# A Novel Miniaturized Flame Ionization Detector for Portable Gas Chromatography

**Chunhui Deng, Xiuhan Yang, Ning Li, Yue Huang,** and **Xiangmin Zhang\***

Department of Chemistry, Fudan University, Shanghai 200433, China

### Abstract

A novel miniaturized flame ionization detector (FID) operated by battery, which can be used as a detector in portable gas chromatography (GC) is devised and manufactured. It is characterized by the structure of building blocks, small volume, low energy consumption, and needing only two gases, which can be used for detection of hydrocarbons in portable GC. The miniaturized detector mainly includes a porous metallic diffuser plate, bugle-figuration collector, quartz capillary flame tip, and self-heated system. The miniaturized FID is easy to fabricate and assemble because of its structure of building blocks. The FID response is linear over six orders of magnitude and the detection limit of 0.518 ng for benzene, 0.430 ng for *n*-dodecane, 0.473 ng for naphthalene, and 0.509 ng for *n*-tetradecane.

## Introduction

The flame ionization detector (FID) was developed by McWilliams et al. and Harley et al. (1,2). FID is the most popular and widely used detector of gas chromatographic (GC) instruments. The sample from the analytical column of a GC is pre-mixed with hydrogen make-up gas and burned at the top of a flame tip with air as oxidant. The burning decomposes the organic sample and quantitatively ionizes it. The resulting ions or electrons are collected by an electrode located adjacent to the flame tip. Another electrode located below the flame tip provides a bias potential, so that an electrical field develops between the two electrodes. Current flowing between the two electrodes is proportional to the number of ions or electrons produced and collected (3).

In an FID, a diffusion flame is the most efficient way to ionize the sample. The introduction of air must also be done very smoothly to minimize flicker noise caused by having a flame burning unsteadily and typically resulting from an air inlet that does not bring the air to the flame tip smoothly. In conventional FID, flicker noise is reduced by allowing sufficient vertical distance between the air inlet and flame tip so as to develop laminar air flow. However, a sufficient vertical distance undesirably results in a physically longer detector. In addition, conventional FID uses a high flow rate of hydrogen makeup gas (> 30 mL/min), thus requiring a correspondingly high air flow rate. The high flow rate of hydrogen creates a longer and wider flame, requiring a corresponding physically larger collector, hence increasing overall detector size.

Recently, conventional GC cannot meet with the dramatic reduction in analysis time, which could lead to significant economical advantages for applications involving environmental and chemical process monitoring. It has been demonstrated that column temperature programming of 10°C/s can be obtained by direct resistance heating of capillary columns (4–6). Direct heating and other technologies led to the development of portable, micro, and high-speed GC (7,8). FID is a universal detector used with GC. However, conventional FID with its large size and high fuel consumption is not suitable as the detector for a portable GC. Recently, Fremont and Zimmermann invented a micro-FID with low fuel consumption (9,10). However, the detector is not convenient to fabricate because that process requires silicon micromachining technology. Also, it is unsuitable for the detection of hydrocarbons in portable GC.

In this work, a novel miniaturized FID for portable GC, which mainly included a porous metallic diffuser plate, bugle-figuration collector, quartz capillary flame tip, and self-heated system is devised and manufactured. To evaluate the miniaturized FID, the study of the linear response range and detection limit for hydrocarbon compounds was carried out.

### Description of the miniaturized FID

Figure 1 shows a photograph of the miniaturized FID that was devised and manufactured. A sectional view is shown in Figure 2. It mainly includes a cylindroid cavity, which is made by a stainless steel cylinder, porous metallic plate, novel nozzle, bugle collector, and small inside diameter tube attached to the bottom of the cavity. The tube is a triple channel. In the triple channel, sample, make-up gas passed from a capillary GC column, and complementary hydrogen are mixed, and these gases instantly enter another quartz capillary, which is used as the nozzle. The nozzle, with a 0.25-mm i.d., needs the optimum fuel flow of approximately 15 mL/min. This gives high sensitivity and saves fuel.

Figure 3 is the detail structure of the nozzle. The outer side of the quartz capillary is a stainless steel tube, and the top end of the stainless steel tube is lower (1.0 mm) than the quartz capillary,
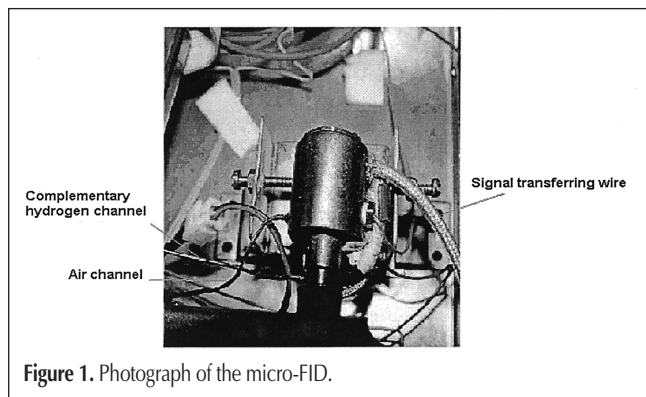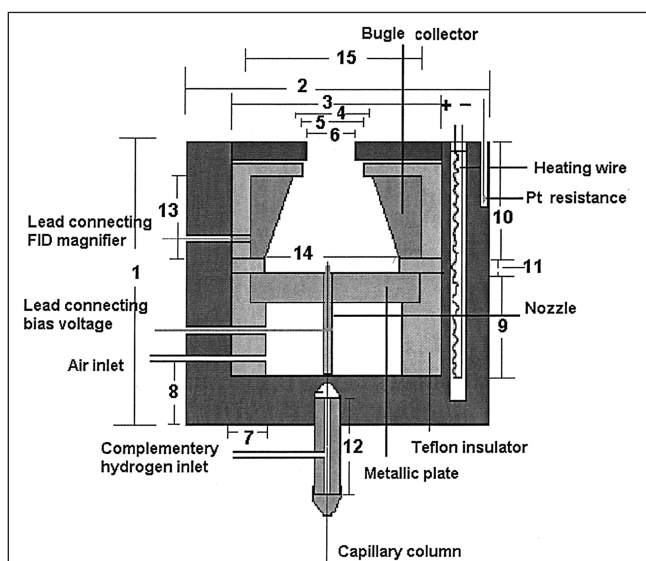


**Figure 1.** Photograph of the micro-FID.



**Figure 2.** Sectional view of the micro-FID: 1, 19.4 mm; 2, 19.0 mm; 3, 11.8 mm; 4, 6.5 mm; 5, 4.3 mm; 6, 2.9 mm; 7, 5.3 mm; 8, 4.0 mm; 9, 4.6 mm; 10, 11.2 mm; 11, 2.3 mm; 12, 15.2 mm; 13, 10.2 mm; 14, 10.1 mm; and 15, 11.0 mm.
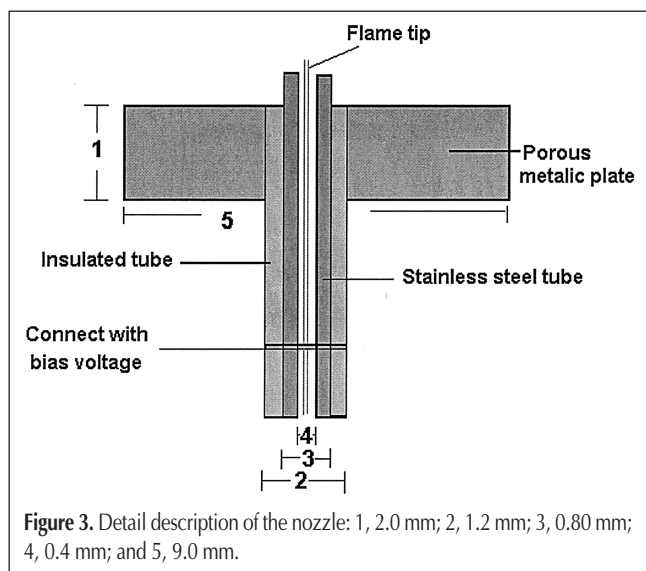


**Figure 3.** Detail description of the nozzle: 1, 2.0 mm; 2, 1.2 mm; 3, 0.80 mm; 4, 0.4 mm; and 5, 9.0 mm.

which is the first electrode. The outside of the stainless steel tube is an insulated tube, and the top end of the insulated tube is lower (0.5 mm) than the stainless steel tube, which passes through the central hole of the porous metallic plate. The lower portion of the steel tube under metallic plate is connected with the bias voltage.

The air needed for combustion is introduced from the air channel beneath the metallic plate, flowing in a direction parallel to the plane defined by the plate. A Teflon shim is placed between the metallic plate and collector. The flame tip is located at the lower end of a 4 to 6 mm (height) collector. The collector, with bugle figuration, can achieve high electrical field strength for efficient collection. This close spacing advantageously minimizes the internal volume of detector. The overall height of the detector assembly may be less than 3 cm. The overall compactness also conserves electrical power to heat the detector to operating temperatures. A porous metallic plate (Figure 3) was used. The metallic plate is made of many small stainless steel balls (200-μm i.d.) by using the technology of powdered metals. Because many holes formed, the plate allows the air to be introduced immediately adjacent the flame tip, thus eliminating the need for a substantial vertical separation between the air inlet and flame tip. The structure reduces overall detector height, allowing for a more compact device.

The heating wire in the channel in ektexine of the detector is used to heat the detector and a Pt resistance was used as the temperature sensor to control the temperature of the micro-FID. Because the thermal mass of the micro-FID is very small, the temperature approximately 250°C is easy to obtain in a very short time (2 min) by heating with the aluminum batteries (18.5 V, 9 Ah).

## Experimental

### Chemicals and reagents

Standard compounds of benzene, *n*-dodecane, *n*-tetradecane, and naphthalene were purchased from Sigma-Aldrich (Deisenhofen, Germany). Dichloromethane (analytical grade) was obtained from Chemical Agent Company (Shanghai, China). Benzene (52.8 mg), *n*-dodecane (53.1 mg), *n*-tetradecane (54.8 mg), and naphthalene (50.1 mg) were weighted. Standard solutions with the concentrations from 5280 to 0.0528 μg/mL for benzene, 5310 to 0.0531 μg/mL for *n*-dodecane, 5480 to 0.0548 μg/mL for *n*-tetradecane, and 5010 to 0.051 μg/mL for naphthalene were prepared.

### Portable GC

Experimental was performed using a portable GC190, which was devised and manufactured by Fudan University and Analytical Science Instrument Company (Shanghai, China). Capillary column of OV-1 (5.0-m × 0.25-mm i.d., 0.25 μm) was used. A split injection of a 0.4-μL sample with a split ratio of 14:1 was employed. The temperature program for GC separation was as follows: initial temperature, 50°C for 30 s, ramp up to 200°C at 2.5°C/s, maintained 200°C for 30 s. Hydrogen (99.999%) was used as carrier gas with a flow rate of 15.0 mL/min. Four replicate analyses for each sample were performed.

## Results and Discussion

### Linearity and detection limit

Figure 4 is the chromatogram of the standard solution of benzene, *n*-dodecane, *n*-tetradecane, and naphthalene. Retention time of benzene, *n*-dodecane, *n*-tetradecane, and naphthalene is 17.2, 26.9, 38.6, and 46.7 s, respectively. A linear equation for the four compounds was obtained by peak height and concentration logarithm (Table I). A linear response for each compound is clearly observed over a range of almost six orders of magnitude with a slope of 0.99.

The detection limit is defined as the hydrocarbon concentration that generates signal equivalent to three times noise. The detection limits for the four compounds are also listed in Table I. The data in Table I show that low detection limits could be obtained.

### Repeatability

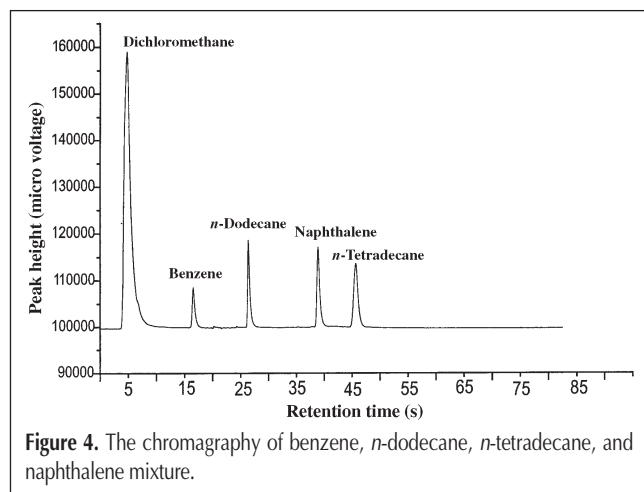Four replicate analyses of standard solutions were performed at the same conditions. The values of peak height obtained were used for calculation of the relative standard deviation (RSD). RSD values of benzene, *n*-dodecane, naphthalene, and *n*-tetradecane were 0.34%, 0.21%, 0.46%, and 0.29%, respectively.

## Conclusion

The present micro-FID with small volume was operated with battery, which needs two gases (air and hydrogen). It is very easy to fabricate and assemble because of its structure of building blocks. The novel FID has an excellent detection limit and linear response, which can be used as the detector for a portable GC.

**Figure 4.** The chromagraphy of benzene, *n*-dodecane, *n*-tetradecane, and naphthalene mixture.

### Table I. Linear Equations and Detection Limits for Benzene, *n*-Dodecane, Naphthalene, and *n*-Tetradecane

|  | Linear equation | $R^2$ | Linear range | Detection limit (ng) |
|---|---|---|---|---|
| Benzene | $Y = 0.8207X + 2.0766$ | 0.9999 | $10^6$ | 0.518 |
| *n*-Dodecane | $Y = 0.9994X + 2.0900$ | 0.9994 | $10^6$ | 0.430 |
| Naphthalene | $Y = 0.8948X + 1.8848$ | 0.9991 | $10^6$ | 0.473 |
| *n*-Tetradecane | $Y = 0.8762X + 1.8247$ | 0.9986 | $10^6$ | 0.509 |

## References

1. I.G. Mcwillian and R.A. Dewar. Flame ionization detector for gas chromatography. *Nature* **181:** 760 (1958).
2. J. Harley, W. Nell, and V. Pretorius. Flame ionization detector for gas chromatography. *Nature* **181:** 177–78 (1958).
3. D.G. McMinn and H.H. Hill. *Detectors for Capillary Chromatography.* John Wiley & Sons, New York, NY, 1992, pp. 7–21.
4. V. Jain and J.B. Phillips. Fast temperature programming on fused-silica opentubular capillary columns by direct resistive heating. *J. Chromatogr. Sci.* **33:** 55–59 (1995).
5. E.U. Ehrmann, H.P. Dharmasena, K. Carney, and E.B. Overton. Novel column heater for fast capillary gas chromatography. *J. Chromatogr. Sci.* **34:** 533–39 (1996).
6. M.E. Hail and R.A. Yost. Compact gas chromatograph probe for gas chromatography/mass spectrometry utilizing resistively heated aluminum-clad capillary columns. *Anal. Chem.* **61:** 2410–16 (1989).
7. R. Sacks, H. Smith, and M. Nowak. High-speed gas chromatography. *Anal. Chem.* **70:** 29A–37A (1998).
8. C. Henry. Taking the show on the road—portable GC and GC/MS. *Anal. Chem.* **69:** A195–A200 (1997).
9. S. Zimmermann, S. Wischhusen, and J. Müller. Micro flame ionization detector and micro flame. *Sens. Actuators, B* **63:** 159–66 (2000).
10. S. Zimmermann, P. Krippner, A. Vogel, and J. Müller. Miniaturized flame ionization detector for gas chromatography. *Sens. Actuators, B* **83:** 285–89 (2002).