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Fast detection of fluoroacetamide in body fluid using gas chromatography–mass spectrometry after solid-phase microextraction

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Abstract

A novel method for fast determination of fluoroacetamide, a kind of organic fluorine pesticide, in blood and urine samples was developed with acetamide as an internal standard using gas chromatography/mass spectrometry (GC/MS) after solid-phase microextraction (SPME) technique. The SPME was performed by immersing a PDMS fiber of 100 μm coating thickness in a sample solution for 25 min at 70 °C with $(CH_3CH_2)_4NBr$ to improve the extraction efficiency. After a GC sample injection, the extracted fluoroacetamide was desorbed from the fiber for 4 min to perform the GC/MS detection with a HP-PLOT Q capillary column. The analytical conditions were optimized by examining systematically, the effects of experimental parameters on the ratio of characteristic ion peak areas of fluoroacetamide to acetamide. Under optimal conditions, the ratio was proportional to the concentration of fluoroacetamide ranging from 5.0 to 90 μg/ml with a detection limit of 1.0 μg/ml. The average recovery of fluoroacetamide in blood sample was 92.2%. The established method could be used for the fast and convenient measurement of fluoroacetamide in poisoned sample.

Keywords: Fluoroacetamide

1. Introduction

Fluoroacetamide is a kind of organic fluorine pesticide. It possesses tremendous toxicity and has been recognized as an illicit rodenticide. It is often accidentally eaten and used to poison human and cattle in some countries due to its simple and cheap synthesis, tastelessness, and high solubility in water. In recent years, fluoroacetamide has become one of the most common poisonous substances occurring in criminal cases. Thus, its identification and quantitative determination have attracted considerable concern. Unfortunately, to our best knowledge, a convenient and effective method for this purpose has not been reported except the one presented by Allender, for determination of sodium fluoroacetate, an analog of fluoroacetamide [1].

In general, fluoroacetamide can be qualitatively detected by the sulfur–indigo reaction [2–5]. This method was further developed by Logan et al. in 1990 [5]. The detection procedure first uses methanol/water mixture to extract the fluoroacetamide from the sample. At present, it has been able to detect qualitatively, fluoroacetamide down to 10 μg [4]. Because of the low efficiency and sensitivity, this method has shown many limits such as suffering from interferences, false positivity, and false negativity. In 1997, Feng and Yu [6] presented a gas chromatography (GC)/FID and GC/NPD determination method. This method possessed high sensitivity and could detect 5 μg fluoroacetamide in 1 ml blood or 1 g tissue sample with a recovery of 80–85%. However, this method was unsuitable for the detection of fluoroacetamide in the poisoned tissue sample. For a poisoned tissue sample, the fluoroacetamide must first be extracted using acetone and water mixture and ethyl acetate in strong acidity, and then derived with *N*,*N*-diethyl-*p*-benzene diamine. The derivative was further purified with silicon–magnesium

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sorbent and then detected using GC/NPD method with a recovery up to 80%. This procedure was similar to that presented previously with GC/ECD [1]. Obviously, these methods are time-consuming and induce uncertainty associated to final results. Therefore, it is necessary to develop a new feasible method for the identification and quantitative determination of fluoroacetamide.

Sodium fluoroacetate, an analog of fluoroacetamide, is also a frequently and illegally used rodenticide. The methods for its determination have been developed using gas–liquid chromatographic [7–10], high performance liquid chromatographic [11], and fluoride electrode analytical [12,13] techniques. These methods required some type of derivatization of sodium fluoroacetate and lacked adequate sensitivity for the detection of small amounts of sodium fluoroacetate [14,15]. In 1995, Guan et al. [15] established a direct derivatization method in aqueous phase for fluoroacetate determination by GC/NPD. This method simplified the pretreatment procedure for fluoroacetate determination. With the development of analytical techniques, GC/mass spectrometry GC/(MS) [16,17] and nuclear magnetic resonance (NMR) spectroscopy [18,19] have been employed for the identification of sodium fluoroacetate. These works have displayed that GC/MS is an efficient technique, and is playing an important role in toxicant analysis [20–24].

Solid-phase microextraction (SPME) technique was firstly presented by Pawllszyn [25,26]. It has been well known as a method to integrate sampling, extraction, concentration, and sample introduction into a single step [27,28]. The sample preparation by SPME prior to GC analyses can be more advantageous than conventional solvent extraction [29–31]. This technique has begun to find diverse applications in many areas of forensic science [24,32,33], including toxicant analyses [34–36]. This work studies the application of GC/MS in the identification and quantitative determination of fluoroacetamide. Coupling it with SPME technique, a SPME–GC/MS method for convenient analysis of fluoroacetamide is established. In this work, acetamide is selected as an internal standard to optimize the SPME conditions for GC/MS determination of fluoroacetamide. The proposed method can satisfy the requirement for convenient quantitative measurement of fluoroacetamide in poisoned sample.

2. Experimental

2.1. Reagents

All reagents used were of analytical grade quality or higher. Fluoroacetamide and acetamide were obtained from the Forensic Medical Examination and Identification Center of Public Security Ministry of China. Their stored solutions were prepared at a concentration of 2.0 mg/ml. Tetraethylammonium bromide was obtained from Aldrich (USA). Methanol, acetonitrile, and acetone were purchased from Tedia Company Inc. (USA). Water was 18.2 MΩ quality from a Milli-Q system. The standard blood samples for SPME–GC/MS determination contained 22.5 μg/ml acetamide, 0.05 M tetraethylammonium bromide, and 5, 15, 30, 45, 60, or 90 μg/ml fluoroacetamide.

2.2. Apparatus

All samples were analyzed with a HP GCD GC/MS system with an electron ionization chamber (HP company, USA). SPME fibers of 100 μm coating of polydimethylsiloxane/divinylbenzene and manual fiber holders were purchased from Supelco (PA, USA).

SPME fibers were introduced directly into the injection port of the GC/MS to thermally desorb analytes for 4 min. The injection port and the detector of the GC/MS were maintained at 200 °C. Gas chromatographic separations were performed on a 25 m HP-PLOT Q capillary column with 0.32 mm inner diameter and 0.22 μm film thickness, which was maintained at the initial column temperature of 100 °C for 1 min, heated to 200 °C at the rate of 10 °C/min, and then held at 200 °C for 14 min. Helium gas (99.999%) was used as carrier gas at a flow rate of 0.8 ml/min. The MS was operated in scan mode at a rate of 1.5 scan/s over the range of 10–200 amu with a solvent delay of 5 min.

2.3. Sample preparation for extraction of fluoroacetamide

Ninety microliters of 2.0 mg/ml fluoroacetamide and 45 μl of 2.0 mg/ml acetamide were added into 2.0 ml of blood. The pH value of the mixed solution was adjusted to 9.0 with ammonia. The proteins were then precipitated by the addition of 10% zinc phosphate. After centrifugation, the mixture was filtrated with hydrophilic membrane of 0.2 μm pore diameter and followed by adding 2.0 ml of 0.1 M tetraethylammonium bromide in the filtrate and adjusting the pH value to 7.0 with phosphoric acid. The final volume was controlled to be about 4 ml (sample A).

For a urine sample, 90 μl of 2.0 mg/ml fluoroacetamide, 45 μl of 2.0 mg/ml acetamide, and 2.0 ml of 0.1 M tetraethylammonium bromide were added into 2.0 ml urine. After the mixture was filtrated with hydrophilic membrane of 0.2 μm pore diameter, a sample of about 4 ml (sample B) was obtained. The control was prepared by mixing 90 μl of 2.0 mg/ml fluoroacetamide, 45 μl of 2.0 mg/ml acetamide, and 2.0 ml of 0.1 M tetraethylammonium bromide with 2.0 ml water. After the mixture was filtrated with the hydrophilic membrane, sample C was obtained.

For a real sample, 45 μl of 2.0 mg/ml acetamide was added into 2.0 ml poisoned sample. The mixture was further treated and mixed with 2.0 ml of 0.1 M tetraethylammonium bromide for analysis via SPME process.

2.4. SPME

The partition coefficient of the analyte between the coating and the sample matrix K_{fs}, and the volume of the coating

V_f, are the important factors that affect the sensitivity and recovery of the analytical method [26,28]. The volume of the coating depends on the fiber length and the coating thickness, which are usually fixed. When the volume of the sample solution V_s, is very large ($V_s \gg K_{fs}V_f$), the amount of analyte extracted by the fiber coating is not related to the sample volume [28]. This work selected a fiber length of 1.0 cm and a V_s value of 4 ml for the SPME. Thus, the sensitivity and recovery was decided by the partition coefficient, which was related to the SPME conditions. Under optimal conditions, the amount of fluoroacetamide absorbed by the coating was directly related to its concentration in the sample.

Prior to SPME, the sample solution was kept at 70 °C and stirred in a whirlpool mixer for 2.0 min. The fiber was then immersed in the solution through the needle of the SPME syringe assembly. The depth of 1.0 cm of the fiber head soaked in solution was controlled by adjusting the plunger. After an extraction time of 25 min, the needle of the SPME syringe assembly was taken out for GC/MS analysis.

3. Results and discussion

3.1. Selection of internal standard

Although sodium fluoroacetate and fluoroacetic acid are the analogs of fluoroacetamide, and they can also be completely separated from fluoroacetamide by gas chromatography, it cannot be used as an internal standard for practical applications, due to the metabolism of fluoroacetamide in body into fluoroacetic acid. Both fluoroacetamide and fluoroacetic acid can be found simultaneously in the body fluid after poisoned by fluoroacetamide for 20 h. Both the poisoned time and the pretreatment of the sample affect their relative contents and repeatability. Thus, for a quantitative purpose, we must look for other analogs as the internal standard. Acetamide possesses the structure and extraction property similar to those of fluoroacetamide. It can also be completely separated from fluoroacetamide by gas chromatography (Fig. 1) and is not related to the metabolism of fluoroacetamide in body. Thus, it is a suitable internal standard for quantitative detection of fluoroacetamide by GC/MS. The total ion current scan gave a retention time of 14.09 min for fluoroacetamide, and a retention time of 15.89 min for acetamide (Fig. 1). Fortunately, the retention time of fluoroacetic acid at the same conditions was 16.68 min, thus, it did not interfere the detection of fluoroacetamide. The GC/MS spectrum corresponding to the retention time of 14.09 min showed the molecular and fragment ion peaks of fluoroacetamide at *m*/*z* of 77, 44, and 33. The characteristic ion peaks of the interior marker at the retention time of 15.89 min were at *m*/*z* 59 and 44, respectively (Fig. 2).

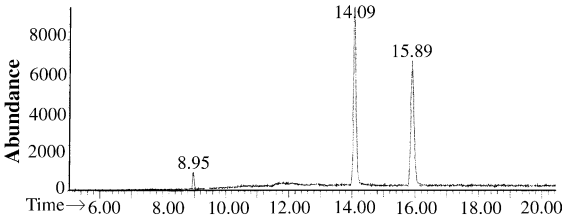

Fig. 1. GC/MS total-ion chromatogram of 22.5 μg/ml fluoroacetamide and 22.5 μg/ml acetamide in blood sample.

3.2. Optimization of SPME conditions for determination of fluoroacetamide

The analytical method based on SPME has two steps: extracting analytes and desorbing them into analytical instruments. Most important experimental parameters are determined largely during extraction. The extraction efficiency is decided by the partition coefficient of the analyte between the coating and the sample matrix, and the extraction time. This work used three samples (A, B, and C) in duplicate to examine the effects of extraction system, temperature and time, pH of extraction solution, desorption time on the GC/MS sensitivity, and the ratio of the average characteristic ion peak areas of fluoroacetamide to acetamide.

3.2.1. Selection of extraction system

When the solutions of 2.0 ml containing 0.18 mg fluoroacetamide and 0.09 mg acetamide, are mixed with (a) 2.0 ml methanol, (b) 0.3 ml methanol + 1.7 ml acetonitrile, (c) 2.0 ml of 2.5 M NaCl, or (d) 2.0 ml of 0.1 M tetraethylammonium bromide, respectively, the GC/MS total-ion chromatograms showed different responses of fluoroacetamide and acetamide. The abundances of the characteristic ion peaks for both fluoroacetamide and acetamide changed with the difference in the extraction system. The analytical results were listed in Table 1. The presence of salt resulted in an abundant increase of characteristic ion peak of fluoroacetamide from twice to thrice (systems a, b, and c). The ratio of the peak areas of fluoroacetamide to acetamide showed a small increase. Thus, the effects of solution polarity on the partition coefficients of fluoroacetamide and acetamide were similar. Changing the solution polarity could not improve

Table 1
Systems used for sample extraction, the ratio of the characteristic ion peaks of fluoroacetamide and acetamide, and average abundance of characteristic ion peak of flouroacetamide measured using SPME

Systems for sample extraction[a]	$S_{fluoroacetamide}/S_{acetamide}$	Average $A_{fluoroacetamide}$ value
a	0.909	1076
b	1.070	1661
c	1.152	3165
d	5.268	5965

[a] The samples were prepared by mixing 2.0 ml solutions containing 0.18 mg fluoroacetamide and 0.09 mg acetamide with (a) 2.0 ml methanol, (b) 0.3 ml methanol + 1.7 ml acetonitrile, (c) 2.0 ml of 2.5 M NaCl, or (d) 2.0 ml of 0.1 M tetraethylammonium bromide.

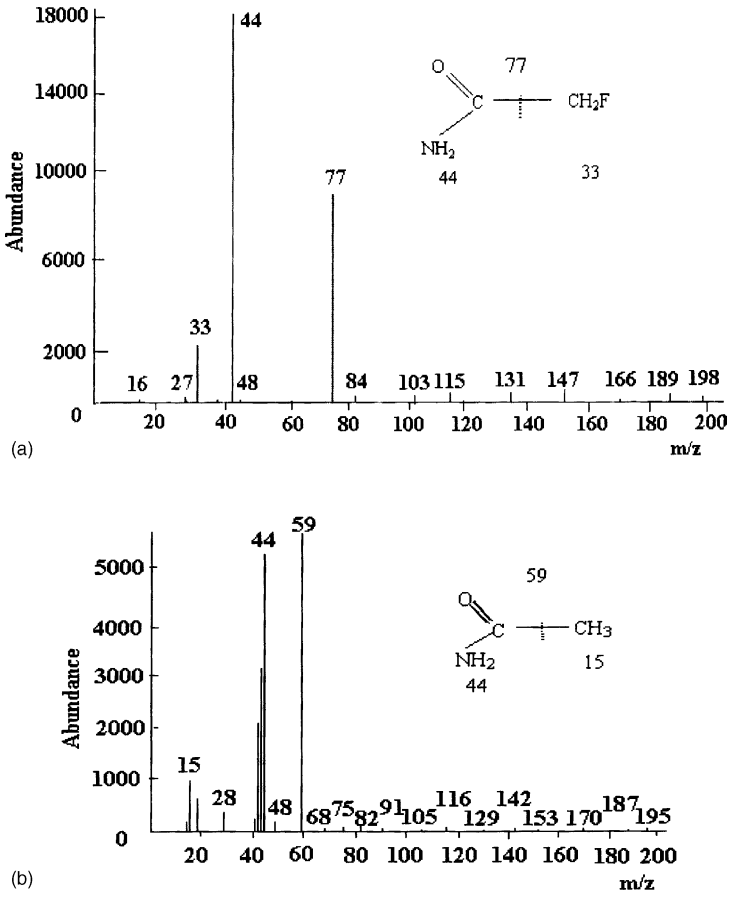

Fig. 2. GC/MS spectra of (a) fluoroacetamide and (b) acetamide in blood sample.

obviously the analytical sensitivity. After tetraethylammonium bromide was added to this system, both the abundance of the characteristic ion peak of fluoroacetamide and the ratio of the peak areas of fluoroacetamide to acetamide increased greatly. The coating on the fiber was nonpolar. There was a high surface tension at the interface between the nonpolar coating and the polar solution. The presence of hydrophobic tetraethylammonium cation decreased the surface tension, which increased the partition coefficients of fluoroacetamide and acetamide in the coating. The change in the partition coefficient of fluoroacetamide was larger than that of acetamide due to high polarity of fluoroacetamide. Thus, more fluoroacetamide molecules were adsorbed on the fiber surface. This work added tetraethylammonium bromide to the extraction system to improve the analytical sensitivity.

3.2.2. Effect of concentration of tetraethylammonium bromide

Fig. 3a shows the dependence of the peak area ratio and the abundance of the characteristic ion peak of fluoroacetamide on the concentration of tetraethylammonium bromide. At a concentration of 0.05 M, the peak area ratio and abundance correspond to the maximum values of 5.267 and 5965, respectively. At concentrations higher than 0.05 M, the partition coefficient of fluoroacetamide decreased due to the adsorption of more tetraethylammonium molecules in the coating, which decreased the amount of fluoroacetamide adsorbed in the coating. So, 0.05 M tetraethylammonium bromide was used for the SPME.

3.2.3. Effect of solution pH

The partition coefficients of both fluoroacetamide and acetamide depended on the solution pH, which affected the absorption capacity of the fiber coating. The maximum peak area ratio occurred at pH 7.0, which was used as the optimal pH value of the extraction system.

3.2.4. Temperature of the extraction system

The partition coefficient is an important thermodynamic parameter. Thus, it is related to the extraction temperature. The partition coefficient usually decreases with increasing temperature. At equilibrium, the SPME sampling method has the maximum sensitivity, but for practical purposes, extraction time is always limited. Thus, the effect of temperature on the rate of extraction is also very important because, the mass transport rate of analytes from the sample matrix into the fiber coating is faster at higher temperatures. The compositive effect of these two factors gave the results as shown in Fig. 3b. The optimal extraction temperature was at 70 °C.

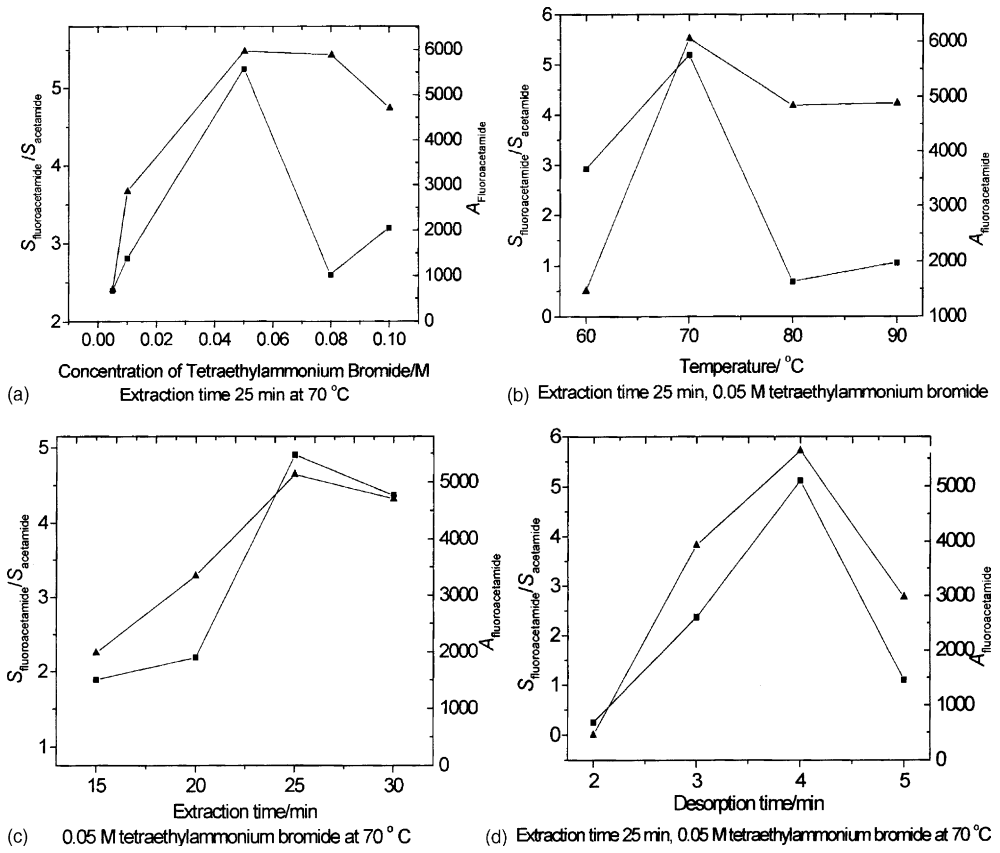

Fig. 3. Effects of (a) tetraethylammonium bromide concentration, (b) temperature, (c) extraction time, and (d) desorption time, on the ratio of peak areas of fluoroacetamide to acetamide (■) and average abundance of fluoroacetamide (▲) for SPME in sample C.

3.2.5. Dependence of sensitivity on extraction time

At the extraction temperature of 70 °C, with increasing extraction time the amount of fluoroacetamide absorbed in the fiber coating increased, the abundance of the characteristic ion peak also increased. The change for a larger molecule was more obvious. So the peak area ratio of fluoroacetamide to acetamide increased (Fig. 3c). The extraction reached equilibrium and the maximum sensitivity was 25 min. After an extraction time of 25 min, however, both abundance and the peak area ratio decreased slightly due to more difficult desorption during GC/MS analysis. So the extraction time of 25 min was selected for the SPME process.

3.3. Effect of desorption time

Desorption is closely related to the efficiency of chromatographic separation and the precision of quantitative analysis and has a great influence on the full utilization of the potential of SPME. At a desorption temperature of 200 °C, the effect of desorption time on the sensitivity time was showed in Fig. 3d. The peak area ratio and the abundance of fluoroacetamide increased with increasing desorption time up to 4 min. The further increase of desorption time resulted in some fluoroacetamide molecules to be lost, thus the response decreased. This work selected a desorption time of 4 min at which the absorbed components could be completely desorbed from the coating.

3.4. Calibration curve and detection limit

With increasing fluoroacetamide concentration in blood samples from 5 to 90 μg/ml, the ratio of the average characteristic ion peak areas of fluoroacetamide to acetamide increased linearly. The correlation coefficient of the plot of this ratio versus fluoroacetamide concentration was above 0.99 (six-point calibration). At the signal-to-noise ratio of three, the detection limit of the SPME–GC/MS analysis was 1.0 μg/ml.

3.5. Recovery and precision

In order to confirm the validity of this method, known amounts of fluoroacetamide was spiked into the blood and urine samples, and their recoveries were calculated to be between 80.7 and 103.5%. The average recovery was 92.2% with a relative standard deviation of 11.2%. At the fluoroacetamide concentration of 20 μg/ml, the relative standard deviation for five continuous determinations by the proposed method was 4.5%, while it was 5.3% for five determinations with a 2 h interval within 1 day. The inter-day

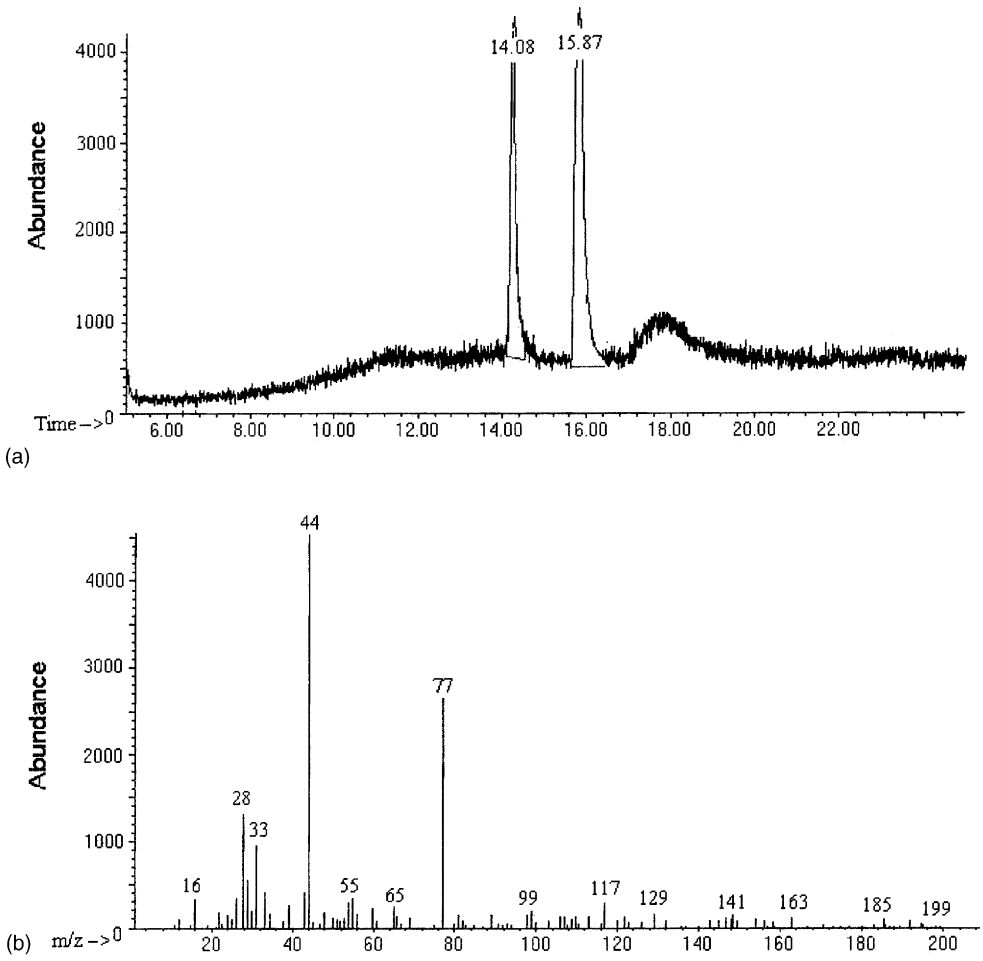

Fig. 4. (a) Total ion current scan of a blood sample from a woman's heart and (b) mass spectrum corresponding to fluoroacetamide in the poisoned sample in presence of 22.5 μg/ml acetamide.

variation coefficient at this concentration among 5 days was 6.2%. Thus, this method could be used for the identification and quantitative determination of fluoroacetamide in poisoned sample.

3.6. Application to the analysis of real sample

A blood specimen was obtained from the heart of a poisoned woman's body in a real case. After 45 μl of 2.0 mg/ml internal standard was added into the 2.0 ml sample, it was treated according to the method described in Section 2.3 and was then analyzed with the proposed SPME–GC/MS method. Fig. 4 shows its total-ion chromatogram and the mass spectrum corresponding to fluoroacetamide in the poisoned sample. The total-ion chromatogram shows two peaks corresponding to fluoroacetamide and acetamide at the retention times of 14.08 and 15.87 min, respectively. The peak at 14.08 min gives the molecular and fragment ion peaks at *m*/*z* 77, 44, and 33, indicative of the presence of fluoroacetamide in the poisoned woman's body, which is in agreement with the result obtained using the sulfur–indigo reaction.

From the ratio of the characteristic ion peak areas of fluoroacetamide to internal standard and the calibration curve, a fluoroacetamide concentration of 17.6 μg/ml in the blood specimen was obtained. The relative standard deviation for three determinations was 4.6%.

4. Conclusions

This work proposes an efficient method for the identification and quantitative determination of fluoroacetamide. The SPME is an ideal sample preparation technique for the extraction of fluoroacetamide from a complex sample. The addition of tetraethylammonium bromide into the extraction system increases the extraction efficiency. The best operating SPME conditions are at pH 7.0 and 70 °C with presence of 0.05 M tetraethylammonium bromide and an extraction time of 25 min. The desorption time is 4 min at 200 °C. This method simplifies the sample pretreatment process and possesses the advantages of convenience, speed, environmental-friendly nature, and non-interference. It is selective and sensitive, and can be applied to the analysis

of practical samples. We believe that this method provides a useful tool for the determination of fluoroacetamide and the detection of relative criminal cases.

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References

[1] J.W. Allender, J. Anal. Toxicol. 14 (1990) 45.
[2] H.Z. Wang, Jiangxi J. Crim. Technol. 2 (1987) 7.
[3] J.Z. Zhang, Chin. J. Crim. Technol. 4 (1987) 20.
[4] J.X. Zhu, L. Man, J. Zhu, Chin. J. Forensic Med. 14 (1998) 220.
[5] B.K. Logan, D.T. Stafford, I.R. Tebbet, J. Anal. Toxicol. 14 (1990) 154.
[6] S.Z. Feng, Z.S. Yu, Chin. J. Forensic Med. 15 (1999) 91.
[7] R. Sawyer, B.G. Cox, E.J. Dixon, J. Thomsen, J. Sci. Food Agric. 18 (1967) 287.
[8] H.M. Stevens, A.C. Moffat, J.T. Drayton, Forensic Sci. 8 (1976) 131.
[9] H.M. Stahr, J. Assoc. Off. Anal. Chem. 60 (1977) 1434.
[10] I. Okuno, D.L. Meeker, R.R. Felton, J. Assoc. Off. Anal. Chem. 65 (1982) 1102.
[11] C.R. Ray, L.O. Post, J.C. Reagor, J. Assoc. Off. Anal. Chem. 64 (1981) 19.
[12] J.O. Egekeze, F.W. Oehme, Toxicol. Lett. 4 (1979) 461.
[13] J.A. Peters, K.J. Baxter, Bull. Environ. Contam. Toxicol. 11 (1974) 177.
[14] I. Okuno, G.E. Connolly, P.J. Savarie, C.P. Breidenstein, J. Assoc. Off. Anal. Chem. 67 (1984) 549.
[15] F.Y. Guan, L. Liu, Y. Lou, Environ. Chem. 14 (1995) 196.
[16] H.H. Casper, T.L. McMahon, G.D. Paulson, J. Assoc. Off. Anal. Chem. 68 (1985) 722.
[17] T. Vartiainen, P. Kauranen, Anal. Chim. Acta 157 (1984) 91.
[18] M.A. Kirms, L.M. Kirms, J. Forensic Sci. 47 (2002) 573.
[19] F.Y. Guan, Z.C. Miao, Y. Lou, Chin. Sci. Bull. 39 (1994) 154.
[20] S. Gentili, A. Torresi, R. Marsili, M. Chiarotti, T. Macchia, J. Chromatogr. B 780 (2002) 183.
[21] A. Ribeiro, M.H. Neves, M.F. Almeida, A. Alves, L. Santos, J. Chromatogr. A 975 (2002) 267.
[22] A. Navalon, A. Prieto, L. Araujo, J.L. Vilchez, J. Chromatogr. A 975 (2002) 355.
[23] W.J. Wu, D.L. Ashley, C.H. Watson, Anal. Chem. 74 (2002) 4878.
[24] T.A. Brettell, K. Inman, N. Rudin, R. Saferstein, Anal. Chem. 73 (2001) 2735.
[25] C.L. Arthur, J. Pawliszyn, Anal. Chem. 62 (1990) 2145.
[26] D. Louch, S. Motlagh, J. Pawliszyn, Anal. Chem. 64 (1992) 1187.
[27] K.D. Buchholz, J. Pawliszyn, Anal. Chem. 66 (1994) 160.
[28] Z. Zhang, M.J. Yang, J. Pawliszyn, Anal. Chem. 66 (1994) 844A.
[29] T. Kumazawa, X. Lee, M. Tsai, H. Seno, A. Ishii, K. Sato, Jpn. J. Forensic Toxicol. 13 (1995) 25.
[30] Y. Iwasaki, M. Yashiki, N. Nagasawa, T. Miyazaki, T. Kojima, Jpn. J. Forensic Toxicol. 13 (1995) 189.
[31] K. Ameno, C. Cuke, S. Ameno, Can. Soc. Forensic Sci. 29 (1996) 43.
[32] K.P. Kirkbride, G. Klass, P.E. Pigou, J. Forensic Sci. 43 (1998) 76.
[33] K.G. Furton, L. Wu, J.R. Almirall, J. Forensic Sci. 45 (2000) 857.
[34] K.E. Kongshaug, S. Pedersen-Bjergaard, K.E. Rasmussen, M. Krogh, Chromatographia 50 (1999) 247.
[35] J.T. Liu, K. Hara, S. Kashimura, M. Kashiwagi, M. Kageura, J. Chromatogr. B 758 (2001) 95.
[36] C.J. Koester, B.D. Andresen, P.M. Grant, J. Forensic Sci. 47 (2002) 1002.