API-MS Transfer Capillary Flow: Examination of the Downstream Gas Expansion

Sebastian Klopotowski[1]; Yessica Brachthaeuser[1]; David Mueller[1]; Hendrik Kersten[1]; Walter Wissdorf[1]; Valerie Derpmann[1]; Sonja Klee[1]; Klaus J. Brockmann[1]; Uwe Janoske[2]; Hauke Gregor[2]; Thorsten Benter[1]

[1]Physical & Theoretical Chemistry
[2]Mechanical Engineering
University of Wuppertal, Germany
Institute for Pure and Applied Mass Spectrometry

Introduction

The application of atmospheric pressure ionization (API) necessitates a pressure reduction in differential pumping stages before mass analysis. One common approach for the first reduction step from atmospheric pressure to the low mbar range is realized by critically operated capillaries.
The pressure ratio between capillary entrance and first pumping stage is generally greater than 0.5 leading to choked flow conditions. In fluid dynamical terms the gas flow out of the capillary is a transonic overexpansion. Stationary shock waves (Mach discs), depending on capillary bore and background pressure, are thus expected. The interaction between the gas jet leaving the capillary and downstream ion optical devices (skimmers / ion funnels) could potentially lead to significant ion losses.
The capillary is either actively tempered or heated by a hot dry gas stream.
We used an automated experimental setup to analyze in detail the flow conditions downstream of the transfer capillary exit at various temperatures and pressures.

Methods

Experimental Methods

Measurement Chamber:
Home built vacuum recipient with mounting for a transfer capillary, evacuated by a flow controlled rotary pump

Temperature Probe:
Micro thermocouple (Type K: Ni/CrNi) with 0.25 mm diameter, mounted on a positioning stage behind the capillary exit

Capillary:
Home built glass capillary with internal Pt100 measuring resistor, length 18 cm, inner diameter 0.5 mm. Enveloped with tantalum wire (0.1 mm) for current heating

XY-Stage:
Moveable platform adjusted by two I²C controlled, motorized thread rods

Contact Microfone:
Directly attached close to capillary exit, signals recorded by computer audio interface

Pressure Control:
MKS Type 252E Exhaust Valve Controller between measurement chamber and rough pump with matching butterfly valve.

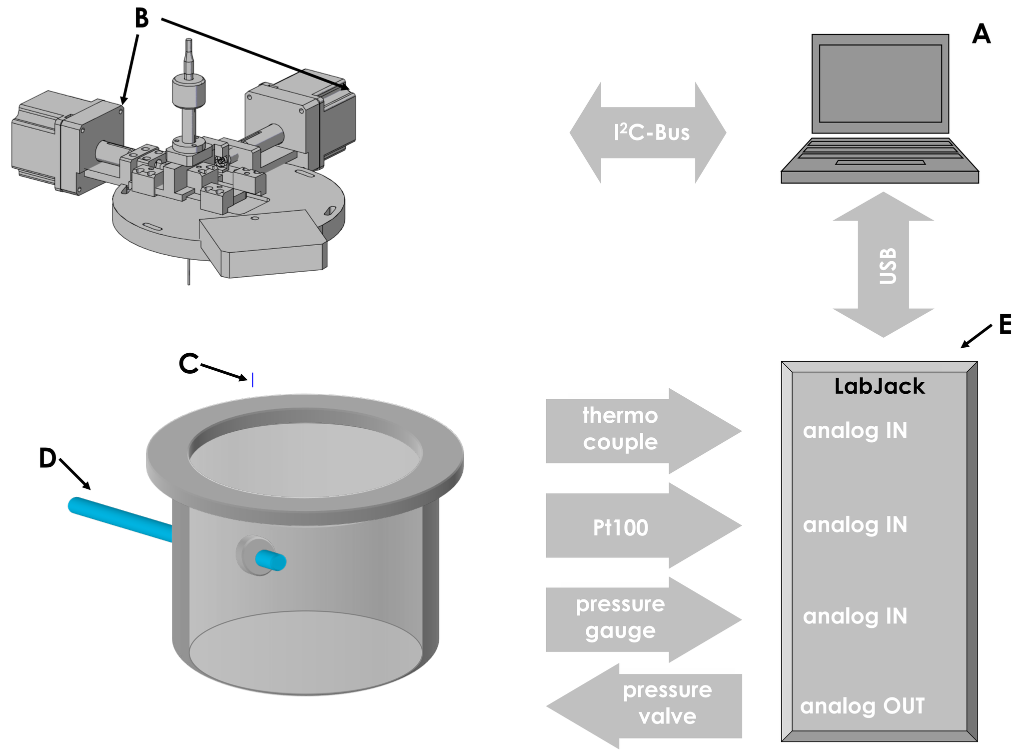
Data Acquisition:
Labjack U12 A/D D/A Interface

Numerical Methods

Computational Fluid Dynamics (CFD) :
Basic numerical calculations performed with OpenFOAM

Fast-Fourier-Transformation (FFT) :
Sound spectrum analysis performed by gnuOCTAVE and plotted with gnuPlot

Experimental Setup



- Automated measuring equipment is computer controlled. Software is written in Python programming language (version 3.1) using Qt 4.7 for graphical user interface.
- Data are collected with an AD/DA device that additionally generates the control voltage for the pumping stage. Stepper motors, driven via I²C-Interface, move xy-stage with mounted thermocouple.
- Spatial resolution is 0.1mm per iteration, maximal measured area is two by two centimeters.
- The contact microphone, clipped onto the capillary, is not shown here. It is directly connected to the PC sound interface

A: Computer with Python interpreter
B: Stepper Motors
C: Thermocouple
D: Heated capillary with temp. sensor
E: Labjack U12 AD/DA Device

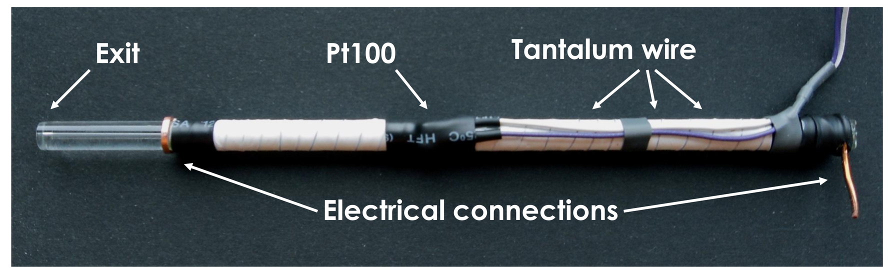


Figure 1a) Heatable transfer capillary with built-in thermo resistor and tantalum filament; the gas flow enters from the right.

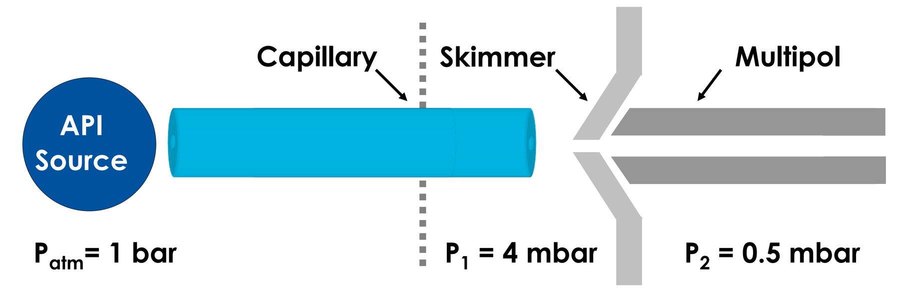


Figure 1b) Schematic representation of the first differential pumping stage, the skimmer diameter is about 1.5 mm, the distance from the capillary exit to the skimmer is 1 to 2 mm

Sound Measurements

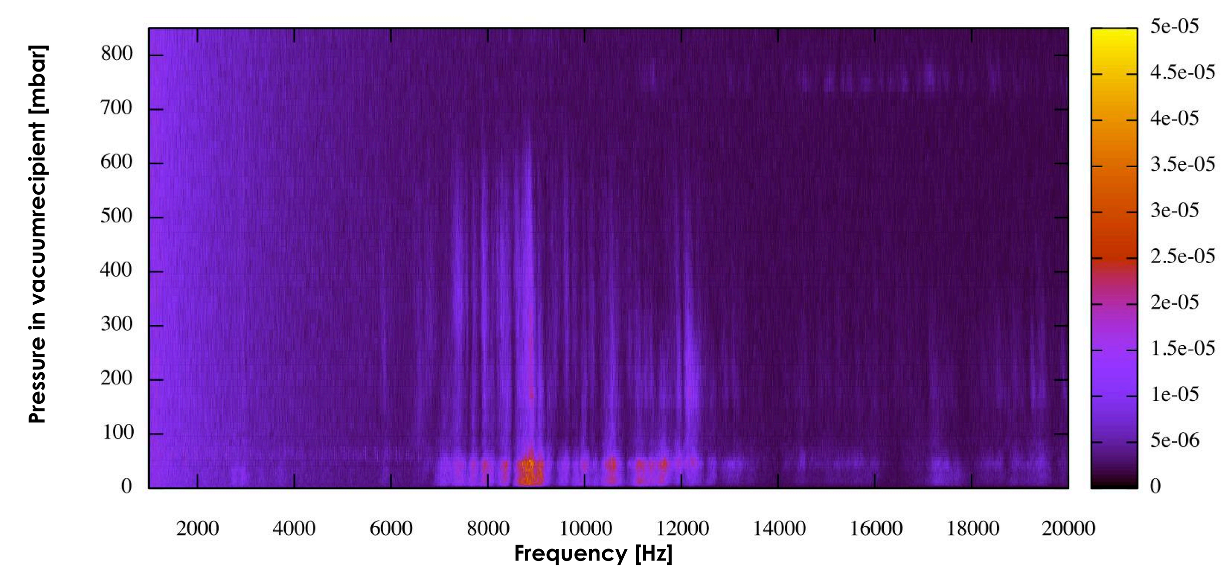


Figure 2) Sound emission as function of pressure in the vacuum recipient at 85°C capillary temperature.

Temperature Distribution behind Capillary Exit

a.) T = 26°C / p_1 = 3 mbar

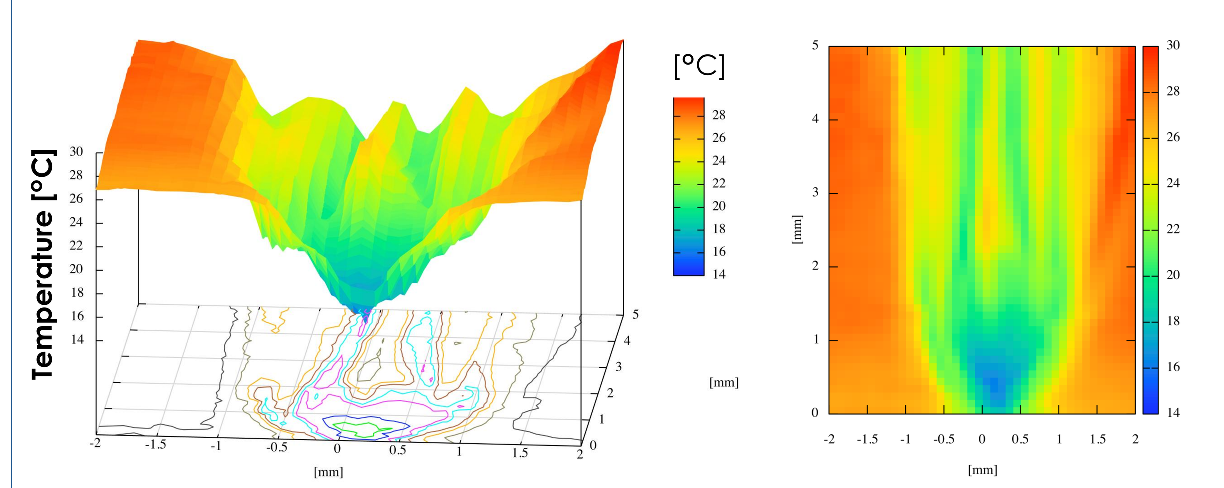


Figure 3a) Results of the temperature measurements in a transonic jet. First shock front is identifiable approximately 2.3 mm behind the capillary exit, width the of gas jet is 2.5 mm

b.) T = 85°C / p_1 = 2 mbar

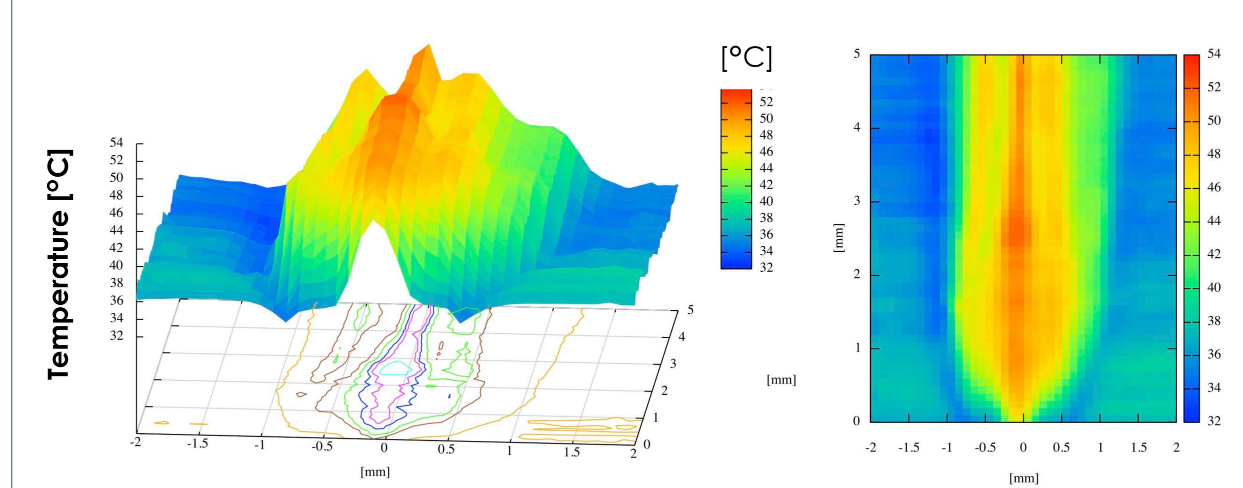


Figure 3b) Results of the temperature measurements in a transonic jet. First shock front is identifiable approximately 2.4 mm behind the capillary exit, width of the gas jet is roughly 2.5 mm

c.) T = 100°C / p_1 = 20 mbar

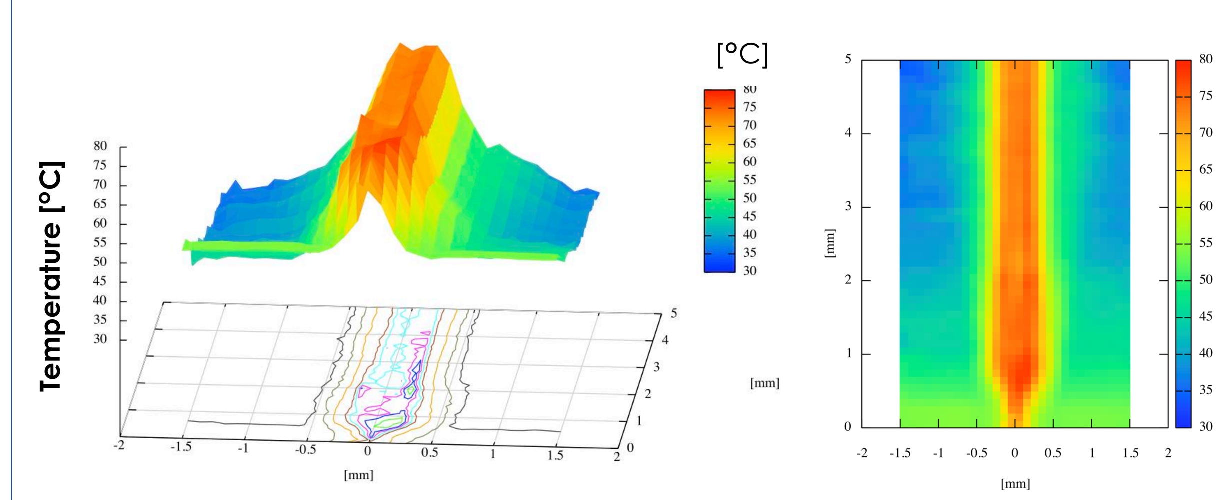


Figure 3c) Results of the temperature measurements in a transonic jet. First shock front is identifiable approximately 1.0 mm behind the capillary exit, width of the gas jet is roughly 1 mm

Axial Temperature Profile

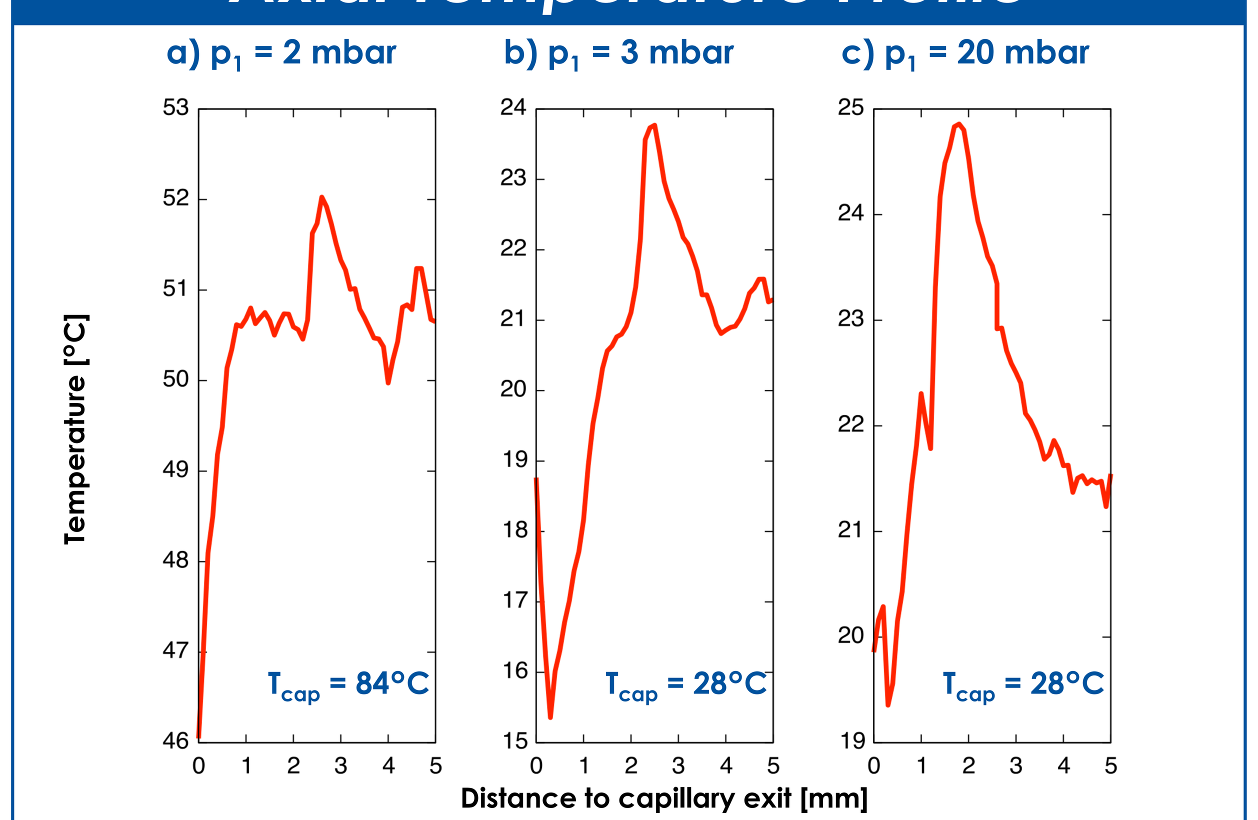


Figure 4) Temperature distribution along the jet axis for different background pressures. Shock waves are recognizable by the transient temperature increases.

Position of Mach Disc

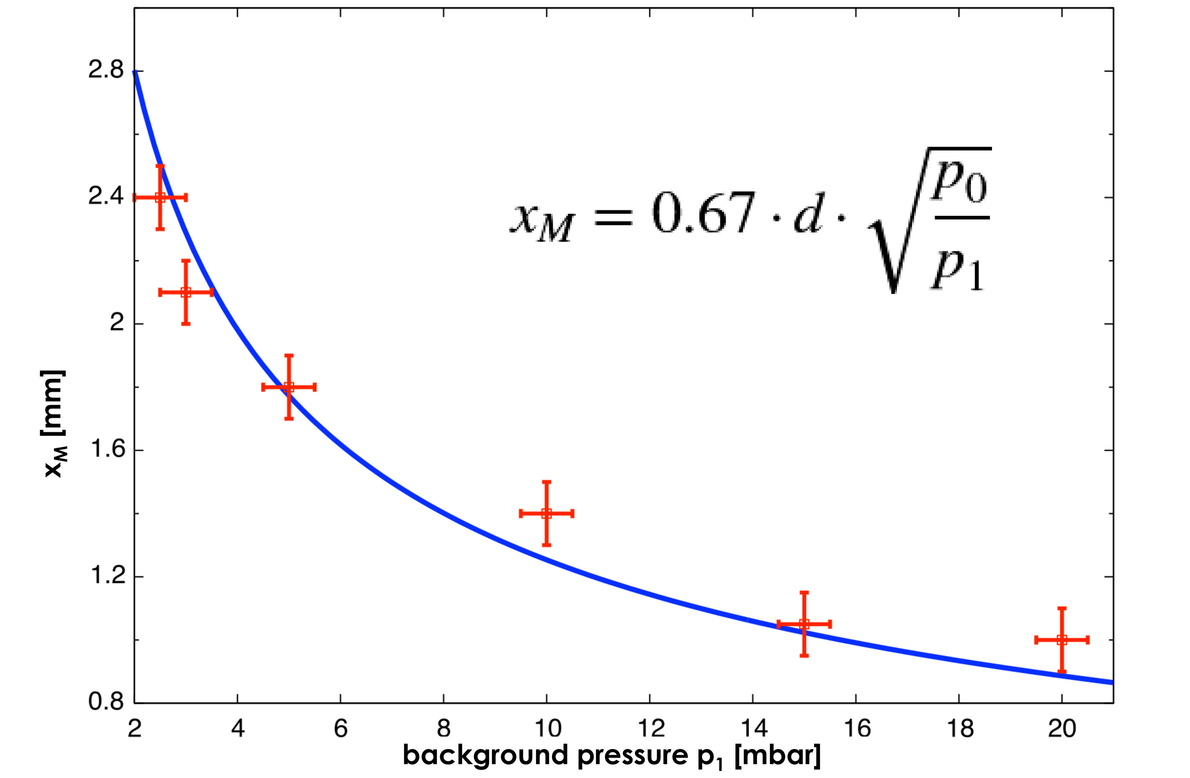


Figure 5) Position of mach disc as a function of background pressure ($p1$) compared to theoretical values[6,7]. x_M is the distance from the capillary exit to the mach disc, p_0 = 140 mbar is the pressure at the capillary exit, d is the capillary diameter (capillary temperature = 100°C)

Choked Flow

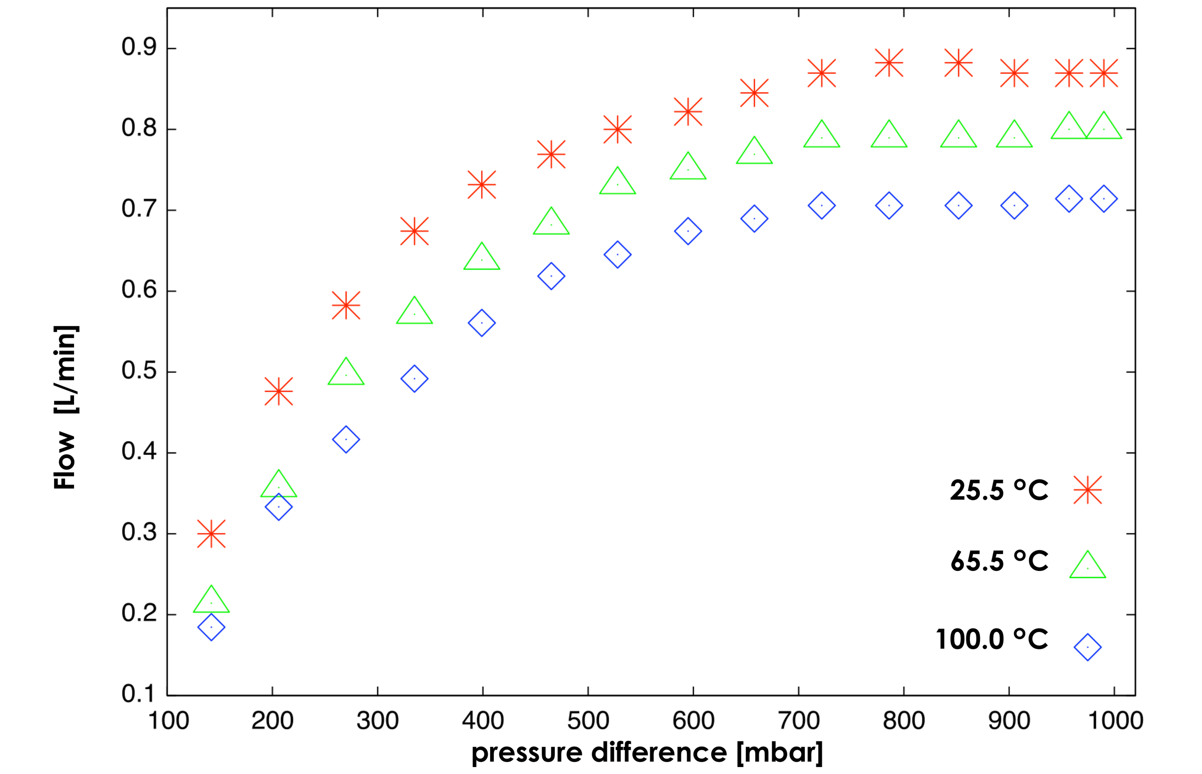


Figure 6) Choked flow through the capillary for three different temperatures. Flow rate increases up to a pressure ratio close to 0.5 and becomes constant and independent of the vacuum recipient pressure

Conclusions

- A remarkably narrow and stable gas jet leaves the transfer capillary and can still be measured at some centimeters distance from the exit
- The jet diameter is inversely dependent on the vacuum recipient pressure
- Under typical operating conditions the jet diameter is two to three times wider than the skimmer bore
- Stationary shock waves are observed as sudden increases in the jet temperature, as predicted by numerical calculations
- The Mach disc appears at the expected location irrespective of the capillary wall temperature
- The measured maximum jet core temperature is only marginally lower than the controlled capillary wall temperature
- The minimum temperature in the expansion zone is higher than predicted by the calculations even if the effect of radiation heating by the capillary is considered

➢ *Interference between jet and skimmer have to be expected*

- With rising temperature and viscosity the gas throughput decreases to values that also result from theoretical calculations
- The gas flow is heated effectively even when only the last few cm of the capillary are tempered

➢ *There is not much doubt that the capillary flow is turbulent.*

- Transfer capillaries emit characteristic sound spectra, which are dependent on the wall temperature and gas flow
- The frequency spectrum varies with wall temperature but not with recipient pressure
- The highest signal intensities were observed at a recipient pressure of 30 mbar

- Preliminary CFD calculations give an adequate, qualitative picture of the overexpanded flow; additional investigations are underway

Literature

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