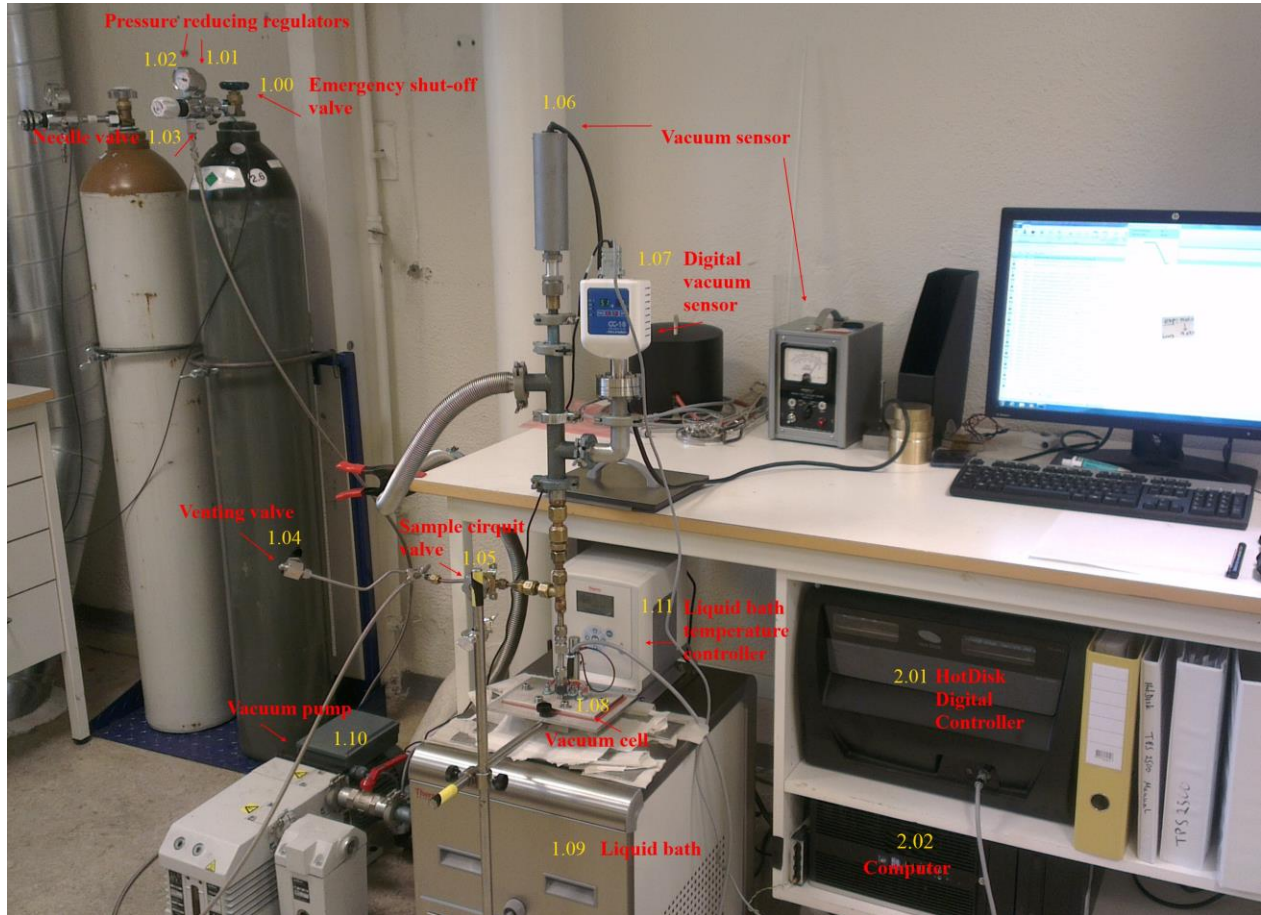
Figure 5- Thermal conductivity setup with gas supply, vacuum sensors and computer analyser. Location: Finlab.

As shown in Figure 4 and Figure 5, compressed gas (N2 or He at approximately 200 bar) from a gas bottle is supplied after opening the shut-off valve (1.00). A maximal allowed pressure is set with the pressure regulators (max 1.4 bar (absolute)) (1.01) and (1.02). Experiments are preformed usually at 1.2...1.4 bar (absolute). Next follows a needle valve (1.03) for extra security, and a venting valve (1.04). The venting valve is there in order to ensure that pressure can be released if it is too high, or for venting before gas change. By turning the pressure regulator (1.02) up, or turning it down and venting through (1.04) one can regulate the pressure. By opening up the sample circuit valve (1.05) the system is supplied with gas.

The porous medium is located inside the pressure cell (1.08), which again is lowered in the liquid bath (1.09). Before one starts any measurements the porous medium must be evacuated. This is because the porous powder contains humid air which can interfere with the measurement. Evacuation is monitored through the vacuum pressure indicators (1.06) and (1.07). In order to evacuate, the sample circuit valve (1.05) is closed, the vacuum pump (1.10) is started and its valve is opened. When vacuum reaches $10^{-2}$ torr (0.13 bar), the valve on the vacuum pump (1.10) is closed and the sample circuit valve (1.05) is opened. Gas is then purged into the system.

To ensure that all humidity is gone, the porous medium must also be activated. In order to heat it, the liquid bath controller (1.11) is then set to 150 degrees Celsius. The evacuation process is then repeated five times over.

The HotDisk Digital Controller TPS2500S (2.01) governs the measurements together with a timetable set up in the software on the computer (2.02).

The filling and emptying procedure is explained short further down. An additional "Procedure for running experiments" summarizes all important steps for carrying out safe measurements and is added in Appendix G.
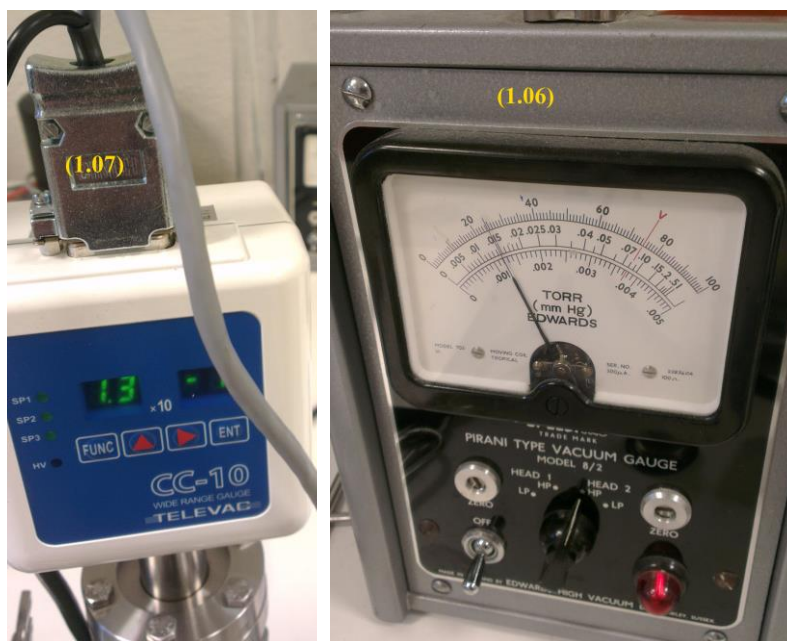


Figure 6 Vacuum pressure indicators

The components installed at the conductivity rig are listed in Table 1 below. The list can be used to identify the main instrument parameters.

Table 1 Sensor actor list conductivity rig

| Sensoric und Actoric Permeability Rig | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| **NTNU – Trondheim** Norwegian University of Science and Technology     :¦:Energilab | | | | | | | | | | |
| Lfd. | P&ID | measurement position | | measure-/actuating variable | | | device specification | | | signal | comments |
| Nr. | Nr. | medium | position | sensor / device | S/A | EMSR | device typ | measure-/ control value | U / I / P | range | |
| | | | | | | | (z.B. producer, name (typ).) | | [V, A,W] | | |
| 1 | 1.00 | compressed N2/He | gas bottle | shut-off valve | A | | Yara Praxair | 200bar | bar | | |
| 2 | 1.01 | compressed N2/He | gas bottle | Pressure reducing regulator | A/S | PIC | Yara Praxair | 200bar | bar | p_max=315 bar | |
| 3 | 1.02 | compressed N2/He | gas bottle | Pressure reducing regulator | A/S | PIC | Yara Praxair | operating @ < 0.5 bar | bar | p_max=5 bar | |
| 4 | 1.03 | compressed N2/He | gas bottle | Needle valve | A | | Yara Praxair | operating @ 0.2/0.4 bar | bar | p_max= 60 bar | |
| 5 | 1.04 | compressed N2/He | between gas bottle/sample circuit | Venting valve | A | | Swagelok SS 43GS 6mm | operating @ 0.2/0.4 bar | bar | 0.5bar | |
| 6 | 1.05 | compressed or evacuated N2/He | between venting valve/sample holder | Sample circuit valve | A | | Danfoss | operating @ 0.2/0.4 bar | bar | 0.5bar | |
| 7 | 1.06 | compressed or evacuated N2/He | above sample holder | Vacuum pressure indicator | S | PIR | SPEEDIVAC | 0.005…1 torr | torr | 0.005…1 torr | 1 torr =0.013 bar |
| 8 | 1.07 | compressed or evacuated N2/He | above sample holder | Vacuum pressure indicator | S | PIR | TELEVAC CC-10 | $10^{-9}...10^{-3}$ torr $10^{-3}...10^{-1}$ torr $10^{-1}...10^{1}$ torr $10^{1}...10^{3}$ torr | torr | $10^{-9}...10^{3}$ torr | can log to pc |
| 9 | 1.08 | compressed or evacuated N2/He | in liquid bath | Vacuum pressure cell (sample inside) | - | | HotDisk Vacuum cell 11502 | operating @ 0.2/0.4 bar | bar | Tmax =200°C p_max=2.5 bar | |
| 10 | 1.09 | Oil | floor | Liquid bath | - | | AC200 | | | | |
| 11 | 1.10 | compressed or evacuated N2/He | floor next to liquid bath | Vacuum pump | A | | Oerlikon AF-16-25 | $p=10^{3}...5·10^{-3}$ | torr | $p=10^{3}...5·10^{-3}$ torr | $<=10^{-4}$ mbar (given from producer) |
| 12 | 1.11 | | on top of liquid bath | liquid bath temperature controller | A/S | TIC | Thermo Scientific AC 200 | T=243…423 | K | T=238…473K | |
| 13 | 2.01 | | in desk next to liquid bath | Hotdisk digital measurement controller | A/S | DRC | HotDisk Th. Const. Analyser TPS 2500S | | | | Bridge: Keithley 2400, Off balance: Keithley 2700 (6.5 digits resolution) |
| 14 | 2.02 | | in desk next to liquid bath | Computer | A/S | DRA | HotDisk Th. Const. Ana. Version 7.0.16 | Thermal conductivity k | W/mK | 0.005-500 W/mK | Repeatability +-0.5% , Thermal conductivity uncertainty=+-5% |

**Filling of new powder**

1. If rig is running, set liquid bath to 22 degrees and close shut-off and needle valve. Open the sample circuit valve and vent the system to ambient pressure.
2. When liquid bath has been at 22 degrees for 10 minutes, shut of the bath.
3. Remove wires from pressure cell.
4. Then remove the pressure cell from the bath.
5. Remove the pressure cell lid and disconnect the inner wires.
6. Control possible leakages and damages at the gasket
7. Wear glasses, hand gloves and dusk mask for protection.
8. Remove silicone gasket and take out the sample/sample holder.
9. Weigh the sample holder and measure sample height.
10. Empty sample holder and remove sensor.
11. Clean vacuum pressure cell, sensor, rubber sealing, and sample holder.
12. Weigh the sample holder without any powder in it. One can now tell how much mass the powder has lost during evacuation.
13. Pour in a new powder sample using a funnel.
14. Weigh new powder and measure filling height.
15. If the powder consists of small light grains, tape aluminum foil over the sample in order to prevent powder loss during evacuation.
16. Put sample holder back into the pressure cell.
17. Lubricate the sample holder and its lid with vacuum grease if necessary.
18. Connect wires and close the lid.
19. Mount the pressure cell into the liquid bath.
20. Cover the gap between pressure cell and bath to prevent oil vaporization.
21. Then start new experiment (described below)

The dismantling, empting and filling of the cylinder, which contains the powder sample is shown more in detail in figure 7.
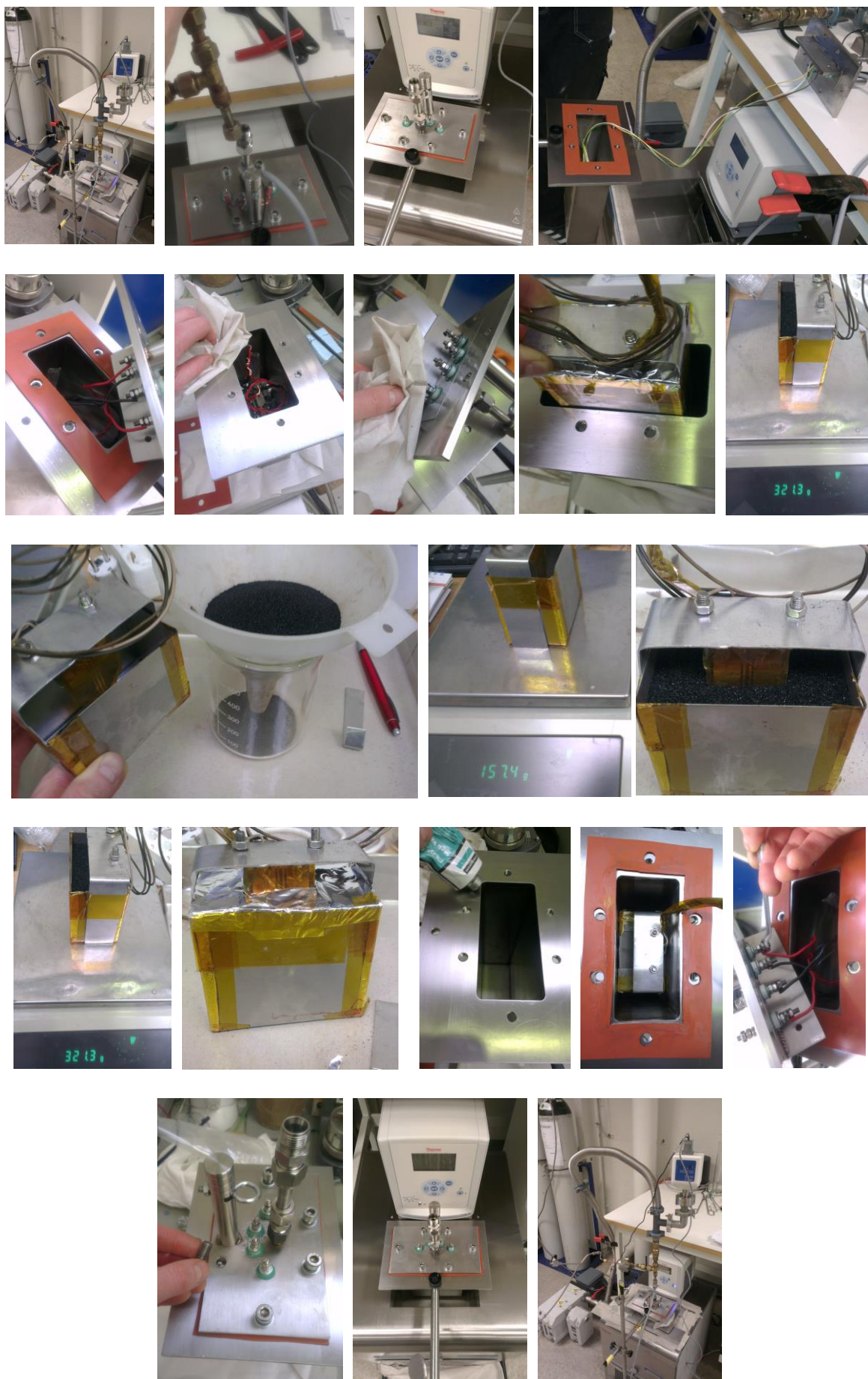
Figure 7 Dismantling, emptying filling and mantling.

**Experimental procedure**

1. Restart liquid bath and computer.
2. Use glasses and gloves.
3. All valves are closed.
4. Set liquid bath (1.09) to 150 degrees and start vacuum pump (1.10).
5. Open the shut-off valve.
6. Adjust pressure with the pressure regulator (1.02). Pressure must be <0.5bar.
7. Open needle valve (1.03).
8. Open venting valve (1.04) and regulate (1.02) to desired pressure (0.4 or 0.2 bar). Then close the venting valve (1.04).
9. Open sample circuit valve (1.05). Gas now flows into the pressure cell (1.08).
10. Wait 10 seconds and close sample circuit valve (1.05)
11. Open valve on the vacuum pump (1.10).
12. When vacuum is below $10^{-2}$torr (0.13 bar), close vacuum pump valve (1.10).
13. Repeat point 9-12 six times.
14. Open sample circuit valve (1.05) and turn off vacuum pump (1.10).
15. Close needle valve (1.03) and wait one hour before starting the experiment.

# 5   EVACUATION FROM THE EXPERIMENT AREA

Evacuate at signal from the alarm system or local gas alarms with its own local alert with sound and light outside the room in question, see 6.2
Evacuation from the rigging area takes place through the marked emergency exits to the assembly point, (corner of Old Chemistry Kjelhuset or parking 1a-b.)
**Action on rig before evacuation**:

1. Shut off the gas (N2 or He) supply (1.00) marked with sign "Emergency shut-off valve"
2. Power off the electrical supply.

# 6   WARNING

## 6.1   Before experiments

E-mail with information about the planned experiment to: iept-experiments@ivt.ntnu.no
The e-mail should contain the following items:
- Name of responsible person:
- Experimental setup/rig:
- Start Experiments: (date and time)
- Stop Experiments: (date and time)

You should get the approval back from the laboratory management before start up. All running experiments are notified in the activity calendar for the lab to be sure they are coordinated with other activity.

## 6.2   Nonconformance

**FIRE**
For fires which you are not able to put out with locally available fire extinguishers, activate the nearest fire alarm and evacuate area. Be then available for fire brigade

and building caretaker to detect fire place.
If possible, notify:

| NTNU | SINTEF |
|---|---|
| Labsjef Morten Grønli, tlf: 918 97 515 | Labsjef Harald Mæhlum tlf 930 149 86 |
| HMS: Erik Langørgen, tlf: 91897160<br>HMS: Bård Brandåstrø, tlf918 97 257 | Forskningssjef Mona Mølnvik 93008868 |
| Instituttleder: Olav Bolland: 91897209 | |
| NTNU Sintef Beredskapstelefon | 800 80 388 |

**GASALARM**

At a gas alarm, close gas bottles immediately and ventilated the area. If the level of gas concentration does not decrease within a reasonable time, activate the fire alarm and evacuate the lab. Designated personnel or fire department checks the leak to determine whether it is possible to seal the leak and ventilate the area in a responsible manner.
Alert Order in the above paragraph.

**PERSONAL INJURY**
- First aid kit in the fire / first aid stations
- Shout for help
- Start life-saving first aid•

**CALL 113** if there is any doubt whether there is a serious injury
**Other Nonconformance (AVVIK)**
**NTNU:**
Reporting nonconformance, Innsida, avviksmelding:
https://innsida.ntnu.no/lenkesamling_vis.php?katid=1398
**SINTEF:**
Synergi

# 7 ASSESSMENT OF TECHNICAL SAFETY

## 7.1 HAZOP

The experiment set up is divided into the following nodes:

| Node 1 | Sections filled with pressurized N2/He. |
|---|---|

**Attachments: skjema: Hazop_mal**
**Conclusion: Safety taken care of**

## 7.2 Flammable, reactive and pressurized substances and gas

Contains the experiments Flammable, reactive and pressurized substances and gas

| NO | YES. Explosion document have to be made and or documented pressure test, (See 7.3) |
|---|---|

**Attachments: n.a.**
**Conclusion: n.a.**

## 7.3 Pressurized equipment

**Contain the set up pressurized equipment?**

| | YES, but working at a total pressure of max 1.5 bar |
|---|---|

*Attachments:* YES. **Attachment B** Instructions TPS Vacuum /Backfilled insert Cell for Circulation Bath 11502 (2013/02/25)

Pressure in rig never exceeds 1,5 bar$_{absolute}$ respective 0,5 bar$_{relative}$, and will therefore cause no risk, since the working medium is only N2 or He @ -30…150 °C.

**Conclusion*: all devices downstream from valve (1.02) are working around ambient pressure***

### 7.4 Effects on the environment (emissions, noise, temperature, vibration, smell)

| NEI | YES |
|---|---|

**Conclusion:** n.a.

### 7.5 Radiation

| NEI | JA, Radiation source need to have an own risk assessment |
|---|---|

**Attachments: n.a.**
**Conclusion: n.a.**

### 7.6 Usage and handling of chemicals.

| NEI | JA, Do a risk assessment of the use |
|---|---|

**Attachments: MSDS**
**Conclusion:** The operator needs to use during **filling and emptying of material**:
Protection glasses
Laboratory hand gloves
Dust mask
The operator needs to use during **experiments**:
Glasses and laboratory hand gloves.

### 7.7 El safety (need to deviate from the current regulations and standards.)

| NEI | JA, El safety have to be evaluated |
|---|---|

**Attachments:** none
**Conclusion:** only regular devices with 220V are used

## 8 ASSESSMENT OF OPERATIONAL SAFETY

For ensuring that established procedures cover all identified risk factors that must be taken care of through procedures and ensure that the operators and technical performance have sufficient expertise.

### 8.1 Prosedure HAZOP

The method is a procedure to identify causes and sources of danger to operational problems.
**Attachments:** HAZOP_MAL_Prosedyre
**Conclusion: procedure is uncomplicated and easy to understand → Safety taken care of**

### 8.2 Operation and emergency shutdown procedure

The operating procedure is a checklist that must be filled out for each experiment.
Emergency procedure should attempt to set the experiment set up in a harmless state by unforeseen events.

**Attachments:** Procedure for running experiments
**Emergency shutdown procedure:**

1. Shut off the gas (He or N2) supply by closing valve (1.00) marked with sign "Emergency shut-off valve".
2. Power off the liquid bath.

### 8.3 Training of operators

A Document showing training plan for operators
  *What are the requirements for the training of operators?*
  • *What it takes to be an independent operator*
  • *Job Description for operators*
  **Attachments***: Training program for operators*

### 8.4 Technical modifications

- Technical modifications made by the Operator
  ○ Yes

- What technical modifications give a need for a new risk assessment; (by changing the risk picture)?
  ○ If more pressure is needed (>1,5 bar), or other types of samples with higher safety requirements than already described is used.

**Conclusion: All modifications can be made by the operator**

### 8.5 Personal protective equipment

- It is mandatory use of eye protection when handling fine powder.
- Use gloves when handling fine powder.
- Use of respiratory protection apparatus when handling fine powder.

**Conclusion: always glasses, dust mask and gloves nearby during powder handling.**

### 8.6 General Safety

- Monitoring: operator must start the experiment. After start: experiment can execute itself without operator presence.

**Conclusion: The operator is allowed to leave during the experiment.**

### 8.7 Safety equipment

- Warning signs, see the Regulations on Safety signs and signaling in the workplace

### 8.8 Special actions.

## 9   QUANTIFYING OF RISK - RISK MATRIX

*See Chapter 13 "Guide to the report template".*
The risk matrix will provide visualization and an overview of activity risks so that management and users get the most complete picture of risk factors.

| IDnr | Aktivitet-hendelse | Frekv-Sans | Kons | RV |
|------|---------------------|------------|------|-----|
| Xx | *Pipes are torn; N2 or He@<0,5 bar stream out* | 1 | B2 | B2 |
| | *Liquid bath not covered; water is spilled into it* | 1 | B1 | B1 |

**Conclusion** : *Participants will make a comprehensive assessment to determine whether the remaining risks of the activity / process is acceptable. Barriers and driving outside working hours e.g.*

## 10   CONCLUSJON

The rig is built in good laboratory practice (GLP).

Experiment unit card get a period of **24 months**
Experiment in progress card get a period of **24 months**

# 11   REGULATIONS AND GUIDELINES

Se http://www.arbeidstilsynet.no/regelverk/index.html
- Lov om tilsyn med elektriske anlegg og elektrisk utstyr (1929)
- Arbeidsmiljøloven
- Forskrift om systematisk helse-, miljø- og sikkerhetsarbeid (HMS Internkontrollforskrift)
- Forskrift om sikkerhet ved arbeid og drift av elektriske anlegg (FSE 2006)
- Forskrift om elektriske forsyningsanlegg (FEF 2006)
- Forskrift om utstyr og sikkerhetssystem til bruk i eksplosjonsfarlig område NEK 420
- Forskrift om håndtering av brannfarlig, reaksjonsfarlig og trykksatt stoff samt utstyr og anlegg som benyttes ved håndteringen
- Forskrift om Håndtering av eksplosjonsfarlig stoff
- Forskrift om bruk av arbeidsutstyr.
- Forskrift om Arbeidsplasser og arbeidslokaler
- Forskrift om Bruk av personlig verneutstyr på arbeidsplassen
- Forskrift om Helse og sikkerhet i eksplosjonsfarlige atmosfærer
- Forskrift om Høytrykksspyling
- Forskrift om Maskiner
- Forskrift om Sikkerhetsskilting og signalgivning på arbeidsplassen
- Forskrift om Stillaser, stiger og arbeid på tak m.m.
- Forskrift om Sveising, termisk skjæring, termisk sprøyting, kullbuemeisling, lodding og sliping (varmt arbeid)
- Forskrift om Tekniske innretninger
- Forskrift om Tungt og ensformig arbeid
- Forskrift om Vern mot eksponering for kjemikalier på arbeidsplassen (Kjemikalieforskriften)
- Forskrift om Vern mot kunstig optisk stråling på arbeidsplassen
- Forskrift om Vern mot mekaniske vibrasjoner
- Forskrift om Vern mot støy på arbeidsplassen


Veiledninger fra arbeidstilsynet
se: http://www.arbeidstilsynet.no/regelverk/veiledninger.html

# 12 DOCUMENTATION

- Tegninger, foto, beskrivelser av forsøksoppsetningen
- Hazop_mal
- Sertifikat for trykkpåkjent utstyr
- Håndtering avfall i NTNU
- Sikker bruk av LASERE, retningslinje
- HAZOP_MAL_Prosedyre
- Forsøksprosedyre
- Opplæringsplan for operatører
- Skjema for sikker jobb analyse, (SJA)
- Apparaturkortet
- Forsøk pågår kort

# 13 GUIDANCE TO RISK ASSESSMENT TEMPLATE

**Kap 7 Assessment of technical safety.**
Ensure that the design of the experiment set up is optimized in terms of technical safety.
Identifying risk factors related to the selected design, and possibly to initiate re-design to ensure that risk is eliminated as much as possible through technical security.
This should describe what the experimental setup actually are able to manage and acceptance for emission.

**7.1 HAZOP**
The experimental set up is divided into nodes (eg motor unit, pump unit, cooling unit.). By using guidewords to identify causes, consequences and safeguards, recommendations and conclusions are made according to if necessary safety is obtained. When actions are performed the HAZOP is completed.
(e.g. "No flow", cause: the pipe is deformed, consequence: pump runs hot, precaution: measurement of flow with a link to the emergency or if the consequence is not critical used manual monitoring and are written into the operational procedure.)

**7.2 Flammable, reactive and pressurized substances and gas.**
*According to the Regulations for handling of flammable, reactive and pressurized substances and equipment and facilities used for this:*

| |
|---|
| **Flammable material**: Solid, liquid or gaseous substance, preparation, and substance with occurrence or combination of these conditions, by its flash point, contact with other substances, pressure, temperature or other chemical properties represent a danger of fire. |

| |
|---|
| **Reactive substances**: Solid, liquid, or gaseous substances, preparations and substances that occur in combinations of these conditions, which on contact with water, by its pressure, temperature or chemical conditions, represents a potentially dangerous reaction, explosion or release of hazardous gas, steam, dust or fog. |

| |
|---|
| **Pressurized** : Other solid, liquid or gaseous substance or mixes havinig fire or hazardous material response, when under pressure, and thus may represent a risk of uncontrolled emissions |

Further criteria for the classification of flammable, reactive and pressurized substances are set out in Annex 1 of the Guide to the Regulations "Flammable, reactive and pressurized substances"

http://www.dsb.no/Global/Publikasjoner/2009/Veiledning/Generell%20veiledning.pdf
http://www.dsb.no/Global/Publikasjoner/2010/Tema/Temaveiledning_bruk_av_farlig_stoff_Del_1.pdf

Experiment setup area should be reviewed with respect to the assessment of Ex zone

• Zone 0: Always explosive atmosphere, such as inside the tank with gas, flammable liquid.
• Zone 1: Primary zone, sometimes explosive atmosphere such as a complete drain point
• Zone 2: secondary discharge could cause an explosive atmosphere by accident, such as flanges, valves and connection points

**7.4 Effects on the environment**

With pollution means: bringing solids, liquid or gas to air, water or ground, noise and vibrations, influence of temperature that may cause damage or inconvenience effect to the environment.

Regulations: http://www.lovdata.no/all/hl-19810313-006.html#6

NTNU guidance to handling of waste:http://www.ntnu.no/hms/retningslinjer/HMSR18B.pdf


**7.5 Radiation**

Definition of radiation

| |
|---|
| **Ionizing radiation:** Electromagnetic radiation (in radiation issues with wawelength <100 nm) or rapid atomic particles (e.g. alpha and beta particles) with the ability to stream ionized atoms or molecules. |
| **Non ionizing radiation**: Electromagnetic radiation (wavelength >100 nm), og ultrasound$_1$ with small or no capability to ionize. |
| **Radiation sources**: All ionizing and powerful non-ionizing radiation sources. |
| **Ionizing radiation sources**: Sources giving ionizing radiation e.g. all types of radiation sources, x-ray, and electron microscopes. |
| **Powerful non ionizing radiation sources**: Sources giving powerful non ionizing radiation which can harm health and/or environment, e.g. class 3B and 4. MR$_2$ systems, UVC$_3$ sources, powerful IR sources$_4$. |
| $_1$Ultrasound is an acoustic radiation ("sound") over the audible frequency range (> 20 kHz). In radiation protection regulations are referred to ultrasound with electromagnetic non-ionizing radiation. |
| $_2$MR (e.g. NMR) - nuclear magnetic resonance method that is used to "depict" inner structures of different materials. |
| $_3$UVC is electromagnetic radiation in the wavelength range 100-280 nm. |
| $_4$IR is electromagnetic radiation in the wavelength range 700 nm - 1 mm. |

For each laser there should be an information binder (HMSRV3404B) which shall include:
• General information
• Name of the instrument manager, deputy, and local radiation protection coordinator
• Key data on the apparatus
• Instrument-specific documentation
• References to (or copies of) data sheets, radiation protection regulations, etc.
• Assessments of risk factors
• Instructions for users

• Instructions for practical use, startup, operation, shutdown, safety precautions, logging, locking, or use of radiation sensor, etc.
• Emergency procedures

See NTNU for laser: http://www.ntnu.no/hms/retningslinjer/HMSR34B.pdf

**7.6 Usage and handling of chemicals**.

In the meaning chemicals, a element that can pose a danger to employee safety and health

See: http://www.lovdata.no/cgi-wift/ldles?doc=/sf/sf/sf-20010430-0443.html

Safety datasheet is to be kept in the HSE binder for the experiment set up and registered in the database for chemicals.

## Kap 8 Assessment of operational procedures.

Ensures that established procedures meet all identified risk factors that must be taken care of through operational barriers and that the operators and technical performance have sufficient expertise.

**8.1 Prosedure Hazop**

Procedural HAZOP is a systematic review of the current procedure, using the fixed HAZOP methodology and defined guidewords. The procedure is broken into individual operations (nodes) and analyzed using guidewords to identify possible nonconformity, confusion or sources of inadequate performance and failure.

**8.2 Procedure for running experiments and emergency shutdown.**

Have to be prepared for all experiment setups.

*The operating procedure has to describe stepwise preparation, startup, during and ending conditions of an experiment. The procedure should describe the assumptions and conditions for starting, operating parameters with the deviation allowed before aborting the experiment and the condition of the rig to be abandoned.*

*Emergency procedure describes how an emergency shutdown have to be done, (conducted by the uninitiated),*

*what happens when emergency shutdown, is activated. (electricity / gas supply) and*

*which events will activate the emergency shutdown (fire, leakage).*

## Kap 9 Quantifying of RISK

Quantifying of the residue hazards, Risk matrix

To illustrate the overall risk, compared to the risk assessment, each activity is plo tted with values for the probability and consequence into the matrix. Use task IDnr.

Example: If activity IDnr. 1 has been given a probability 3 and D for consequence the risk value become D3, red. This is done for all activities giving them risk values.

In the matrix are different degrees of risk highlighted in red, yellow or green. When an activity ends up on a red risk (= unacceptable risk), risk reducing action has to be taken

| CONSEQUENSES | Svært alvorlig | E1 | E2 | E3 | E4 | E5 |
|---|---|---|---|---|---|---|
| | Alvorlig | D1 | D2 | D3 | D4 | D5 |
| | Moderat | C1 | C2 | C3 | C4 | C5 |
| | Liten | B1 | B2 | B3 | B4 | B5 |
| | Svært liten | A1 | A2 | A3 | A4 | A5 |
| | | Svært liten | Liten | Middels | Stor | Svært Stor |
| | | PROBABILITY | | | | |

The principle of the acceptance criterion. Explanation of the colors used in the matrix

| Farge | | Beskrivelse |
|---|---|---|
| Rød | | Unacceptable risk Action has to be taken to reduce risk |
| Gul | | Assessment area. Actions has to be concidered |
| Grønn | | Acceptable risk. Action can be taken based on other criteria |

# Attachment to Risk Assessment report

## [Conductivity rig: thermal conductivity measurements of porous media in a packed bed]

| Project name | Conductivity rig: conductivity measurements in porous media |
|---|---|
| Project leader | Erling Næss/Christian Schlemminger |
| Organization | NTNU |
| HSE-koordinator | Erik Langørgen |
| Head of Department | Olav Bolland |
| Rig name | Conductivity rig |
| Plassering | VATlab |
| Room number | FinLab |
| Rig responsible | Jan Georg Henriksen/Christian Schlemminger |

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| **Node:  1** Sections filled with pressurized N2 or He, vacuum, and liquid bath containing hot/cold oil. |            |            |            |            |            |            |            |

| Ref | Guideword | Causes | Consequences | Safeguards | Recommendations | Action | Date/ Sign |
|-----|-----------|--------|--------------|------------|-----------------|--------|------------|
| 1 | No flow | valves closed /Pipes blocked etc. Gas bottle is empty. | Can't conduct experiment/wrong measurement values No safety hazards | Always conduct experiment according to procedure | | Check all valves. If not ok; dismantle & check for blockage | |
| 2 | Reverse flow | n.a. | | | | | |
| 3 | More flow | High pressure <5bar | Torn pipes (most likely not) | Always check pressure before starting experiment. Keep sample circuit valve closed. Close Needle Valve (1.03) | Check if instruments states zero before exp. Open venting valve. | Close (if not already) sample circuit valve). Adjust pressure regulator and vent out pressure | |
| 4 | Less flow | Blockage in pipes or leakage. | Pressure chamber not flushed→Air and moist may still be inside. No safety hazards. | Clean pipes regularly. Make sure everything is tight and sealed. | Set pressure to the system and spray sealing's with soap water | Check for leaks. If not ok-> Dismantle and check for blockage. | |
| 5 | More level | Amount of filled powder is too much | Powder floats out of sample holder→ could be evacuated/ plug the pipes. | Make sure to fill the proper height. Have filter inside piping system. | | Dismantle setup and refill powder | |
| 6 | Less level | Amount of filled powder is too less | Wrong measurements | Make sure to measure probing depth. | Put in the correct probing depth in | Dismantle setup and refill powder | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Project:**<br>**Node: 1** Sections filled with pressurized N2 or He, vacuum, and liquid bath containing hot/cold oil. | | | | | | **Page** | |
| | | | | | | | |
| **Ref** | **Guideword** | **Causes** | **Consequences** | **Safeguards** | **Recommendations** | **Action** | **Date/ Sign** |
| | | | | | Analyser software. | | |
| 7 | No vacuum when evacuating. | Vacuum pump valve not open/Sample circuit valve not closed/leakage. | Wrong measurement values (moist still inside powder)<br>No safety hazards | Conducti experiment according to procedure. Always close the sample circuit valve during evacuation. | Always check pressure before and after valves are turned on | Check valves. Check for leaks with soap water. If not ok→dismantle and clean setup and start over. | |
| 8 | Some vacuum (but not enough | leakage | Wrong measurement values (moist still inside powder)<br>No safety hazards | Screw the pressure cell lid on firm (but not too firm). Use vacuum grease. | Always check pressure before and after valves are turned on | Stop the leakage. Clean setup and screw sealing's tighter together. | |
| 9 | More temperature | Oil in liquid bath increases in volume→high-level warning. | Measurement stops. Slippery floor. Evaporated oil. | Make sure no warning comes during evacuation. If also no oil is evaporated it is a good sign. Cover bath. | Test @ 150 degrees: if high level warning not sho within an hour it is ok. | Raise the pressure cell. | |
| 10 | Less temperature | Oil in liquid bath decreases in volume→/low-level warning. | Measurement stops | Have enough oil in the bath/lower the pressure cell far enough. | Test @ 150 degrees: if high level warning not sho within an hour it is ok. | Lower pressure cell. | |
| 11 | More viscosity | n.a. | | | | | |
| 12 | Less viscosity | n.a. | | | | | |
| 13 | Composition | Spilling water into | Boiling water, oil | Cover bath. Keep bath at | | Put lid on. | |

| Project:  Node: 1 Sections filled with pressurized N2 or He, vacuum, and liquid bath containing hot/cold oil. | | | | | | Page | |
|---|---|---|---|---|---|---|---|
| Ref | Guideword | Causes | Consequences | Safeguards | Recommendations | Action | Date/ Sign |
|  | Change | the oil. | bursts out. | room temperature between experiments. |  |  |  |
| 14 | Contamination | n.a. |  |  |  |  |  |
| 15 | Relief | n.a. |  |  |  |  |  |
| 16 | Instrumentation | Damage or failure zeroing | See 1;3;4;5;6;7;8;9;10 | See 1;3;4;5;6;7;8;9;10 | See 1;3;4;5;6;7;8;9;10 | See 1;3;4;5;6;7;8;9;10 |  |
| 17 | Sampling | See 5 and 6 | See 5 and 6 | See 5 and 6 | See 5 and 6 | See 5 and 6 |  |
| 18 | Corrosion/erosion | n.a. |  |  |  |  |  |
| 19 | Service failure | n.a. |  |  |  |  |  |
| 20 | Abnormal Operation | See 1;3;4;5;6;7;8 | Can't use values | Always conduct according to same procedure – no shortcuts | Take your time | Turn off and recalibrate instruments. Go through checklist |  |
| 21 | Maintenance | Rig not run properly | maintenance | Always follow same procedure |  | Change whatever needs to be changed |  |
| 22 | Ignition | Non-flammable oil/gas. | - | - | - | - |  |
| 23 | Spare equipment | Not used |  |  |  | Store in C165 |  |
| 24 | Safety |  |  |  |  |  |  |

- **ATTACHMENT B – PRESSURE TESTING CERTIFICATE**

**Pressure testing sertificate: mail from vacuum cell supplyer.**

**From:** Daniel Cederkrantz [mailto:daniel.cederkrantz@hotdisk.se]
**Sent:** 15. februar 2013 11:04
**To:** Erling Næss
**Cc:** Henrik Otterberg
**Subject:** Re: NTNUs HotDisk system

Hi Erling,

Thank you for this information. Regarding point 1: The bottom will most likely be welded on the new design. Regarding point 2: The plan is to have two glide connection with a the space in-between them filled with vacuum grease to keep them lubricated and vacuum tight. I will investigate the possibility to use a electrical heater, but this would increase the complexity of the device. I'll see what the workshop thinks about this.

The requested dome for reducing the icing of the cryo probe is now integrated in the design, with a flange diameter in the range of 120-150 mm, matching the ISO flange. The N2 and vacuum connections will be extended and standard 3/8" tubs will be used for easy Swagelok integration.
I have also confirmed the inner diameter of the powder sample holder to 60 mm and the inner tube for cables and suspension of the copper cell will likely be made of stainless steel and have an outer diameter or 20 mm and a wall thickness of 1,5 mm.

I've also been in further contact with our Canadian supplier of the vacuum cell to use with the oil bath, and I got the attached photos showing their testing of the device. As you can see on the pictures the type of sealing has been changed to silicone gasket, which they claim to be superior to the old gasket.
Regarding maximum pressure they say the following: "It is designed to our local max pressure, which does not require certification of "15 PSI" (1 bar).  You may recommend to confirm what the non-certified max pressure is for the clients country."
You mention 5 bar, but I have no recollection or mentioning this, but I certainly remember promising to determine the maximum pressure, which according to the above should be 1 bar above ambient without any special certifications.

Sincerely,
Daniel
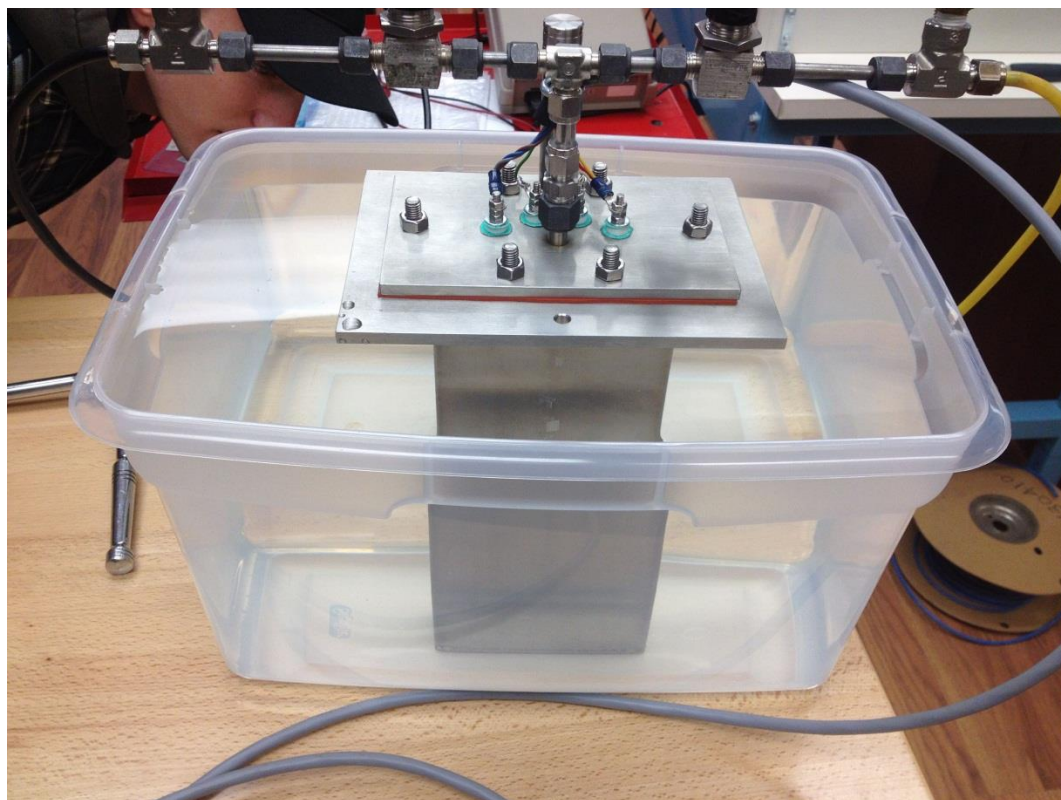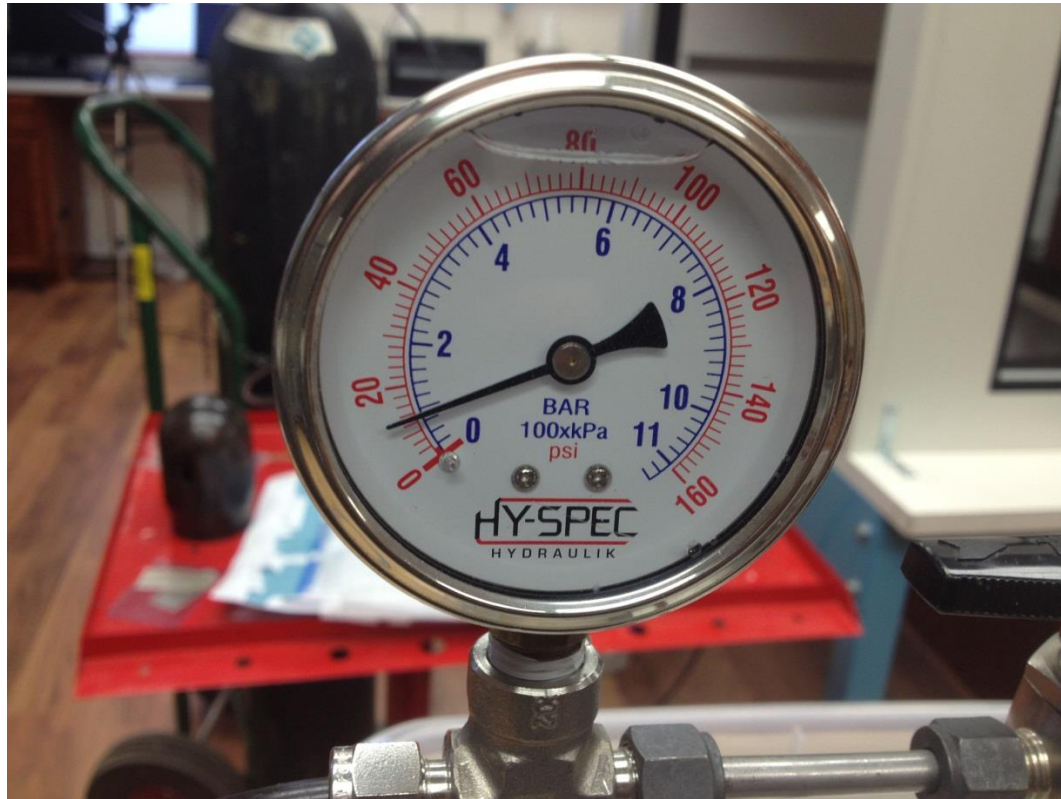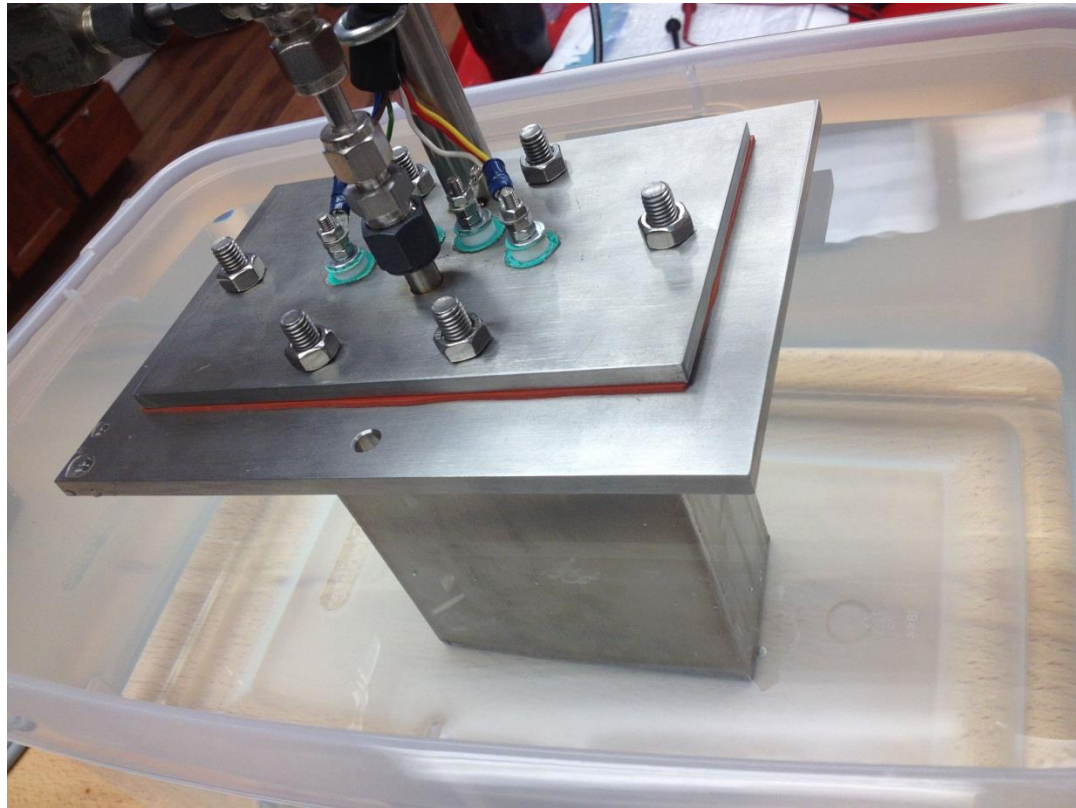
# TPS Vacuum / Backfilled Insert Cell for Circulating Bath 11502

## Maximum temperature: 200°C

Written by: Dale Hume
Last Reviewed: 2013/02/25

# TPS Vacuum / Backfilled Insert Cell for Circulating Bath
*Instructions for Setup and Use*

## Summary

TPS Vacuum / Backfilled Insert Cell for Circulating Bath for measuring thermal properties up to 200°C.

### Required Materials

- Insert Sample Holder, with integrated PEEK High Temperature Adaptor
- Mica Board Insulation
- Clamping Bar for the TPS sensor
- Clamping Bar Knurled Knobs
- TPS Sensor (*not included*)
- Top / Bottom Sample Plates
- Top / Bottom Sample Plates Knurled Knobs
- Circulating Insert
- Valves and Gauges (*not included*)



**Figure 1a.** Required Materials
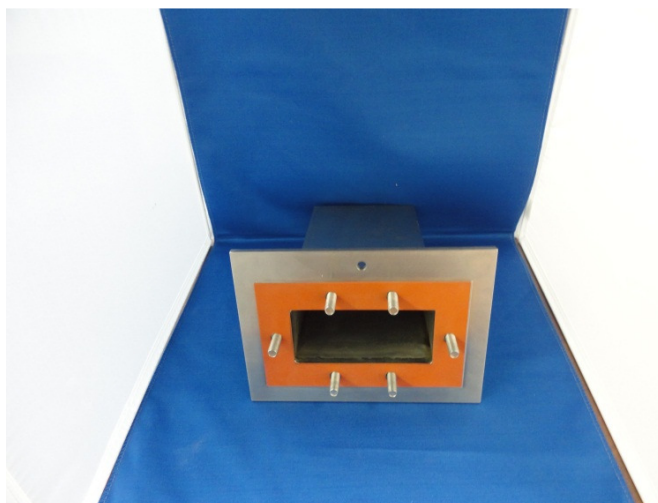


**Figure 1b.** Required Materials

## Insert Procedure

1. The TPS sensor / sample insert comes pre-assembled with the PEEK High Temperature Adaptor (*see Figure 2a*). The electrically insulating Mica Board Insulation is placed on top of the PEEK Adaptor first and then the Clamping Bar is placed on top of the Mica and the PEEK Adaptor (*see Figure 2b*).

**Figure 2a.** TPS PEEK High Temperature Adaptor positioned on sample / sensor insert
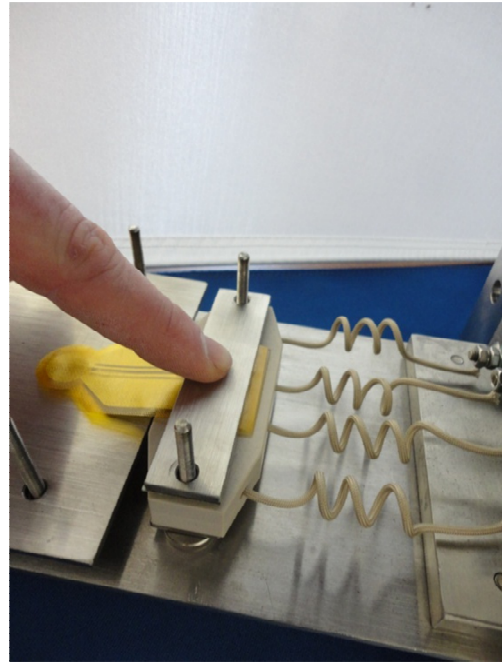


**Figure 2b.** Positioning of TPS Sensor, Mica Board Insulation and Clamping Bar

2.  To position the TPS sensor in the final stage of assembly of the High Temperature TPS Sensor Holder, align the four metal contact pads of the TPS sensor with the metal contact pads (*screws*) of the TPS PEEK High Temperature Adaptor.

To assist with TPS sensor alignment, a positioning guide has been conveniently machined into the Adaptor surface. The TPS sensor should be positioned within the guide boundaries with the sensor contact pads facing down toward the contact pads of the PEEK adaptor.

To ensure best contact, position the TPS sensor and using your finger press firmly on the middle of the Clamping Bar and tighten each knurled knob included with the Clamping Bar.



**Figure 3.** Positioning the TPS Sensor

3.  For optimal horizontal alignment of the sensor holder and the upper and lower sample plates, the knurled knobs can be adjusted for the best horizontal testing position of TPS Sensor, based on available sample thickness.
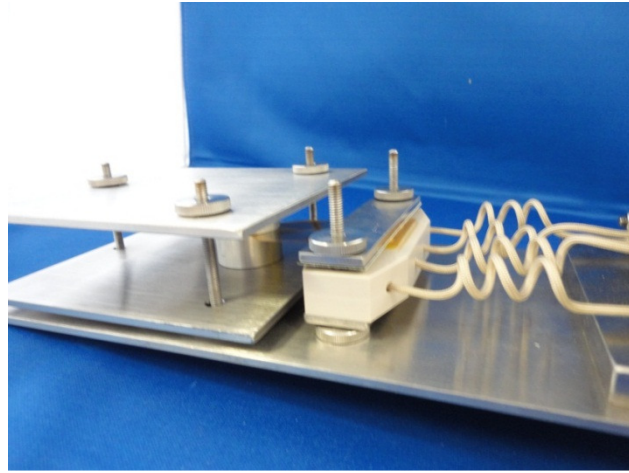


4.  Once the sample is set, the testing insert can be positioned inside the circulator insert; the silicone gasket must be in place between the sample insert and circulator insert for sealing. The assembly is then evenly tightened with bolts.
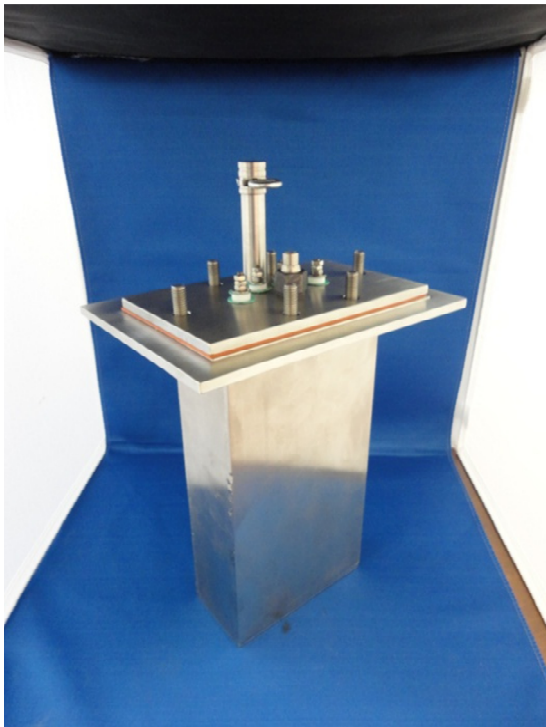


5.  Once the assembly is sealed, vacuum and pressure (*maximum pressure of 15 PSI*) can be applied as needed with the use of the included tube nut (*Parker CPI 6BZ, Tube O.D. 3/8*).

6. Your insert will arrive with TPS controller wires already attached; for reference the controller wires are attached as follows (*referencing the figure below*):

- Post (*Far Left*): **Brown/Grey/Blue**
- Post (*Inside Left*): **Green**
- Post (*Inside Right*): **Pink**
- Post (*Far Right*): **White/Red/Yellow**
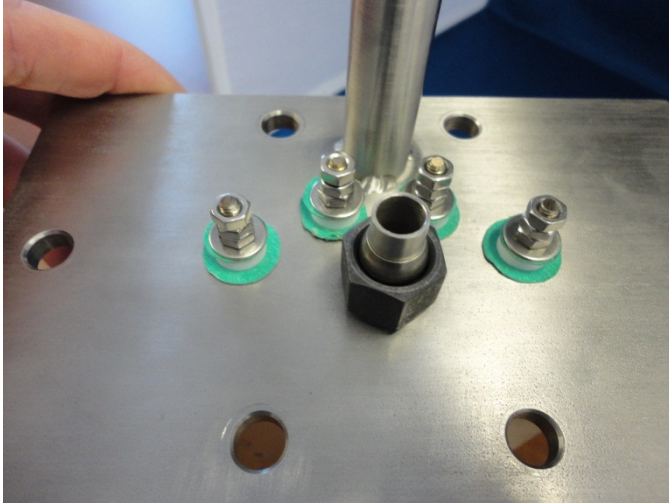


## *Suggestion*

***Prior to making measurements on an unknown sample, first measure the Stainless Steel training materials first included with your TPS System to verify that everything is properly connected.***

## Circulator Procedure

1. The assembled circulator insert is now ready to be attached to the assembly stand.

2.  By adjustment of the assembly stand, the insert can now be positioned and lowered into the fluid bath of the circulator. Ensure that there is space left between the insert and the bottom of the fluid bath to isolate any vibrations that could affect thermal property measurements.



## *<u>Notes</u>

-   ***It is recommended that a small hole be drilled into the HD Liquid Sample Holder block to allow for the TPS HD Thermocouple Sensor to be used***. **If this is undertaken, care should be taken to ensure that that the hole is drilled into the block away from the sample fill area of the Holder.**

- **ATTACHMENT C - GLASS BEADS SPECIFICATIONS.**

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Cleaning, Care, Aids  /  Aids  /  Glass beads





**Glass beads**

Reflex beads made of soda-lime glass. Specific weight approx. 2.5 g/cm$^3$.
**Technical Specifications:**
Apparent density depending on size 1.46-1.51 kg/l. Refraction index 1.5188. Chemical composition: $SiO_2$ 72.5 %; $Na_2O$ 13.0 %; $K_2O$ 0.2 %; CaO 9.6 %; MgO 4.22 %; $Al_2O_3$ 0.58 % $Fe_2O_3$ 0.11 %.

| Article | Ø (mm) | Pack Qty. | Price |
|---|---|---|---|
| A553.1 | 0,25-0,5 | 1 kg | 19.80 € |
| A554.1 | 0,75-1,0 | 1 kg | 19.80 € |
| A555.1 | 1,25-1,65 | 1 kg | 19.80 € |
| A556.1 | 1,7-2,1 | 1 kg | 19.80 € |
| A557.1 | 2,85-3,45 | 1 kg | 19.80 € |

Accessories

Glass beads ▶

● PRODUCT AVAILABILITY IN STOCK   ■ ARTICLE SOON AVAILABLE   ▲ ARTICLE NOT AVAILABLE

Additional information
• Tables

Add this page to service-bookmarks

- **ATTACHMENT D – MOF SPECIFICATIONS.**

# SIGMA-ALDRICH

## SAFETY DATA SHEET

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## 1. IDENTIFICATION OF THE SUBSTANCE/MIXTURE AND OF THE COMPANY/UNDERTAKING

### 1.1 Product identifiers

Product name : **Basolite® F300**

| | | |
|---|---|---|
| Product Number | : | 690872 |
| Brand | : | Aldrich |
| CAS-No. | : | 1195763-37-1 |

### 1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Manufacture of substances

### 1.3 Details of the supplier of the safety data sheet

| | | |
|---|---|---|
| Company | : | Sigma-Aldrich Norway AS |
| | | Filipstad Brygge 1 |
| | | N-0252 OSLO |

| | | |
|---|---|---|
| Telephone | : | +47 23 176000 |
| Fax | : | +47 23 176010 |
| E-mail address | : | eurtechserv@sial.com |

### 1.4 Emergency telephone number

Emergency Phone # : Giftinformasjonssentralen 22 59 13 00

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## 2. HAZARDS IDENTIFICATION

### 2.1 Classification of the substance or mixture

**Classification according to Regulation (EC) No 1272/2008 [EU-GHS/CLP]**
Acute toxicity, Oral (Category 4)
Skin irritation (Category 2)
Eye irritation (Category 2)
Specific target organ toxicity - single exposure (Category 2)

**Classification according to EU Directives 67/548/EEC or 1999/45/EC**
Harmful by inhalation, in contact with skin and if swallowed. Harmful: possible risk of irreversible effects through inhalation, in contact with skin and if swallowed. Irritating to eyes and skin.

### 2.2 Label elements

**Labelling according Regulation (EC) No 1272/2008 [CLP]**
Pictogram

Signal word          Warning

Hazard statement(s)

| | |
|---|---|
| H302 | Harmful if swallowed. |
| H315 | Causes skin irritation. |
| H319 | Causes serious eye irritation. |
| H371 | May cause damage to organs. |

Precautionary statement(s)

| | |
|---|---|
| P260 | Do not breathe dust/ fume/ gas/ mist/ vapours/ spray. |
| P305 + P351 + P338 | IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. |

---

Supplemental Hazard          none
Statements

**According to European Directive 67/548/EEC as amended.**
Hazard symbol(s)



R-phrase(s)
R20/21/22                     Harmful by inhalation, in contact with skin and if swallowed.
R36/38                        Irritating to eyes and skin.
R68/20/21/22                  Harmful: possible risk of irreversible effects through inhalation, in contact
                              with skin and if swallowed.

S-phrase(s)
S26                           In case of contact with eyes, rinse immediately with plenty of water and
                              seek medical advice.
S36/37                        Wear suitable protective clothing and gloves.

Caution - this mixture contains a substance not yet fully tested.

### 2.3    **Other hazards** - none

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## 3.     **COMPOSITION/INFORMATION ON INGREDIENTS**

### 3.2    **Mixtures**
Synonyms                      :   Iron 1,3,5-benzenetricarboxylate
                                  Fe-BTC

Formula                       :   $C_9H_3FeO_6$
Molecular Weight              :   262,96 g/mol

| Component | Classification | Concentration |
|---|---|---|
| **Iron-1,3,5-benzenetricarboxylate** | | |
| | Acute Tox. 4; Skin Irrit. 2; Eye Irrit. 2; H302, H315, H319 Xn, R22 - R36/38 | - |
| **Methanol** | | |
| CAS-No.             67-56-1 <br> EC-No.             200-659-6 <br> Index-No.           603-001-00-X <br> Registration number  01-2119433307-44-XXXX | Flam. Liq. 2; Acute Tox. 3; STOT SE 1; H225, H301 + H311 + H331, H370 <br> F, T, R11 - R23/24/25 - R39/23/24/25 | 3 - 10 % |
| **Diiron trioxide** | | |
| CAS-No.             1309-37-1 <br> EC-No.             215-168-2 | Skin Irrit. 2; Eye Irrit. 2; STOT SE 3; H315, H319, H335 Xi, R36/37/38 | < 10 % |

For the full text of the H-Statements and R-Phrases mentioned in this Section, see Section 16

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## 4.     **FIRST AID MEASURES**

### 4.1    **Description of first aid measures**

**General advice**
Consult a physician. Show this safety data sheet to the doctor in attendance.

**If inhaled**
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

**In case of skin contact**
Wash off with soap and plenty of water. Take victim immediately to hospital. Consult a physician.

**In case of eye contact**
Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

**If swallowed**
Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

**4.2 Most important symptoms and effects, both acute and delayed**
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

**4.3 Indication of any immediate medical attention and special treatment needed**
no data available

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**5. FIREFIGHTING MEASURES**

**5.1 Extinguishing media**

**Suitable extinguishing media**
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

**5.2 Special hazards arising from the substance or mixture**
Carbon oxides, Iron oxides

**5.3 Advice for firefighters**
Wear self contained breathing apparatus for fire fighting if necessary.

**5.4 Further information**
no data available

---

**6. ACCIDENTAL RELEASE MEASURES**

**6.1 Personal precautions, protective equipment and emergency procedures**
Use personal protective equipment. Avoid dust formation. Avoid breathing vapors, mist or gas. Ensure adequate ventilation. Evacuate personnel to safe areas. Avoid breathing dust.

**6.2 Environmental precautions**
Prevent further leakage or spillage if safe to do so. Do not let product enter drains.

**6.3 Methods and materials for containment and cleaning up**
Pick up and arrange disposal without creating dust. Sweep up and shovel. Keep in suitable, closed containers for disposal.

**6.4 Reference to other sections**
For disposal see section 13.

---

**7. HANDLING AND STORAGE**

**7.1 Precautions for safe handling**
Avoid contact with skin and eyes. Avoid formation of dust and aerosols.
Provide appropriate exhaust ventilation at places where dust is formed.

**7.2 Conditions for safe storage, including any incompatibilities**
Store in cool place. Keep container tightly closed in a dry and well-ventilated place.

**7.3 Specific end uses**
no data available

---

**8. EXPOSURE CONTROLS/PERSONAL PROTECTION**

**8.1 Control parameters**

**Components with workplace control parameters**

**8.2 Exposure controls**

**Appropriate engineering controls**
Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

### Personal protective equipment

#### Eye/face protection
Face shield and safety glasses Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

#### Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

The selected protective gloves have to satisfy the specifications of EU Directive 89/686/EEC and the standard EN 374 derived from it.

#### Body Protection
Complete suit protecting against chemicals, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

#### Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face particle respirator type N99 (US) or type P2 (EN 143) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

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## 9.    PHYSICAL AND CHEMICAL PROPERTIES

### 9.1    Information on basic physical and chemical properties

| | | |
|---|---|---|
| a) | Appearance | Form: solid<br>Colour: brown |
| b) | Odour | odourless |
| c) | Odour Threshold | no data available |
| d) | pH | no data available |
| e) | Melting point/freezing point | no data available |
| f) | Initial boiling point and boiling range | no data available |
| g) | Flash point | no data available |
| h) | Evaporation rate | no data available |
| i) | Flammability (solid, gas) | no data available |
| j) | Upper/lower flammability or explosive limits | no data available |
| k) | Vapour pressure | no data available |
| l) | Vapour density | no data available |
| m) | Relative density | no data available |
| n) | Water solubility | no data available |
| o) | Partition coefficient: n-octanol/water | no data available |
| p) | Autoignition temperature | no data available |
| q) | Decomposition temperature | no data available |
| r) | Viscosity | no data available |

| s) | Explosive properties | no data available |
| --- | --- | --- |
| t) | Oxidizing properties | no data available |

### 9.2 Other safety information

Bulk density 160 - 350 kg/m3

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## 10. STABILITY AND REACTIVITY

### 10.1 Reactivity
no data available

### 10.2 Chemical stability
no data available

### 10.3 Possibility of hazardous reactions
no data available

### 10.4 Conditions to avoid
no data available

### 10.5 Incompatible materials
Strong oxidizing agents

### 10.6 Hazardous decomposition products
Other decomposition products - no data available

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## 11. TOXICOLOGICAL INFORMATION

### 11.1 Information on toxicological effects

**Acute toxicity**
no data available

**Skin corrosion/irritation**
no data available

**Serious eye damage/eye irritation**
no data available

**Respiratory or skin sensitization**
no data available

**Germ cell mutagenicity**
no data available

**Carcinogenicity**

IARC: 3 - Group 3: Not classifiable as to its carcinogenicity to humans (Diiron trioxide)

**Reproductive toxicity**
no data available

**Specific target organ toxicity - single exposure**
no data available

**Specific target organ toxicity - repeated exposure**
no data available

**Aspiration hazard**
no data available

**Potential health effects**

| | |
| --- | --- |
| **Inhalation** | Toxic if inhaled. Causes respiratory tract irritation. |
| **Ingestion** | Toxic if swallowed. |
| **Skin** | Toxic if absorbed through skin. Causes skin irritation. |
| **Eyes** | Causes serious eye irritation. |

**Signs and Symptoms of Exposure**
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

**Additional Information**
RTECS: Not available

## 12. ECOLOGICAL INFORMATION

**12.1 Toxicity**
no data available

**12.2 Persistence and degradability**
no data available

**12.3 Bioaccumulative potential**
no data available

**12.4 Mobility in soil**
no data available

**12.5 Results of PBT and vPvB assessment**
no data available

**12.6 Other adverse effects**
no data available

## 13. DISPOSAL CONSIDERATIONS

**13.1 Waste treatment methods**

**Product**
Offer surplus and non-recyclable solutions to a licensed disposal company. Dissolve or mix the material with a combustible solvent and burn in a chemical incinerator equipped with an afterburner and scrubber.

**Contaminated packaging**
Dispose of as unused product.

## 14. TRANSPORT INFORMATION

**14.1 UN number**
ADR/RID: -                    IMDG: -                    IATA: -

**14.2 UN proper shipping name**
ADR/RID:    Not dangerous goods
IMDG:        Not dangerous goods
IATA:        Not dangerous goods

**14.3 Transport hazard class(es)**
ADR/RID: -                    IMDG: -                    IATA: -

**14.4 Packaging group**
ADR/RID: -                    IMDG: -                    IATA: -

**14.5 Environmental hazards**
ADR/RID: no                  IMDG Marine pollutant: no        IATA: no

**14.6 Special precautions for user**
no data available

## 15. REGULATORY INFORMATION

This safety datasheet complies with the requirements of Regulation (EC) No. 1907/2006.

**15.1 Safety, health and environmental regulations/legislation specific for the substance or mixture**
no data available

**15.2 Chemical Safety Assessment**
no data available

## 16. OTHER INFORMATION

**Text of H-code(s) and R-phrase(s) mentioned in Section 3**

| | |
|---|---|
| Acute Tox. | Acute toxicity |
| Eye Irrit. | Eye irritation |
| Flam. Liq. | Flammable liquids |
| H225 | Highly flammable liquid and vapour. |
| H301 + H311 + H331 | Toxic if swallowed, in contact with skin or if inhaled |
| H302 | Harmful if swallowed. |
| H315 | Causes skin irritation. |
| H319 | Causes serious eye irritation. |
| H335 | May cause respiratory irritation. |
| H370 | Causes damage to organs. |
| Skin Irrit. | Skin irritation |
| STOT SE | Specific target organ toxicity - single exposure |
| F | Highly flammable |
| R11 | Highly flammable. |
| R22 | Harmful if swallowed. |
| R23/24/25 | Toxic by inhalation, in contact with skin and if swallowed. |
| R36/37/38 | Irritating to eyes, respiratory system and skin. |
| T | Toxic |
| Xi | Irritant |
| R36/38 | Irritating to eyes and skin. |
| R39/23/24/25 | Toxic: danger of very serious irreversible effects through inhalation, in contact with skin and if swallowed. |
| Xn | Harmful |

**Further information**

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

- **ATTACHMENT F HAZOP MAL PROSEDURE**

| Project: Node: 1 | | | | | | Page | |
|---|---|---|---|---|---|---|---|
| | | | | | | | |
| Ref# | Guideword | Causes | Consequences | Safeguards | Recommendations | Action | Date/Sign |
| | Not clear procedure | Procedure is to ambitious, or confusingly | Fail in measurement point | Simple procedure **Note: already applied** | | | |
| | Step in the wrong place | The procedure can lead to actions done in the wrong pattern or sequence | Pressurized N2 or He <5 bar can leak out. | no parts can be connected in wrong direction and fail positions **Note: already applied** | | | |
| | Wrong actions | Procedure improperly specified | Fail in measurement point; Pressurized N2 or He <5 bar can leak out. | Marks at setup sign clearly the actions, linked to the procedure steps, **Note: already applied** | | | |
| | Incorrect information | Information provided in advance of the specified action is wrong | Fail in measurement point; Pressurized N2 or He <5 bar can leak out. | Procedure is written clearly and prevents misunderstanding. Contact to project leaders is accessible. **Note: already** | | | |

| Project:<br>Node: 1 | | | | | | Page | |
|---|---|---|---|---|---|---|---|
| | | | | | | | |
| Ref# | Guideword | Causes | Consequences | Safeguards | Recommendations | Action | Date/Sign |
| | | | | **applied** | | | |
| | Step missing | Missing step, or step requires too much of operator | Fail in measurement point; Pressurized N2 or He <5 bar can leak out. | Proven procedure is applied and Operator training in necessary **Note: already applied** | | | |
| | Step unsucessful | Step has a high probability of failure | Fail in measurement point; Pressurized N2 or He <5 bar can leak out. | Operator training **Note: already applied** | | | |
| | Influence and effects from other | Procedure's performance can be affected by other sources | Fail in measurement point; | Notification of measurements to responsible person **Note: clear mention in procedure** | | | |

- **ATTACHMENT G PROCEDURE FOR RUNNING EXPERIMENTS**

| Experiment, name, number: | Date/ |
|---|---|
| Conductivity rig: measuring conductivity in porous media | Sign |
| **Project Leader:** | |
| Erling Næss/Christian Schlemminger | |
| **Experiment Leader:** | |
| Jan Georg Henriksen/Christian Schlemminger | |
| **Operator, Duties:** | |
| Jan Georg Henriksen/Christian Schlemminger | |

| | Conditions for the experiment: | Completed |
|---|---|---|
| 1 | Experiments should be run in normal working hours, 08:00-16:00 during winter time and 08.00-15.00 during summer time. Experiments outside normal working hours shall be approved. | |
| 2 | The operator must start the experiment. After start operator may leave and experiment will run itself. | |
| 3 | An early warning is given according to the lab rules, and accepted by authorized personnel. | |
| 4 | Be sure that everyone taking part of the experiment is wearing the necessary protecting equipment and is aware of the shut down procedure and escape routes. | |
| | | **Carried out** |
| | **Filling of new powder** | |
| | | |
| 5 | If rig is running, set liquid bath to 22 degrees and close shut-off and needle valve. Open the sample circuit valve and vent the system to ambient pressure. | |
| 6 | When liquid bath has been at 22 degrees for 10 minutes, shut of the bath. | |
| 7 | Remove wires from pressure cell. | |
| 8 | Then remove the pressure cell from the bath. | |
| 9 | Remove the pressure cell lid and disconnect the inner wires. | |
| 10 | Remove rubber sealing and take out the sample/sample holder. | |
| 11 | Use glasses eye protection, gloves and dusk mask | |
| 12 | Weigh the sample holder and measure sample height. | |
| 13 | Empty sample holder and remove sensor. | |
| 14 | Clean vacuum pressure cell, sensor, rubber sealing, and sample holder. | |
| 15 | Weigh the sample holder without any powder in it. One can now tell how much mass the powder has lost during evacuation. | |
| 16 | Pour in a new powder sample using a funnel. | |
| 17 | Weigh new powder and measure filling height. | |
| | | |
| 18 | If the powder consists of small light grains, tape aluminum foil over the sample in order to prevent powder loss during evacuation. | |
| 19 | Put sample holder back into the pressure cell. | |
| 20 | Lubricate the sample holder and its lid with vacuum grease. | |
| 21 | Connect wires and close the lid. Connect to computer. | |

| 22 | Mount the pressure cell into the liquid bath. | |
|---|---|---|
| 23 | Cover the gap between pressure cell and bath to prevent oil vaporization. | |
| 24 | Then start new experiment (described below) | |
| | | |

| | **Experimental procedure** | |
|---|---|---|
| 25 | Restart liquid bath and computer. | |
| 26 | Use glasses and gloves. | |
| 27 | All valves are closed. | |
| 28 | Set liquid bath (1.09) to 150 degrees and start vacuum pump (1.10). | |
| 29 | Open the shut-off valve. | |
| 30 | Adjust pressure with the pressure regulator (1.02). Pressure must be <0.5bar. | |
| 31 | Open needle valve (1.03). | |
| 32 | Open venting valve (1.04) and regulate (1.02) to desired pressure (0.4 or 0.2 bar). Then close the venting valve (1.04). | |
| 33 | Open sample circuit valve (1.05). Gas now flows into the pressure cell (1.08). | |
| 34 | Wait 10 seconds and close sample circuit valve (1.05) | |
| 35 | Open valve on the vacuum pump (1.10). | |
| 36 | When vacuum is below $10^{-2}$torr (0.13 bar), close vacuum pump valve (1.10). | |
| 37 | Repeat point 37-40 six times. | |
| 38 | Open sample circuit valve (1.05) and turn off vacuum pump (1.10). | |
| 39 | Close the needle valve (1.03) and wait 1 hour before starting the experiment. | |
| | **End of experiment** | |
| 40 | Close all valves starting with the shut-off valve (1.00) | |
| 41 | Open venting valve (1.04) and sample circuit valve (1.05) and vent the system to ambient pressure. | |
| 42 | Turn of liquid bath (1.09) | |
| 43 | Empty sample holder (if not used afterwards) and cover liquid bath. | |
| | **Also:** | |
| 44 | Remove all obstructions/barriers/signs around the experiment. | |
| 45 | Tide up and return all tools and equipment. | |
| 46 | Tidy and clean work areas. | |
| 47 | Return equipment and systems back to their normal operation settings | |
| 48 | **To reflect on before the next experiment and experience useful for others** | |
| 49 | Was the experiment completed as planned and on scheduled in professional terms? | |
| 50 | Was the competence which was needed for security and completion of the experiment available to you? | |
| 51 | Do you have any information/ knowledge from the experiment that you should document and share with fellow colleagues? | |

# ⬛ NTNU

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- ## ATTACHMENT H TRAINING OF OPERATORS

| Experiment, name, number: | |
|---|---|
| **Thermal conductivity measurements in porous media** | |
| **Project Leader:** | 22-05-2013 *Ql..ff* Date/Sign |
| **Christian Schlemminger / Erling Næss** | |
| **Operator** | |
| **Jan Georg Henriksen** | |

| | **Knowledge to EPT LAB in general** | |
|---|---|---|
| | Lab<br>- Access<br>- Routines and rules<br>- Working hour | |
| | Knowledge about the evacuation procedures. | |
| | Activity calendar for the Lab | |
| | Early warning, iept-experiments@ivt.ntnu.no | |
| | | |
| | **Knowledge to the experiments** | |
| | MSDS | ✓ |
| | Use of personal safety equipment | ✓ |
| | Procedures for the experiments | ✓ |
| | Emergency shutdown | ✓ |
| | Nearest fire and first aid station | ✓ |
| | Practical training with project leader | ✓ |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |

I hereby declare by my signing, that I have reviewed and understood the HSE regulations, have been given appropriate training for running this experiment and am aware of my personal responsibility by working in EPT laboratories.

**Operatør**

Dato  22/5 - 2013

Signert

- **ATTACHMENT I FORM FOR SAFE JOB ANALYSIS**

| SJA name: | |
|---|---|
| Date: | Location: |
| Mark for completed checklist: | |

| Participators: | | |
|---|---|---|
| | | |
| SJA-responsible: | | |

Specification of work (What and how?):

Risks associated with the work:

Safeguards: (plan for actions, see next page):

Conclusions/comments:

| Recommended/approved | Date/Signature: | Recommended/approved | Date/Signature: |
|---|---|---|---|
| SJA-responsible: | | HSE responsible: | |
| Responsible for work: | | Other, (position): | |

| HSE aspect | Yes | No | NA | Comments / actions | Resp. |
|---|---|---|---|---|---|
| **Documentation, experience, qualifications** | | | | | |
| Known operation or work? | | | | | |
| Knowledge of experiences / incidents from similar operations? | | | | | |
| Necessary personnel? | | | | | |
| **Communication and coordinating** | | | | | |
| Potential conflicts with other operations? | | | | | |
| Handling of an eventually incident (alarm, evacuation)? | | | | | |
| Need for extra assistance / watch? | | | | | |
| **Working area** | | | | | |
| Unusual working position | | | | | |
| Work in tanks, manhole? | | | | | |
| Work in ditch, shaft or pit? | | | | | |
| Clean and tidy? | | | | | |
| Protective equipment beyond the personal? | | | | | |
| Weather, wind, visibility, lighting, ventilation? | | | | | |
| Usage of scaffolding/lifts/belts/ straps, anti-falling device? | | | | | |
| Work at hights? | | | | | |
| Ionizing radiation? | | | | | |
| Influence of escape routes? | | | | | |
| **Chemical hazards** | | | | | |
| Usage of hazardous/toxic/corrosive chemicals? | | | | | |
| Usage of flammable or explosive chemicals? | | | | | |
| Risk assessment of usage? | | | | | |
| Biological materials/substances? | | | | | |
| Dust/asbestos/dust from insulation? | | | | | |
| **Mechanical hazards** | | | | | |
| Stability/strength/tension? | | | | | |
| Crush/clamp/cut/hit? | | | | | |
| Dust/pressure/temperature? | | | | | |
| Handling of waste disposal? | | | | | |
| Need of special tools? | | | | | |
| **Electrical hazards** | | | | | |
| Current/Voltage/over 1000V? | | | | | |
| Current surge, short circuit? | | | | | |
| Loss of current supply? | | | | | |
| **Area** | | | | | |
| Need for inspection? | | | | | |
| Marking/system of signs/rope off? | | | | | |
| Environmental consequences? | | | | | |
| **Key physical security systems** | | | | | |
| Work on or demounting of safety systems? | | | | | |
| **Other** | | | | | |

- **ATTACHMENT J APPARATURKORT UNITCARD**

## Apparatur/unit

Dette kortet SKAL henges godt synlig på apparaturen!*This card MUST be posted on a visible place on the unit!*

| Faglig Ansvarlig (Scientific Responsible) | Telefon mobil/privat (Phone no. mobile/private) |
|---|---|
| **Christian Schlemminger** / Erling Næss | **41063418** / 91897970 |
| **Apparaturansvarlig** (Unit Responsible) | **Telefon mobil/privat** (Phone no. mobile/private) |
| Christian Schlemminger/ **Jan Georg Henriksen** | 41063418 / **994 32 522** |
| NTNU – Sintef Beredskapstelefon | 800 80 388 |
| **Sikkerhetsrisikoer** (Safety hazards)<br><br>• **Pressurized He/N2**<br>• **Hot/cold liquid bath** | |
| **Sikkerhetsregler**  (Safety rules)<br><br>Use eye protection glasses.<br><br>**Do not touch liquid bath and/or nearby components.** | |
| **Nødstopp prosedyre**<br>(Emergency shutdown)<br>*Close gas shut-off valve signed as "Emergency shut-off valve"*<br><br>*Disable power supply to liquid bath* | |

**Her finner du** (Here you will find):

| **Prosedyrer** (Procedures) | **above computer screen**, in FinLab. |
|---|---|
| **Bruksanvisning** (User's manual) | **in computer desk** in the FinLab. |

**Nærmeste** (nearest)

| **Brannslukningsapparat** (fire extinguisher) | **beside elevator** |
|---|---|
| **Førstehjelpsskap** (first aid cabinet) | **beside elevator** |

| | |
|---|---|
| **NTNU**<br>**Institutt for energi og prosessteknikk** | **SINTEF Energi**<br>**Avdeling energiprosesser** |
| **Dato** | **Dato** |
| | |
| **Signert** | **Signert** |

• **ATTACHMENT K FORSØK PÅGÅR KORT**

# Forsøk pågår!
# Experiment in progress!

Dette kort skal settes opp før forsøk kan påbegynnes This card has to be posted before an experiment can start

| Ansvarlig / Responsible<br>**Christian Schlemminger** / Erling Næss | Telefon work/mobile/home<br>**41063418** / 91897970 |
|---|---|
| Operatører/Operators<br>**Jan Georg Henriksen** / Christian Schlemminger<br>**994 32 522 /** 41063418 | Forsøksperiode/Experiment time(start – slutt) |
| Project leaders signature | Project<br>**Thermal conductivity measurements.** |
| NTNU – Sintef Beredskapstelefon | 800 80 388 |

**Kort beskrivelse av forsøket /** Short description of the experiment

There is a new conductivity rig located in the FinLab in the VATLab. It is a swedish HotDisk setup and utilizes a small power (<2W) to heat up a sample of porous material and then measure the temperature transient. From the measured temperature differences the HotDisk Software then calculates the thermal conductivity.

Thermal conductivity's for various porous media are then found. These values can be used to verify theoretical models.

**Relaterte farer** / related hazards

Hot/cold liquid bath and nearby components→ do not touch!
Liquid bath may evaporate oil → be aware of slippery floor!
Vacuum pump causes noise.

Pressurized Helium or Nitrogen can cause noise
Pressurized Helium or Nitrogen may cause tubes to crack

Don't eat and drink in the area

**NTNU**
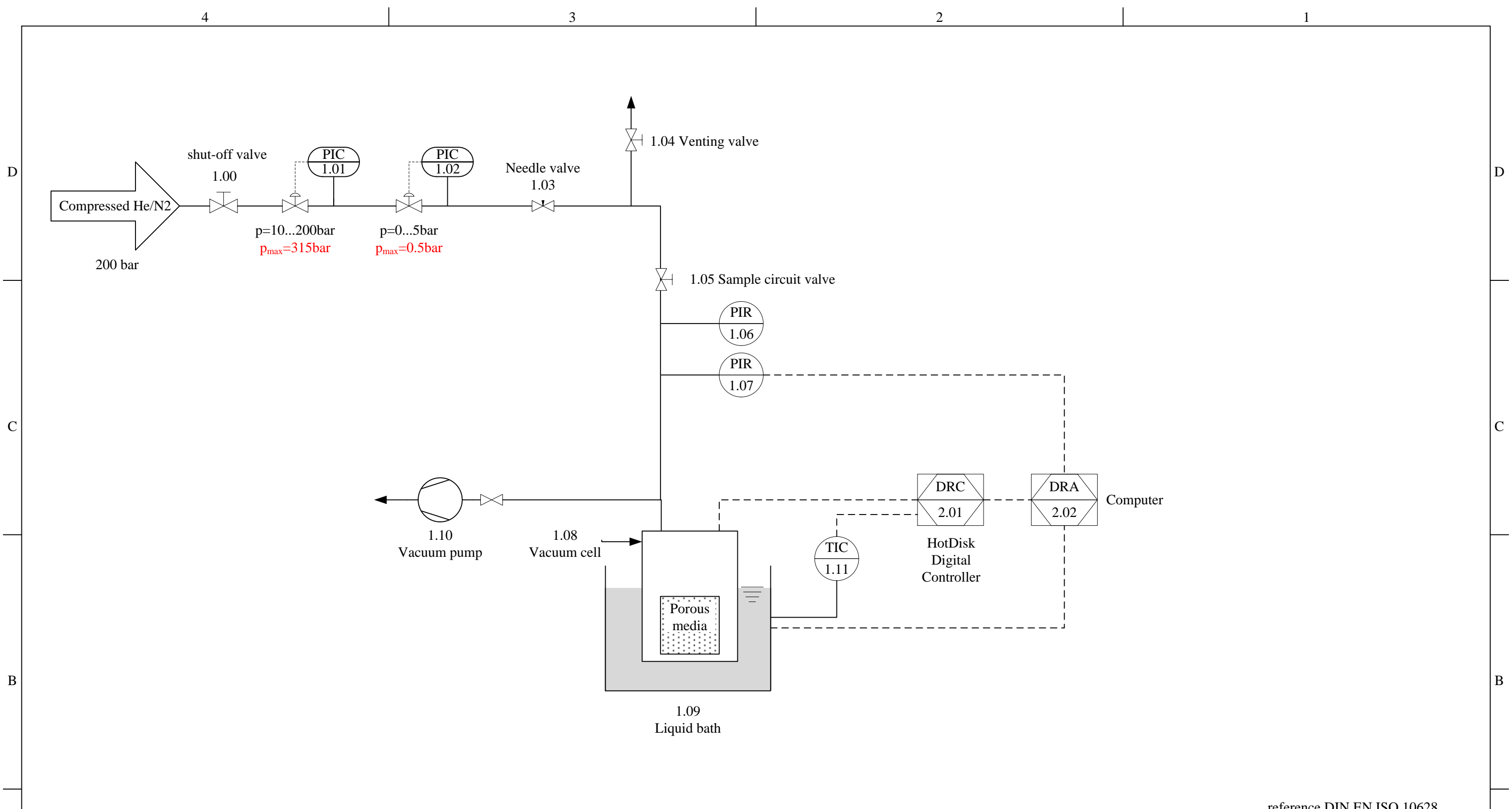**Institutt for energi og prosessteknikk**

**SINTEF Energi**
**Avdeling energiprosesser**

**Dato**

**Dato**

**Signert**

**Signert**

| | | | |
|---|---|---|---|
| | | 4 | 3 | 2 | 1 | | |

Compressed He/N2

200 bar

shut-off valve
1.00

PIC
1.01

p=10...200bar
p$_{max}$=315bar

PIC
1.02

p=0...5bar
p$_{max}$=0.5bar

Needle valve
1.03

1.04 Venting valve

1.05 Sample circuit valve

PIR
1.06

PIR
1.07

DRC
2.01

DRA
2.02

Computer

HotDisk
Digital
Controller

TIC
1.11

1.10
Vacuum pump

1.08
Vacuum cell

Porous
media

1.09
Liquid bath

reference DIN EN ISO 10628

# FORSØK PÅGÅR

Enhet (unit) og bygg/romnr. (building/room no.):

| NTNU-E 302 | C165C | 1. etg |

Laboratorium      Finlab 1

## Dette kortet SKAL henges opp før forsøk kan starte!
### *This card MUST be posted on the unit before the experiment startup!*

| | |
|---|---|
| **Apparatur (Unit)**<br>**Thermal conductivity rig** | **Dato godkjent (Date Approved)**<br>31. mai 2013 |
| **Prosjektleder (Project Leader)**<br>Erling Næss | **Telefon mobil/privat (Phone no. mobile/private)**<br>918 97 970 |
| **Apparaturansvarlig (Unit Responsible)**<br>Erling Næss | **Telefon mobil/privat (Phone no. mobile/private)**<br>918 97 970 |

**Godkjente operatører (Approved Operators)**

| Navn/Name | Telefon/Phone | Mobil |
|---|---|---|
| Schlemminger, Christian | | 410 63 418 |
| Henriksen, Jan Georg | | 99432522 |

**Prosjekt (Project)**

Thermal conductivity measurements in porous media

**Forsøkstid / Experimental time (start - stop))**

31.05.2013 - 31.05.2014

**Kort beskrivelse av forsøket og relaterte farer (Short description of the experiment and related hazards)**

- Hot/cold liquid bath and nearby componets do not touch!
- Liquid bath may evaporate il, be aware of slippery floor!
- Vacuum pump causes noise.
- Pressurized Helium or Nitrogen can cause noise
- Pressurized Helium or Nitrogen may cause tubes to crack
- Don't eat and drink in the area

**NTNU**
**Institutt for energi og prosessteknikk**

Dato   4/6 – 2013

Signert

**NTNU**    **SINTEF**

# APPARATURKORT

Enhet (unit) og bygg/romnr. (building/room no.):

| NTNU-E 302 | C165C | 1. etg |

Laboratorium          Finlab 1

## Dette kortet SKAL henges godt synlig ved maskinen!
## *This card MUST be posted on a visible place on the unit!*

| **Apparatur (Unit)** | **Dato Godkjent (Date Approved)** |
|---|---|
| **Thermal conductivity rig** | 31. mai 2013 |
| **Prosjektleder (Project Leader)** | **Telefon mobil/privat (Phone no. mobile/private)** |
| Erling Næss | 918 97 970 |
| **Apparaturansvarlig (Unit Responsible)** | **Telefon mobil/privat (Phone no. mobile/private)** |
| Erling Næss | 918 97 970 |

**Sikkerhetsrisikoer (Safety hazards)**
- Pressurized He/N2
- Hot/cold liquid bath

**Sikkerhetsregler (Safety rules)**
- Use eye protection glasses.
- Do not touch liquid bath and/or nearby components

**Nødstopp prosedyre (Emergency shutdown)**
- Close gas shut-off valve signed as "Emergency shut-off valve"
- Disable power supply to liquid bath

| **Her finner du (Here you will find):** | |
|---|---|
| **Prosedyrer (Procedures)** | At the control desk |
| **Bruksanvisning (User manual)** | At the control desk |
| **Brannslukningsapparat (Fire extinguisher)** | 1. etasje TermiskLab (nord) |
| **Førsthjelpsskap (First aid cabinet)** | 1. etasje TermiskLab (nord) |

**NTNU**

**Institutt for energi og prosessteknikk**

Dato   4/6 – 2013

Signert   *[signature]*

NTNU          SINTEF