



Selective removal of carbon dioxide contained in the effluent from ion chromatography suppressors using a new non-vacuum device



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ABSTRACT

In this paper, a new CO₂ gas removal device optimized to selectively remove CO₂ gas contained in the effluent from suppressors used in ion chromatography (IC) under non-vacuum conditions is described. This device consists of a closed vessel equipped with gas permeable tubing (GPT) and a CO₂ adsorbent. During operation, the CO₂ adsorbent adsorbs CO₂ gas in the vessel, creating CO₂ partial pressure difference between the inside of the GPT and the vessel. The CO₂ gas contained in the effluent being pumped into the GPT is selectively removed from the effluent based on the diffusion of the CO₂ associated with the CO₂ partial pressure difference. The purpose of this study is to optimize the IC operating conditions with the aim of selectively removing HCO₃⁻ (CO₃²⁻) contained in the effluent and reducing the electrical conductivity of the effluent under non-vacuum conditions. The electrical conductivity of the effluent and the signal intensity of the water dip is decreased by approximately 25 μS/cm (from 30 to 5 μS/cm) and by approximately twentieth, respectively, using the optimized CO₂ remover. In addition, the anion detection limit achieved in IC instruments with a CO₂ remover is on the order of a few ppb.

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1. Introduction

In 1975, Small et al. introduced ion chromatography (IC) [1], which is now widely used in engineering, science, as well as other fields. Most ion chromatographs are equipped with a suppressor and an electrical conductivity detector (ECD). Alkaline hydroxide solutions, such as LiOH, NaOH, and KOH, and carbonate buffer solutions have been mainly used as the eluents for IC anion analysis. Although the detection sensitivity is improved using the hydroxide eluents, the preparation and maintenance of pure hydroxide eluents is very difficult [2]. While an ion chromatograph equipped with an in-line hydroxide generator has been reported [3], the system is very expensive. Because the sensitivity of IC with the carbonate buffer eluents is lower than that with hydroxide eluents, a post-suppression degassing method for the removal of the CO₂ contained in the effluent from the suppressor [4–7] was developed in the 1980s for improving the detection performance. However, because this method uses gas permeable tubing (GPT), such as a silicone rubber [8,13] or poly(tetrafluoroethylene) (PTFE) [4,6,7], it has two problems. First, the peak band of the sample eluted

from the separation column is broadened by diffusion through the GPT. Second, the GPT is constrained by its pressure and chemical resistance. In 2002, Saari-Nordhaus *et al.* reported a new post-suppression degassing device using Teflon AF tubing, which was developed in the 1990s, for the GPT to overcome these problems [9–16]. This device consisted of a suppressor and a CO₂ remover. The suppressor removed the cations contained in the effluent, and the HCO₃⁻-(CO₃²⁻) in the effluent was converted to CO₂ (H₂CO₃) gas. The CO₂ remover consisted of a closed vessel equipped with Teflon AF tubing and a CO₂ adsorbent. The CO₂ gas was selectively removed from the suppressed effluent via the diffusion of CO₂ from the inside of the GPT to the vessel, due to the CO₂ partial pressure difference between the inside of the GPT and the vessel created by adsorption of CO₂ gas in the vessel by the CO₂ adsorbent. Although the properties of the CO₂ remover, such as the water content of the adsorbent, the temperature, and the quantity of the adsorbent, possibly have an effect on the amount of CO₂ removed, the paper by Saari-Nordhaus *et al.* does not discuss these concerns in any detail.

This paper describes the introduction of a practicable CO₂ gas removal device existing of a commercially available polypropylene-nonwoven fabrics pack (PNFP) for tea leaves, which is packed with a substantially dry OH-type anion-exchange (AE) resin. The purpose of this study is to optimize the operating conditions, including the temperature, quantity of adsorbent, *etc.*, with the aim of selectively

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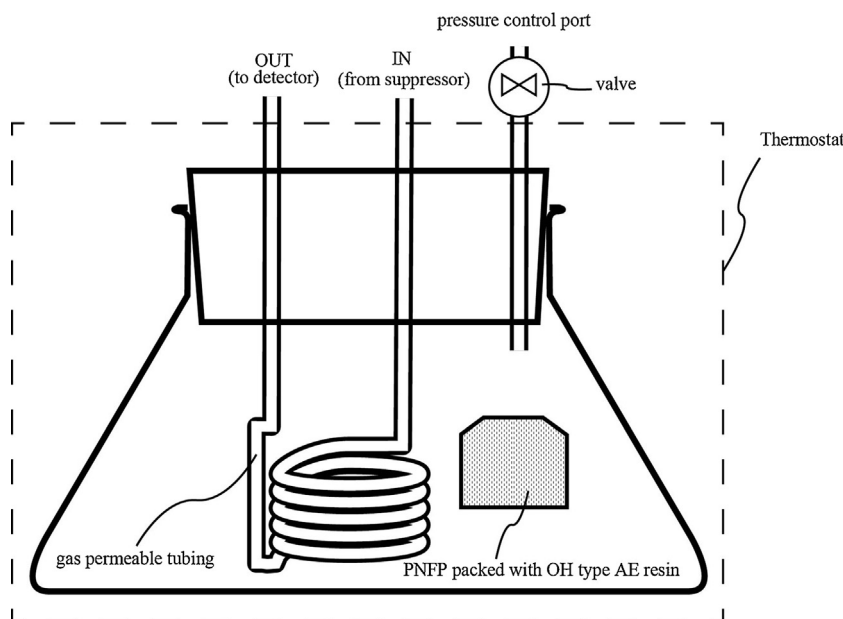


Fig. 1. Schematic of a CO₂ remover.

removing HCO₃⁻ (CO₃²⁻) contained in the effluent and reducing the electrical conductivity of the effluent under non-vacuum conditions.

2. Materials and methods

2.1. Preparation of the OH-type anion exchange resin

Amberlite IRA402BL (Cl type, Dow Chemical, 0.50–0.65 mm, 1.25 eq/L) was obtained from Organo Corp. The Cl type IRA402BL was exchanged to an OH-type using aqueous 2–4% NaOH as the regeneration solution. The exchanged resin was washed with pure water produced using an SA-2000E1 (<1 μS/cm, EYELA), then was with acetone, and substantially dried using a reduced-pressure drying method.

2.2. CO₂ adsorbent

A polypropylene-nonwoven fabric pack (PNFP) packed with the substantially dried OH-type AE resin was used as the CO₂ adsorbent. The quantity of resin packed in PNFP was 20 g, unless otherwise indicated.

2.3. CO₂ remover

A diagram of the CO₂ remover is shown in Fig. 1. The device consists of a closed vessel (conical flask, 300 mL) equipped with a GPT (AF2400 tubing, I.D.: 0.25 mm, O.D.: 0.5 mm, length: 4 m, U-VIX Corp., Tokyo, Japan) and the PNFP packed with AE resin. In addition, because the pressure increases as the temperature in the vessel increases, the vessel is equipped with a port and a valve for release of the internal pressure such that atmospheric pressure can be maintained. The temperature in the vessel is controlled using a heater installed outside of the vessel. In the present study, the temperature in vessel was 37 °C unless otherwise indicated. The valve was left open until the temperature of the vessel reached the set temperature and then closed.

2.4. System for estimation of CO₂ removal

A diagram of the system used to estimate the CO₂ removal is shown in Fig. 2. The system consists of a pump (1 mL/min, DP-8020, Tosoh, Tokyo, Japan), a suppressor (SA8010 [18], Nichiri, Chiba, Japan), a CO₂ remover, and an ECD (ICA-2000, TOA DKK, Tokyo, Japan). Two types of carbonate buffer solutions (1 L) were prepared and pumped into the system: type (A) solution consisted of 12 mM NaHCO₃ + 0.6 mM Na₂CO₃ (ca. 850 μS/cm, pH 8.9) and could not represent the HCO₃⁻ (CO₃²⁻) peak contained in the sample; type (B) solution was comprised of 1.8 mM Na₂CO₃ + 1.7 mM NaHCO₃ (ca. 435 μS/cm, pH 10), which is commonly used as a carbonate eluent for IC. The Na ions were removed from the pumped carbonate buffers with the aid of the suppressor, and then CO₂ gas was removed from the suppressor effluent with the aid of the CO₂ remover. The electrical conductivity of the CO₂ remover effluent was detected using the ECD.

First, the quantity of OH-type AE resin required for 12 h of continuous operation was determined by measuring the electrical conductivity of the effluent from the CO₂ remover containing a PNFP packed with different amounts of OH-type AE resin amounts (1, 2, 5, and 10 g). Second, the effect of the water content in the OH-type AE resin on the adsorption of CO₂ gas in the vessel was investigated by measuring the electrical conductivity of the effluent from the CO₂ remover when different quantities (1, 2, 5, and 10 mL) of pure water were added to 10 g of the OH-type AE resin packed in the PNFP. Third, the effect of the GPT length on the removal of CO₂ from the suppressor effluent pumped into the GPT was investigated by measuring the electrical conductivity of the effluent from the CO₂ remover using GPTs of varying lengths (0, 1, 2, 3, 4, and 5 m). Fourth, the effects of the PNFP and the temperature in the vessel on the removal of CO₂ from the suppressor effluent were investigated by measuring the electrical conductivity of the effluent from the CO₂ remover when the pressure and the temperature in the vessel without the PNFP were 7.9 kPa and 50 °C and with the PNFP were 100 kPa and 50 °C and 100 kPa and 80 °C, respectively. Finally, the decrease in the electrical conductivity using the CO₂ remover under optimized conditions was calculated by determining the difference in the electrical conductivities with and without the optimized CO₂ remover.

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