

A novel optical sensor for uranium determination

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Abstract

A metal ion indicator, Alizarin Red S, was tested for its potential use in uranium selective optode membrane. The water-soluble indicator was lipophilized in the form of an ion pair with tetraoctylammonium bromide, and subsequently immobilized on a triacetyl cellulose membrane. The membrane responds to uranium ions, giving a color change from yellow to violet in acetate buffer pH 5. This optode has a linear range of $(1.70\text{--}18.7) \times 10^{-5}$ M of UO_2^{2+} ions with a limit of detection of 5×10^{-6} M. The response time of optode was within 6 min depending on the concentration of UO_2^{2+} ions. The sensor can readily be regenerated with hydrochloric acid solution (0.01 M). The optode is fully reversible. © 2004 Elsevier B.V. All rights reserved.

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1. Introduction

Many of the samples requiring analysis for uranium content are intensely radioactive and an analytical method for the constant monitoring for the uranyl ion in solution, amongst others, is desirable. A convenient method for the detection of these impurities in liquid effluents would help enormously in environmental monitoring of such waters and their management.

Optical sensors are suited to this type of application as they may easily be incorporated into low-cost, easy-to-use kits; yet at the same time, they can offer the selectivity and sensitivity necessary for environmental monitoring [1].

Uranyl ions in aqueous solution can be excited with laser and some methods based on time-resolved luminescence have been reported in literature. Wangen et al. [2] discussed the application of a fiber-optic-based time-resolved laser-induced luminescence sensor for uranium in the range of 10^{-4} to 10^{-9} M. The inorganic cation quenchers such as Fe^{2+} , Pb^{2+} and Ag^+ pose problems because they can permeate the Nafion membrane. It was also necessary that the flow optode was

equilibrated in a uranyl solution for 30 min before the signal was obtained [2]. Later, an optode was reported based on laser-induced fluorimetry capable of determining ultra low levels of uranium in a nuclear reprocessing plant [3]. Moulin et al. used time-resolved laser-induced fluorescence for uranium and nitrate remote sensing in the nuclear fuel cycle [4]. DeNeufville et al. examined a remote sensing of laser-induced UO_2^{2+} ion fluorescence as a potential indicator of uranium occurring in geological samples at the Earth's surface [5]. These approaches, although sensitive, suffer from the need for expensive instrumentation.

Arsenazo III is the most sensitive reagent for the spectrophotometric determination of uranium [6–8]. Several uranium-sensitive optical sensors have been reported in the literature, using this indicator. Petrova et al. used membranes made of various polyamide materials [9]. They used immobilized Arsenazo III on membrane discs for determination of uranium ion in batch ($0.1\text{--}2 \text{ mg L}^{-1}$) and dynamic modes ($0.01\text{--}0.10 \text{ mg L}^{-1}$). One of the shortcomings of this method was the long time of 35 min required for contacting the membrane with the uranyl ions in order to obtain a stable response [9]. Baylor and Buchanan applied Arsenazo III for construction of a remote probe for the detection of uranium in ground water [10]. Arsenazo III was affixed to a piece of cotton cloth

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and attached to an optical fiber for monitoring the change in reflectance when exposed to a uranium solution. The sensor was irreversible and the linear range was $0.5\text{--}0.1\text{ mg L}^{-1}$. The only interferences examined were zinc and humic acid. Collins et al. discussed the application of Arsenazo III to the remote spectrophotometric determination of very low levels of uranium (VI) in water ($<20\text{ ppb}$) [11]. Their device utilized a renewable reagent system by pumping the Arsenazo III reagent to a Nafion micro tube for sampling uranyl ions in groundwater, followed by delivery of the complexed cation to a light emitting diode (LED)-based fiber-optic flow cell for monitoring uranium. The response time reported was 20 min for diffusion of uranyl ions into the membrane. Calcium and thorium were the main interferences. Shvoeva et al. prepared a sensor for uranium (VI) from polyacrylonitrile fibre impregnated with a finely dispersed anion-exchange resin immobilized by Arsenazo I [12]. Miller and Hirschfeld presented a review in fiber optic chemical sensors for industrial and process control [13].

However, one of the main shortcoming of most of the reported uranyl ion optical sensors is their long response time. Thus, sensitive and simple uranium optodes with relatively short response times are still demanding.

The indicators were originally developed for use in water solutions. Their immobilization into or onto a solid support is a key issue for their application in optical sensing [14]. A number of techniques have been developed for the immobilization of water-soluble indicator dyes into hydrophobic matrices [14–16].

The most commonly used method is lipophilization of the dye molecule, by chemical modifications through synthesis of a lipophilic derivative of the dye [14,17].

Alizarin Red S is a metallochromic ligand that selectively reacts with the actinide and lanthanide metal ion series [18]. We have prepared and characterized an optical sensor membrane on triacetylcellulose based on the lipophilic form of Alizarin Red S in order to evaluate its potential use for uranium ion sensing. The reaction takes place within few minutes and a color change occurs from yellow to violet, which is spectrophotometrically detected.

2. Experimental

2.1. Reagents

All reagents were the best available analytical reagent grade. Alizarin Red S was obtained from Merck. Tetraoctylammonium bromide (TOABr), dimethylformamide (DMF) and ethylene diamine were also obtained from Merck.

A stock solution of $10^{-2}\text{ M UO}_2^{2+}$ ion was prepared by dissolving 0.2511 g of $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (BDH) in 50 ml of buffer solution. Working standard solutions of lower concentrations were prepared by suitable dilution of the stock solution. Different buffer solutions were prepared from either citric acid, acetic acid, (tris-hydroxymethyl)amino-methane

and KHP and pH adjustments were made with hydrochloric acid (1 M) and sodium hydroxide solution (1 M) to achieve the desired pH.

2.2. Apparatus

A Metrohm 632 pH-meter with a Metrohm double junction glass electrode was used for pH adjustment. A Shimadzu 1601PC UV–vis spectrophotometer was used for recording the visible spectra and absorbance measurements. The sensing membrane was placed and fixed in a disposable plastic cuvette and all measurements were performed in a batch mode.

The membrane was first exposed to the buffer solution and the absorbance was measured at 547 nm. Then the sample solution was added and the absorbance at 547 nm was again measured after 10 min.

2.3. Synthesis of the ion pair

The Alizarin Red S-TOABr ion pair (Fig. 1) was prepared by the following procedure. Alizarin Red S (0.5 g) and 0.5 g of TOABr were separately dissolved in 5 ml of dimethylformamide and then mixed. Then 1 ml of 0.1 M HCl was added to the mixture. At pH 3, only the sulphonic group is dissociated, and thus is capable of forming an ion pair, whilst the chelate-forming groups are protected. Dilution of the mixture with water results in the precipitation of the ion pair in the form of a viscous oil. The ion pair was extracted by dichloromethane and dried at 70°C .

2.4. Preparation of the sensor membrane

The immobilized indicator on triacetylcellulose was prepared according to the following procedure [19].

The transparent triacetylcellulose membranes were produced from waste photographic film tapes that were previously treated with commercial sodium hypochlorite

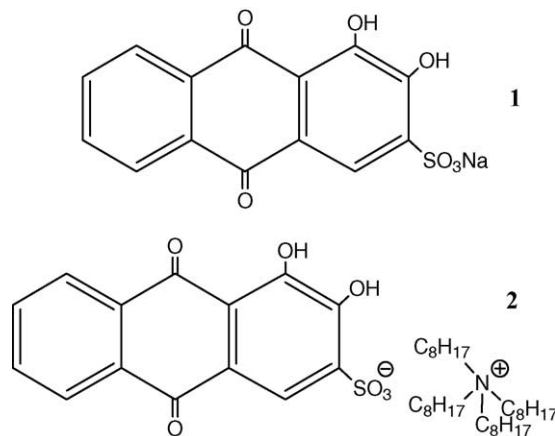


Fig. 1. Chemical structure of Alizarin Red S [1] and the Alizarin Red S-tetraoctylammonium ion pair [2].

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