Characterization of API-MS Inlet Capillary Flow: Examination of Transfer Times and Choked Flow Conditions

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Introduction

Challenge:

To understand the complex flow dynamics inside inlet capillaries, gain knowledge on the gas flow through such devices is necessary

State of Knowledge:

The gas flow is governed by parameters such as capillary wall temperature, pressure difference and gas viscosity, which directly affect essential flow characteristics

Experimental:

- A custom transfer capillary made of fused silica was attached to a custom built vacuum recipient. Atmospheric pressure laser ionization (APLI) was used for the generation of temporally and spatially well defined ion packets inside the transfer capillary. The ions were detected by a faraday cup, connected to an electrometer. The ionization region was shifted along the capillary main axis. Ion transfer times were measured with an oscilloscope, triggered by the excimer laser used for ionization
- A number of glass capillaries of various lengths and inner diameters were fitted with heating elements. The mass flow through the temperature controlled inlet capillaries was measured as a function of pressure difference and wall temperature with a drum-type gas meter

Methods

Experimental Setup

Measurement Chamber:

Home built vacuum recipient with mounting for a transfer capillary, evacuated by a rotary pump

Capillary:

Home built glass and quartz capillary, length: 6 to 22 cm, inner diameter: 0.4 – 0.6 mm; Enveloped with tantalum wire (0.1 mm) for current heating

Laser:

ATL Atlex KrF-Excimer Laser@248 nm

Current Measuremnent:

Keithley 610C Electrometer

MS:

Bruker MicrOTof

Current Measurement:

Keithley 610C Electrometer

Flow Meter:

Ritter TG-1 Drum-Type Gasmeter

Temperature Probe:

Micro thermocouple with 0.25 mm diameter

Pressure Control:

MKS Type 252E Exhaust Valve Controller between measurement chamber and rough pump

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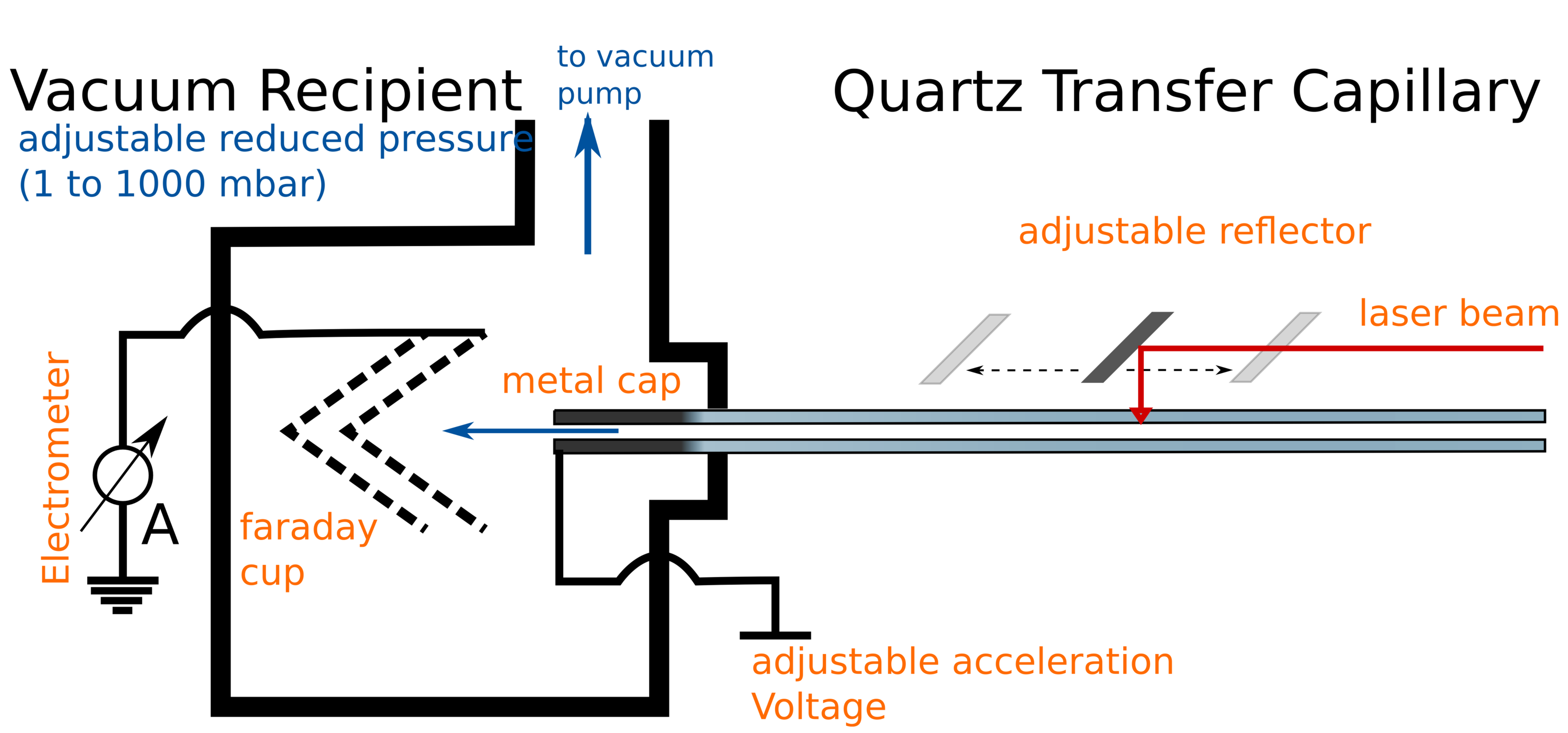
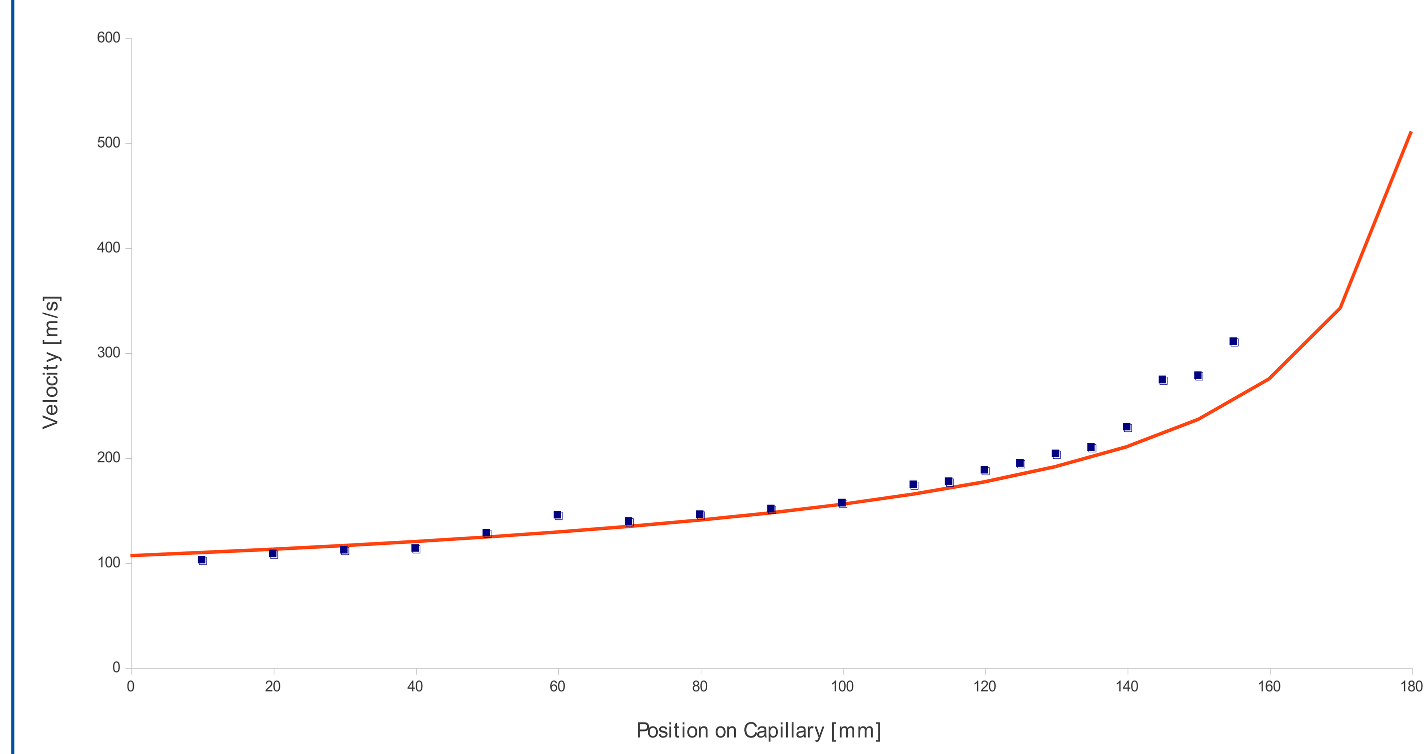
Experimental Setup



Ion Velocity

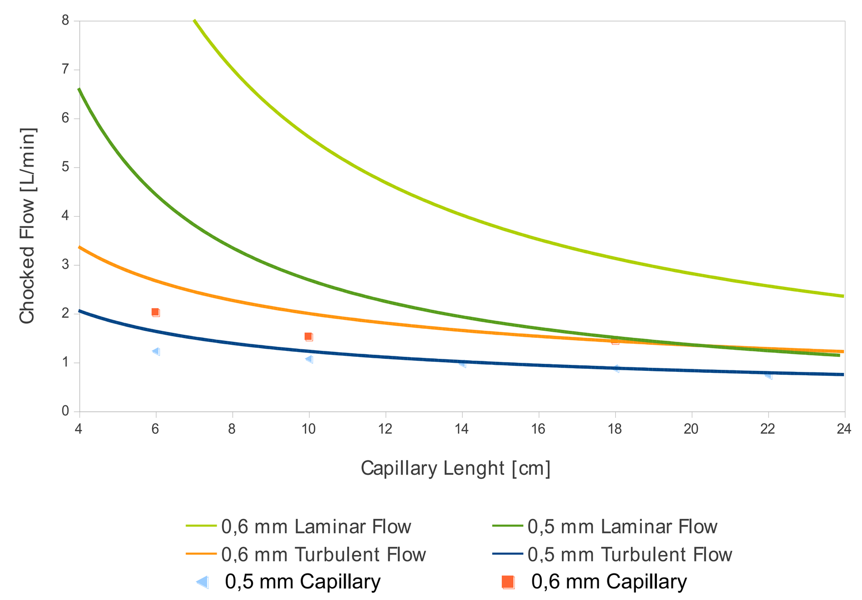


The generated ion packets travel with the bulk gas through the capillary. The transfer time of these ions was measured as a function of the distance between the ionization region varied along the main capillary axis

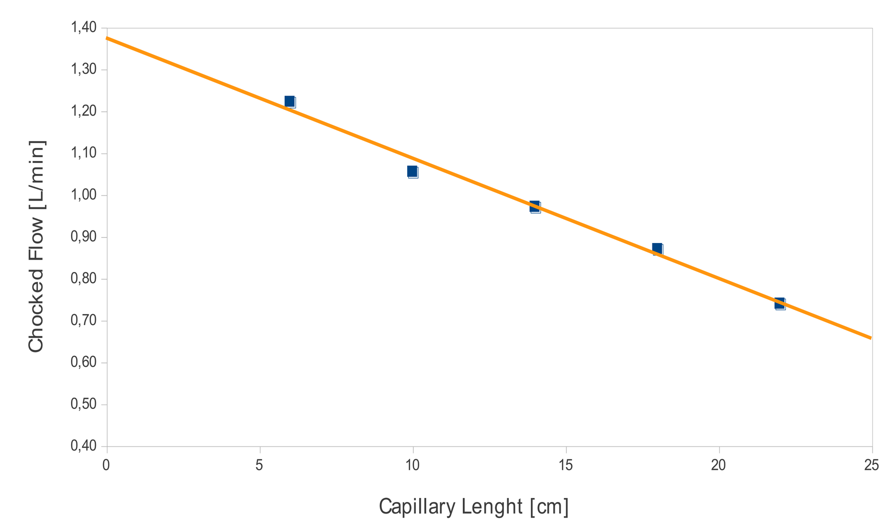
Ion velocity was calculated from measured transfer times. Theoretical fluid dynamic velocity was calculated for turbulent flow conditions

Ion velocity in a quartz capillary, inner diameter 0.4 mm, length 18 cm; REMPI analyte toluene, ionized KrF excimer laser; time of flight measured with an oscilloscope triggered by the laser

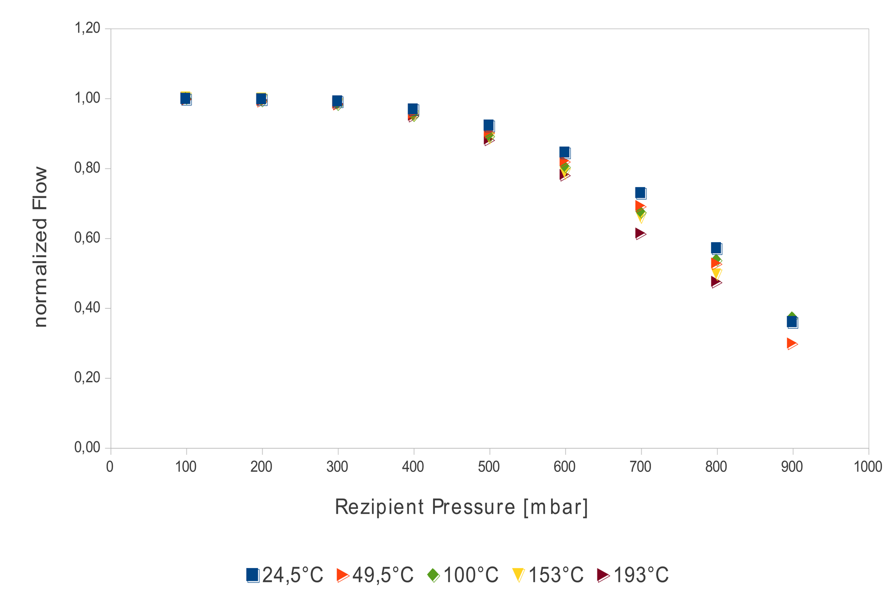
Choked Flow & Temperature Dependency



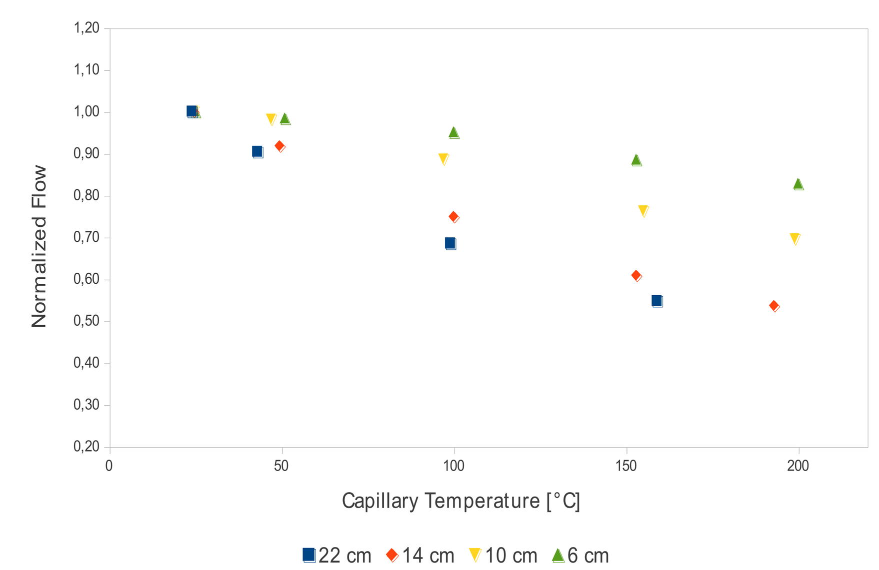
Measured flow at choked conditions for a 0.5 mm and 0.6 mm capillary, compared to theoretical calculations for laminar and turbulent flow conditions.



Absolute flow through a 0.5 mm inner diameter capillary at choked flow conditions

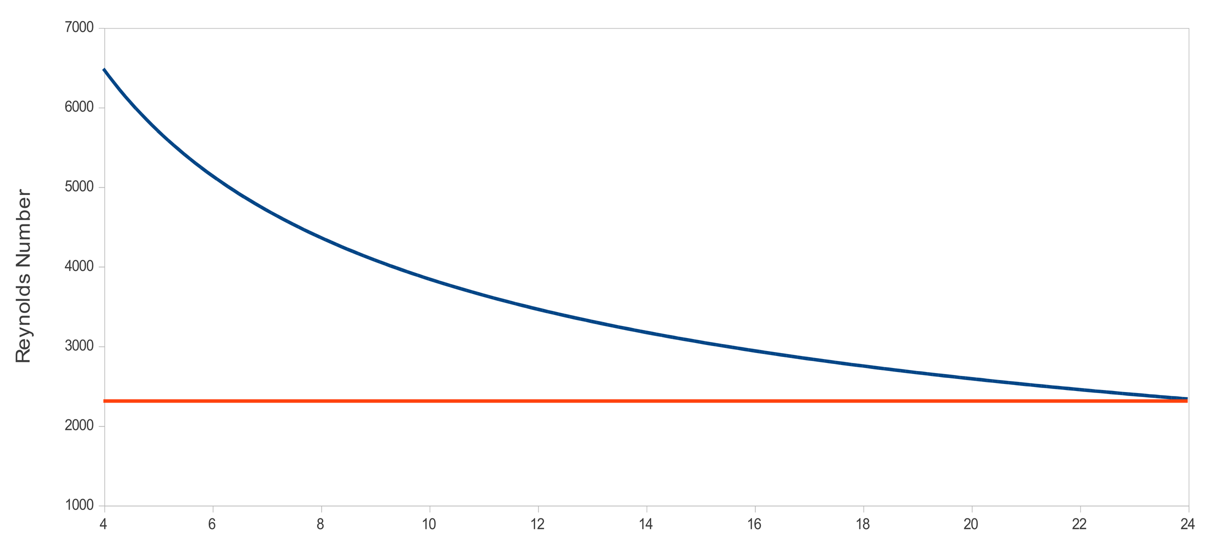


Normalized flow through a 0.5 mm inner diameter capillary and 10 cm length, for five different temperatures



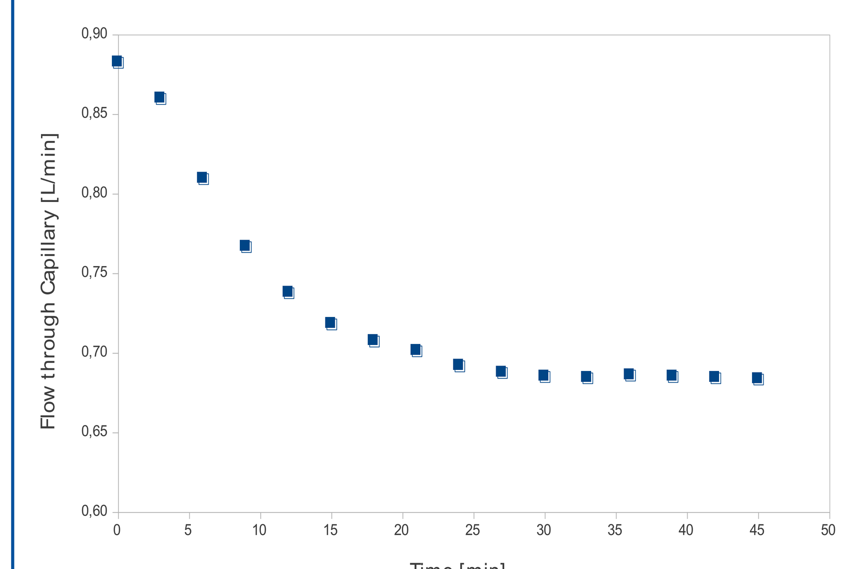
Temperature dependent choked flow through four different capillaries with 0.5 mm inner diameter, but different length

Reynolds Numbers

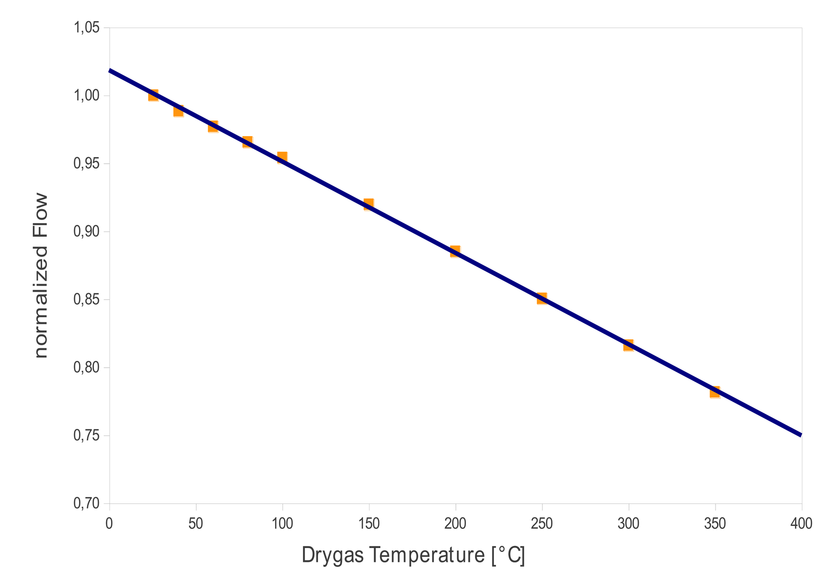


The Reynolds Number of a 0.5 mm capillary decreases with increasing capillary length, but is still greater than 2300 (turbulence criteria)

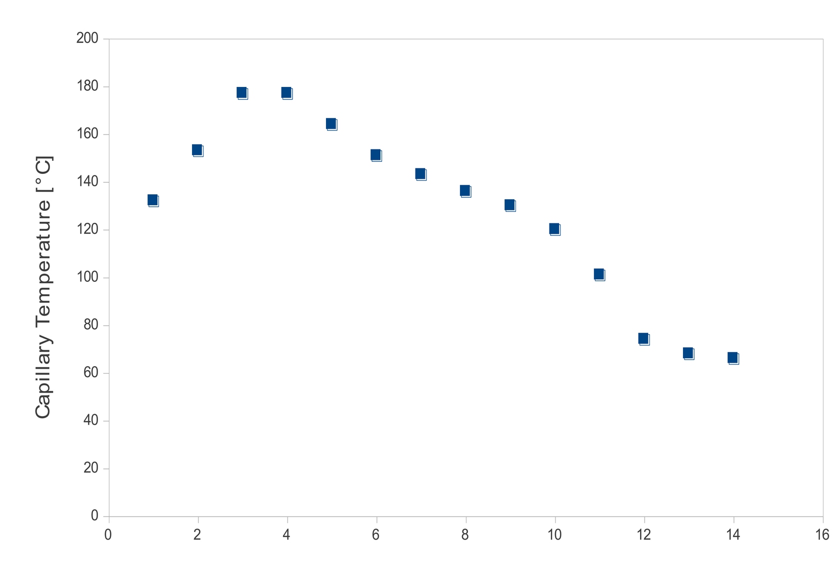
Behavior of Bruker micrOTOF



Absolute flow into the micrOTOF, on warming from room temperature with 4 L/min dry gas flow at 350°C



Normalized flow through inlet capillary with 4 L/min dry gas for different dry gas temperatures



Temperature profile along the Bruker micrOTOF inlet capillary measured with a flow of 4 L/min dry gas at 350°C

Conclusions

- The experimental setup allows a detailed insight into the fluid dynamical transport properties of inlet capillaries
- The measured transfer times are in very good agreement with those predicted by fluid dynamical equations
- Choked flow conditions are temperature independent
- Comparisons of the choked mass flow as well as the velocity of the fluid with theoretical models for the laminar and turbulent flow cases clearly show that transfer capillaries are generally operated at fully developed turbulent conditions
- Heated dry gas reduce fluid flow up to 25%
- Fluid dynamical models describe well the flow through capillaries with large length/diameter ratio, shorter wider capillaries are not approximated very well
- Turbulent flow is associated with significant ion wall losses, particularly in the case of metal capillaries, however this is not observed
- Further studies are necessary to explain the observed high ion transmissions of glass and metal inlet capillaries

Literature

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