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Note

Determination of butylamine in air samples by isotachophoresis

LARS HANSEN*, JAN SOLLENBERG and KATE WIBERG

Research Department, National Board of Occupational Safety and Health, S-171 84 Solna (Sweden)

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Butylamine (1-aminobutane) is a liquid with an ammoniacal odour and a boiling point of 78°C that is used industrially for the production of pharmaceuticals, dyestuffs, rubber chemicals and insecticides. It is a potent skin, eye and mucous membrane irritant and direct contact with the skin can cause blistering in severe cases. In Sweden a threshold limit value of 5 ppm (15 min) for butylamine in the work room environment has been proposed.

For the determination of butylamine in air the U.S. National Institute for Occupational Safety and Health (NIOSH) has suggested a method that involves sampling on sulphuric acid-treated silica gel in glass tubes and determination by gas chromatography¹. As part of our investigations on analytical methods for amines used in industry, we present here an alternative method in which the air is collected in gas wash-bottles made of glass and containing HCl (0.1 mol/l), followed by analysis by isotachophoresis (ITP).

EXPERIMENTAL

Static gaseous butylamine standards were prepared in sacks. A thin sheet of alumina covered with a film of polyester (size 100 × 80 cm) was folded and the edges were fixed by adhesive tape on both sides thus forming a sack to contain up to 100 l. The air for dilution was led into the sack by a pump after passing through a gas volume meter. A PTFE tube was fixed on to the wall of the sack with PTFE nuts and kept tight with a rubber gasket. Liquid amine was injected into the sack with a microlitre syringe. Thus standard mixtures of butylamine in air in known concentrations could be produced. From the sack sample could be taken by bubbling the gas mixture through an absorption solution consisting of 10 ml of HCl (0.1 mol/l) in a glass wash-bottle (30 ml). The flow-rate was 500 ml/min as determined by a rotameter. The pumps used were MSA Model G portable pumps (Mine Safety Appliances, Pittsburgh, PA, U.S.A.). The sampling time was 15 min.

After sampling it was necessary to concentrate the absorption solution to increase the sensitivity of the analysis. This was done by transferring 5 ml of the solution to a polypropylene tube, which was placed in an alumina cylinder in the heating block of a thermostated electrical oven. The solution was evaporated to dryness at 80°C by passing a gentle stream of nitrogen over the surface of the solution. The residue was dissolved in 500 µl of distilled water and an aliquot was injected into the isotachophoretic instrument.

The injection volume was 1–20 μl and the instrument was a Tachophor 2127 (LKB, Bromma, Sweden) equipped with a PTFE capillary tube (200 mm \times 0.5 mm I.D.). The instrument had a conductivity detector. A calibration graph was constructed and was linear in the working range of 1–50 nmol.

The leading electrolyte was KOH (10 mmol/l) in 0.4% hydroxypropylmethylcellulose and L-valine. The latter, which was the counter ion, was added to pH 8.8. The terminating electrolyte was tris(hydroxymethyl)aminomethane (20 mmol/l) and HCl (5 mmol/l), pH 8.4.

The separation was performed at 140 μA and was generally completed within 10 min.

RESULTS AND DISCUSSION

The practical detection limit for butylamine by this isotachophoretic technique was about 1 nmol injected into the instrument. Analytically we could separate two butylamine isomers from each other and from some other low-molecular-weight amines (Fig. 1).

The overall recovery for a typical experiment was $108.8 \pm 9.3\%$ ($n = 6$). By "overall recovery" we mean the ratio of the butylamine concentration determined by isotachopheresis to the calculated value of the concentration in the sack. The uncertainty of the determinations includes errors from the gas standard preparation, sampling, concentration of absorption solution and analysis.

The concentration of the butylamine standard in air in this experiment was 1 μl per 100 l, corresponding to 2.5 ppm. To study the efficiency of the absorption, two gas wash-bottles were coupled in series and samples were taken at a flow-rate of 500 ml/min. After sampling, the absorption solutions from both bottles were analysed. At this flow-rate 95.2% of the amine collected was found in the first bottle. The loss was acceptable as the sampling volume at a reduced flow-rate would not give sufficient amine at lower concentrations of butylamine in air.

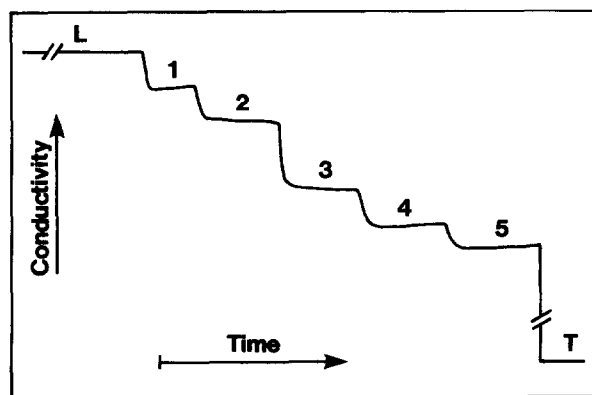


Fig. 1. Isotachopheretic separation of a series of amines (10 nmol of each). 1 = Methylamine; 2 = ethylamine; 3 = allylamine; 4 = *n*-butylamine; 5 = *tert.*-butylamine. L = leading electrolyte; T = terminating electrolyte. Arrows = relative conductivity and time (the length of the latter corresponds to 0.5 min of recording time).

Comparisons were also made of sampling efficiency with impingers made of glass or polystyrene. As no detectable differences were noticed, the presumed risk of adsorption of the amine on to glass during the sampling procedure could be neglected. On the other hand, glass tubes should be avoided in the concentration step. Notable losses were found on evaporating the acidic solution from the butylamine hydrochloride produced. This was probably due to the affinity of the amine for the glass walls. With tubes made of polypropene we obtained excellent results if the temperature of the thermostated oven was 70°C and the stream of nitrogen was about 150 ml/min. The time of evaporation was then about 3 h for a sample volume of 1 ml.

REFERENCE

- 1 National Institute for Occupational Safety and Health, *Manual of Analytical Methods*, Vol. 4, U.S. Department of Health, Education and Welfare, Washington, DC, 2nd ed., 1978, Method 138.