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Development of clamp-on type thermal mass flow meter using near infrared absorption method for micro flow applications

KRISS 한국표준과학연구원

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Content

Contents

Measurement of micro liquid flow rate

❑ Medical drug injection

- Small amount of drug
  (pain treatment : 21.6 ug/day, insulin pump : 70 nL/min)
- Patient treatment through accurate drug injection monitoring and control
- Limitation of infusion and syringe pumps
  (low accuracy, environment dependency)

❑ Chemical analysis(chromatography)

- Micro flow liquid chromatography (250 – 600 μL/min)
- Column diameter : 0.25 – 1 mm, total volume : 2-10 μL
- Accuracy flow rate control and monitoring

❑ Semiconductor and fuel cell industries

- Semiconductor process (precursor delivery, cleaning..)
- Fuel flow rate inside fuel cell

Infusion and syringe medical pumps

Liquid chromatography

Fuel cell

Micro flow meters

- ❑ Coriolis mass flow meter
- High accuracy (±0.2 %, maximum flow rate : 5 g/h)
- Expensive price, relatively heavy size

Coriolis flow meter

- ❑ Thermal mass flow meter
- Low flow rate limit : nL/min order
- 10 % flow rate accuracy, low price and high portability

Thermal mass flow meter

- ❑ Ultrasonic flow meter
- Can measure in-situ condition(none contact)
- Low accuracy in micro flow rate

Ultrasound flow meter

➢ No commercial flowmeter which can measure micro flow rate in-situ condition.

Flow meters	Coriolis	Thermal mass	Ultrasonic
Accuracy	*****	***	**
Price	*	****	**
Micro flow rate	*****	*****	*
In situ measurement	none	none	****

Laser based flow measurement tool

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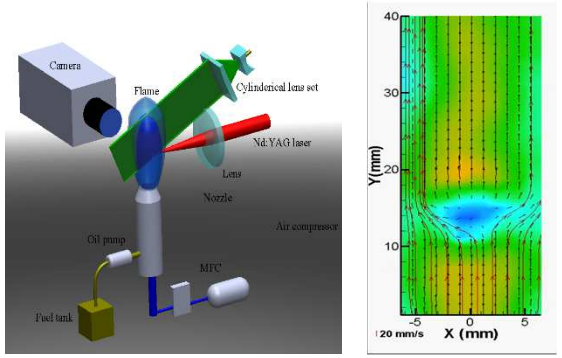
❑ LDA(Laser Doppler Anemometry)
- Flow velocity measurement using Doppler effect
- Point measurement, seeding particle necessary

Laser Doppler anemometry

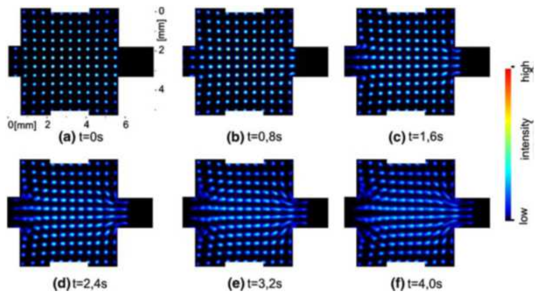
❑ PIV(Particle Image Velocimetry)
- Most popular flow velocity field visualization method
- Seeding particle necessary



Particle Image Velocimetry

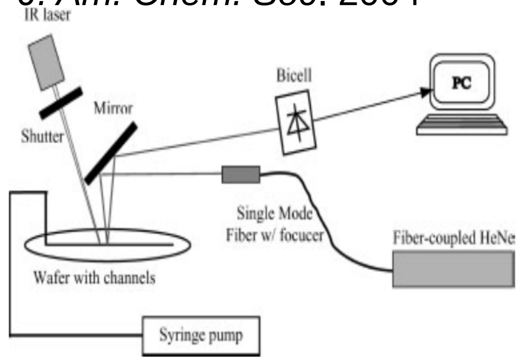
❑ MTV(Molecular Tagging Velocimetry)
- Visualization of Raman scattering signal
- Simultaneous measurement of concentration and velocity
- Low signal intensity

➢ Seeding particle is necessary

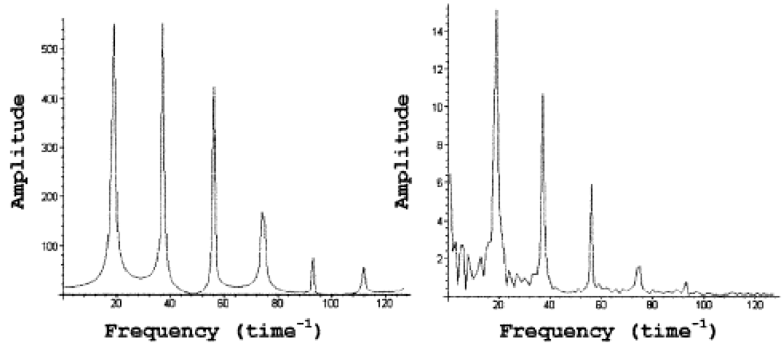


Molecular Tagging Velocimetry

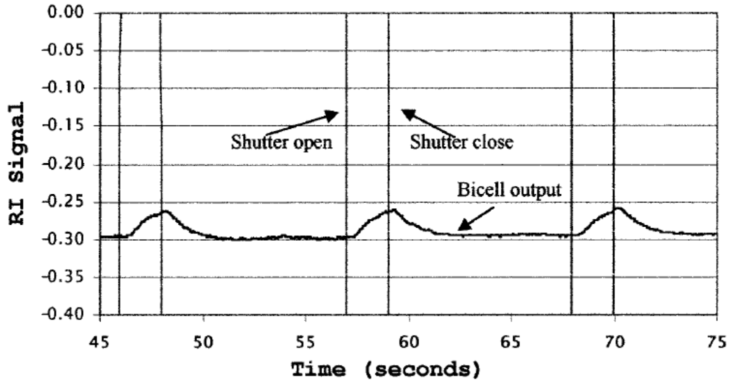
- Micro-interferometric back-scatter and phase detection
- D. A. Markov et al, Nanoliter-scale non-invasive flow-rate quantification using micro-interferometric back-scatter and phase detection, *Fresenius J. Anal Chem*. 2001
- D. A. Markov et al, Noninvasive fluid flow measurements in microfluidic channels with backscatter Interferometry, *Electrophoresis* 2004
- D. A. Markov et al, Label-Free Molecular Interaction Determinations with Nanoscale Interferometry, *J. Am. Chem. Soc*. 2004



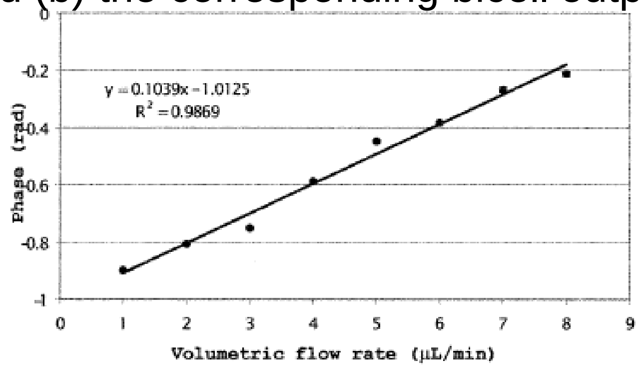
Experimental setup



Fourier spectrum of (a) the shutter control waveform, and (b) the corresponding bicell output



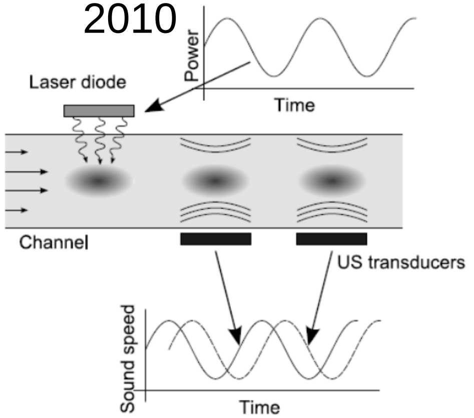
Waveforms of the shutter and bicell output



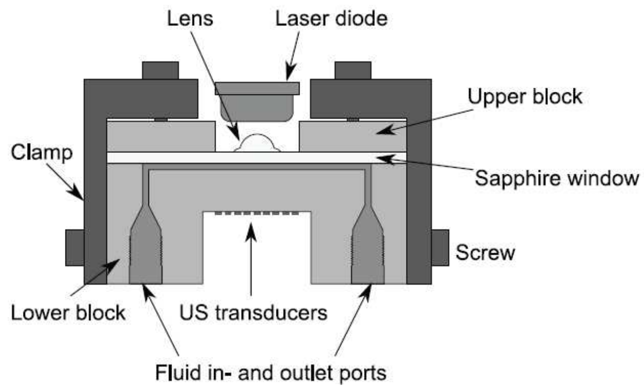
Calibration curve for the flow rate with the heating zone 1 mm away from the detection region

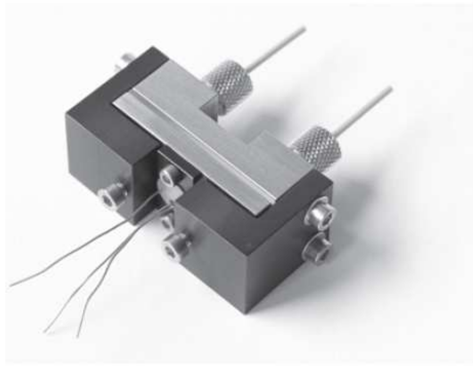
In-situ measurement of micro flowrate without seeding

- Micro-interferometric back-scatter and phase detection
- L. F. G Geers et al, A liquid-independent volume flow measurement principle, MST 2010

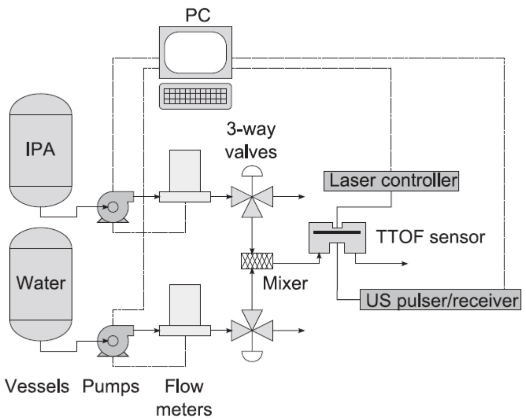


Schematics of principle

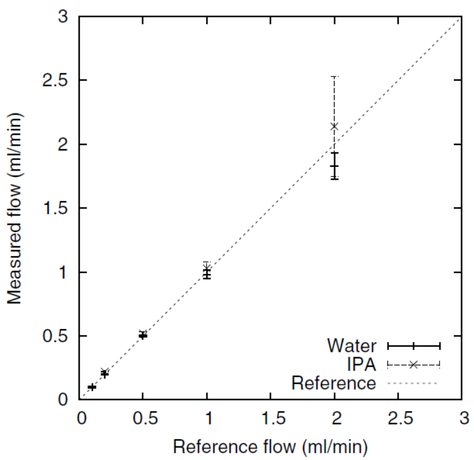




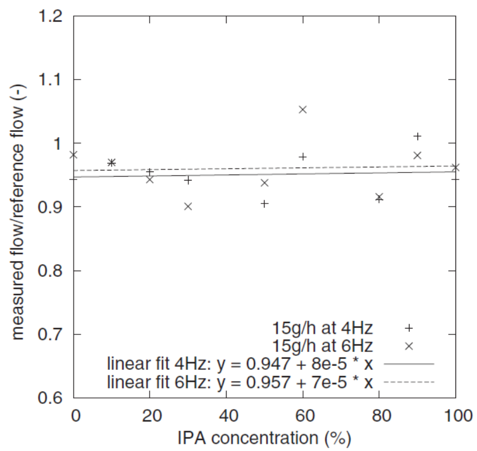
The flow meter(a) Schematics (b) Current unit



Schematic of the test rig



Flow measurement data for pure liquids at different flow rates.



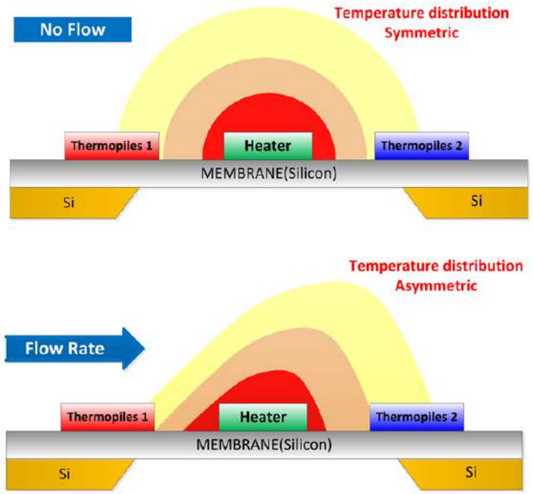
Ratio between measured and reference flow rate for IPA–water mixtures

Thermal mass flow meter with IR absorption

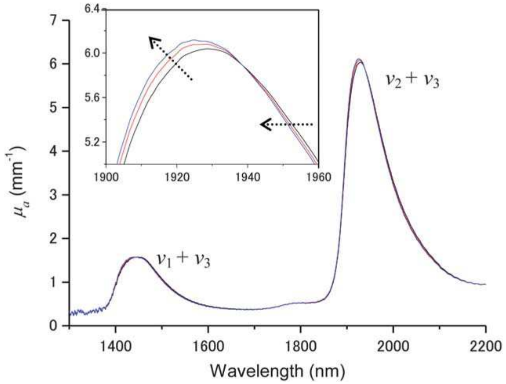
- Thermal mass flow meter
  - Temperature profile change for different flow rate
  - Temperature difference between upstream and downstream positions → obtaining flow rate

- IR absorption and temperature
  - Spectra of IR absorption change for different temperature
  - IR absorption spectra can be used for temperature measurement

How about replacing the heater and thermometer in a thermal mass flow meter using the IR absorption method?



Working principle of thermal mass flowmeter

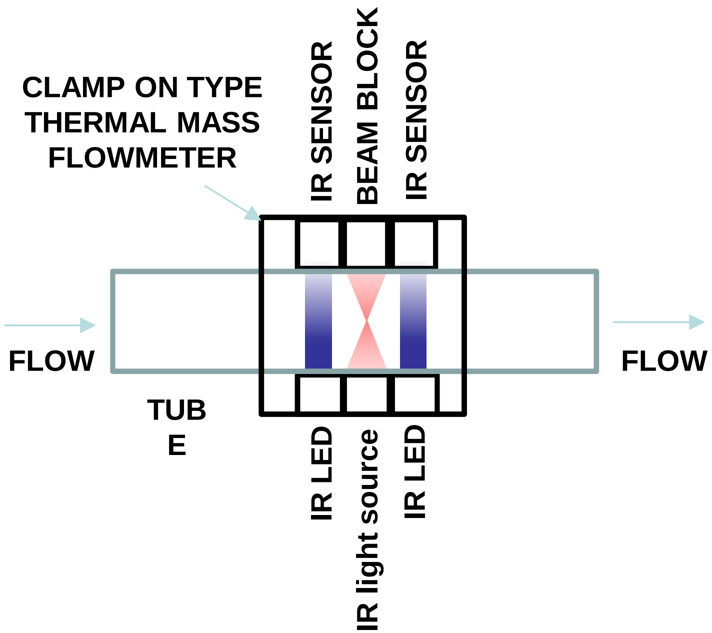


Spectra of the absorption coefficient : 30, 35 and 40 °C (Lab on a chip, 2011)

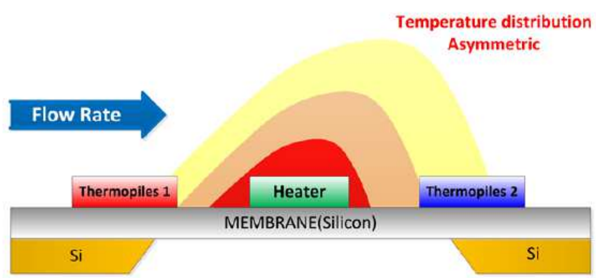
Purpose

- ❑ in-situ measurement of micro flow rate using near infrared absorption method
- \- Measurement of flow temperature inside of tube using IR absorption method
- \- Water heating inside of tube using IR laser
- \- Obtaining flow rate from the IR absorption method in-situ condition

➢ Development of clamp on type of thermal mass flow meter which can measure flow rate in-situ condition



Schematic of clamp on type thermal mass flowmeter



Schematic of conventional thermal mass flowmeter

In-situ measurement of micro flow rate using FTNIR (Fourier Transform Near Infrared Spectroscopy)

(S.H. Lee et al., In situ measurement of micro flow rate using near infrared absorption method, *Optics Express*, Vol. 26, 2018)

Research Article Vol. 26, No. 13 | 25 Jun 2018 | OPTICS EXPRESS 17078

Optics EXPRESS

In situ measurement of micro flow rate using near infrared absorption method

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Experimental setup

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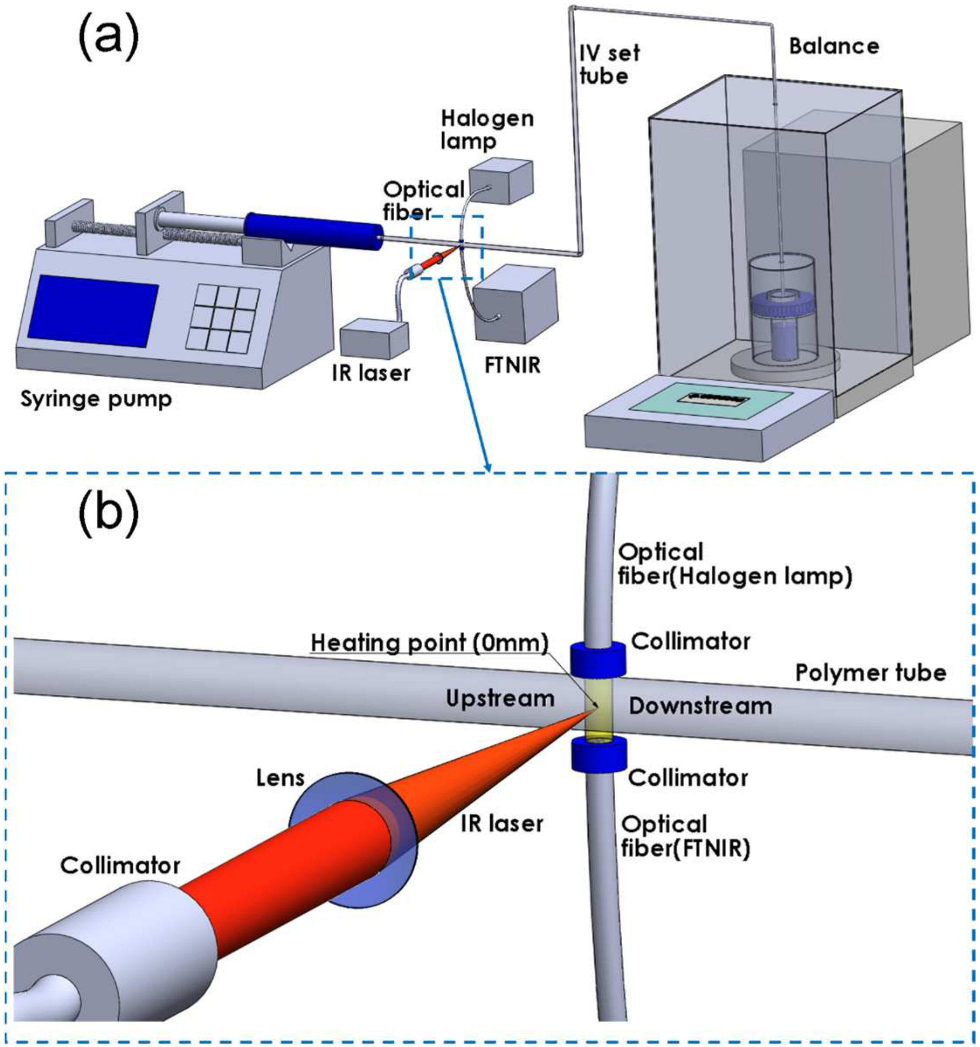
- ❑ Reference flow rate measurement
  - Flow generation : syringe pump (1-100 mL/h)
  - IV-set polymer (3 mm Dia.) tube
  - Balance (Mettler-Toredo, max : 81 g, resolution : 0.005 mg)

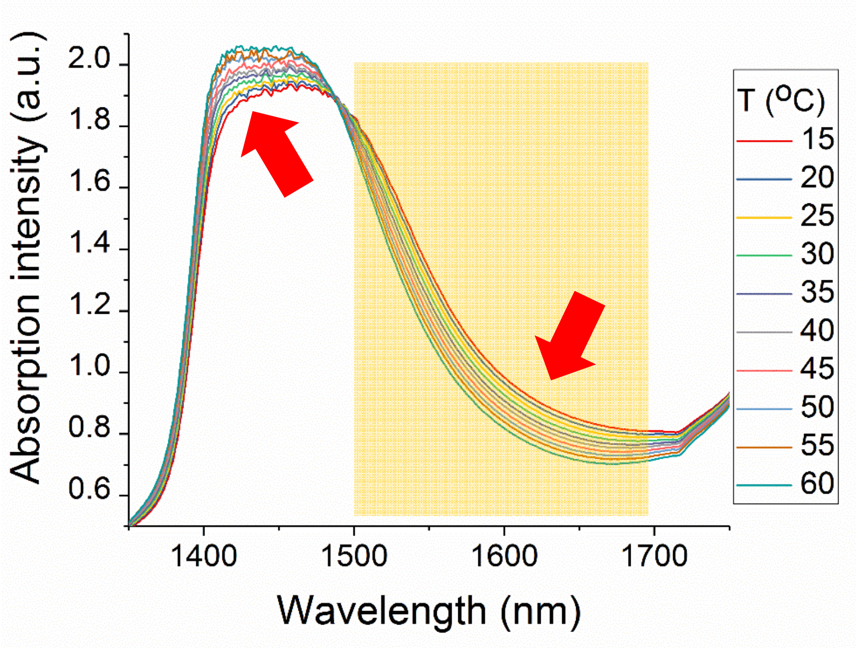
- ❑ IR absorption measurement
  - IR laser heating : 1450 nm wavelength, 500 mW, 50 mm focal length lens
  - FTNIR absorption measurement : 360 nm – 2500 nm wavelength, Halogen lamp
  - Transverse FTNIR measuring position



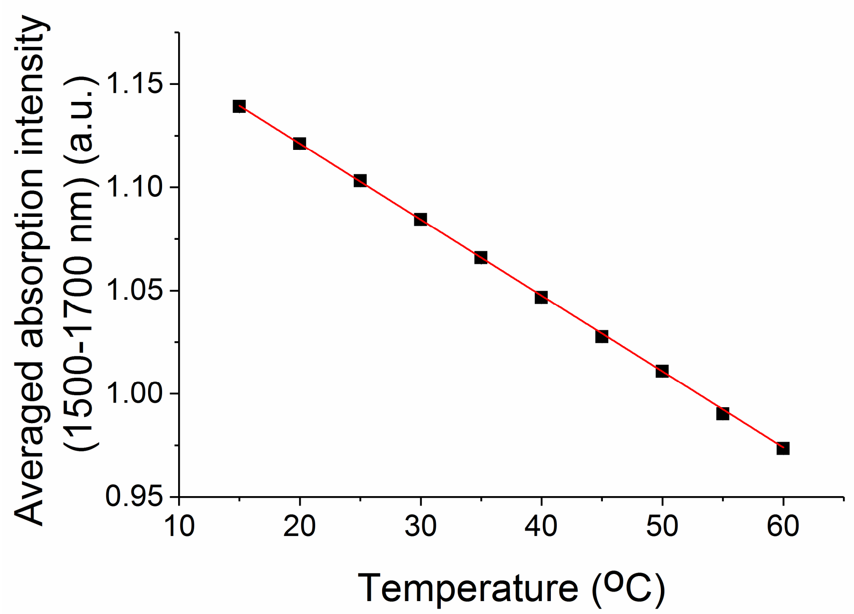
(a) Experiment setup (b) magnification of measuring part

Temperature measurement using FTNIR

- Reference temperature measurement
  - Control temperature using heating circulator (10-60 °C)
  - Obtaining reference temperature : PT 100 sensor
  - Absorption spectra change for different reference temperature
  - Linear calibration curve between ref. temperature and averaged absorption signal (1500 – 1700 nm)



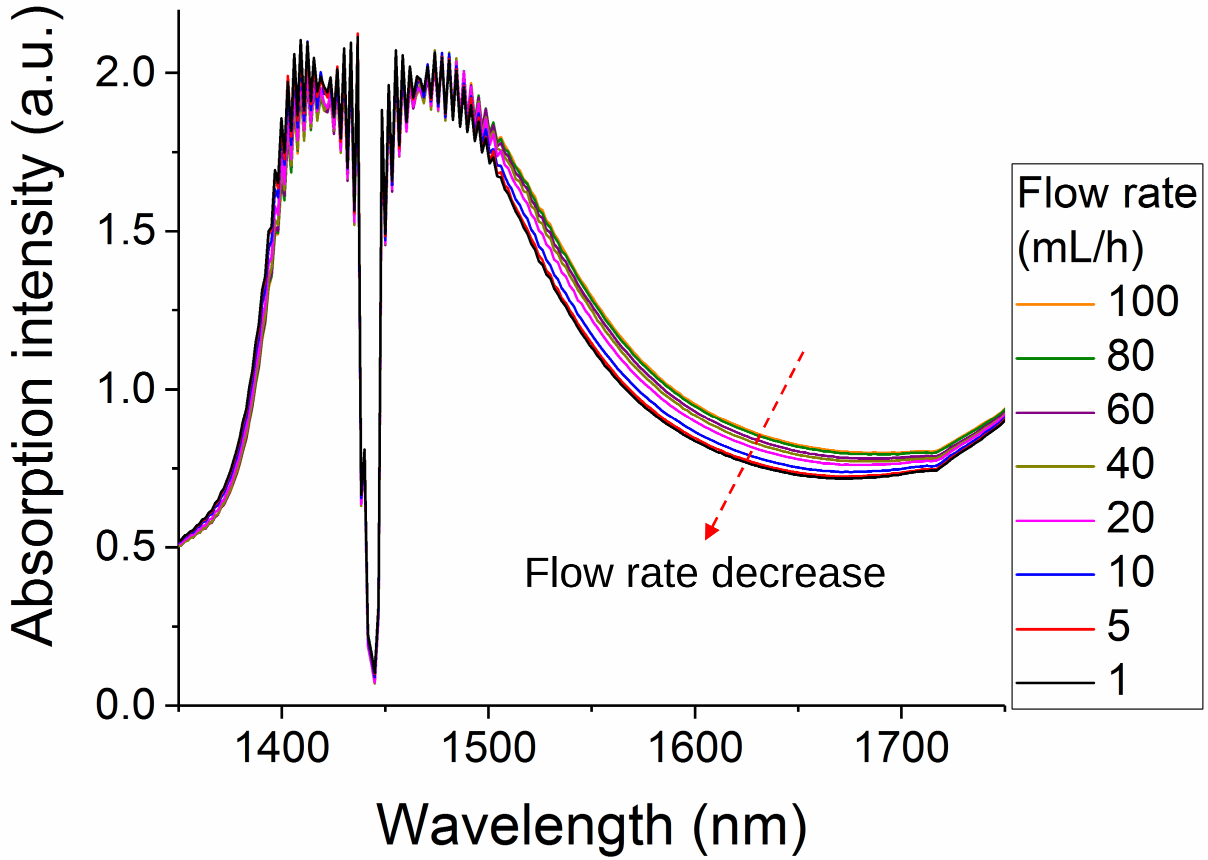
Absorption spectra for different temperature



Absorption intensity according to temperature

Flow rate change with laser heating

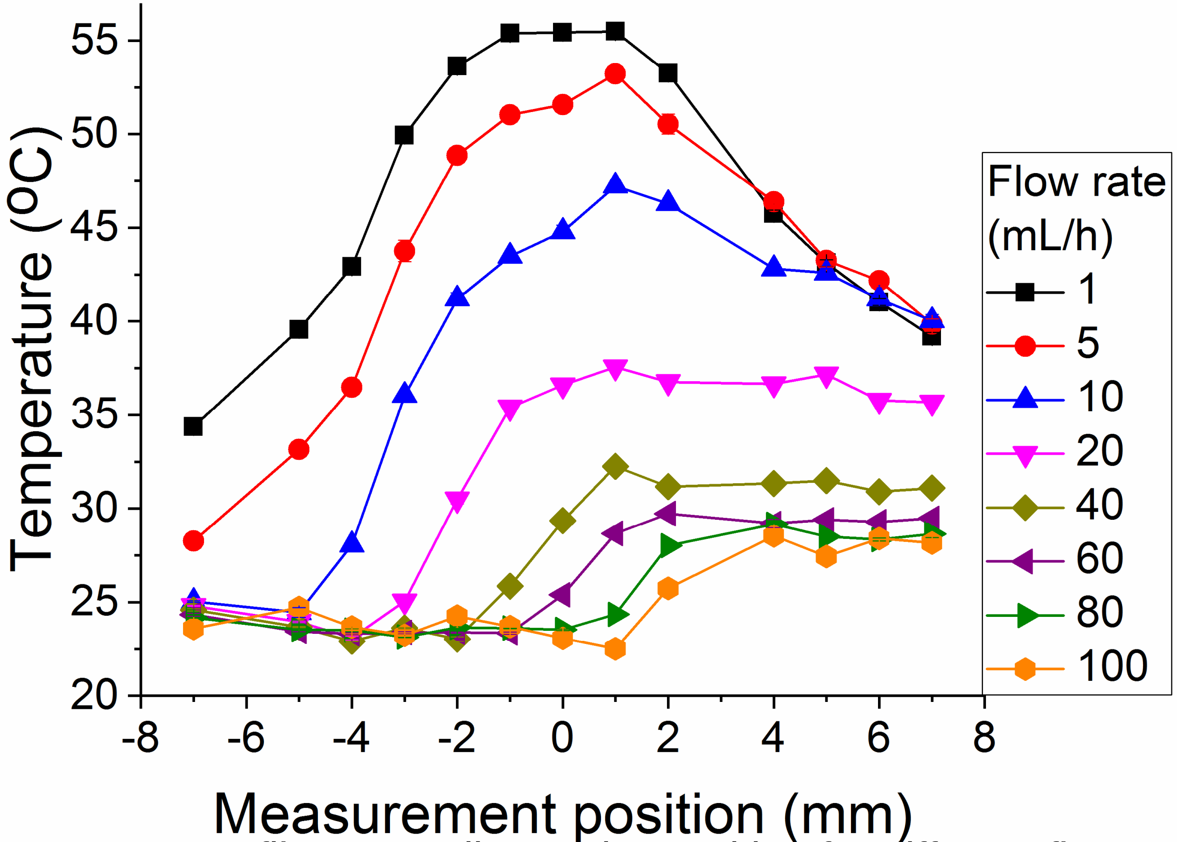
- ❑ Flow rate and FTNIR absorption spectrum
- \- Change of absorption spectrum with change of flow rate
- \- The temperature increases as the flow rate decreases



Absorption spectra for different flow rate at the 1 mm position

Temperature profile with laser heating

- ❑ Temperature profile for different flow rate and positions
- Quantitative temperature obtained from calibration curve
- Temperature profile shift to downstream with increasing flow rate
- Above 20 mL / h, the difference in temperature profile is small



Temperature profiles according to the position for different flow rates

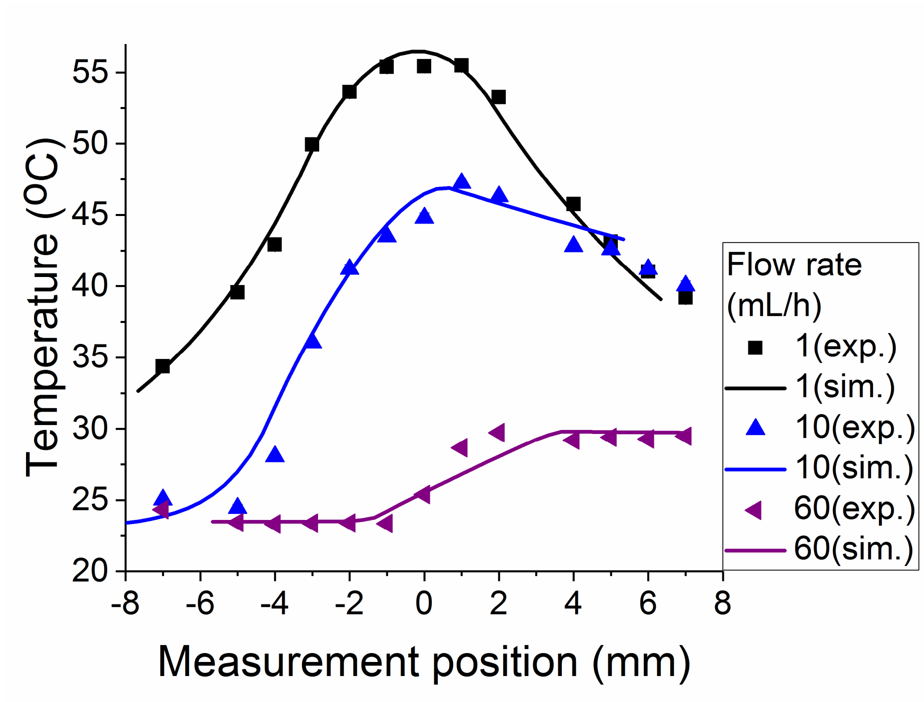
- Comparison between exp. and sim. results
  - Conductive and convective heat transfer
  - Good agreement of experimental and simulation results
  - More dominant convection with increasing flowrate

Governing equations

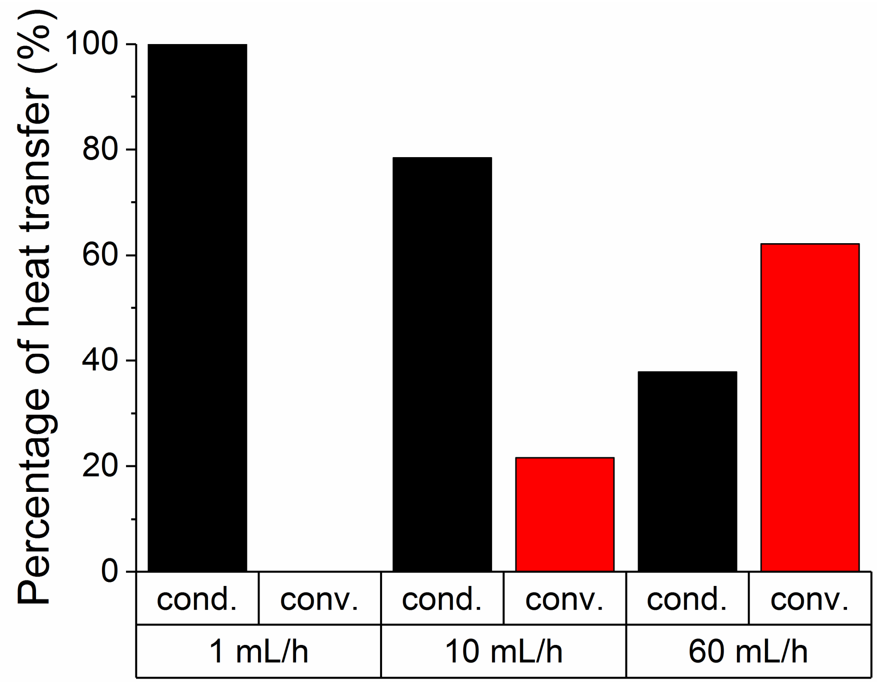
$$\dot{q}_{laser} + \dot{q}_{conv,i-1\rightarrow i} = \dot{q}_{cond,i\rightarrow i-1} + \dot{q}_{cond,i\rightarrow i+1} + \dot{q}_{loss}$$

$$\dot{q}_{laser} + \dot{m}C_p(T_i - T_{i-1}) = KA\frac{T_i - T_{i-1}}{\Delta x} + KA\frac{T_i - T_{i+1}}{\Delta x} + hP\Delta x(T_i - T_\infty)$$

$$T_{i=1} = T_\infty,\ T'_{t=N} = 0$$



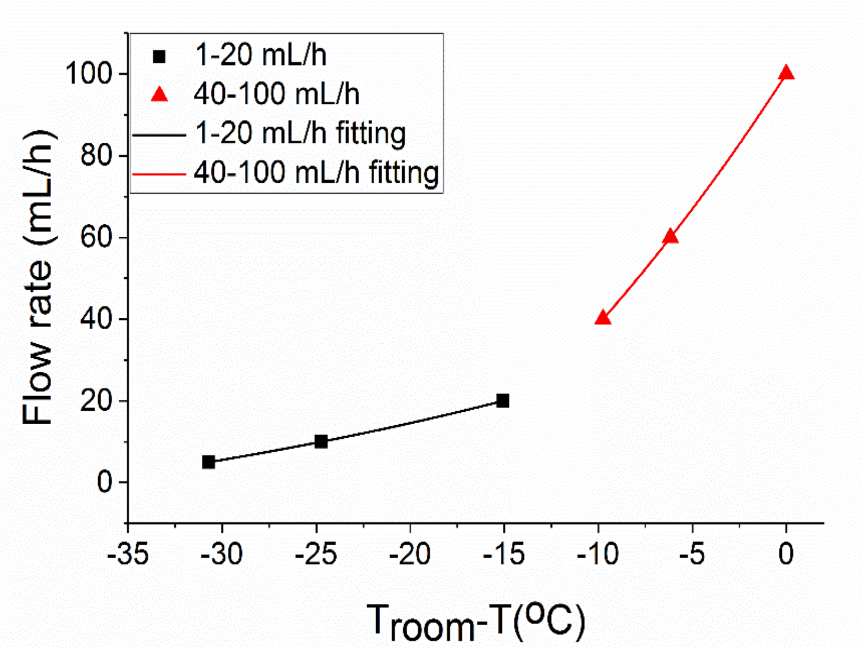
Comparison of temperature profiles between the experimental and simulation results



Calculated percentage of heat transfer (conduction and convection)

Quantitative flow rate measurement using NIR method

- Calibration curve between temperature difference(ΔT) and flow rate
  - Two exponential curves based on the flow rate ranges (1-20 mL/h and 40-100 mL/h)
  - The two flow rate ranges (1-20 mL/h and 40 mL/h) are significantly influenced by conduction and convection, respectively
  - We measure the quantitative flow rate by using the calibration curve.

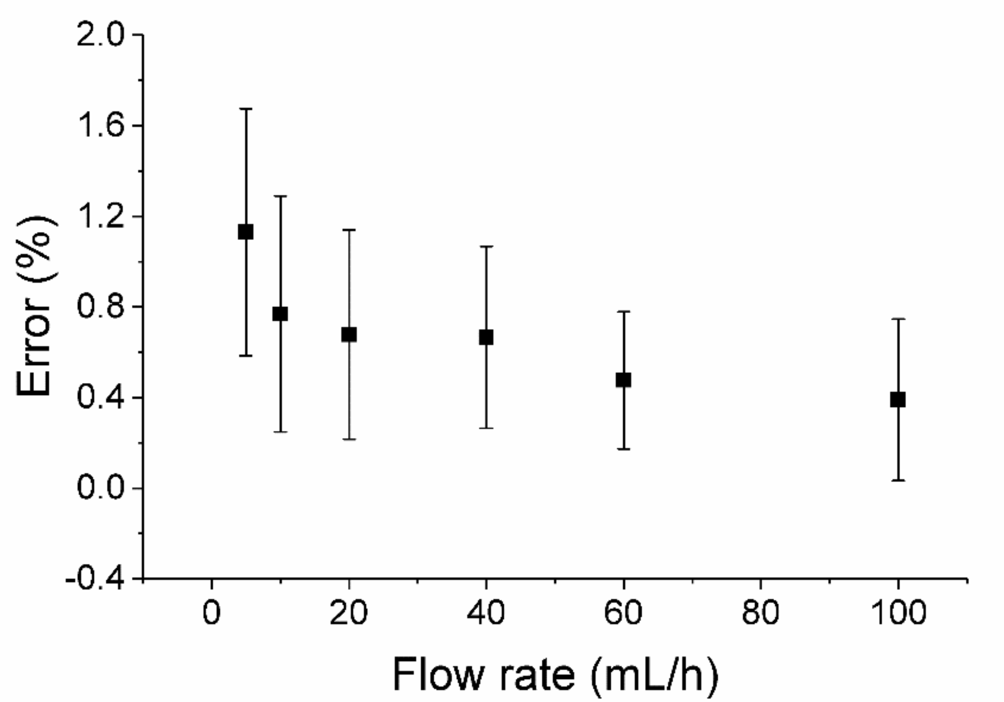


Flow rate according to the difference of room temperature (Troom, 22.5 oC) and temperature(T) at 1 mm position with exponential curve fitting for different flow rate ranges (1-20 mL/h, 40-100 mL/h)

Quantitative flow rate measurement using NIR method

- Error of NIR absorption method according to the flow rate with measurement uncertainty
- The reference flow rate was obtained from the gravimetric flow standard system in KRISS
- The error according to the flow rate is less than 1.2 % (0.05 mL/h) and the uncertainty is less than 1 % (0.39 mL/h) at the 1-100 mL/h flow rate

$$Error = \frac{(q_{NIR} - q_{REF})}{q_{REF}} \times 100$$



Error of NIR absorption method according to the flow rate with measurement uncertainty

Conclusions (FTNIR)

- In-situ measurement of micro flow rates was conducted using the NIR absorption method.
- We used the IR absorption method to measure the temperature under in-situ conditions instead of using a contact temperature sensor.
- The temperature profiles were obtained by the NIR absorption method via laser heating for different flow rates.
- The shape of the temperature profile changes resulting from conduction and convection heat transfer were presented based on the range of flow rates.
- A simulation was conducted considering conductive and convective heat transfer for analysis of temperature profiles.
- The calibration curves between the flow rate and the difference in room temperature (Troom, 22.5 °C) and the temperature (T) obtained from the NIR absorption method exhibit two exponential curves based on the flow rate ranges (1–20 mL/h and 40–100 mL/h).
- The error and the uncertainty of the NIR absorption method were approximately 1.2 % and 1% in the 1–100 mL/min flow rate range, respectively.
- Thus, we confirmed for the first time that the NIR absorption method quantitatively measures the flow rate in the in-situ condition.

In-situ measurement of micro flow rate using laser diode

(S.H. Lee et al., Practical methodology for in situ measurement of micro flow rates using laser diode absorption sensors, *Metrologia*, In press)

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Practical methodology for in situ measurement of micro flow rates using laser diode absorption sensors

Lee Seok Hwan[1], Seongchong Park[2], Joohyun Lee[2] and Woong Kang[2]

Accepted Manuscript online 19 June 2019 • © 2019 BIPM & IOP Publishing Ltd

Laser diode based flow measurement

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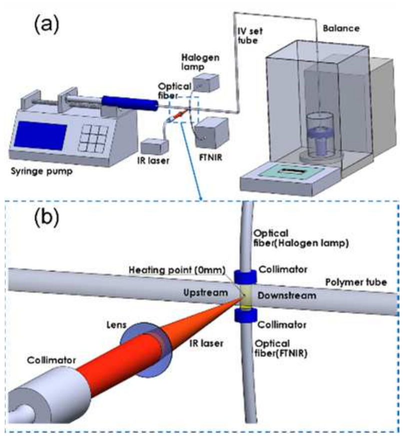
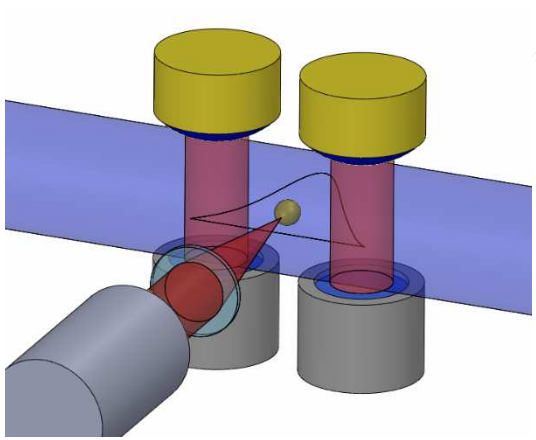
❑ In-situ measurement of micro flow rate using IR diode

- Portable instrument for micro flow rate measurement
- IR diode($10) is much cheaper than FTNIR($40,000)
- Compact size(less than 5 mm)
- Accurate measurement of flow rate using temperature at up and downstream





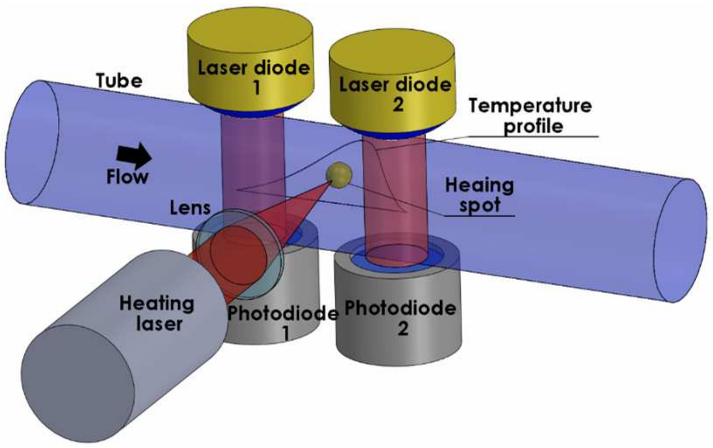
Measurement setup using FTNIR ($40,000)

Measurement setup using IR diode ($10)

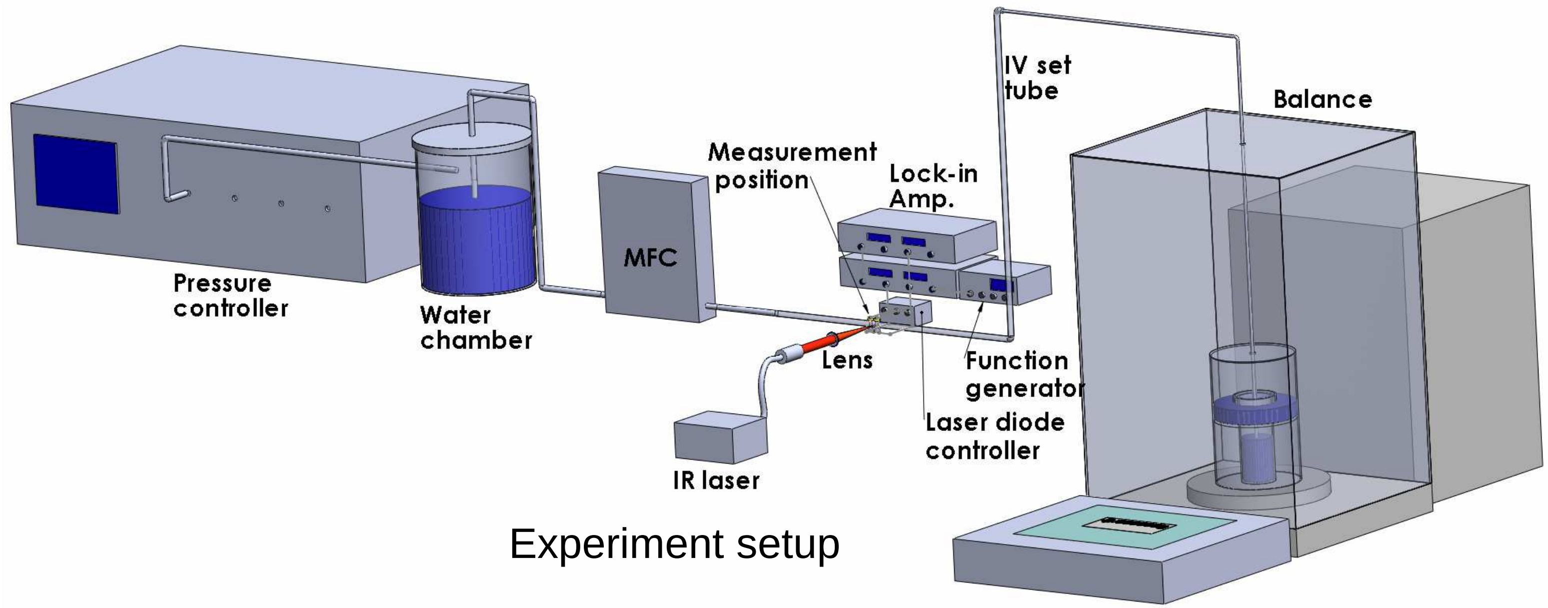
Experimental setup

- ❑ Reference flow rate measurement
- Flow generation : Coriolis MFC
- IV-set polymer (3 mm Dia.) tube
- Balance (Mettler-Toredo, max : 81 g, resolution : 0.005 mg)
- Laser diode : 1550–1650 nm, 2 mW, d=5 mm
- Lock-in Amp. : to enhance S/N
- Heating laser : 1450 nm, 500 mW

Laser diode

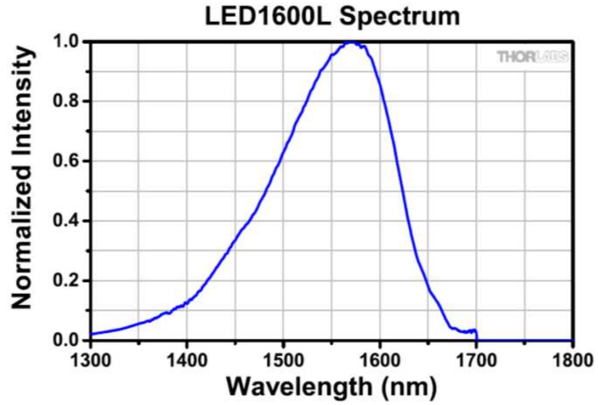


Magnification of measuring part

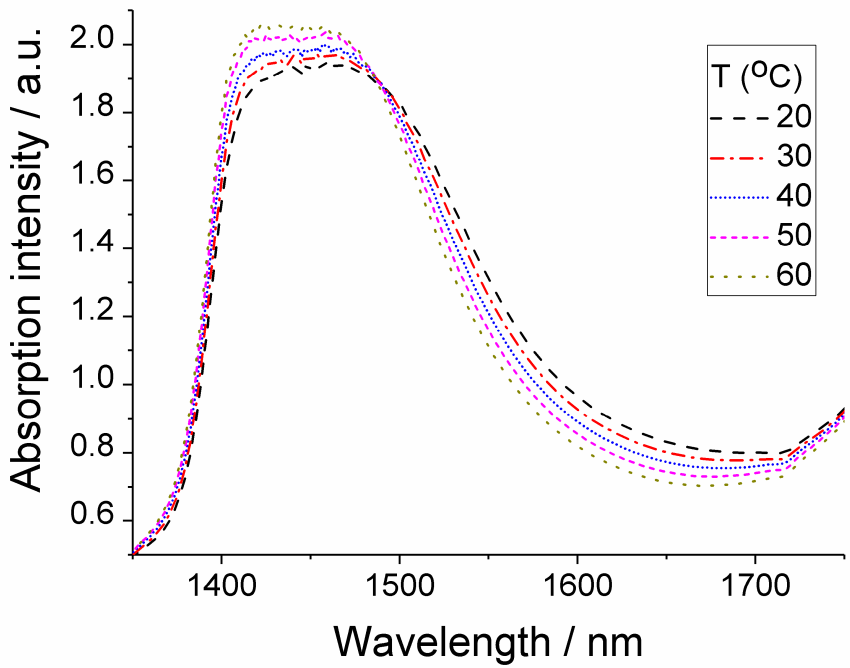


Experiment setup

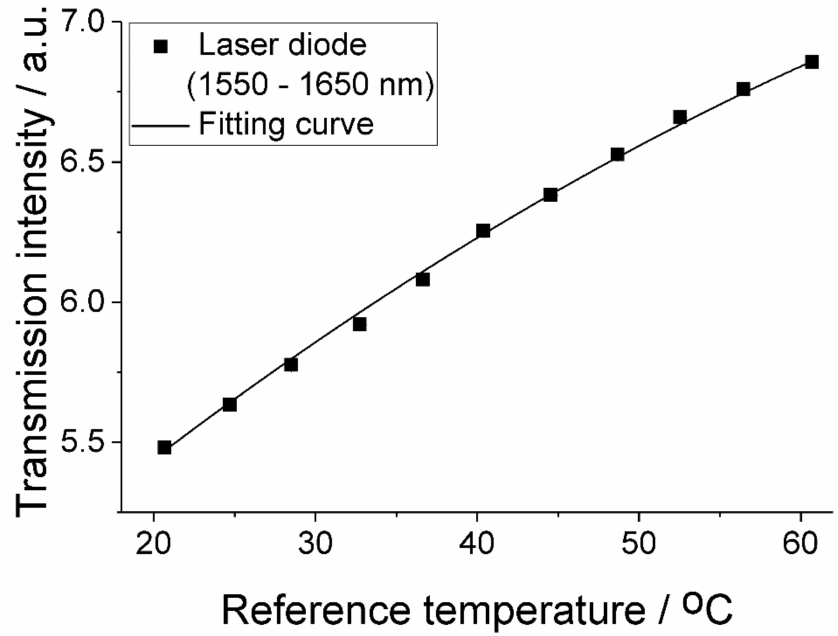
- Temperature measurement using laser diode
- Light from 1550 to 1650 nm with a laser diode
- Calibration using temperature controlled bath
- Uncertainty of temperature measurement : 0.001 %



Spectrum of laser diode

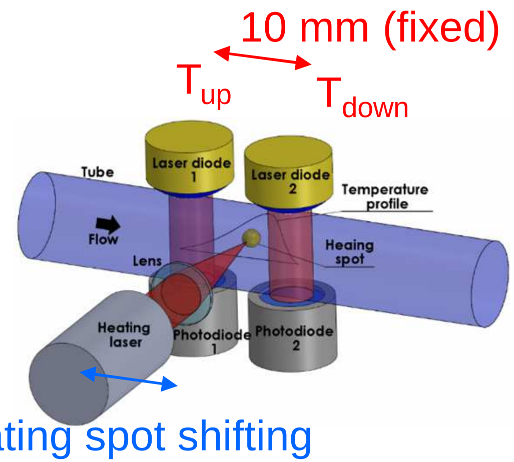


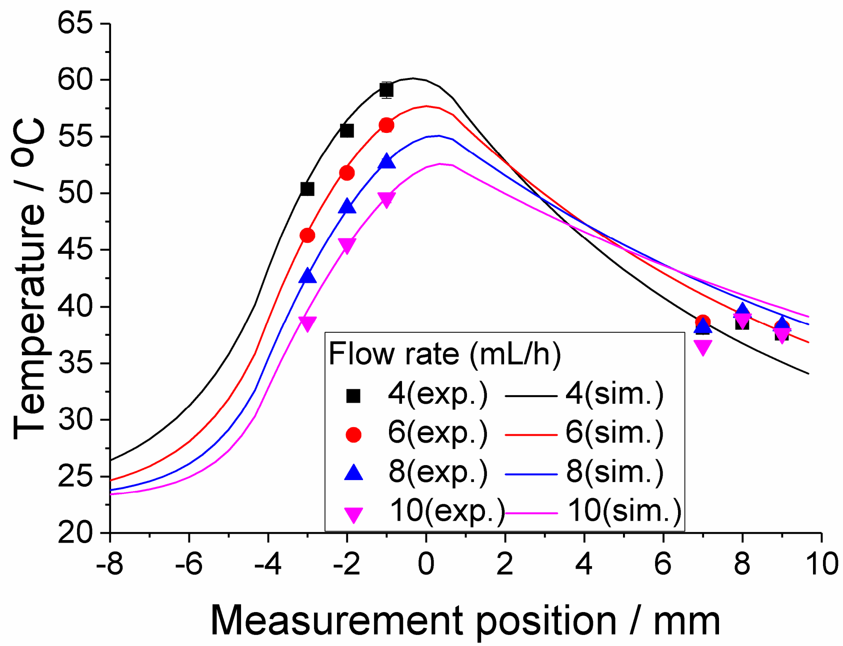
Absorption spectra for different temperature



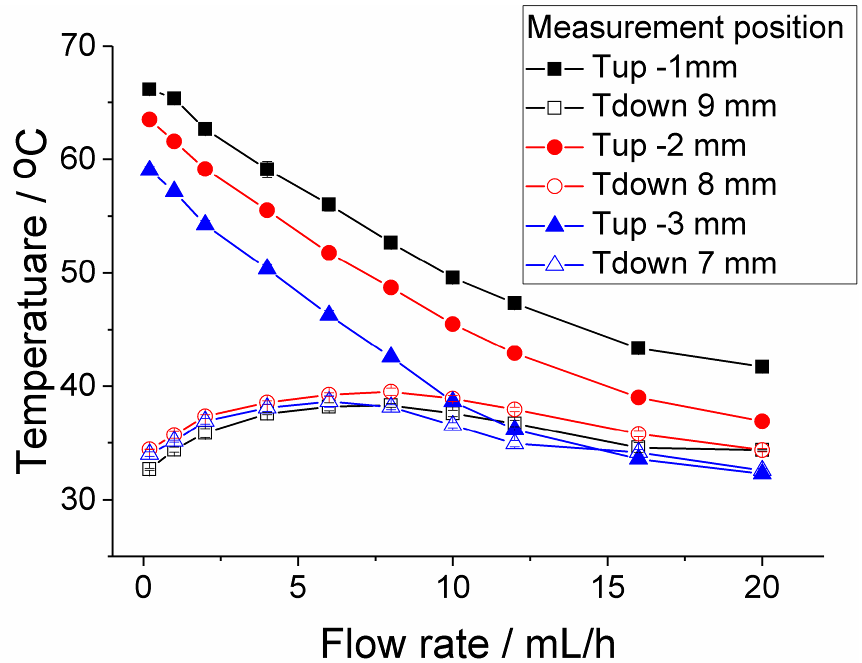
Transmission intensity according to temperature for laser diode based temperature measurement

- Different measurement positions
- A heated spot created with a heating laser
- Changing the position of heating spot
- Increase of flow reduced the temperature of the upstream
- Temperature change not large with flow rate in downstream





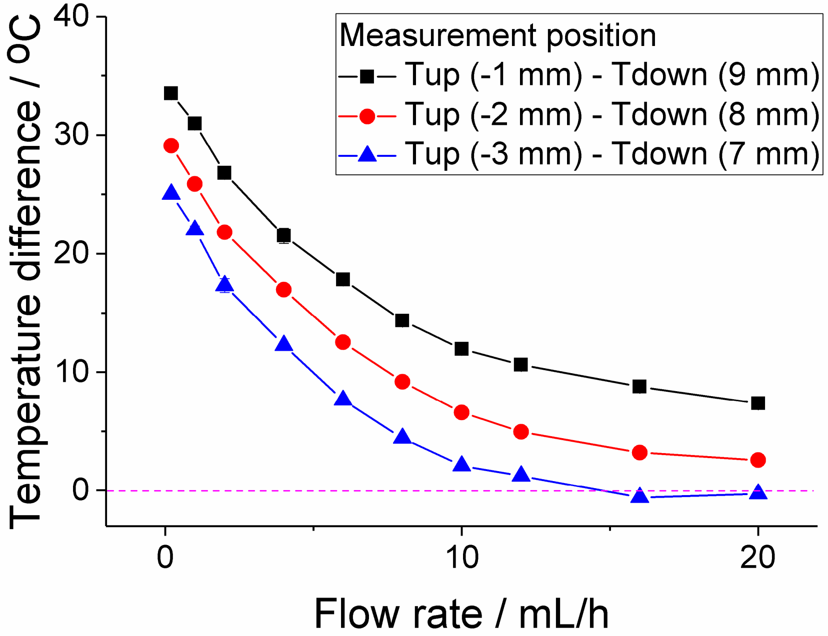
Comparison of temperature profiles



Temperatures measured for different measurement positions

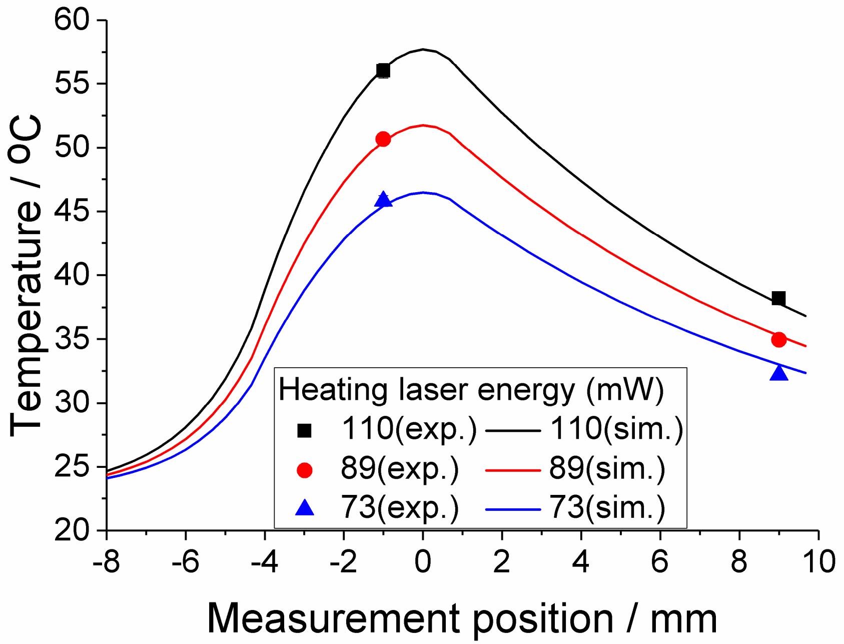
Temperature difference with flow rate

- ❑ Different measurement position
- Temperature difference decreases exponentially
- When the measurement position is moved from -1 mm (upstream) and 9 mm (downstream) to -3 mm (upstream) and 7 mm (downstream), the curve shifts downward with almost the same slope.
- Where the upstream measurement position is close to the laser heating position, we can measure a wider range of flow rates.

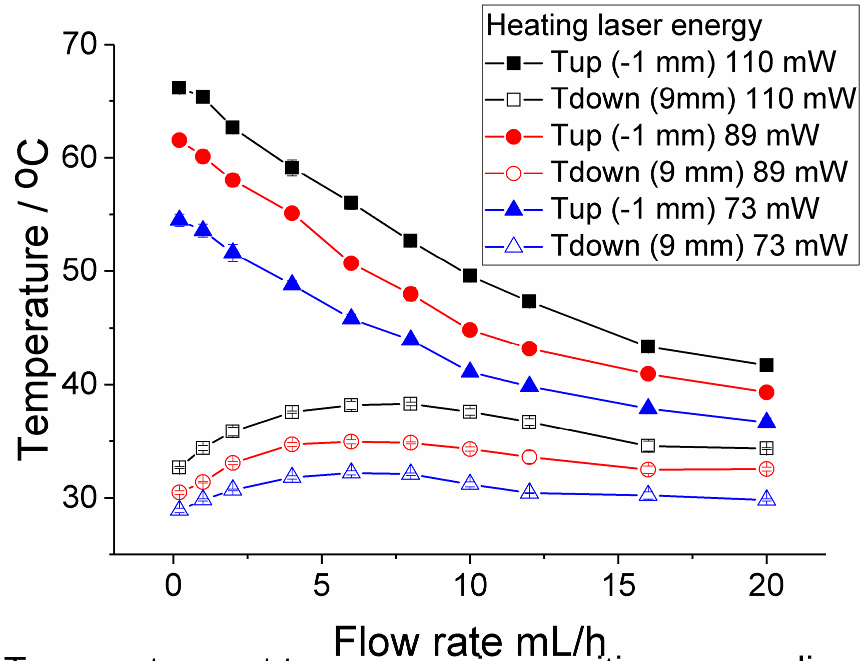


Temperature difference measured upstream and downstream

- Different heating laser energies at the fixed measurement position (-1,9)
  - When the heating laser energy increases, the maximum temperature increases
  - When the flow rate increases, the temperature upstream of the heated spot decreases, and there is little change in the temperature downstream.
  - As the heating laser energy decreases, the temperature curve according to the flow rate at each position is shifted downward.



Comparison of temperature profiles for different heating laser energies



Temperatures at two measuring positions according to the flow rates for different heating laser energies

Temperature difference for different heating energies

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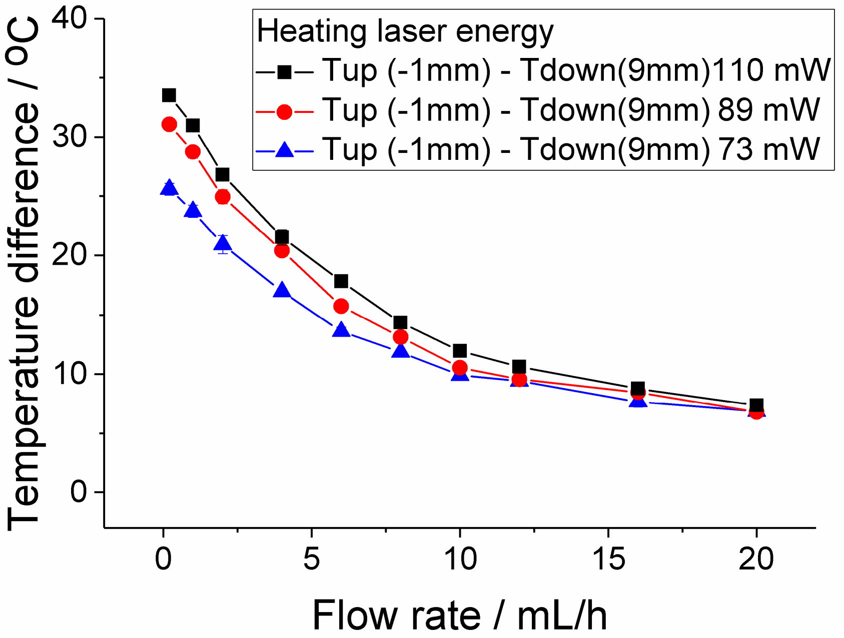
- Different heating laser energies
  - The heating laser energy decreases, the slope of the temperature curve decreases.
  - The higher the heating laser energy, the better the accuracy of the flow measurement.
  - The lowest flow rate measured in this study is 0.2 ml/h, but if the laser energy is controlled, lower flow rates can be measured in the future.



Temperatures difference according to the flow rates for different heating laser energies

Uncertainty analysis

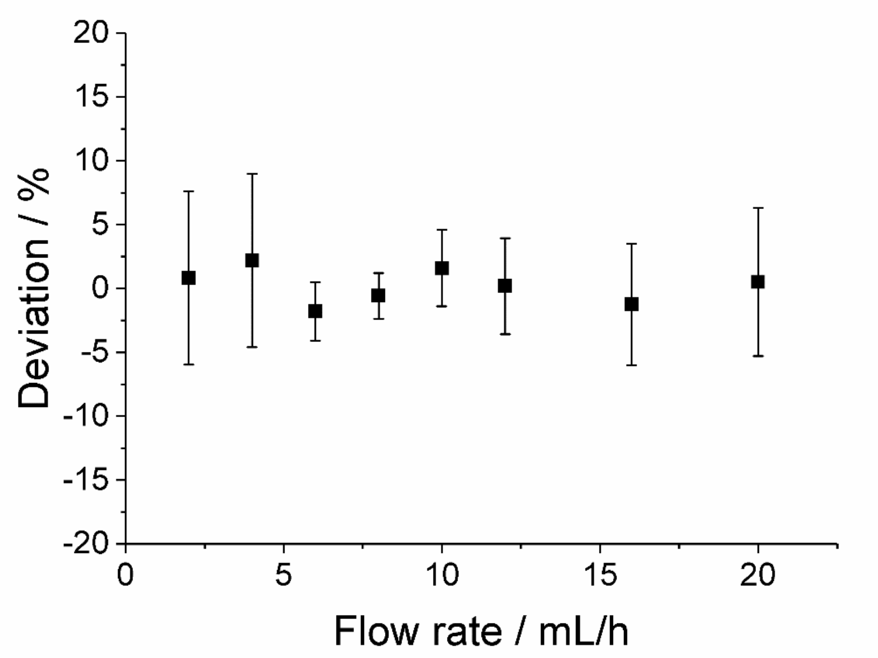
- Mathematical model is based on the comparison calibration $E = \frac{\dot{m}_{DUT} - \dot{m}_{REF}}{\dot{m}_{REF}}$
- The relative uncertainty of E

$$u(E) = \sqrt{\begin{array}{c} u(E_A)^2 + (c_{rW} u_r(\delta W))^2 + (c_{rt} u_r(\delta t))^2 + (c_{r\varepsilon} u_r(\delta \varepsilon))^2 \\ + \left(c_{r\rho_{DUT}} u_r(\delta \rho_{DUT})\right)^2 + \left(c_{rq_{DUT}} u_r(\delta q_{DUT})\right)^2 \end{array}}$$

Quantity (Xi)	Uncertainty factor	Value of uncertainty	$C_i = \frac{\partial f}{\partial x_i}$	Uncertainty contribution (k = 2)	Degree of freedom
Mass	u(δW)	5.17E-04	1.96E-04	1.03E-04	289
Time	u(δt)	9.80E-04	3.27E-07	1.09E-06	210
Density	u(δρ)	3.75E-03	9.83E-07	3.75E-06	107
Indicator	$u(\delta q_{DUT})$	3.46E-04	1.00E+00	3.46E-02	354
Thermometers	u(δT)	3.33E-05	1.00E+00	1.67E-06	1140
Temperature curve fitting	u(δTcurve)	1.30E-03	1.00E+00	2.17E-04	183
Buoyancy correction	u(δε)	2.89E-06	-9.80E-04	2.88E-06	3873
Relative error	$u(E_A)$	2.90E+00	1.00E+00	1.94E-01	4
				$u_c(E)$=1.14 % U(E) = 2.28% $\nu_{eff}(E)$=4853 k=2.0	

Main sources of uncertainty for the laser diode flowmeter calibration for 6 ml/h flow rate

- Deviations in the LD flowmeter readings according to the flow rate with measurement uncertainty
- The maximum measurement uncertainty was 6.8% at a 1 ml/h flow rate, and the minimum measurement uncertainty was 1.78% at 8 ml/h.
- The performance of the LD flowmeter developed in this study was confirmed to be close to that of a commercial thermal mass flowmeter (10%, full scale).



Deviations in the LD flowmeter readings according to the flow rate with measurement uncertainty

Conclusions

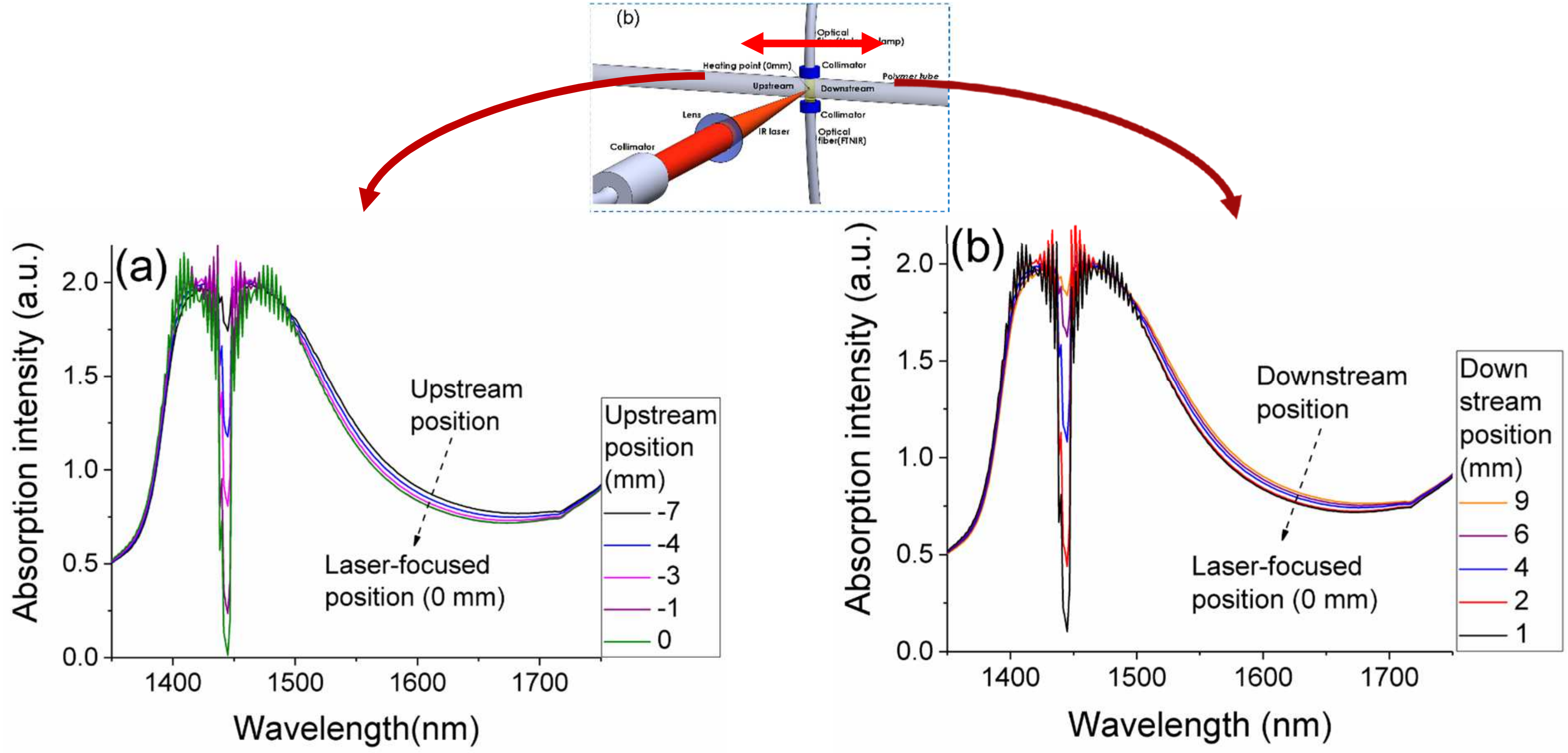
- A laser diode-based flowmeter based on this infrared absorption method that can measure in-situ micro flow rates from 0.2 to 20 ml/min using a simple diode laser and a photo detector was developed.
- We measured the temperature profiles of the flow rate by changing the temperature measurement position and the heating laser energy upstream and downstream of the heated spot, and compared the obtained values with the simulation results.
- The flow measurement range was adjusted according to the temperature measurement position. Increasing the energy of the heating laser also improved the measurement accuracy in the lower flow range.
- The developed flowmeter was calibrated by the gravimetric method, and the maximum measurement uncertainty was 6.8%, which is similar to that the conventional commercial contact thermal mass flowmeter. The lowest flow measurement uncertainty was 1.78% at a flow rate of 8 ml/h.
- Thus, it was confirmed that the flow rate can be measured through the temperature difference gauged using a simple diode laser set.

표준이 올라가면 생활이 즐거워 집니다!

Leading Group in Fluid Flow and Acoustics Metrology

Thank you for your attention

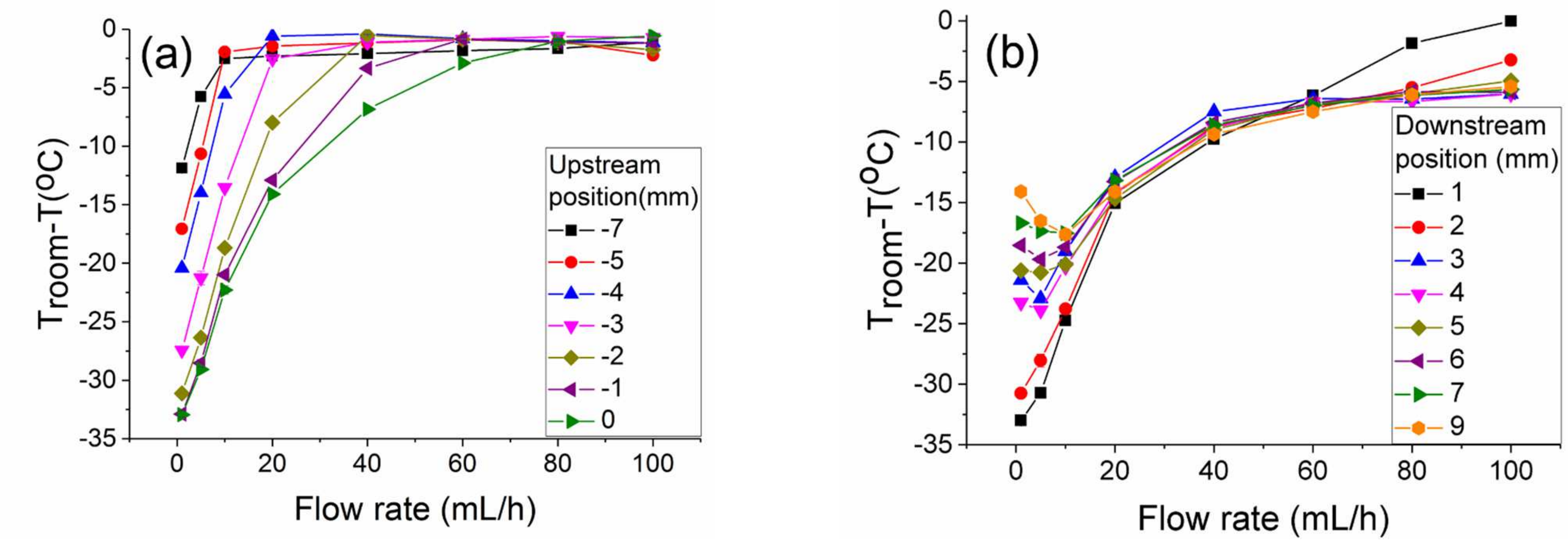
- NIR absorption heating
  - 1450 nm : absorption of water(O-H band), transmission of polymer tube
  - Focusing laser : tens of μm hot spot generation
  - Spectral change in the range of 1500-1700 nm according to laser heating
  - Temperature increase more and more near hot spot



Absorption spectra at upstream positions

Absorption spectra at downstream positions

- Temperature difference(ΔT) between measuring position and room temperature for varied flow rate
- Varied ΔT according to the measuring position in up and downstream
- Linearly increase of ΔT with increasing flow rate up to 20 mL/h @ -1 mm
  : conduction is dominant effect
- Gradually decreasing the slope of ΔT from 20 mL/h to 100 mL/h @ -1 mm
  : influence of convection increases



Difference between room temperature (Troom, 22.5 °C) and temperature (T) according to the flow rate for different positions (a) upstream and (b) downstream with IR laser heating at the 0 mm position