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Analysis of trace impurities in neon by a customized gas chromatography

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ABSTRACT

Excimer lasers, widely used in the semiconductor industry, are crucial for analyzing the purity of premix laser gases for the purpose of controlling stable laser output power. In this study, we designed a system for analyzing impurities in pure neon (Ne) base gas by customized GC. Impurities in pure neon (H_2 and He), which cannot be analyzed at the sub-μmol/mol level using commercial GC detectors, were analyzed by a customized pulsed-discharge Ne ionization detector (PDNeD) and a pressurized injection thermal conductivity detector using Ne as the carrier gas (Pres. Inj. Ne-TCD). From the results, trace species in Ne were identified with the following detection limits: H_2, 0.378 μmol/mol; O_2, 0.119 μmol/mol; CH_4, 0.880 μmol/mol; CO, 0.263 μmol/mol; CO_2, 0.162 μmol/mol (PDNeD); and He, 0.190 μmol/mol (Pres. Inj. Ne-TCD). This PDNeD and pressurized injection Ne-TCD technique thus developed permit the quantification of trace impurities present in high-purity Ne.

1. Introduction

Neon (Ne), obtained by air liquefaction, is separated from other gases by fractional distillation; it is used for making common Ne advertising signs, accounting for its highest use [1]. Recently, Ne has been widely used in the semiconductor industry; high-purity Ne has been widely used as a buffer gas in excimer lasers, which are widely used in high-resolution photolithography machines; high-resolution photolithography is one of the critical technologies for the manufacturing of microelectronic chips.

Instead of helium (He), when Ne is used as a buffer gas in an excimer or excimer-like laser [2], output power and laser efficiency substantially increase. However, Ne contains trace contaminants from extreme ultraviolet (EUV) absorbers, particularly, H_2, N_2, O_2, H_2O, and CO_2; these absorbers decrease the number of the photons detected from any scattering event via the quenching of excited Ne_2 molecules or by the absorption of scintillation light [3]. With further increase in impurity concentration, lasing decreases and breaks off [4,5]. High-purity Ne also contains impurities, such as H_2O, H_2, O_2, N_2, CH_4, CO, CO_2, He, and C_nH_m, which either degrade the performance of the excimer laser machine used in the semiconductor process or shorten its life. Particularly, when a Ne purifier is used, most of these impurities can be reduced to the nmol/mol levels. However, He cannot be purified because it is inert.

Typically, GC equipped with a pulsed-discharge helium ionization detector (PDHeD) using a helium-discharge ionization detector (HDID) is employed for analyzing the impurities in high-purity Ne [6–10]. However, both high-concentration Ne and H_2 are eluted at the same retention time; hence, it is difficult to analyze H_2 without using a cryogenically cooled column. Furthermore, He impurities present in Ne cannot be measured by a PDHeD. Atmospheric pressure ionization mass spectrometry (APIMS) can also analyze impurities present in Ne [11–13]. However, it is expensive, thereby not feasible for industrial gas applications.

In this paper, we designed a new GC method, with a GC system comprising a pulsed-discharge Ne ionization detector (PDNeD) and a thermal conductivity detector (TCD) with a pressurized loop injector. Ne used as the carrier gas was equipped with a sample introduction device without air contamination and a data acquisition computer. This self-modified GC system can be used for analyzing H_2, O_2, N_2, CH_4, CO, CO_2, and He in Ne at room temperature by a single injection.

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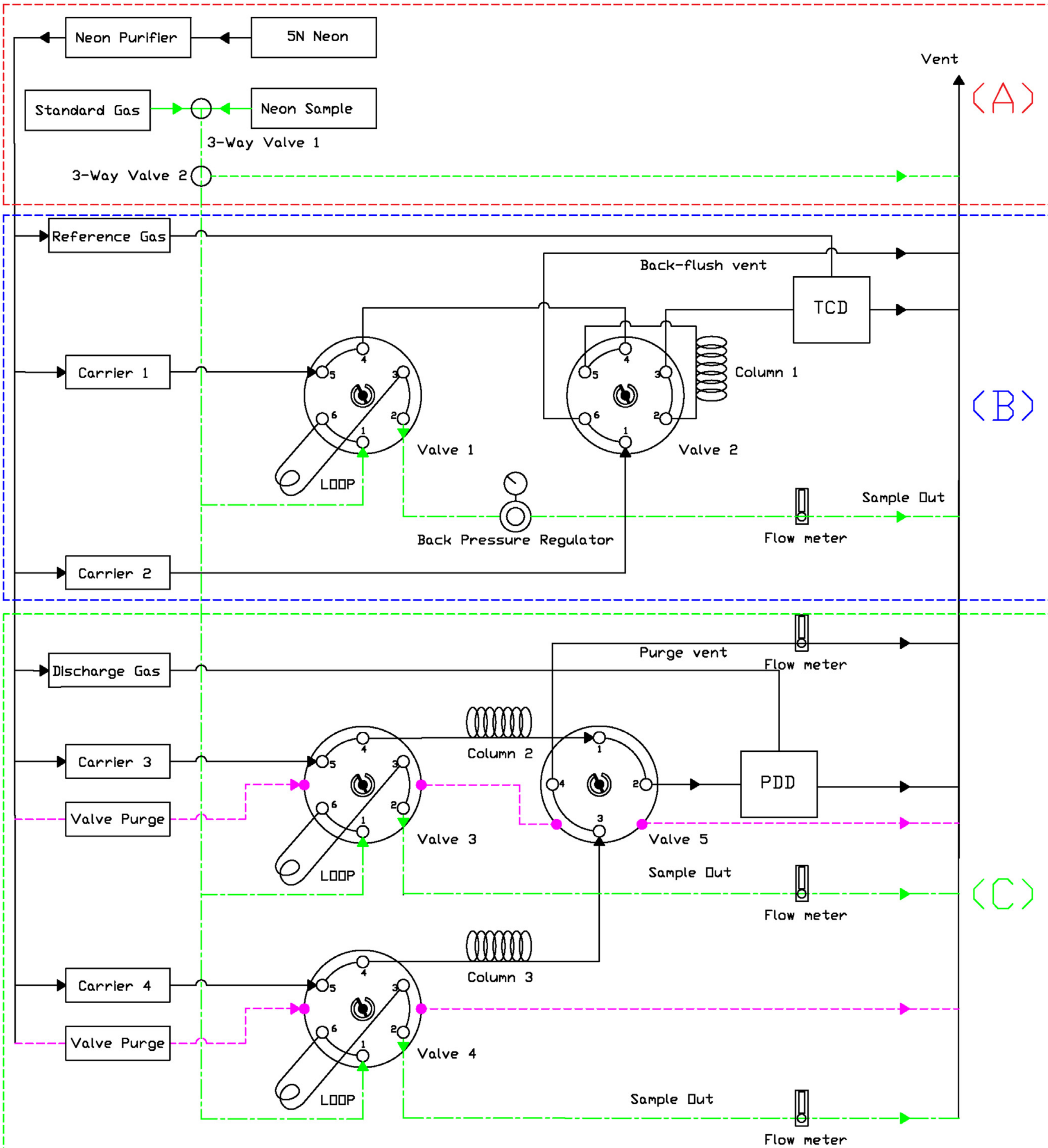

Fig. 1. Schematic of the plumbing, valves, and column configuration of the GC system analyzing high-purity Ne. (A), (B), and (C) are the sample introduction stage, TCD side, and PDNeD side, respectively.

2. Materials and methods

2.1. Reagents

For testing the reliability of analyzing Ne impurities by the GC system with two detectors, PDNeD and TCD with Ne carrier gas, few standard mixtures were prepared: helium pure gas with 5 µmol/mol of impurities, such as H_2, O_2, N_2, CH_4, CO, and CO_2 (Cylinder #: D232844) and two nitrogen gases with 5.04 µmol/mol of helium(Cylinder #: ES0001321) and 109.76 µmol/mol of helium(Cylinder #: ME5380). produced by the KRISS(Korea Research Institute of Standards and Science, Daejon, Korea). As well as Ne pure gas with 10 µmol/mol of H_2, O_2, N_2, CH_4, CO, and CO_2 (Cylinder #: NK30803) and Pure Ne gas (Cylinder # HK2871) was produced by Deokyang (Deokayng Co., Ltd. Ulsan, Korea).