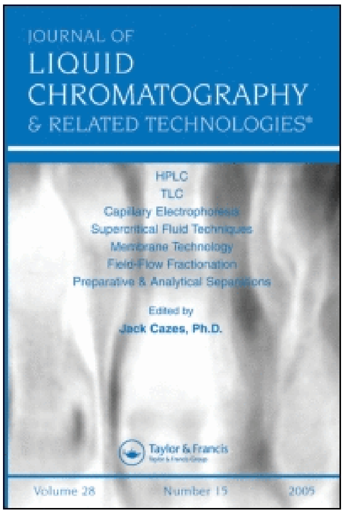

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HIGH PERFORMANCE LIQUID CHROMATOGRAPHIC DETERMINATION OF N-METHYL-2-PYRROLIDONE IN WATER

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ABSTRACT

N-Methyl-2-pyrrolidone was determined in water using High Performance Liquid Chromatography. A determination required less than 10 minutes using an octadecyl reversed phase column and isocratic elution with methanol:water (10/90, V/V). Detection was at 205 nm. Sub-part-per-billion concentrations were determined without preconcentration by injecting a 500 µL sample into the chromatograph.

INTRODUCTION

N-Methyl-2-pyrrolidone (NMP) is a versatile solvent (1-5). It is very miscible with water and is recommended for use as a solvent for pesticides (1), resins (2), vinyl coatings (3), or as a general reaction solvent (4). NMP is also recommended as a solvent for some inorganic salts (5).

Since NMP is recommended for so many uses, sensitive methods are needed to determine its concentration in a variety of matrices. Methods used to determine NMP include Thin Layer Chromatography (6,7) UV Spectrometry (8) and Gas Chromatography (9-11). Scoggins and Skurcenski (11) developed a G.C. method to determine the NMP in waste water. Although that method is sensitive, the method requires preconcentration of NMP prior to its GC determination. The HPLC procedure outlined in this paper is easy, rapid and requires no sample preconcentration prior to NMP determination.

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EXPERIMENTAL

Reagents and Apparatus: N-Methylpyrrolidone (Burdick and Jackson Laboratories, Inc., Muskegon, MI), HPLC grade methanol and HPLC grade water (Fisher Scientific, Pittsburgh, PA) were used without further purification. The HPLC apparatus consisted of a Waters Associates (Medford, MA) model M6000A pump and a model 450 variable wavelength detector. A 4.5 x 250 mm, 5 µm, C-18 column (IBM Instrument, Yalesville, CT) was used. A Rheodyne (Cotati, CA) model 7125 injector equipped with either a 100 µL or 500 µL sample loop was used. Chromatographic peaks were recorded on a Hewlett-Packard (Avondale, PA) model 3380A integrator. Signals were filtered through a Spectrum Scientific Corp. (Newark, DE) 1021A filter prior to being sent to the recorder.

Chromatographic Conditions: The mobile phase consisted of 10% methanol in water. A flow rate of 1.5 mL minute^{-1} was used and detection was at 205 nm. The electronic filter was set at a cut-off frequency of 0.02 Hertz and a gain of 1. The parameters set on the integrator were a sensitivity of 0.30 mV/V and an attenuation of 8.

RESULTS AND DISCUSSION

Trace analysis using High Performance Liquid Chromatography is plagued by noise problems. Baseline noise can limit the sensitivity of a technique. Noise is minimized in the method outlined in this paper through the use of an electronic filter. It was found that as a cut-off frequency of 0.02 Hertz, the optimum filtering was obtained. At a higher cut-off frequency, peaks were integrated in an inconsistent manner as noise spikes sometimes triggered the beginning or ending of peak integrations, while at a lower cut-off frequency, peak shapes were distroted.

NMP was detected adequately at 205 nm. Peak areas were increased by decreasing the wavelength, but the noise level was increased beyond the amount that could be filtered out by the electronic filter. Above 205 nm, the sensitivity decreased. The

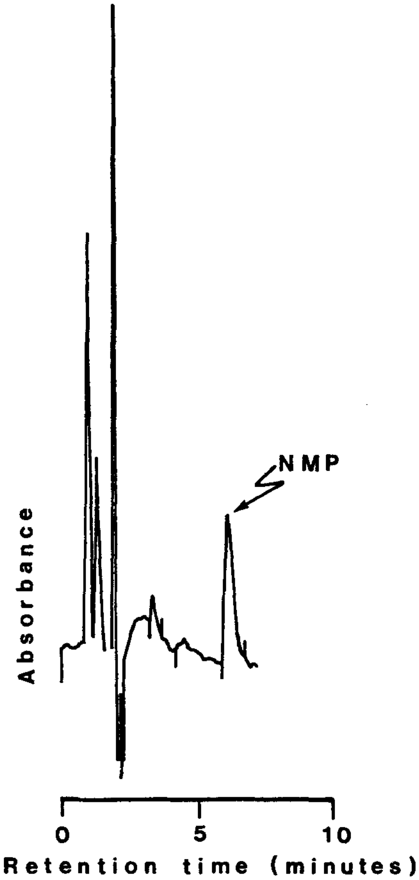

Figure 1. Chromatogram of 100 ppb of N-methyl-2-pyrrolidone (NMP) in water. Chromatographic conditions: 100 µL injection, isocratic elution with methanol:water (10/90, V/V), separation on a C-18 column, detection at 205 nm. The tic marks indicate the beginnings (negative) and endings (positive) of integrations. No attempt was made to determine the identities of the other peaks in the chromatogram.

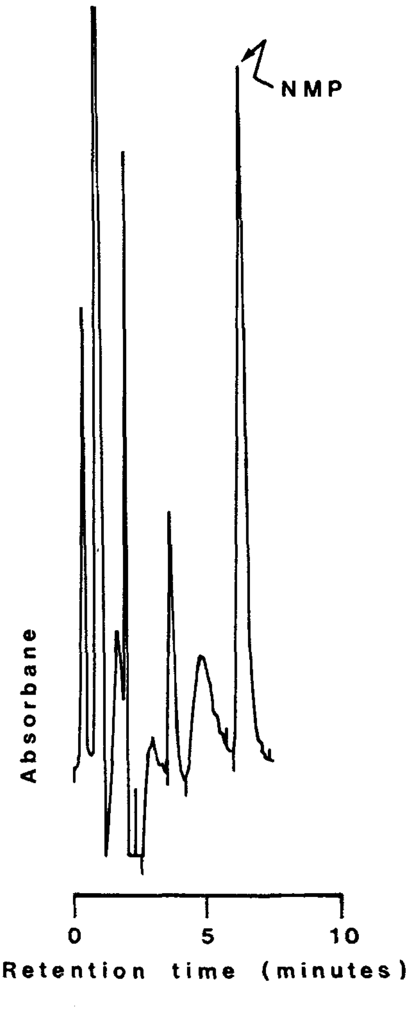

Figure 2. Chromatogram of 100 ppb of N-methyl-2-pyrrolidone (NMP) in water. Chromatographic conditions: 500 µL injection, all other conditions the same as Figure 1.

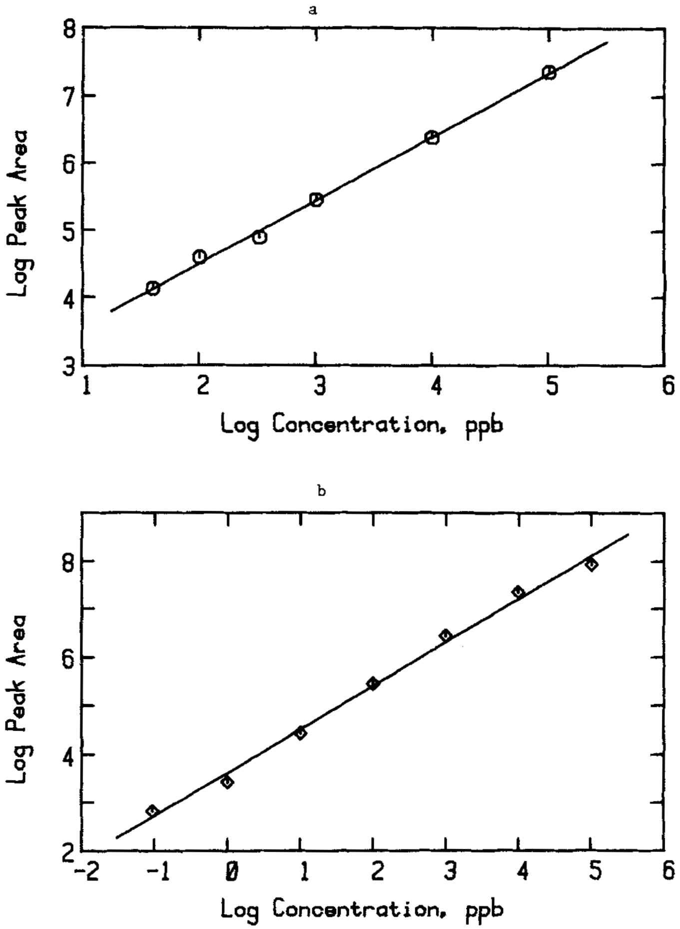

Figure 3. Calibration graphs for the determination of N-methyl-2-pyrrolidone in water. (a) 100 µL injection, (b) 500 µL injection. Chromatographic conditions as outlined in Figure 1. Each point represents the average of three or four determinations.

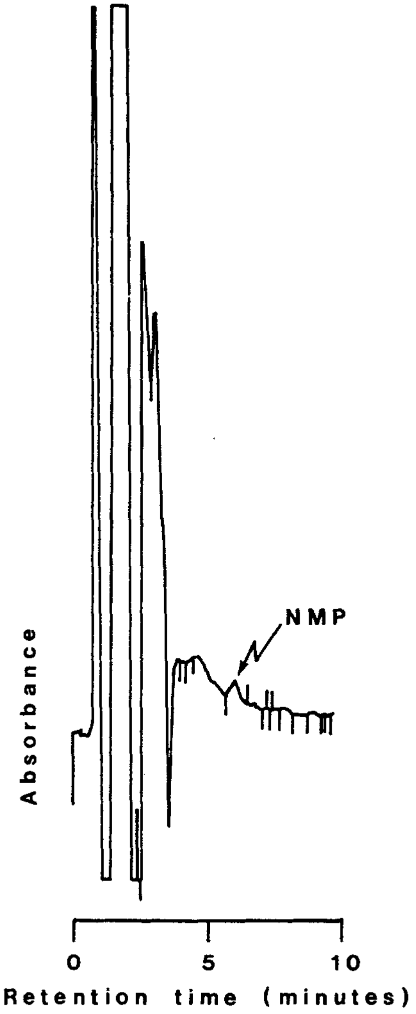

Figure 4. Chromatogram of 2 ppb N-methyl-2-pyrrolidone (NMP) in a cooling water sample. Chromatographic conditions are the same as in Figure 2.

assignment of the NMP peak on the chromatogram was done through a comparison of peak areas for a series of standards.

Initial work utilized a 100 µL injection volume to determine NMP. It was found, however, the signal using that sample size was not sufficient for the concentration of NMP in a real sample. By increasing the injection volume to 500 µL, an acceptable signal was obtained. Chromatograms of 100 ppb of NMP in water determined using 100 µL and 500 µL injection volumes are presented in Figures 1 and 2. It is seen that the 500 µL injection resulted in a significantly higher absorbance for the NMP peak.

Calibration graphs for both injection volumes are found in Figure 3. The 100 µL injection volume yielded a linear response over more than three orders of magnitude with a correlation coefficient of 0.998. The 500 µL injection volume was tested over six orders of magnitude and the response was also linear with a correlation coefficient of 0.997.

The method was tested by applying it to a sample of cooling water from a resin tank reactor. This sample was determined to contain 2 ppb NMP. A representative chromatogram of this sample is presented in Figure 4.

CONCLUSIONS

High Performance Liquid Chromatography has been shown to be an effective technique in determining trace amounts of N-methyl-2-pyrrolidone in water. Using injection volumes of 100 µL or 500 uL yielded calibration graphs with linear responses from 0.1 ppb to 100 ppm.

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