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Ammonium-N Determination by Including Dialysis with Technicon AutoAnalyzer II.

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Abstract

Inclusion of original dialysis system with Technicon AutoAnalyzer II did not show smooth flow which resulted in non reproducible peaks. Air flow across the dialysis membrane disturbed the air bubble pattern. The chemistry of main Ammonium-N system was affected by 0.1M HCl in the acceptor stream. Therefore, 0.01M hydrochloric acid was found suitable in the accepter stream of the dialysis.

Introduction

The ammonium-N can be separated from interferences either by distillation or gas phase dialysis. Crowther and Evans (1980) proposed an automated distillation-spectrophotometry procedure for determining ammonium in water describing that the distillation pre-treatment step for ammonium analysis was automated and coupled with an automated phenate-hypochlorite spectrophotometric step. Two buffers (Phosphate, pH 7 and borate, pH 9.5) were evaluated as pH controls for the distillation step.

The gas dialysis using a polytetrafluroethene (PTFE) gas permeable membrane as pre-treatment in the determination of nitrogen by flow injection analysis (FIA) has become a method of wide use in the research laboratories throughout the world.

Aoki *et al.* (1986) used tubular microporous PTFE in a continuous flow system. They separated the ammonia generated in alkaline sample solution with this membrane. Hara *et al.* (1988) used gas dialysis to the determination of residual concentration of ammonium ions in water purified by distillation or deioninsation and to natural water analysis. Schulze *et al.* (1988) proposed that gas separation is more satisfactory for determining sub-micromolar levels of ammonium in sea water, certain surface waters, arctic and glacial ice. Similarly, Nakata *et al.* (1988) found that bromocresol purple (pH 6.8) as acceptor solution gives the maximum sensitivity in the flow injection analysis system. Keeping in view the need of dialysis system as a pre-treatment step with Technicon AutoAnalyzer II, the study was undertaken to see its effect on Ammonium-N determination.

Materials and Methods

Experiment was carried out to investigate the effect of inclusion of dialysis system with Technicon AutoAnalyzer II on ammonium-N determination in the Department of AFE, University of Glasgow. The glassware used in this experiment was first washed with hot water and soaked overnight in a 2 percent solution of Decon 90 (Decon Laboratories limited). These were then washed with hot water, rinsed twice with deionized water and finally dried in an oven at 70 C.

Ammonium nitrogen was measured by a modification of the indophenol green method using a complexing reagent to prevent interferences due to the precipitation of hydroxides in the reagent system. With the inclusion of a sodium nitroprusside catalyst, the sensitivity of the method was such that ammonium could be determined in the range of 0 to 1 ppm and with care 0 to 0.1 ppm (Brown, 1973). This method is applicable to water samples and a wide range of soil extractant solutions, acid digests of plant or soil material. The schematic diagram of the flow system is shown in Figure 1 . The dialysis system was included with AutoAnalyzer Manifold for determining NH_4-N as shown in Fig. 2.

Reagents: Analar grade reagents and deionized water were used throughout.

Alkaline phenol: Sodium hydroxide (22.5 g) was dissolved in about 800 ml deionized water in 1 litre dark glass bottle and the resulting solution was degassed. Fifty gram phenol was weighed in 1 litre beaker and approximately 600 ml sodium hydroxide solution was added and stirred with glass rod to dissolve the phenol. The solution was returned to the bottle and the volume was made to 1 litre with degassed water and mixed gently.

Complexing reagent: Fifty gram potassium sodium tartrate and 50 g sodium citrate were dissolved in 800 ml deionized water and degassed. Sodium nitroprusside (1.2 g) was weighed in a 100 ml beaker. Fifty ml of degassed water was added to the beaker and stirred gently with a magnetic stirrer. The resulting solution was added to the citrate-tartrate solution. Thirty percent Brij-35 (0.5 ml) was added and volume was made to 1 litre. The solution was then mixed gently.

Sodium hypochlorite solution (0.5%): Fifty ml sodium hypochlorite solution (12% w/v available chlorine) was diluted to 1 litre with degassed deionized water and mixed gently.

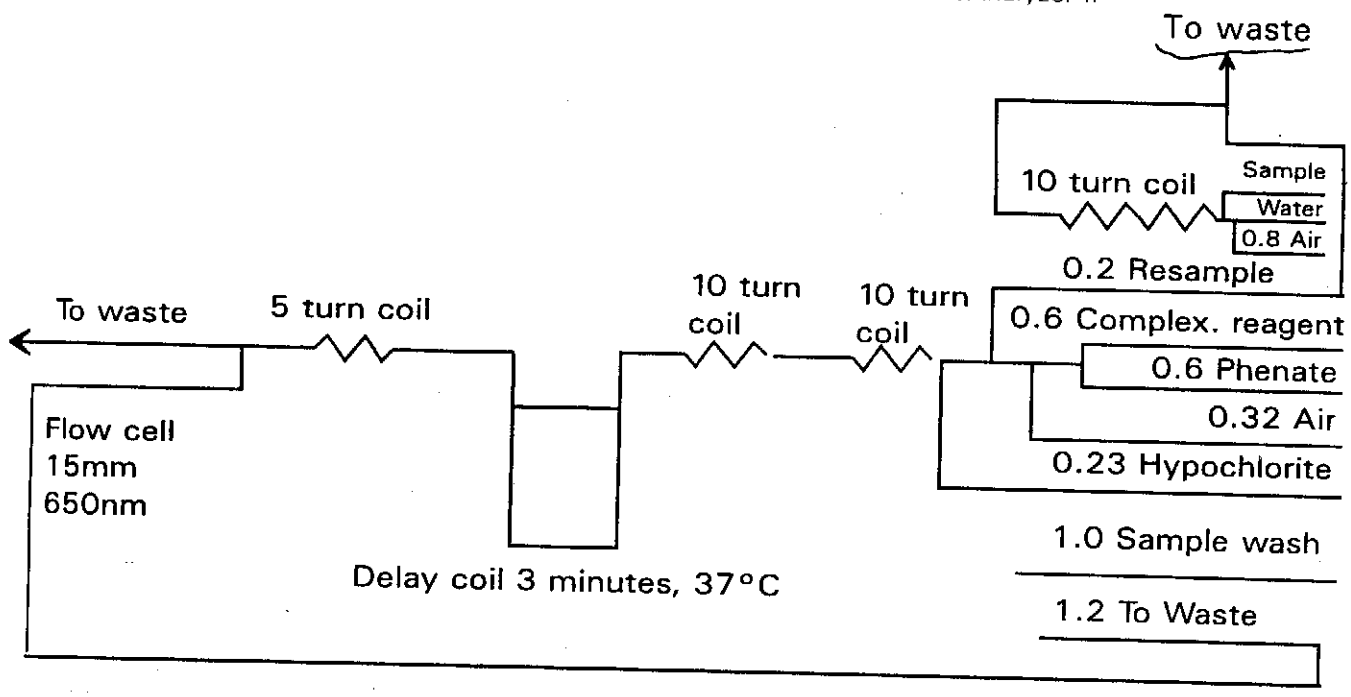

Fig. 1: AutoAnalyzer manifold for determining NH_4-H

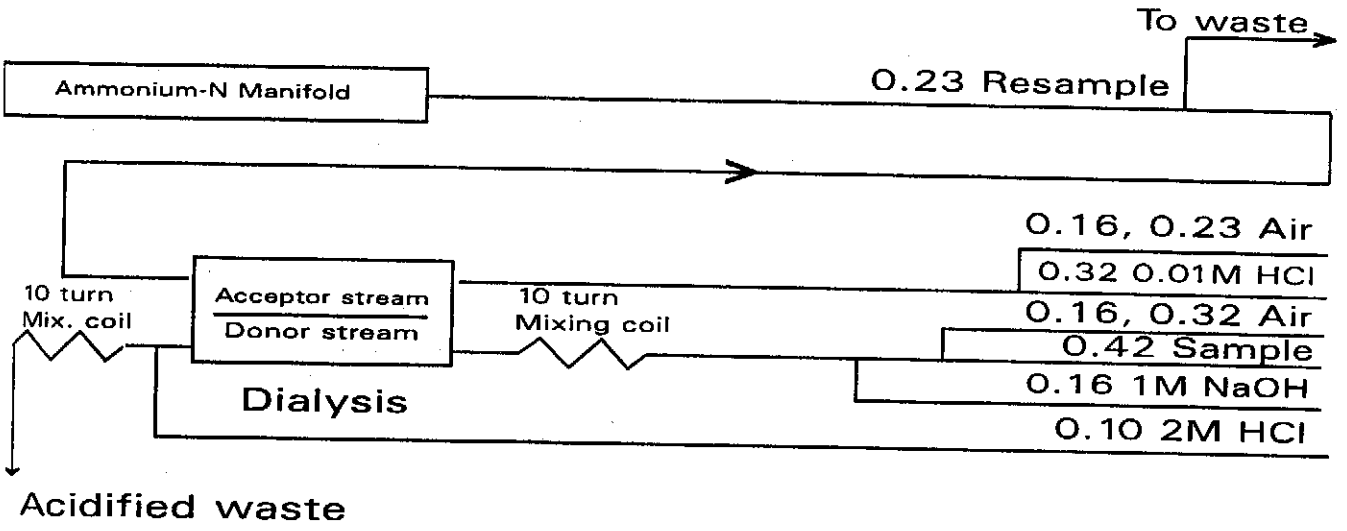

Fig. 2: Original dialysis system

Ammonium-N standard stock solution (1000 mg N l^{-1}): Ammonium sulphate was dried for an hour at 110 °C in the oven and cooled in a desiccator. Dried ammonium sulphate (4.718 g) was dissolved in deionized water and the volume was made to 1 litre. The solution was stored at 2°C. Working standards were prepared by dilution in the appropriate extracting solutions.

Working solutions of organic nitrogen compounds: The required weights of 10 amino acids, galactosamine, glucosamine and urea were transferred to 25 ml volumetric flasks carefully and volume was made to the mark with deionized water. The strength of each solution was 1000 mg N l^{-1}

Ammonium-N working solution (100 mg N l^{-1}): Ten ml of 1000 mg l^{-1} NH4-N stock solution was pippeted into 100 ml flask by a bulb pipette and the volume was made with deionized water up to the mark.

Ammonium-N working standards (1 mg N l^{-1}): One ml of 100 mg l^{-1} NH4-N working solution was added to each of three 100 ml volumetric flasks and the volume was made with deionized water, 2M KCl and 0.5M K_2SO_4, respectively.

Zero ammonium-N working solutions: Deionized water, 2M KCl and 0.5M K_2SO_4 were analysed as zero ammonium-N solutions with their appropriate set of samples.

Organic compound samples without added ammonium-N: One ml of each organic nitrogen compound stock solution (1000 mg N l^{-1}) was diluted separately into 100 ml volumetric flasks with water, 2M KCl and 0.5M K_2SO_4. Each solution contained 10 mg N l^{-1}.

Organic compound sample with added ammonium-N: One ml of ammonium-N (100 mg N l^{-1}) and 1 ml of organic compound solution (1000 mg N l^{-1}) were added together by an automatic pipette into three 100 ml volumetric flasks for each compound. These were then diluted with water, 2M KCl and 0.5M K_2SO_4 to produce solutions containing 1 mg l^{-1} NH4-N and 10 mg l^{-1} organic nitrogen.

1M NaOH: Forty gram sodium hydroxide was dissolved in 500 ml of deionized water in 1 litre volumetric flask. After cooling, the volume was made upto the mark with deionized water.

2M HCl: One hundred and seventy ml concentrated HCl was diluted with deionized water and volume was made upto the mark in 1 litre volumetric flask.

1M HCl: Eighty five ml concentrated HCl was diluted with deionized water in 1 litre volumetric flask upto the mark.

0.01M HCl: Ten ml of 1M HCl acid were pippeted into 1 litre volumetric flask and volume was made upto the mark with deionized water.

Technicon silicon rubber membrane (product No. 157-13129) of the Technicon Corporation, New York, USA was used in the dialysis system. Dialysis system included with NH_4-N main manifold of Technicon AutoAnalyzer II comprised of dialysis block (6" dialyzer of the Technicon Corporation, New York, USA).

Procedure: The ammonium-N manifold as shown in Fig. 1 was used for NH_4-N determination in water, 2M KCl and 0.5M K_2SO_4 and also to determine the possible interference of organic nitrogen compounds. The samples were run at the rate of 40 per hour and the colour was developed in the water bath at 38°C. The colour intensity was measured at 650 nm. The calibration graph for NH_4-N is linear from 0 to 5 mg NH_4-N. The solutions were analysed for the determination of NH_4-N in organic N solution with and without NH_4-N in water, 2M KCl and 0.5M K_2SO_4 using 0 and 1 mg l^{-1} NH_4-N working standards and blank solutions.

Results and Discussion

The dialysis system shown in Fig. 2 worked on the mechanism explained below:

The donor stream of the dialysis was alkaline, therefore, the ammonium in the sample was converted to NH_3 which passed across the membrane and trapped in the acid acceptor stream and again converted to NH_4-N. As there is liquid and air bubbles in the donor stream, it is likely to happen that NH_3 either can pass directly from the liquid across the membrane or first move into the air bubbles and then pass through the membrane. It is also possible that NH_3 adopts both paths for crossing the membrane.

Table 1: Effect of sample acid concentration on NH_4-N measurement

NH_4-N (1 mgl^{-1}) in	NH_4-N measured
Water	1.00
0.01M HCl	1.01
0.1M HCl	0.80

Results of several time repeated analysis for the ammonium-N (Table 1) revealed that 0.1M HCl acid affected the chemistry of the main manifold. Therefore, 0.01M HCl should be used in the acceptor stream of dialysis system during determining NH_4 N by including dialysis system with Technicon AutoAnalyzer II. It was also noted that system (Figure II) did not show smooth flow. Peaks were not reproducible. Adding wetting agent (Brij-35) in the donor and acceptor streams showed some improvements. Mass flow of air across the dialysis membrane disturbed the air bubble pattern. Therefore, the air bubbles were eliminated from the acceptor side of the dialysis.

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