Available online at www.sciencedirect.com
SCIENCE DIRECT®
Journal of Chromatography A, 1045 (2004) 247–252
JOURNAL OF CHROMATOGRAPHY A
www.elsevier.com/locate/chroma

Determination of azide as the 3,5-dinitrobenzoyl derivative by capillary electrophoresis

Masataka Ohashi*, Yoshimi Kitada, Shunsuke Imai

Nara Prefectural Institute for Hygiene and Environment, 57-6 Ohmori-cho, Nara 630-8131, Japan

Received 20 February 2004; received in revised form 11 June 2004; accepted 16 June 2004

Available online 10 July 2004

Abstract

A simple, rapid and reliable capillary electrophoresis method with a photodiode array detector was developed for determination of azide as the 3,5-dinitrobenzoyl derivative in drink samples fortified with sodium azide. Sample preparation was simple and rapid because no more than a simple dilution of samples is needed after quick derivatization. Separation was carried out using a buffer system comprising 25 mM phosphate buffer and 4 mM cetyltrimethylammonium hydroxide at pH 3.0. Methyl benzoate was selected as the internal standard (IS). This study investigated the influence of the concentration of phosphate buffer and electroosmotic flow (EOF) modifier, and the buffer pH on migration time and signal response. The optimized method made it possible to determine azide within 5 min. The limit of detection was determined to be 1.9 μg/ml with $S/N > 3$. The quantitation range was 6.5–323 μg/ml. By the method recoveries of azide in drink samples fortified with sodium azide were investigated. Mean recovery values ranged from 93.6 to 105.8% and results were satisfactory. In addition, no interference was observed in electropherograms of drink samples fortified with sodium azide. Thus, by this method, azide in drink samples can be determined rapidly with high recoveries and good selectivity despite extremely simple sample preparation.

Keywords: Fruit juices; Soft drinks; Milk; Wine; Tea; Coffee; Food analysis; Azide; 3,5-Dinitrobenzoylazide

1. Introduction

Sodium azide is normally a white powder that is highly soluble in water: it has been used as preservative, antimicrobial agent and a propellant for inflation of automobile protective safety airbags. On the other hand, sodium azide has been a toxic compound implicated in many poisoning cases [1–6]. In humans, it is rapidly absorbed through the skin, after ingestion, and by inhalation together with hydrazoic acid. Sodium azide causes hypotension, headaches, vomiting, convulsions and at worst, death. Case reports show that the lethal dose is considered to be 13.5 mg/kg body weight [4]. In vitro studies of rat liver mitochondria have shown that sodium azide acts as an uncoupler of oxidative phosphorylation and as an inhibitor of cytochrome oxidase; moreover, it blocks energy transfer [7,8].

In 1998, azide-poisoning cases occurred one after another in Japan. In those cases, sodium azide was spiked in the hot water of an electric kettle. Drink samples are very likely contaminated with sodium azide because of the chemical's high water solubility. If drink samples that are possibly contaminated with sodium azide are submitted for toxicological analyses at a research institute, they should be analyzed with a reliable, yet rapid analytical method.

Terpinski described the determination of azide using a direct spectrophotometric method, which is based on the reaction of sodium azide with cerium(IV) ammonium nitrate [9]. However, that method is unreliable for precise determinations in sample matrices [6]. Klug and Schneider used colorimetric methods based on a color reaction with ferric chloride and volumetric measurement of nitrogen liberated by iodine reaction with hydrazoic acid [3]. However, both methods also lack specificity and sensitivity. Moreover, several methods for determining sodium azide were described using gas chromatography–mass spectrometry (GC–MS) [10–12], ion chromatography (IC) [13,14], high-performance liquid chromatography (HPLC) [5,6,15,16], and capillary electrophoresis (CE) with indirect ultraviolet (UV) detection [17,18]. GC–MS methods have advantages in terms of

* Corresponding author. Tel.: +81 742 20 2884; fax: +81 742 27 0634.
E-mail address: m.ohashi@ihe.pref.nara.jp (M. Ohashi).

0021-9673/$ – see front matter © 2004 Elsevier B.V. All rights reserved.
doi:10.1016/j.chroma.2004.06.030

sensitivity and specificity, but present some disadvantages in terms of time consumption. They are also technically complicated because of the chemical derivatization [10,11]. The problem of the GC–MS method for determining azide via phase-transfer-catalyzed pentafluorobenzylation is that recovery from samples such as orange juice containing matrices are somewhat low [12]. IC methods have been utilized to determine inorganic anions, but the methods lack specificity because they use a conductivity detector. HPLC methods determine azide as 3,5-dinitrobenzoyl derivatives using a UV detector. Azide, because of its extremely strong nucleophilicity, reacts quickly with a 3,5-dinitrobenzoyl chloride (DNBC) and forms a 3,5-dinitrobenzoyl azide (DNBA) with strong UV absorption. This derivatization improves specificity. However, HPLC spend much time on equilibration of the column. On the other hand, CE is a powerful separation technique that can provide high-resolution efficiency. Only several minutes are required to equilibrate the CE system; thereafter, sample preparation is extremely simple because no more than a simple dilution of samples is usually needed. However, CE methods with indirect UV detection lack specificity.

The present study attempts development of a simple, rapid, and reliable CE method with a photodiode array detector for determination of azide as a 3,5-dinitrobenzoyl derivative in drink samples fortified with sodium azide. In addition, we investigated the influence of the concentration of phosphate buffer and electroosmotic flow (EOF) modifier, and buffer pH on migration time and signal response.

2. Experimental

2.1. Chemicals

Hydrochloric acid solution (1 mol/l), sodium hydroxide solution (1 mol/l), 3,5-dinitrobenzoyl chloride (DNBC), methyl benzoate (BAMe), tetramethylammonium bromide (TMAB), *n*-octyl trimethylammonium bromide (OTAB), tetra-*n*-butylammonium hydroxide (TBAH) solution (0.5 mol/l), sodium azide, phosphoric acid, acetonitrile, *n*-hexane and acetone, which were all analytical grade, were obtained from Wako (Osaka, Japan). Tetrapropylammonium bromide (TPAB) was obtained from Tokyo Kasei Kogyo (Tokyo, Japan). The cetyltrimethylammonium hydroxide (CTAH) (10% in water) was obtained from Aldrich (Milwaukee, WI, USA). Water was purified with a Milli-Q purification system (Millipore, Bedford, MA, USA). All solutions were prepared in purified water.

2.2. Instrumentation

All CE experiments were performed with a P/ACE 5510 system (Beckman Coulter, Fullerton, CA, USA) equipped with a photodiode array detector, an autosampler and a temperature controller (15–40 ± 0.1 °C). The capillary temperature was examined at 25 °C. The sample was injected in pressure mode at 3.45 kPa for 1 s. The system was operated at −10 kV in a constant-voltage mode. Detection was performed by monitoring absorbance at 225 nm. The instrument was equipped with a data-handling system comprising a Compaq personal computer and 'P/ACE station' software. Separation was performed in a fused-silica capillary (75 μm i.d.; effective length, 30 cm; total length, 37 cm; Beckman Coulter).

The NMR spectra were recorded in C^2HCl_3 with $CHCl_3$ (^1H $\sigma = 7.27$ ppm, ^{13}C $\sigma = 77.0$ ppm) as an internal standard using a Unity-Inova 500 spectrometer (Varian, CA, USA). The IR spectra, the UV spectra, and the MS spectra were taken using an FTIR-4100 spectrometer (Shimadzu, Kyoto, Japan), a UV-2200A spectrometer (Shimadzu) and a JMS SX102 spectrometer (JEOL, Tokyo, Japan), respectively. Mass spectra were obtained using field desorption (FD) ionization.

2.3. Confirmation of the derivative identity

Corresponding to the procedure of Munch-Peterson [19], a few grams of 3,5-dinitrobenzoyl azide (DNBA) were prepared as follows.

A DNBC solution (2.30 g, 10 mmol) in acetonitrile (3 ml) was added, dropwise, to a stirred solution of sodium azide (0.78 g, 12 mmol) in distilled water (10 ml) at room temperature. Thereupon, DNBA separates immediately as a white precipitate. After 5 min, DNBA was separated on a suction filter, washed with water, and dried in air. The yield of the crude product was 2.25 g (94.9%). It was recrystallized from acetone and *n*-hexane. It is freely soluble in acetone, acetonitrile, chloroform and toluene, soluble in methanol and insoluble in water. The melting point of DNBA is 104–105 °C.

Spectra data of DNBA are shown as the following: ^1H NMR (σ, ppm) 9.28 (t, $J = 2.2$ Hz, 1 H), 9.17 (d, $J = 2.2$ Hz, 2H); ^{13}C NMR (σ, ppm) 169, 149, 134, 129, 123; IR (KBr) 3106, 2230, 2155, 1692, 1630, 1547, 1348, 1257, 1170, 924, 745, 731, 716; UV [λ_{max} ($\log \varepsilon$) in acetonitrile] 223 (4.54); MS (FD) 237 (M+).

2.4. Electrophoretic procedure

Fresh capillaries were used for each buffer system to avoid hysteresis effects. The electrolyte was prepared by mixing 25 mM phosphoric acid and 25 mM sodium hydroxide solution containing 4 mM CTAH, respectively, using a pH meter (HM-26S; TOA) to adjust pH 3.0; the electrolyte was filtered through a 0.45-μm membrane filter (Millipore, Bedford, MA, USA). Each new capillary was treated with a high-pressure rinse (138 kPa) of 1 M hydrochloric acid, purified water, 1 M sodium hydroxide solution and purified water for 5 min, respectively, and then with the electrolyte for 10 min. The capillary was rinsed with the electrolyte for 0.5 min following every sample run.

2.5. Standard preparation

We selected a proper EOF modifier by determining the isolated and identified DNBA solution. The solution was prepared as follows.

To 2 ml of DNBA solution (912 μg/ml) in acetonitrile was added purified water and adjusted to final volume of 10 ml with purified water. Subsequently, the solution was filtered through a 0.45-μm membrane filter. Concentration of this solution was corresponding to 50 μg/ml in terms of sodium azide concentration.

On the other hand, after selection of the proper EOF modifier, standard solutions were prepared when needed as follows.

To 1 ml of sodium azide solution (10–500 μg/ml) in purified water was added 1.8 ml of acetonitrile. After 0.1 ml of 10% (w/v) DNBC solution in acetonitrile was added, the solution was stirred by a vortex mixer for 30 s at room temperature and 0.1 ml of BAMe solution (3000 μg/ml) was added in acetonitrile as the IS. The solution was brought to a final volume of 10 ml by adding purified water; then it was filtered through a 0.45-μm membrane filter. The percentage of acetonitrile in the solution consequently became 20% (v/v). If 0.8 ml of acetonitrile is added to the sodium azide solution and the above operations are performed, the percentage of acetonitrile in the prepared solution becomes 10% (v/v).

2.6. Sample preparation

Green tea, coffee, red wine, milk, soft drinks, and orange juice were used as samples: 50 μg sodium azide and then 1.8 ml acetonitrile were added to 1 ml of each sample. After 0.1 ml of 10% (w/v) DNBC solution in acetonitrile was added, the solution was stirred by a vortex mixer for 30 s at room temperature. Then, 0.1 ml of BAMe solution (3000 μg/ml) in acetonitrile was added as the IS. The solution was brought to a final volume of 10 ml by adding purified water; it was then filtered through a 0.45-μm membrane filter. However, a precipitate formed in the cases of milk, soft drinks and orange juice. Therefore, the suspension was centrifuged at 2500 × *g* for 1 min and the supernatant was filtered through a 0.45-μm membrane filter. The percentage of acetonitrile in the solution and the suspension consequently became 20% (v/v). The percentage of acetonitrile in the prepared solution becomes 10% (v/v) if 0.8 ml of acetonitrile is added to sample and the above operations are performed.

3. Results and discussion

3.1. Selection of an EOF modifier

Azide reacts quickly with DNBC to form DNBA and we tried to determine azide as DNBA. Selecting of an EOF modifier, we used the isolated and identified DNBA to protect the capillary system from other compounds.

First, we tried to determine DNBA under the condition that the buffer system contained 100 mM phosphate buffer without cationic surfactants at pH 4.5. The applied voltage was set at 10 kV. Under the above condition, we detected DNBA at a migration time of 10.7 min, but could not separate it from a negative peak appearing at a migration time of 9.8 min. The negative peak is derived from acetonitrile.

We tried to determine DNBA using a buffer system containing 100 mM phosphate buffer and 5 mM cationic surfactant, such as a quaternary ammonium salt at pH 4.5 and set the applied voltage at 10 kV potential. We used TMAB, TBAH, TPAB and OTAB as a cationic surfactant. We detected DNBA; however, we could not separate DNBA from acetonitrile.

According to some previous literature, CTAB has been used generally to reverse EOF [20–24]. We also expected a reversal of EOF direction in the case using CTAH. Therefore, we reversed the polarity of the instrument because the EOF was toward the detector. That is, we determined DNBA at −10 kV potential under the same condition except for the applied potential. Thereby, we detected DNBA at a migration time of 3.5 min; moreover, we succeeded in separating DNBA from acetonitrile completely. From this result, we selected CTAH as the proper EOF modifier.

Incidentally, we used the isolated DNBA to prevent influence of other compounds from a capillary system during selection of the EOF modifier. However, after its selection, we prepared standard solutions using the previously mentioned method. We confirmed that azide was converted stoichiometrically into DNBA in the method in spite of such short reaction times as 30 s. The confirmation method was based on an HPLC method reported by Lambert et al. [5].

On the other hand, to improve the reliability of determination, we selected BAMe as the IS because BAMe showed a marked UV absorbance at 225 nm; also, BAMe migrated more quickly than DNBA under the above conditions. That fact shortens the analysis time. In addition, BAMe was separated from acetonitrile as well as DNBA. On the other hand, we did not select ethyl benzoate and sodium benzoate as the IS because they migrated more slowly than DNBA.

3.2. Optimization of analytical conditions

If drink samples that are possibly contaminated with sodium azide are submitted for toxicological analyses at a research institute, they should be analyzed with a reliable, yet rapid analytical method. Thus, it is very important to determine azide as DNBA rapidly and sensitively. Concentration of the phosphate buffer, EOF modifier, and buffer pH generally affect migration time as well as signal response in CE. Therefore, we attempted to investigate their effects on migration time and signal response.

First, we investigated the effect of phosphate buffer concentration on separation using a buffer system containing

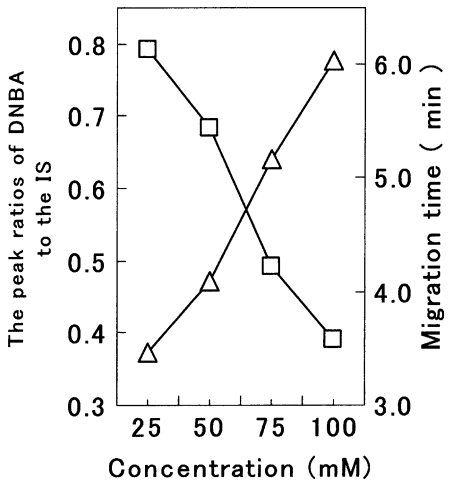

Fig. 1. The peak area ratios of DNBA to the IS and migration time as a function of phosphate buffer concentration. Conditions: electrolyte, 25–100 mM phosphate buffer containing 5 mM CTAH at pH 4.5; DNBA concentration, 18.2 μg/ml; applied voltage, −10 kV; temperature, 25 °C; capillary, fused-silica capillary, 37 cm × 75 μm i.d.; UV detection was at 225 nm. (□) The peak area ratios of DNBA to the IS; (△) migration time of DNBA.

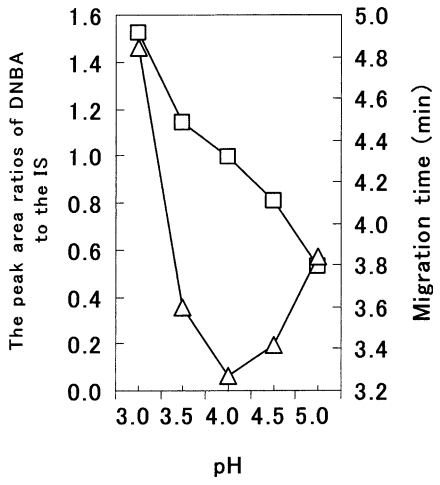

Fig. 2. The peak area ratios of DNBA to the IS and migration time as a function of buffer pH (3–5). Conditions: electrolyte, 25 mM phosphate buffer containing 4 mM CTAH. For other conditions, see Fig. 1. (□) The peak area ratios of DNBA to the IS; (△) migration time of DNBA.

5 mM CTAH. We varied concentration of the phosphate buffer within 25–100 mM concentration range in increments of 25 mM. The pH was set at 4.5. During optimization of the analytical conditions, we used a sodium azide solution of the concentration of 50 μg/ml. Therefore, the concentration of DNBA and the IS in the prepared solution was 18.2 and 30 μg/ml, respectively. The DNBA concentration of 18.2 μg/ml was equivalent to the azide concentration of 3.2 μg/ml. Fig. 1 shows the effect of concentration of phosphate buffer on migration time of DNBA and peak area ratios of DNBA to the IS. Decreasing the concentration of phosphate buffer caused a decrease in migration time of DNBA from 6.0 to 3.5. During this optimization reproducibilities (R.S.D., $n = 6$) were not more than 2.5% on the basis of migration time of DNBA and the IS at all concentrations. This fact indicates that migration time of them shows a little variation.

On the contrary, decreasing the concentration of phosphate buffer caused an increase in the peak area ratios of DNBA to the IS from 0.39 to 0.79. The peak area ratio was maximal at a phosphate buffer concentration of 25 mM; in addition, the migration time of DNBA was minimal. Consequently, 25 mM phosphate buffer was chosen for further investigation.

Secondly, we varied the concentration of CTAH within 0.5 mM and 1–5 mM concentration range in increments of 1 mM to optimize the concentration of CTAH.

When concentration of CTAH was not more than 1 mM, we were unable to separate DNBA from acetonitrile and the IS. On the other hand, when concentration of CTAH was at 2 mM, we succeeded in separating DNBA from acetonitrile and the IS on some level. However, the concentration of 4 mM CTAH was necessary to completely separate DNBA with high concentrations of 500 μg/ml sodium azide. Increasing the concentration of CTAH from 4 mM to 5 mM caused little decrease in a peak ratio of DNBA to the IS from 0.80 to 0.79. Consequently, CTAH concentration was set at 4 mM for further investigation.

Finally, we varied the pH from 3 to 5 in increments of 0.5 to optimize buffer pH. Fig. 2 shows the effect of pH on peak area ratios of DNBA to the IS and migration time of DNBA. Migration time of DNBA increases as pH decreases from 4 to 3. During this optimization reproducibilities (R.S.D., $n = 6$) were not more than 0.5% on the basis of migration time of DNBA and the IS at all pH. This fact indicates that migration time of them shows little variation.

On the other hand, as pH increases, the peak area ratio of DNBA to the IS decreases from 1.5 to 0.5. This increase results from the sensitivity of DNBA to pH. Swarin and Waldo reported that DNBA is stable between pH 3.0 and pH 5.5 [15]. However, as this result showed, we found that increasing pH caused a decrease in the amount of DNBA in the electrolyte under the condition in which a high voltage was applied. The peak area ratio was highest at pH 3.0 between 3.0 and 5.0. Consequently, pH 3.0 was chosen to give preference to the sensitivity over migration time.

As observed above, we optimized the concentration of phosphate buffer and CTAH, and buffer pH. This optimization made it possible to determine DNBA within 5 min and to increase the peak area ratio of DNBA to the IS from 0.39 to 1.5.

3.3. Linearity and limits of detection

Calibration curves for sodium azide showed good linearity ($y = 0.231x + 0.0592$, $r^2 = 0.9999$) in the range of 10–500 μg/ml between the peak area ratio of DNBA to the

IS and concentration. The range of 10–500 μg/ml was corresponding to 6.5–323 μg/ml in terms of azide concentration. The limit of detection was determined to be 3 μg/ml (1.9 μg/ml in terms of azide concentration) with $S/N > 3$. The limit of quantitation was determined to be 10 μg/ml (6.5 μg/ml in terms of azide concentration) with $S/N > 10$. Reproducibilities (R.S.D., $n = 6$) were 0.24 and 0.23% on the basis of migration time of DNBA and the IS, respectively. This fact indicates that migration time of them shows little variation. In addition, reproducibilities were 2.13% on the basis of peak area ratios. Good results were obtained in accuracy studies on both migration times and quantitation. Reproducibilities, on the basis of peak area instead of peak area ratios were 9.21%. Consequently IS considerably improved reproducibilities.

3.4. Recovery of azide in drink samples fortified with sodium azide

According to a case previously reported, the authors infer that a minimum toxic dose for humans is 5000 μg [1]. We assumed that an ingestion of 100 ml of drink fortified with 5000 μg of sodium azide caused intoxication. Therefore, we used control (no sodium azide added) drink samples, which were fortified at 50 μg/ml of sodium azide solution (32.3 μg/ml in terms of azide concentration) to perform a recovery study. We determined the concentrations of azide in the samples and calculated recoveries of azide in the samples. Electropherograms of control green tea and green tea fortified with sodium azide are shown in Fig. 3. As Fig. 3(a) shows, the electropherograms are generally simple; no interference was observed. Fig. 3(b) shows a good resolution of DNBA and the IS.

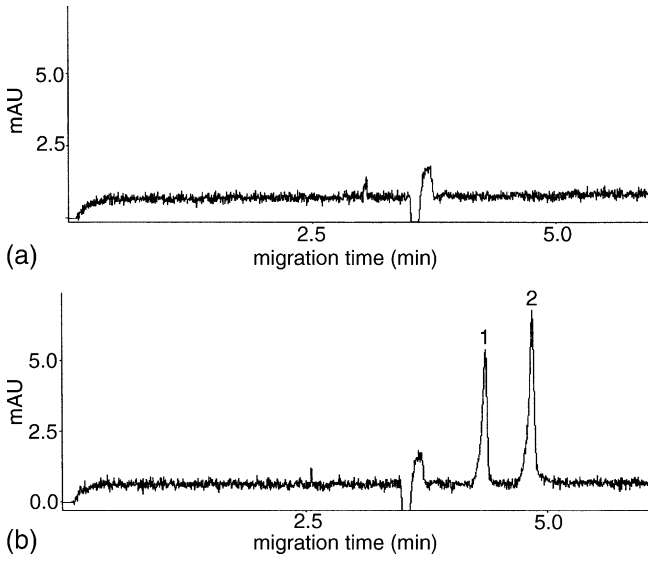

Fig. 3. Electropherograms of (a) control green tea; (b) green tea fortified with sodium azide at 50 μg/ml and the IS: (1) methyl benzoate, (2) DNBA. Conditions: electrolyte, 25 mM phosphate buffer containing 4 mM CTAH at pH 3.0; capillary temperature, 25 °C. For other conditions, see Fig. 1.

Table 1
Recovery of azide in drink samples fortified with sodium azide[a]

Sample	Recovery (%)[b]	
	10% acetonitrile[c]	20% acetonitrile[c]
Green tea	108.6 ± 4.3	100.5 ± 3.8
Coffee	108.7 ± 2.1	97.5 ± 1.6
Red wine	100.3 ± 3.0	103.3 ± 4.3
Milk	78.0 ± 6.2	104.5 ± 4.3
Soft drink	80.2 ± 11.9	105.8 ± 4.9
Orange juice	79.7 ± 3.0	93.6 ± 2.7

[a] Drink samples were fortified at 50 μg/ml of sodium azide solution. The concentration was corresponding to 32.3 μg/ml in terms of azide concentration.
[b] Average ± R.S.D., $n = 6$.
[c] The percentage of acetonitrile in the prepared solution.

As the figures show, the analytes are observed as neat electrophoretic peaks at a migration times of 4.5 min for the IS and 4.9 min for DNBA, respectively; no interference is present. In electropherograms of other samples, no interference is present.

Red wine generally contains a small percentage of ethanol. DNBC reacts with ethanol to form ethyl 3,5-dinitrobenzoate. However, nucleophilicity of azide is much stronger than that of ethanol; also, DNBC reacts preferentially with azide. After the reaction is completed, the excess of DNBC reacts with ethanol. The resultant ethyl 3,5-dinitrobenzoate was detected at a migration time of 5.6 min; the derivative did not interfere with DNBA determination.

Table 1 shows recoveries of azide in several drinks fortified with sodium azide. Results show that when the percentage of acetonitrile is 10%, the recoveries of azide in green tea, coffee, and red wine are nearly 100%, but the recoveries of azide in milk, soft drinks, and orange juice are near 80%. On the other hand, when the percentage of acetonitrile is 20%, the mean recovery values of azide ranged from 93.6 to 105.8%: those results are satisfactory. Apparently, an increase in the percentage of acetonitrile can minimize absorption of DNBA to a precipitate.

4. Conclusion

A simple, rapid and reliable capillary electrophoresis method with a photodiode array detector was developed for determining azide as a 3,5-dinitrobenzoyl derivative in drink samples fortified with sodium azide. Sample preparation was simple and rapid because no more than a simple dilution of samples is needed after quick derivatization. The method uses a buffer system comprising 25 mM phosphate buffer and 4 mM CTAH at pH 3.0 and methyl benzoate as the IS. By this method, the mean recovery values of azide in drink samples fortified with sodium azide ranged from 93.6 to 105.8%. In addition, no interference was observed in electropherograms of drink samples fortified with

sodium azide. Thus, high recovery and good selectivity were achieved even though sample preparation was extremely simple and rapid. This method has marked advantages in terms of feasibility and rapidity in comparison with other analytical methods.

References

[1] S.G.N. Richardson, C. Giles, C.H.J. Swan, J. Clin. Pathol. 28 (1975) 350.
[2] T. Senda, K. Nishio, Y. Hori, A. Baba, Y. Suzuki, Y. Asari, K. Tsuchimoto, Chudoku Kenkyu 14 (2001) 339.
[3] E. Klug, V. Schneider, Z. Rechtsmed. 98 (1987) 129.
[4] J.D. Howard, K.J. Skogerboe, G.A. Case, V.A. Raisys, E.Q. Lacsina, J. Forensic Sci. 35 (1990) 193.
[5] W.E. Lambert, M. Piette, C.V. Peteghem, A.P. De Leenheer, J. Anal. Toxicol. 19 (1995) 261.
[6] P. Marquet, S. Clement, H. Lotfi, M-F. Dreyfuss, J. Debord, D. Dumont, G. Lachatre, J. Anal. Toxicol. 20 (1996) 134.
[7] K. Bogucka, L. Wojtczak, Biochim. Biophys. Acta 122 (1966) 381.
[8] F. Palmieri, M. Klingerberg, Eur. J. Biochem. 1 (1967) 439.
[9] E.A. Terpinski, Analyst 110 (1985) 1403.
[10] S. Kage, K. Kudo, N. Ikeda, J. Anal. Toxicol. 24 (2000) 429.
[11] M. Kikuchi, M. Sato, T. Ito, M. Honda, J. Chromatogr. B 752 (2001) 149.
[12] A. Miki, M. Nishikawa, H. Tsuchihashi, J. Health Sci. 46 (2000) 81.
[13] P.L. Annable, L.A. Sly, J. Chromatogr. 546 (1991) 325.
[14] R. Kruszyna, R.P. Smith, H. Kruszyna, J. Forensic Sci. 43 (1998) 200.
[15] S.J. Swarin, R.A. Waldo, J. Liq. Chromatogr. 5 (1982) 597.
[16] R. Battaglia, J. Mitiska, Z. Lebensm. Unters. Forsch. 182 (1986) 501.
[17] G.L. Hortin, S.K. Dey, M. Hall, C.A. Robinson, J. Forensic Sci. 44 (1999) 1310.
[18] T. Soga, M. Imaizumi, Electrophoresis 22 (2001) 3418.
[19] J. Munch-Peterson, in: N. Rabjohn (Ed.), Organic Syntheses, vol. 4, Wiley, New York, 1973, p. 715.
[20] V. Pucci, R. Mandrioli, M.A. Raggi, Electrophoresis 24 (2003) 2076.
[21] T. Soga, G.A. Ross, J. Chromatogr. A 837 (1999) 231.
[22] V. Galli, N. Olmo, C. Barbas, J. Chromatogr. A 894 (2000) 135.
[23] M.J. Cugat, F. Borrull, M. Calull, Analyst 125 (2000) 2236.
[24] N.E. Baryla, J.E. Melanson, M.T. McDermott, C.A. Lucy, Anal. Chem. 73 (2001) 4558.