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Transversely illuminated liquid core waveguide based
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Fluorometric flow injection determination of aqueous
ammonium/ammonia

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Transversely illuminated liquid core waveguide based fluorescence detection Fluorometric flow injection determination of aqueous ammonium/ammonia

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Abstract

The analytical performance of a new type of fluorescence detector, based on a transversely illuminated liquid core waveguide (LCW), has been investigated using the determination of $\text{NH}_3/\text{NH}_4^+$ as the 1-sulfonatoisindole. With a very inexpensive combination of a miniature Hg blacklight as an excitation source, a colored plastic sheet as the emission filter, and an integrated blue sensitized photodiode-operational amplifier as the detector (totaling < \$100 in hardware cost), we were able to achieve a limit of detection (LOD) of 35 nM (1.6 pmol) NH_3 with a linear dynamic range up to 60 μM NH_3 . Details of detector construction and performance are given. © 1999 Elsevier Science B.V. All rights reserved.

Keywords: Fluorescence detector; Liquid core waveguide

1. Introduction

The literature reports several studies in which the optical path length in liquid phase absorption measurements has been extended by using a reflective cell, either through the use of a physically reflective wall, or ensuring conditions in which the liquid has a refractive index (RI) greater than that of the conduit material and the

assembly therefore exhibits total internal reflection, i. e. it behaves as a liquid core waveguide (LCW) [1–3]. Until recently, due to the unavailability of any convenient material that has a RI value less than that of water, LCW applications has been largely limited to high RI organic solvents like carbon disulfide [4], or mixed aqueous solvents that contain large amounts of alcohol [2] or glycol [5], limiting the number of practical analytical applications.

Recently, a fluoropolymer based on 2,2-bis(trifluoromethyl)-4,5-difluoro-1,3-dioxole was introduced [6]. It is available as a copolymer with

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tetrafluoroethylene and carries the trade name Teflon® AF. Teflon AF is an amorphous, glassy, perfluorinated copolymer with the important characteristic that throughout the 200–2000 nm wavelength range, it is essentially transparent with a RI (1.29) lower than that of water (1.33). Thus, when such a tube is filled with water, it behaves as an LCW and can efficiently transfer the light launched at one end to another. This unusual and useful property of such tubes has been exploited in long pathlength absorbance spectroscopy [7–10].

Fujiwara et al. were among the pioneers in LCW based absorbance spectroscopy. The first use of LCW for fluorometric measurements was also reported by these authors [11,12]. They used a laser source to excite the fluorophore in an LCW axially and read the fluorescence axially from the other terminus of the tube. With this geometry, very effective means of rejecting the excitation light are required. A further disadvantage of the axial excitation scheme is that low wavelength laser sources necessary for fluorescence excitation are expensive and can not provide excitation over a broad wavelength range. They also used a 'side-view cell' to overcome the excitation rejection problem. In this geometry, the plane of a spirally shaped tubular flow cell is placed next to the photosensitive window of an 'end-on' type photomultiplier tube (PMT). The excitation light is launched axially into the spiral. The fluorescence signal is said to be linearly dependent on the refractive index of the solution. In this case, an expensive large window PMT must be used and a higher background, resulting from leaking of the excitation light from the spiraled LCW, is expected.

We propose an altogether different geometry for an LCW based fluorescence detector. An inexpensive linear light source illuminates the LCW from the side (transverse illumination). Light incident orthogonal to the axis of a wave guide is efficiently rejected. Thus, the exciting radiation does not proceed down the lumen of the LCW. We have measured that about 1 out of 10^6 incident photons actually traverses down the lumen (this occurs, presumably, from scattering by particles in the aqueous phase or from surface imper-

fections in the tube) [13]. In any case, this degree of rejection is better than what many monochromators can provide. On the other hand, if scattering or fluorescent species are present in the solution, a significant portion of the scattered/emitted radiation [the exact extent depends on the numerical aperture (NA) of the LCW] undergoes total internal reflection and proceeds down the lumen of the fiber. This light can be efficiently coupled to a high NA conventional optical fiber placed at the end of the LCW. Because the light is now already available in a optical fiber coupled format (without any focusing optics), detection by a small area inexpensive photodiode detector is facile. There is no great problem associated with rejection of the excitation light as exists in the axial excitation schemes. As a result, neither excitation nor emission monochromators are essential in this unique fiber optic coupled transversely illuminated LCW based flow-through fluorescence detection scheme. For highest sensitivity applications, a simple filter can be incorporated on the emission side to further reject any residual broadband excitation light that propagates down the lumen. The system also has the advantage that illumination can be provided over a large surface area but the resulting luminescence becomes available as a point source.

Fundamental studies related to the dependence of the S/N to the illumination length and illumination volume are discussed in our first report of such a detector. In the present paper we focus on testing the performance of such a detector in an application important to our laboratory, the determination of trace levels of $\text{NH}_3\text{-N}$.

Ammonia is the principal atmospheric base responsible for the neutralization of atmospheric acidity [14]. Ammonium salts are typically the principal constituents of the inhalable fraction of the atmospheric particulate matter and are widely believed to be the primary responsible agents for the degradation of atmospheric visibility [15]. Sensitive and affordable methods for determining $\text{NH}_3\text{-N}$ are essential to improve the time resolution of atmospheric measurements. Traditionally, the indophenol blue reaction [limit of detection (LOD) $\sim 0.6 \mu\text{M}$] and Nessler's reaction (LOD $1.2 \mu\text{M}$) have been used [16]. In 1971, Roth [17]

discovered the ternary reaction of *o*-phthalaldehyde (OPA), a ‘reducing agent’ [borohydride or mercaptoethanol (ME)], and ammonia or primary amino acids, to produce intensely fluorescent products and described its analytical usefulness. Many studies have been done on this fluorometric reaction. The OPA–sulfite–NH₃ fluorometric detection system in particular was developed in this laboratory [18] and has the advantage over the corresponding ME-based detection system that the use of malodorous thiol compounds is avoided and the reaction is much more selective for ammonia compared to amino acids. In the present work, we have used this reaction for testing the detector performance.

2. Experimental

2.1. Reagents

Reagents were prepared as previously reported [18]. Briefly, standard grade *o*-phthalaldehyde (P-1378, Sigma, St. Louis, MO) was used without further purification. The OPA solution (10 mM) was prepared by dissolving 268 mg OPA in 50 ml methanol and diluting with water to 200 ml. The solution can be stored in the refrigerator for 1 week.

Phosphate buffer (0.1 M) was made by dissolving 14.2 g of analytical reagent grade Na₂HPO₄ in 900 ml water, adjusting pH with 2 M NaOH and diluting to 1 l. Sodium sulfite solution (3.0 mM) was prepared daily in the phosphate buffer. Ammonium standards were prepared from a 0.1000 M NH₄Cl stock. Dilute solutions were prepared just prior to use by successive dilution.

All chemicals used were of analytical reagent grade, and freshly deionized water was used throughout all experiments.

2.2. Liquid core waveguide based fluorescence detector

The detector is schematically shown in Fig. 1. It consists of a Teflon[®] AF tube (BioGeneral, San Diego, CA, 0.84 mm i.d., 1.04 mm o.d.,

~115 mm long) that constitutes the LCW flow cell. As shown, at the bottom end it butts up against a large NA fiber optic (1 mm core fused silica, Polymicro Technologies, Phoenix, AZ), at the center of an opaque tee fitting composed of PEEK (P-713, Upchurch Scientific, Oak Harbor, WA). An appropriate length of a stainless steel tubing (5.2 mm o.d., 4.4 mm i.d., ~85 mm long, HTX-6, Small Parts, Miami Lakes, FL) is taken and the terminal ends are carefully deburred to remove all sharp edges. The tube is polished inside with a pipe cleaner and tooth paste until the inside is highly reflective. The tube fits in snug into the head of the nut in the tee fitting, as shown in the figure. On the other side of the tube, a $\frac{1}{4}$ -28-to-10-32 male–male

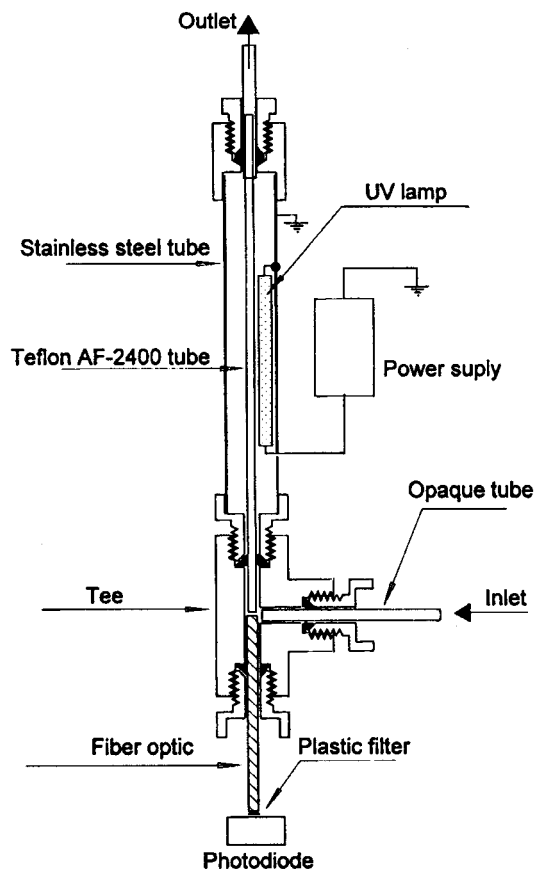


Fig. 1. The liquid–core waveguide fluorescence detector schematically shown. Teflon AF-2400 LCW length 115 mm, 0.84 mm i.d.

chromatography style union is used (P/N 42806, Dionex, Sunnyvale, CA). The $\frac{1}{4}$ -28 threaded side is bored out with an appropriately sized drill bit to snugly accommodate the stainless steel tube. The through hole in this union is drilled out to 1.5 mm bore. Two small holes are drilled through one side of the stainless steel tube, \sim 55 mm apart, to provide exits for the leads to the light source.

The light source is a miniature Hg black light tube (3×50 mm, P/N BF 350-UV1, Digi-Key, Thief River Falls, MN), that is operated by a miniature high voltage power supply (P/N BXA-502, JKL Component Corp., Pacoima, CA) with the primary input supply being 5 V. The total input power to the power supply is 250 mW without the lamp being connected and 950–1000 mW with the lamp connected. The optical lamp output flux is 200–300 $\mu\text{W cm}^{-2}$ at a distance of 1 cm from the lamp. The axial leads of the lamp are cut short to only \sim 1 mm length. Kynar-coated lead wires are inserted through the holes in the stainless steel tube such that both comes out of one end of the tube. These protruding ends are then soldered on to the lamp leads. The lamp is then pushed inside the tube, and the lead wires suitably withdrawn. The insulation is removed from one of the lead wires and it is wrapped around the stainless steel shell and cemented in place with electrically conductive epoxy adhesive to provide a grounded end. The other lead wire is also wrapped around the shell a few turns and the turns epoxied in place with ordinary non conductive epoxy adhesive to secure the lamp firmly in place. The lead wire is connected to the high voltage end of the power supply. The power supply ground is connected to the shell. (Our standard practice is to also provide a third aperture in the stainless steel shell, slightly larger than the other two apertures and located approximately equidistant from the other two. A photodiode is cemented on this hole and can be used to monitor the lamp intensity. No explicit use of this has been made in the present work.)

Because the lamp consumes little power, there are no problems associated with excessive heat-

ing of the lamp or the AF tube; the latter is, of course, also cooled by the flowing fluid.

With the Teflon[®] AF tube and the silica fiber optic already connected to the tee, the lamp-bearing stainless steel shell is guided over the AF tube and firmly inserted into the head of the nut. The AF tube now protrudes out of the free end of the stainless steel shell and the union fitting is now pushed over the end of the AF tube to securely connect to the stainless steel shell. The length of the AF tube is such that it protrudes \sim 2 mm beyond the central partition of the union. An opaque 1/16 in PTFE tube is taken and the terminal end is bored out with a drill bit so that the AF tube can fit in to this end. This is then slipped over the end of the AF tube and secured in place with a 10–32 nut and ferrule. A small disk of blue plastic sheet (No. 856, P/N 60403, Edmund Scientific, Gloucester, NJ) was cemented by UV-Cure adhesive (optical adhesive type 81, Norland Products, New Brunswick, NJ) to the free end of the silica fiber optic. After the disk fully adheres to the optic, it is trimmed to the fiber diameter with a sharp surgical knife.

The photodetector used in the present work is a blue-sensitive integrated photodiode-operational amplifier available in a TO-99 (OPT-301, Burr-Brown, Tucson, AZ) metal can. We used an opaque chromatography style bulkhead fitting with a $\frac{1}{4}$ -28 threaded flat bottom female port on both sides (P/N 38654, Dionex). One side is bored out and the photodetector is securely cemented therein. The bulkhead fitting is secured to the faceplate of the electronics enclosure. In use, the silica fiber optic passes through the through hole of the bulkhead fitting and is secured in place by a $\frac{1}{4}$ -28 nut and a flat bottom ferrule.

The electronic circuit for the detector is shown schematically in Fig. 2. The OPT-301 photodetector/amplifier output signal produces increasing positive outputs with increasing light input. A dual BIFET operational amplifier (TL 082) is used to provide offset and further gain to the primary output. The first stage functions as a summing inverting amplifier. The two 100 K 10-turn potentiometers connected to the first

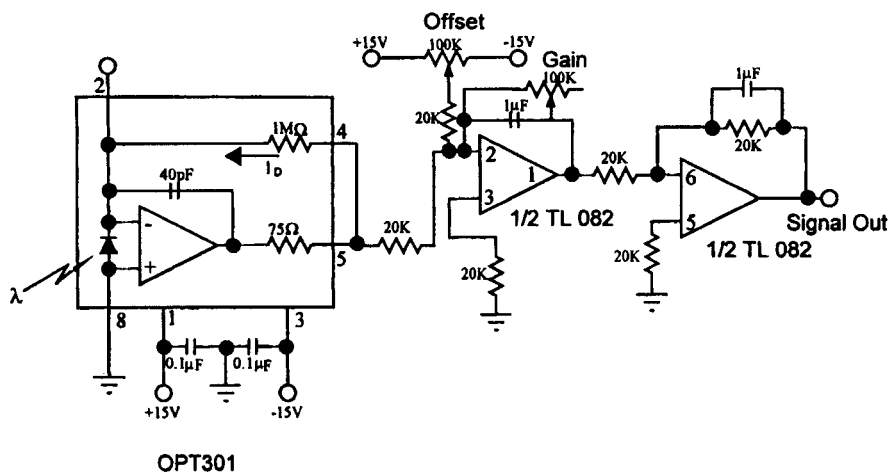


Fig. 2. Electronic schematic for the detector.

stage of the TL 082 IC, respectively provides adjustable offset and variable gain (up to $5 \times$) functions. Since this stage also inverts the signal, the second stage is operated simply as a unity gain inverting amplifier so that the final output is a positive signal that increases with increasing light input. The detector is commercially available from AnalTech (Lubbock, TX).

2.3. Analytical system

The flow system is schematically shown in Fig. 3. A peristaltic pump P (Rabbit, RAININ, Instrument, Emeryville, CA) was used to pump water (W) as carrier at $50 \mu\text{l min}^{-1}$ through an electropneumatically actuated six-port rotary valve V (type 5020P, Rheodyne, Cotati, CA) equipped with a $46 \mu\text{l}$ volume sample loop. The sample S was aspirated by the pump through the valve. The carrier stream mixed with the OPA reagent stream O ($50 \mu\text{l min}^{-1}$) at a low-volume tee. This was followed by a knotted mixing coil M1 ($0.3 \times 250 \text{ mm}$), and merged again with the buffered sulfite reagent stream B ($50 \mu\text{l min}^{-1}$). The next mixing coil M2 ($0.3 \times 1000 \text{ mm}$) was followed by a Teflon reaction coil R ($0.3 \times 1200 \text{ mm}$) kept in a water bath thermostated at $85 \pm 1^\circ\text{C}$. Finally, the fluorescence is detected by the LCW cell; a silica capillary ($7\text{--}10 \text{ cm}$ long, $100 \mu\text{m}$ i.d.) is connected to the cell exit to apply backpressure.

3. Results and discussion

3.1. Excitation source

The maximum excitation wavelength of the fluorescent product from OPA-sulfite- NH_3 system is 365 nm in the pH 11.0 phosphate buffer. A miniature blacklight type Hg line source emitting at 365 nm is perfectly matched for this application. In operation, the lamp appears to be stable such that ratioing the fluorescence signal versus the lamp intensity was not found to be necessary.

3.2. Prevention of bubbles

In a flow system heated as much as 85°C , there is a high likelihood of bubble formation unless

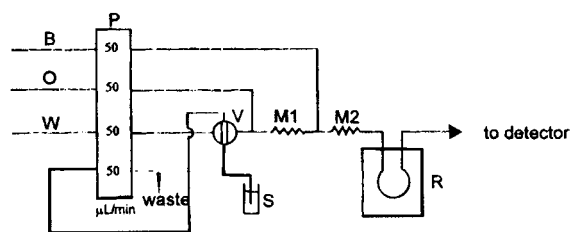


Fig. 3. Flow injection manifold for the determination of $\text{NH}_3/\text{NH}_4^+$. P, peristaltic pump; B, phosphate buffered sulfite; O, *o*-phthalaldehyde; W, water; V, rotary loop injection valve; S, sample; M1, 2, knotted mixing coils; R, heated reactor, thermostated at 85°C .

preventive measures are taken. The presence of a bubble in an LCW cell causes serious problems in much the same way bubbles are problematic in any other optical detection system. It is interesting to note however, that the precise effect of a bubble in the present detector system depends on the position of the bubble. When the bubble is located in the illuminated region of the LCW, it can scatter the excitation light directly to the light detector and thus result in a large background signal. When the bubble is located between the illuminated zone and the silica fiber, the bubble blocks the transmission of the emitted light and a low signal level is observed. For proper operation, the detector cell is built without any dead volume as far as practicable. The cell is positioned vertically with the exit on the top to prevent bubble entrapment. Most importantly, sufficient back pressure is placed at the cell exit to prevent bubble formation in the first place. Occasionally, a small bubble will stick nevertheless on the walls of the LCW tube or the connecting section of AF tube and fiber optics, causing a baseline shift. Such bubbles can be effectively removed by washing the cell with a low surface tension solvent like methanol.

3.3. Performance

The fluorescence intensity was linear with ammonia concentration between the range of 0.2–60 μM with a linear r^2 value of 0.9993. System output for 0.2–1.0 μM NH_4^+ is shown in Fig. 4. The relative standard deviation at the 200 nM level was 1.7% ($n = 11$). Repeated measurements at the 100 nM level are shown in Fig. 5. The baseline oscillations at this level are significant and we therefore conservatively estimate the $S/N = 3$ LOD to be 35 nM. The cause of the baseline variability at this level is not detector noise, but specific contributions from other sources could not be ascertained. Nevertheless, this performance is very comparable to the best LOD of 20 nM obtained with a commercial fluorometer with an optimized long-pass emission filter and a photomultiplier tube detector [18].

In conclusion, dedicated fluorescence detectors based on a transversely illuminated LCW consti-

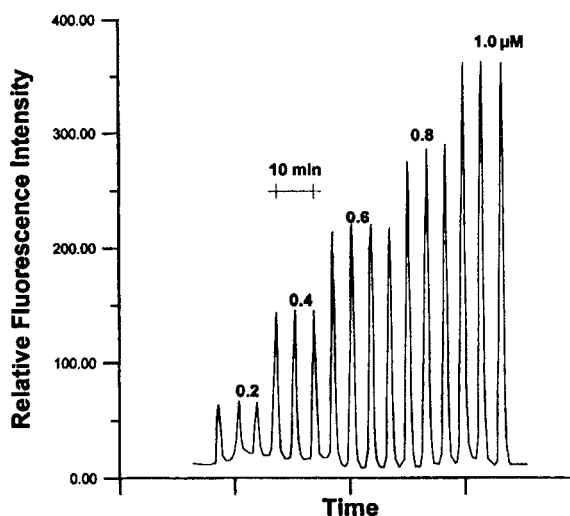


Fig. 4. Typical chart output for $\text{NH}_3/\text{NH}_4^+$ determination. The $\text{NH}_3/\text{NH}_4^+$ -N concentration of the sample in μM is indicated.

tute simple, sensitive, miniature affordable instruments that are especially amenable to portable and flow-through applications. It is remarkable that this photodiode based detector, with a hardware cost more than an order of magnitude less than that of a commercial filter fluorometer, can provide almost the same performance.

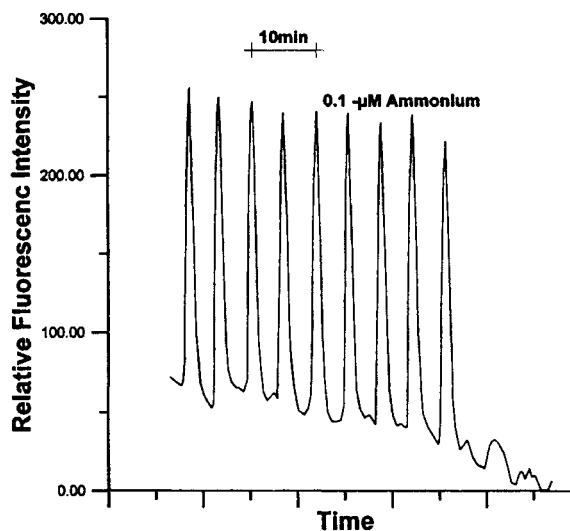


Fig. 5. Typical system output for 0.1 μM ammonium and the background noise level.

References

- [1] P.K. Dasgupta, *Anal. Chem.* 56 (1984) 1401–1403.
- [2] K. Tsunoda, A. Nomura, J. Yamada, S. Nishi, *Appl. Spectrosc.* 44 (1990) 163–165.
- [3] K. Fujiwara, T. Nakamura, T. Kashima, *Appl. Spectrosc.* 44 (1990) 1084.
- [4] K. Fuwa, W. Lei, K. Fujiwara, *Anal. Chem.* 56 (1984) 1640–1644.
- [5] K. Hong, L.W. Burgess, *Proc. SPIE* 2293 (1994) 71–79.
- [6] Dupont Fluoroproducts, AF Teflon amorphous fluoropolymers technical information, Dupont Fluoroproducts, Wilmington, DE, February, 1997.
- [7] R.D. Waterbury, W. Yao, R.H. Byrne, *Anal. Chim. Acta* 357 (1997) 99.
- [8] W. Yao, R.H. Byrne, R.D. Waterbury, *Environ. Sci. Technol.* 32 (1998) 2646.
- [9] P.K. Dasgupta, Z. Genfa, S.K. Poruthoor, S. Caldwell, S. Dong, S.-Y. Liu, *Anal. Chem.* 70 (1998) 4661.
- [10] W. Yao, W.R.H. Byrne, *Talanta* 48 (1999) 277.
- [11] K. Fujiwara, S. Ito, *Trends Anal. Chem.* 10 (1991) 184.
- [12] K. Fujiwara, S. Ito, R.-E. Kojyo, H. Tsubota, R.L. Carter, *Appl. Spectrosc.* 46 (1992) 1032.
- [13] P.K. Dasgupta, Z. Genfa, J. Li, C.B. Boring, S. Jambunathan, R. Al-Horr, *Anal. Chem.* 71 (1999) 1400.
- [14] National Academy of Science, Ammonia, University Park Press: Baltimore, MD, 1979.
- [15] R.L. Poirot, P.R. Wishinski, *Atmos. Environ.* 20 (1986) 1457.
- [16] Standard Methods for examination of Water and Wastewater, 16th ed., American Public Health Association, Washington DC, 1985.
- [17] M. Roth, *Anal. Chem.* 43 (1971) 880.
- [18] Z. Genfa, P.K. Dasgupta, *Anal. Chem.* 61 (1989) 408.

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