

A microchannel concentrator controlled by integral thermoresponsive valves[☆]

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Abstract

A strategy for fabricating a microchannel concentrator having a new type of thermoresponsive valves for the fluid flow switching is described. The function of the valves is based on an actuating flap structure, where the flap is composed of a double-layered sheet, a thermoresponsive hydrogel layer and an acrylic resin layer. Responding to the water swelling/deswelling behavior of the hydrogel layer, the flap bidirectionally bends within several seconds. Likewise, in association with a special valve-sealing concept, two valves showing opposite working (open/close) states were successfully created in a microchip. Consequently, when a microchannel filled with silica gel particles was prepared with two outlet branches having the opposite-working valves, a four-fold enrichment of an analyte was achieved by the single operation of the present concentrator system. © 2007 Elsevier B.V. All rights reserved.

Keywords: Microfluidic device; Thermoresponsive valve; Hydrogel; Concentrator; Adsorption

1. Introduction

Sensitivity of chemical analysis systems depends most of the time on the obtainable detection limit. Concentrating the analyte prior to applying the detection system generally makes the analysis easier [1]. In miniaturized systems, this concept is also true, and fabricating a microscale concentrator is currently a challenging issue aiming to develop the μ TAS technology.

In the past decade, microfluidic device, which is recognized as a microchannel network on/in a small plate, has been broadly used in the biology field such as immunoassays [2,3] and cell manipulations [4,5], and in the material and chemical engineering field such as size-controlled particle production and transformations [6,7]. Microfluidic devices have advantages over the macroscale analogues, which include, for example, less reagent consumption, quick response time, and high throughput [8]. These features are mainly resulted from their large specific

interfacial area, short molecular diffusion distance, and small heat capacity. Recently, microfluidic devices applied for the microscale concentrator were demonstrated, in which the solute concentration depends on the evaporation of solvent [1,9]. The device function was performed by the passage of a gas over a porous membrane behind which the solution flows. We report herein an alternative type of microchannel concentrator, achieving efficient concentration by means of analyte adsorption to and desorption from adsorbents, in association with an effective fluid flow control.

To control the fluid flow, we focused upon thermoresponsive valves for integration in the microchannel concentrator. Stimuli-responsive polymer gels drastically change their solvent swelling/deswelling nature, in response to small changes in the environment such as temperature [10], pH [11], electric field [12], and light field [13–15], etc. Recently, some approaches to microvalves based on this class of polymer gels have been reported. For instance, Beebe et al. [16] prepared a microvalve with a pH-sensitive hydrogel jacket around a post, while Fréchet and coworkers [17,18] prepared monolithic valves for microfluidic chips based on thermoresponsive polymer gels. However, these valves in the precedents cannot achieve opposite working states at a specific environment condition, because they depend on only the simple swelling/deswelling performance of the hydrogels.

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In the current study, we propose a novel *bidirectional-actuating* flap comprising two different polymer layers bonded together; one is a poly(*N*-isopropylacrylamide) (PNIPAAm) hydrogel layer and the other is an acrylic resin layer. PNIPAAm is probably the best-known material of a class of thermoresponsive polymers characterized by a lower critical solution temperature (LCST) of 32 °C [19–23]. Therefore, the PNIPAAm hydrogel layer of the flap shrinks at a temperature above the LCST while swells below the LCST. However, the acrylic resin almost keeps its dimension at all times. Consequently, the flap bends toward opposite directions as a bimetal-like actuator does when the local environmental temperature changes. Based on the bending deformation of the flaps combined with a special channel design, two thermally actuated valves in the chip can achieve opposite working (open/close) states at a certain local environmental temperature.

2. Experimental

2.1. Materials

N-isopropylacrylamide (NIPAAm), α -methylstyrene dimer (retarder, α -MSD), and silica gel (25 μm in diameter, Wakosil 25SIL) were obtained from Wako Chemicals. Poly(vinyl pyrrolidone) (PVP; M_w , 4.0×10^4) and Rhodamine 6G (R6G) were purchased from Tokyo Kasei Kogyo. 1,6-Hexanediol diacrylate (HDDA) and poly(ethyleneglycol) 16-nonylphenylether acrylate (N-177E) were supplied by Dai-ichi Kogyo Seiyaku. A moderate molecular weight, urethane linkage-containing triacrylate monomer (V4263; M_n , ca. 2.0×10^3 , Dainippon Ink and Chemicals), polyvinyl-polypyrrolidone (PVPP; Nacalai Tesque), *N,N*-dimethylacetamide (DMAc, Sigma–Aldrich), and

1-hydroxy cyclohexylphenyl ketone (photoinitiator, trade name Irgacure184, Ciba Geigy) were used as received.

2.2. Chip preparation

The PVP/PVPP modified hydrogel precursor was a mixture of NIPAAm/V4263/PVP/PVPP/DMAc/Irgacure184/ α -MSD (95/5/12.5/75/131/5/0.5 in weight ratio). The conventional hydrogel precursor for comparison was a mixture of NIPAAm/V4263/DMAc/Irgacure184/ α -MSD (95/5/50/5/0.5 in weight ratio). The acrylic resin layer of the flap was made by a mixture (ARP1) of V4263/N-177E/Irgacure184/ α -MSD (80/20/5/0.5 in weight ratio), while all other frameworks of the chips were prepared by a mixture (ARP2) of V4263/HDDA/Irgacure184/ α -MSD (60/40/5/0.5 in weight ratio).

The preparation of the microchannel concentrator was schematically shown in Fig. 1 and Fig. S-1. To fabricate the acrylic resin layer (L2-1) and the gel layer frame (L2-2), the monomer mixtures, ARP1 and ARP2, were separately spin-coated on oriented polypropylene (OPP) films and partially UV-cured through the photomasks, and then washed with ethanol to remove unpolymerized components. After laminating the two partially cured, adhesive layers, the OPP film in contact with the gel layer frame was removed leaving a chamber which was then filled with a drop of the hydrogel precursor. Excessive mixture was pressed out by covering on the chamber with an OPP film, and then the hydrogel precursor was photopolymerized through the identical photomask as L2-1. Finally, the flap layer (L2) was obtained after washing out unreacted hydrogel precursor and PVP with ethanol and deionized water.

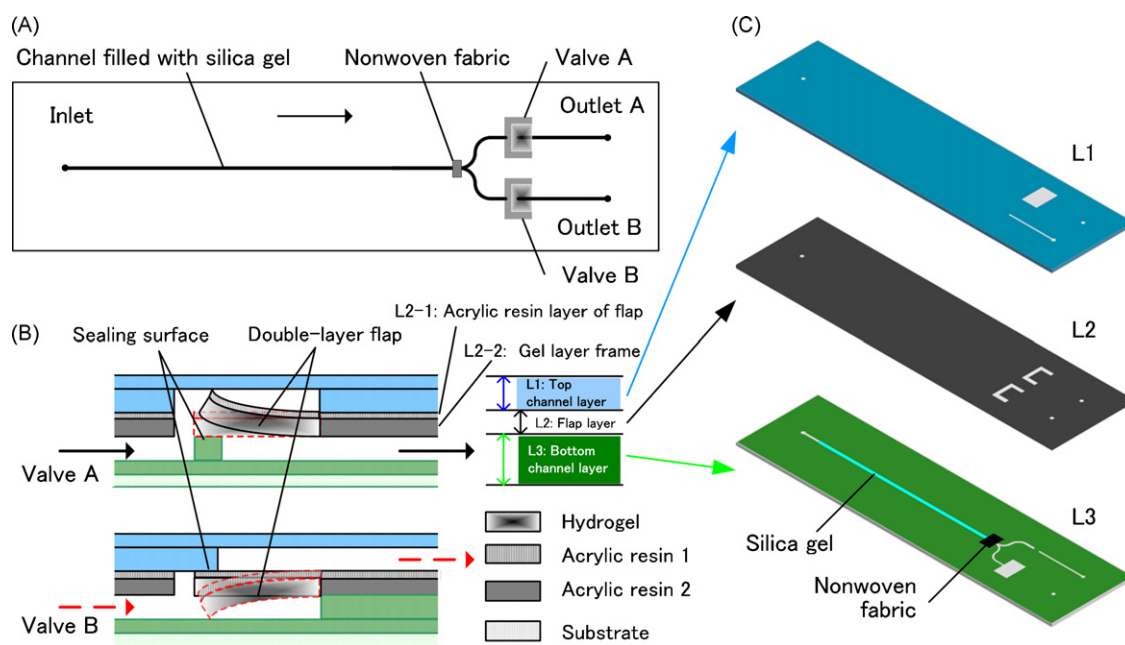


Fig. 1. Schematics of (A) a top view of the overall microchannel concentrator and (B) the cross-sectional views of the thermoresponsive valves at lower (black solid line) and higher temperatures (red dash line). (C) The layout of the fluid control items for each layer before laminating. Single arrows in (B) indicate the directions of fluid flow within the chip. Inlet channel size: 200 μm in depth, 500 μm in width and 60 mm in length. Flap size: 3.0 mm in width and 4.0 mm in length. The thickness of the acrylic resin layer (L2-1) and the hydrogel layer (L2-2) of the flap are 60 and 150 μm , respectively.

To prepare the bottom channel layer (L3), ARP2 was spin-coated on an acrylic plate and UV-cured. ARP2 was spin-coated again on it and then a piece of non-woven fabric was immersed into the coated ARP2. The L3 layer was obtained by the partial curing for the coated ARP2 through a photomask, followed by washing with ethanol. Silica gel particles were then filled in the inlet channel. The top channel layer (L1) was prepared on an OPP film by using the essentially similar procedure as the case of L3. Finally, the three layers of L1, L2 and L3 were laminated, and thoroughly UV-cured to make the adhesion completed.

3. Results and discussion

3.1. Chip design

A microchip that has one inlet channel and two outlet branches was designed, as schematically shown in Fig. 1. The chip is composed of three UV-cured layers, which correspond to a flap layer (L2) sandwiched between top (L1) and bottom (L3) channel layers. Two thermoresponsive double-layered valves are equipped in each outlet channel. As shown in Fig. 1B for the cross-sectional views, the valve parts have special channel structures. The valve A has a sealing surface on the bottom layer (L3) so that the flap can bend only upward, while the valve B has a sealing surface on the top (L1) to bend its flap only downward. At a lower temperature, the hydrogel layer swells causing the flap in the valve A to bend upward and consequently allowing solution to flow through the opened valve A. On the other hand, at a higher temperature, the shrunk hydrogel layer causes the flap to bend downward to open the valve B and solution then passes through this valve toward the outlet B.

3.2. Chip fabrication

The microchannel concentrator was fabricated as follows. To fabricate the flap layer (L2) (see also Fig. S-1), the acrylic resin layer (L2-1) providing a part of flap and the acrylic resin layer (L2-2) giving a gel layer frame were prepared separately via partial curing and then laminated together [24]. Subsequently, a drop of hydrogel precursor was applied in the flap site and photopolymerized through a photomask to complete the flap layer preparation. The bottom layer (L3) was prepared by partial photo-curing through a photomask of the acrylic resin prepolymer including a piece of non-woven fabric, and silica gel particles were filled in its inlet channel. The non-woven fabric was used as a stopper to hold the silica gel particles in the inlet channel. The layers of L2 and L3 were then laminated with the partially cured top layer (L1) that had been prepared by photopolymerization, and, finally, the three-layer chip was thoroughly cured to make the adhesion completed. By installation of adaptor fittings and tubing stuffs, the microchannel concentrator having the inlet channel equipped with adsorbents and the outlet channels equipped with thermoresponsive valves became ready for use. The monomer compositions of the acrylic resin prepolymers are shown in Section 2. Note that the acrylic resin providing a part of flap has a flexible feature, by addition of a non-crosslinkable monomer, more than the resin compris-

ing other device frameworks, to follow the hydrogel motion efficiently.

To obtain a good flap performance, a new idea was given in the PNIPAAm hydrogel layer preparation. Both poly(vinyl pyrrolidone) (PVP) and its lightly-crosslinked counterpart (so-called polyvinyl-polypyrrolidone [25], PVPP) were incorporated in the hydrogel precursor. PVP (a soluble polymer) acts as a micropore-forming reagent through the polymerization-induced phase separation (PIPS) process, so that the resulting gel layer has a microporous structure (Fig. S-2) to show a quick swelling/deswelling performance responding to temperature changes [26–28]. On the other hand, PVPP (an insoluble polymer) is expected to be remained in the gel layer, to prevent the porous hydrogel over-collapsing during deswelling.

3.3. Dynamic responses of double-layer flaps

To get a fundamental insight about the flap deformation, the flap layer (L2, see Fig. 1) itself that has a larger flap (7.5 mm in width; 10 mm in length) was examined by temperature-jump experiments in water bath. Time courses of the flap bending responses are depicted in Fig. 2A, together with those of the flap prepared using a conventional non-porous PNIPAAm hydrogel (for the composition, see Section 2) [29]. The video image showing the side view of the flaps was initially captured after immersing the flap layers into the water bath, and then transformed into a graphic file for further analysis. The deformation of the flaps is expressed in terms of the curvature ($1/r$) at the midlength of the flap, where r is the radius of the osculating circle [30]. When the flap bends toward the acrylic resin layer, we define the curvature is positive (Fig. 2Ba), while as it bends toward the gel layer the curvature is negative (Fig. 2Bb). As shown in Fig. 2A, the flap with the microporous hydrogel layer reveals instantaneous bending responses to temperature switching in both directions between 30 and 60 °C. Of interest is that the flap bending requires only several seconds to reach its equilibrium states. This behavior is in sharp contrast to the slow response of the non-porous hydrogel flap that requires over 10 min to accomplish the bending completion. These results clearly indicate that the microporous nature of the hydrogel layer can help quick response of the flaps significantly. And, no deterioration of the bending performances was found for the hydrogel flap in the repeated experiments for at least 100 times.

3.4. Valve function

The valve function of the flaps integrated in the microchannel concentrator was characterized in terms of the fluid flow switching. The channel temperature was controlled by using a hot plate equipped under the microchip. Since the hot plate needs a longer time to reach the set temperature in the heating and cooling processes, the chip was placed alternately on two aluminium plates preset at 20 and 90 °C, respectively. Water was injected into the microchip at a flow rate of 15 $\mu\text{L}/\text{min}$ and the alternating function of the two-valve system was examined by weighing the collected water through the valves at particular time intervals. Opposite working states of the two valves were successfully

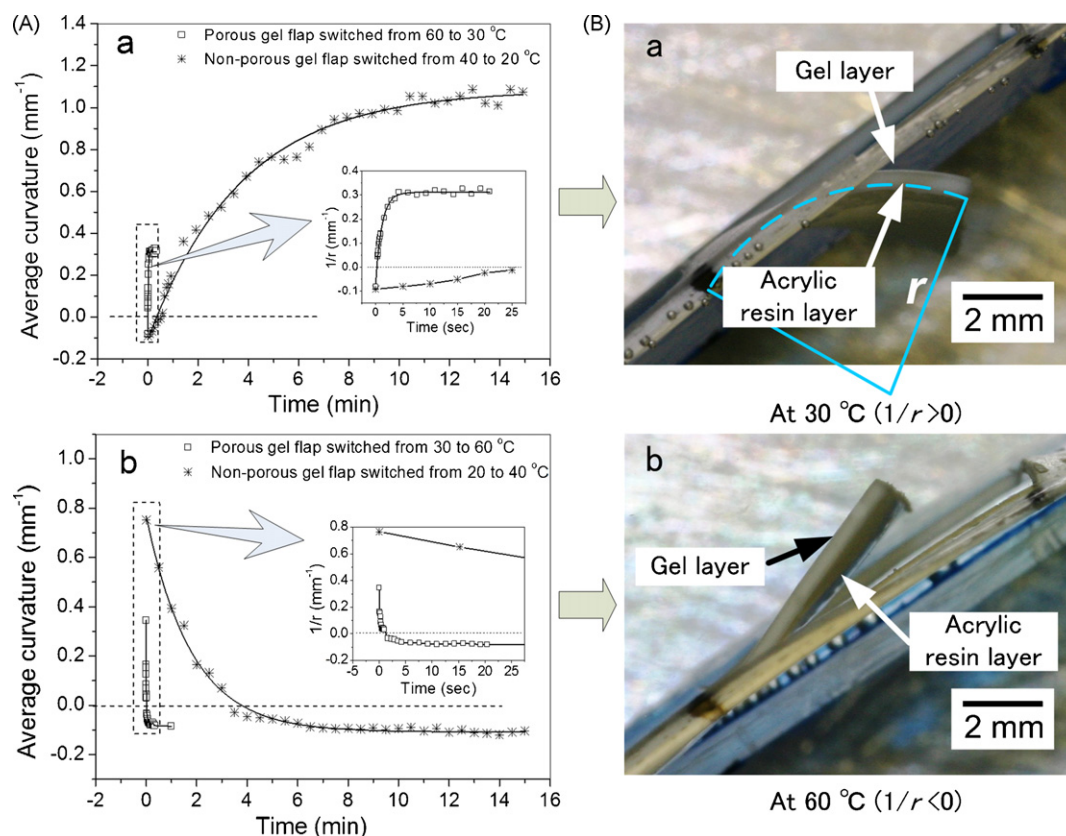


Fig. 2. (A) Time courses of the bending performances of the microporous and non-porous hydrogel flaps in water bath, when the temperature was switched (a) from higher to lower and (b) from lower to higher. Conformation of the flaps is 7.5 mm in width and 10 mm in length. The y-axis indicates the average curvature ($1/r$) of the flaps at the midlength, where r is the radius of the osculating circle. (B) Captured images showing the side view of the flap with porous hydrogel layer in water at (a) 30 and (b) 60 °C.

demonstrated, where at 20 °C the valve A opened and the valve B closed, while they changed to their contrary states at 90 °C. The open/close cycle was repeated for several times with the almost mirror-imaged result as demonstrated in Fig. 3. At this flow rate, the times required for opening and closing the valve A are in the range of 10–20 s. For the valve B, it opens in less than 20 s, but requires about 30 s for the closing. These response times

of the valves are longer than those of the flap itself mentioned above, presumably resulting from the poor thermal conductivity of the acrylic substrate with 1 mm thickness.

The alternating function was then tested at a higher flow rate. This attempt was very suggestive to consider interaction of the valves with the liquid flow. At a flow rate of 50 $\mu\text{L}/\text{min}$, the times in responding to the temperature changes were actually extended, which were much more than those at 15 $\mu\text{L}/\text{min}$.

When the chip was cooled to 20 °C, the valves A and B required ca. 180 and 240 s for their opening and closing, respectively. The flap of the valve B bends upward to the sealing surface as mentioned above and it is anticipated that the fast-flowing water pushes the flap away from the sealing surface in the valve chamber (see Fig. 1B). The force that runs counter to the bending direction of the flap would be a reason why the valve B requires a longer time for its closing. According to this hypothesis, water flow should facilitate the bending of the flap in the valve A upward to open this valve. However, this case also needed a longer time for switching. It can be speculated that the slow response of the valve B affects the behavior of the valve A, that is, long-lasting water leakage through the valve B tends to suppress the response of the valve A.

When the chip was heated to 90 °C, it required 80 s to close the valve A and 50 s to open the valve B. In this case, again, the time for the valve opening was shorter than that for the closing, which seems to result from the assisting from the fast-flowing

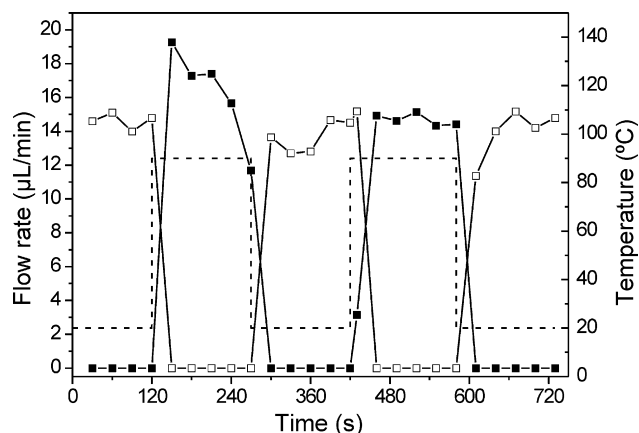


Fig. 3. Monitoring of the alternating function of the two-valve system by weighing the collected water through the valve A (□) and valve B (■) at particular time intervals, where water was injected into the microchip at a flow rate of 15 $\mu\text{L}/\text{min}$. The temperature (—) was varied between 20 and 90 °C.

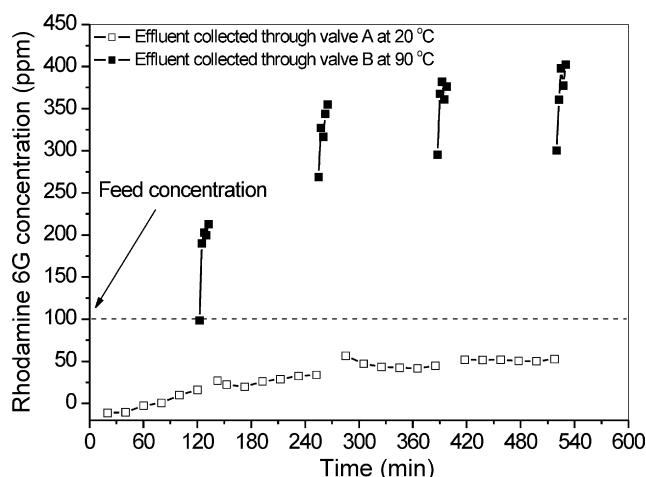


Fig. 4. Rhodamine 6G concentration of the effluents collected through the valve A (□) and B (■) at 20 and 90 °C, respectively, as a function of operation time. The R6G concentrations were determined by means of fluorescence spectroscopy for the diluted samples of the effluents (500 times in volume).

water as the low temperature case. It should be also noted that the switching periods of time at 90 °C (50–80 s) was somewhat shorter than those at 20 °C (180–240 s) for both valves. Currently, no straightforward idea explaining this result is available. Works are in progress to further investigate interactions between the flaps and the liquid flow.

3.5. Concentrator performance

To demonstrate the microchannel concentrator performance, R6G, a typical water-soluble fluorescent molecule, was selected as an analyte. The concentrator was initially fed with 100 ppm aqueous R6G solution at a flow rate of 15 $\mu\text{L}/\text{min}$ for 2 h at 20 °C (adsorption step). Subsequently, while running at the same flow rate, the channel temperature was raised to 90 °C and kept for 12.5 min to desorb the dye from the silica gel embedded in the channel (desorption step). These two steps were alternately conducted for multiple times afterwards. By these processes, effluents were separately collected through the outlet A and B depending on the running temperature at 20 and 90 °C, respectively.

Fig. 4 shows R6G concentration of the effluents collected through the outlet A and B, as a function of operation time. It is evident that the R6G solution was dramatically concentrated in the effluent of the outlet B during the desorption process. The concentration of the outlet B effluent was 2.2–4 times as much as that of the feed solution. On the other hand, diluted solutions of the dye were collected through the outlet A in the adsorption step. It is also interesting to note that the outlet B concentration got increased as the adsorption/desorption cycles have been carried out repeatedly, though the outlet A also increased gradually. This probably indicates that the amount of R6G adsorbed on the silica gel is enhanced as the adsorption/desorption steps are successively applied, so that upon the temperature jump quick release of the increased number of R6G molecules can be observed.

4. Conclusion

We have demonstrated that the microchannel concentrator based on the adsorption/desorption mechanism can be provided, in association with a smart fluid flow control system. The thermoresponsive flaps were successfully performed as a set of switching valves, which revealed opposite working (open/close) states at a certain local environmental temperature. As a consequence, a four-fold enhancement in R6G concentration was achieved by the single operation of the present microchannel concentrator. As our integral valves autonomously change their functions when the adsorption and desorption steps are switched, the chip is well suited for continuous operation if the feedstock is continuously recycled under HPLC pump motivation. A key issue that must be solved for future practical application is to achieve quicker response performance for the valves than the present system. In this work, poor thermal conductivity of the substrate led to the long response time of the valves. Therefore, it can be offered to use laser-beam for pinpoint heating of the valves, instead of the hot plate system. In addition, alteration of the flap conformation might contribute to improve the performances even at a high flow rate, because reducing the size and thickness as well as changing the aspect ratio (length versus width) should exert large influences on the flap motions. Likewise, the concentrating efficiency of the system could be further improved, for example, by further miniaturization of the chip, increasing surface-to-volume ratio of the channel or utilizing an adsorbent having smaller particle size. We believe that the analyte enrichment process based on adsorption/desorption and the smart flow control has a great potential in the field of μTAS technologies.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at [doi:10.1016/j.snb.2007.08.015](https://doi.org/10.1016/j.snb.2007.08.015).

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Biographies

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