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Short communication

Simple technique for joining of capillaries in capillary separation methods

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Abstract

A procedure is described for joining capillaries of the same outer diameters. The ends of the capillaries to be joined are evenly polished and capillaries are placed against one another with polished ends pressed together in a simple Plexiglas holder. The junction obtained does not contribute to separand zone distortion and broadening, does not cause deterioration in the separand resolution and is mechanically stable. © 1998 Elsevier Science B.V.

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1. Introduction

The joining of two capillaries or joining a separation capillary to a detection cell and various kinds of derivatizing cells or micropreparative devices is often required in capillary separation methods (capillary electrophoresis, CE, and micro-HPLC). The capillaries must be joined so that the junction contributes as little as possible to the broadening of the separand zones. The most common and the simplest approach is the joining of two capillaries by a piece of a plastic tubing into which the capillaries are pressed so that their ends touch one another (butt joint). However, it is very difficult to position and maintain the capillaries exactly against one another and the junction is often poorly mechanically stable. Even very small movements of the capillaries during the separation procedure (e.g. when introducing the sample or when applying the separation voltage in CE) cause changes in the position of the capillary ends at the junction.

It has been shown in Ref. [1] that an appreciable zone broadening occurs when the capillary ends are apart by as little as 1 μm and another kind of junction has been proposed. This so-called etched joint employs etching with hydrofluoric acid to produce a conical cavity at the end of one capillary (female end) and to shape the end of the other capillary into a matching cone (male end); the male end is then inserted into the female one and the junction is glued together. This junction is reliable but its preparation is time-consuming and mechanically demanding.

This paper proposes a technique for obtaining a reliable butt joint by evenly polishing the capillary ends and fixing them mechanically in a simple holder. When using polished capillary ends, microscopic cavities are avoided that occur with capillaries that are merely cut to size.

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2. Experimental section

2.1. Capillary joining

The ends of the capillaries to be joined (fused-silica capillaries, 375-μm O.D.×75-μm I.D., Composite Metal Services, UK) were cut at right angles and slightly polished on the flat part of the fused-silica capillary cutter (Chromatography Research Supplies, Cat. No. 205312). To make the capillary ends flat a simple U-shape jig was employed. The cutter was immovably fixed in one arm of the jig. The capillary was placed perpendicular to the cutter in the other arm in such a way that its end could be slightly pressed against the cutter and grind by hand. The capillaries were then washed by passage of isopropanol and distilled water. The junction was then created in a simple holder that consists of two Plexiglas plates (20×30×3 mm) that can be joined together by four bolts. Across the plane of one of the plates, through its center, a V-shaped groove (ca. 350 μm wide and deep) was cut and the capillaries were placed in the groove against one another, their ends pressed together. On fixing the two Plexiglas plates together by the bolts, the position of the two capillaries is fixed. The holder was originally proposed for the alignment of the separation capillary and the detection electrode in CE with amperometric detection [2]; it can be used to fix two capillaries together in the way described in that paper (see Fig. 1 in Ref. [2]).

In the center of the two Plexiglas plates a hole 2 mm in diameter is made at the site of the capillary junction; after fixing the two capillaries, the hole is cemented using a hot glue gun. The capillary junction after fixing in the holder and prior to glueing can be seen in Fig. 1, demonstrating that the capillaries are positioned exactly against one another with their polished ends touching as two parts of a flat ground-glass joint.

2.2. Apparatus for joint testing

The quality of the capillary junction was tested by performing a CE separation on a Crystal 310 CE instrument (ATI Unicam, UK) with a Unicam 4225 UV detector. The experimental conditions are listed in Table 1. Identical separation procedures were carried out with joined capillaries and with an equally long single capillary.

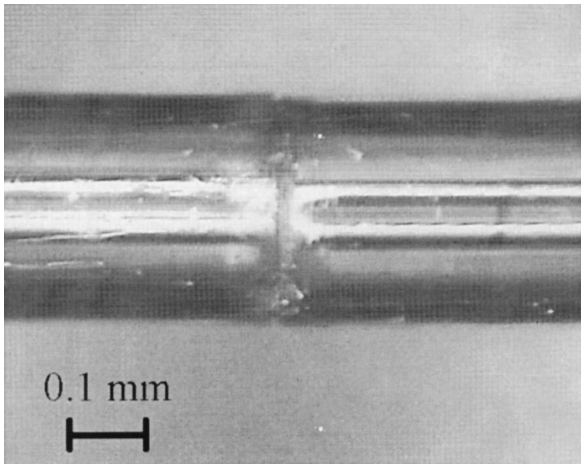

Fig. 1. Microphotograph of the capillary junction in the Plexiglas holder.

3. Results and discussion

3.1. Properties of the junction

The principal characteristics of the junction were tested on a separation of a model mixture of benzoic acid and *o*-nitrophenol. The numbers of theoretical plates, the peak widths at the base and at half-height and the resolution obtained were statistically treated using the ANOVA (analysis of variance) program and compared for the joined and unjoined capillaries; the results are given in Table 2. It can be seen that the junction does not contribute to the value of these parameters and the differences between the two sets of data are mostly statistically insignificant. The only statistically-significant difference was found in the plate count for benzoic acid; however, this parameter was more favorable for the joined capillaries. This deviation may be caused by, e.g., random differences in the properties of the materials used for the production of the joined and unjoined capillaries, by differing amounts of locally-adsorbed substances, by an effect of the capillary orifices on the sample introduction, etc. The appropriate electropherograms are given in Fig. 2.

Table 1
CE experimental conditions

Capillary with junction	Internal diameter: 75 μm Total length: 75 cm Length to detector: 60 cm Length to joint: 51 cm (from inlet)
Capillary without junction	Internal diameter: 75 μm Total length: 75 cm Length to detector: 60 cm
Running buffer	20 m*M* sodium tetraborate
Sample solution	Benzoic acid (0.05 mg/l) *o*-Nitrophenol (0.18 mg/l) in 10 m*M* tris(hydroxymethyl)aminomethane
Applied voltage	20 kV
Injection	Pressure, 10 mbar/6 s
Detection	Photometric, 200 nm

3.2. Mechanical strength and geometric stability of the junction

After one month of use, it can be stated that the mechanical strength of the joined capillaries is quite comparable to that of a capillary without a junction. Due to the Plexiglas fixing holder and to the very good adhesion of the glue to both the capillaries and to the holder material, the junction is also very resistant against breakage. It is tight, as no sign of seepage was observed on prolonged and repeated washing (5×60 min) at the maximum permitted pressure (3000 mbar).

4. Conclusions

A simple and satisfactorily-reliable technique has been proposed for joining of capillaries of the same outer diameters. The junction obtained does not contribute to separand zone distortion and broadening and does not cause deterioration in the separand resolution. The junction can be produced under normal laboratory conditions, without any special equipment. The demands of the operation are comparable to those of common procedures such as the placement of a capillary into the instrument, creating of the detection window and the placement of the capillary into the photometric detector.

The junction was tested only under CE conditions where no pressure is applied. We suppose that the capillaries are mechanically fixed and glued in a Plexiglas holder so that the joint can withstand relatively high pressures. This assumption is supported by the fact, that the junction resisted repetitive pressurised rinsing steps. Moreover, the junction of separation capillary with detection or derivatizing cells is normally placed close to the end of capillary, where the actual pressure is relatively low.

The proposed technique is routinely used in our

Table 2
Comparison of the experimental results obtained for a capillary with and without a junction

Parameter	Capillary with junction[a]	Capillary without junction[a]	Significant difference[b]
Number of plates	140 944 (2668)/139 845 (3210)	126 586 (882)/133 287 (1810)	yes/no
Peak base width (min)	0.193 (0.010)/0.216 (0.010)	0.201 (0.006)/0.217 (0.007)	no/no
Peak half width (min)	0.064 (0.002)/0.066 (0.001)	0.067 (0.001)/0.067 (0.001)	no/no
Resolution	1.063 (0.026)	1.062 (0.017)	no

[a] Benzoic acid/*o*-nitrophenol, mean value from 9 experimental values, standard deviation in parentheses.
[b] For confidence level 95%.

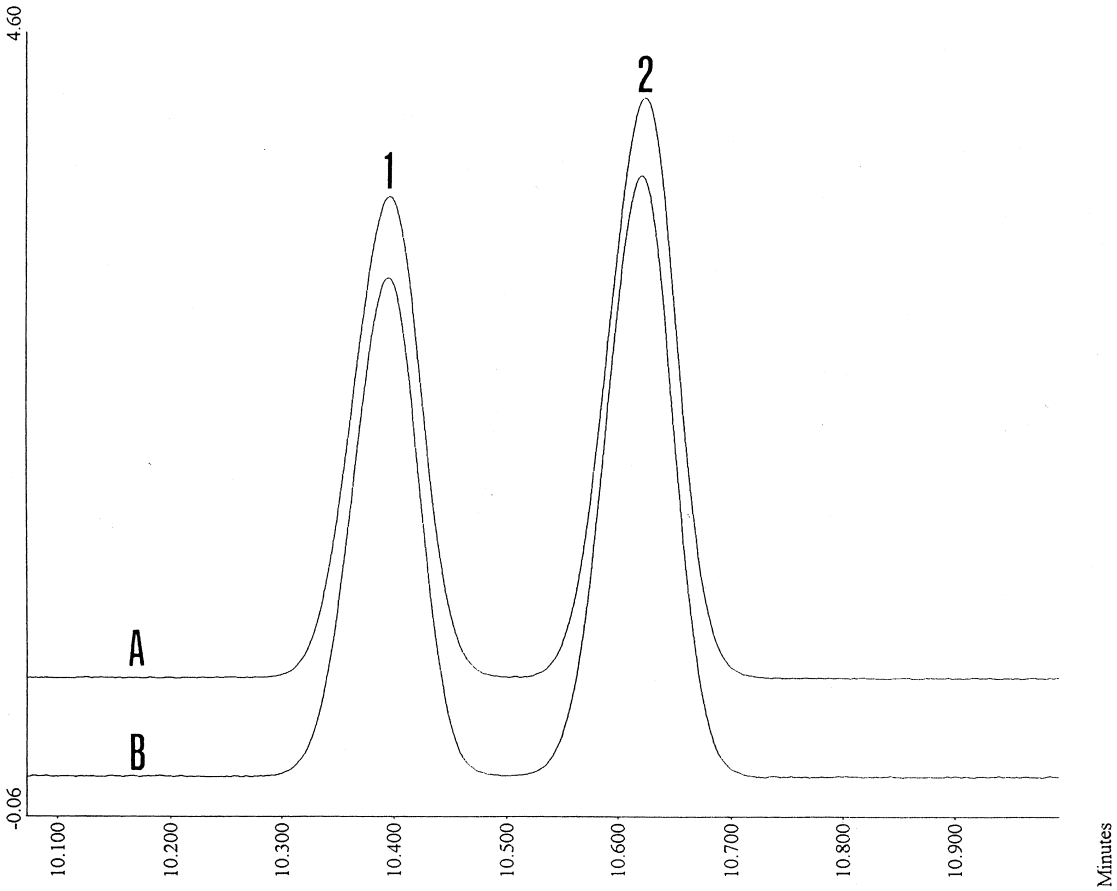

Fig. 2. Electropherogram for the separation of benzoic acid (1) and *o*-nitrophenol (2) in a capillary without (A) and with (B) the junction (the curves are shifted against one another for the sake of lucidity).

laboratory; it follows from our experience that more than 80% of the junctions satisfy all requirements and could be successfully employed.

References

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[2] A.M. Fermier, M.L. Gostkowski, L.A. Colón, Anal. Chem. 68 (1996) 1661.