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Solid phase extraction–ultra performance liquid chromatography for the determination of acrylamide in mainstream cigarette smoke

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A new method was developed to detect acrylamide in mainstream cigarette smoke by solid phase extraction–ultra performance liquid chromatography (SPE–ULPC).

The discovery of the adventitious formation of the potential cancer-causing agent acrylamide in foods has raised much concern in recent years.[1–5] Acrylamide is a neurotoxin, which is easily absorbed by digestive tract or other pathways.[6–9]

It is generally considered that acrylamide is formed by the thermal treatment of a mixture of amino acids and reducing sugars through the Maillard reaction.[10–12]

Analysis methods for acrylamide in foods use GC/MS or LC/MS.[13,14] GC/MS method needs derivatization process and complicated pre-treatment process. Here, we report a new method to detect acrylamide in mainstream smoke by ultra performance liquid chromatography (UPLC). It is superior to the above method in speed, cost and accuracy.

UPLC was used as follows: ACQUITY UPLC™ BEH C_{18} 1.7 μm 2.1×50 mm column; acetonitrile–water (6:94, v/v) as the mobile phase; column temperature, 40 °C; TUV detector was set to 202 nm; analytical time, 6 min.

Acrylamide in cigarette smoke was formed by combustion, because no signal has been found from the unheated tobacco.

The Cambridge filter pad was used to collect acrylamide from smoke in smoking machine. In the pre-treatment process, distilled water was used to extract acrylamide from the Cambridge filter pad (Figure 1). Acetonitrile was also used as extracting solvent, but the effect was poor as there were many impurity peaks in chromatograms (Figure 2).

When acetonitrile is used as eluent in solid phase extraction (SPE) (Figures 1 and 2), its effect is better than that of distilled water (Figure 3). This result should be attributed to the polarity of acrylamide. In SPE process, C_{18} solid phase extraction column is activated by 3 ml of water and 3 ml of acetonitrile before using, and 4 ml of acetonitrile was used to elute the target compound from C_{18} column. For investigation of eluting effect, the eluent is collected per 0.5 ml, *i.e.*, collected from 0 to 0.5 ml, 0.5 to 1 ml, 1 to 1.5 ml, 1.5 to 2 ml, 2 to 2.5 ml, 2.5 to 3 ml, 3 to 3.5 ml and 3.5 to 4 ml of eluent, and detected by UPLC by standard procedure. The result shows that there is almost no signal on the intervals 0 to 0.5 ml and 3 to 4 ml (Figure 4), thus it is only necessary to collect 0.5 to 3 ml of eluent in normal experiment for detection of acrylamide.

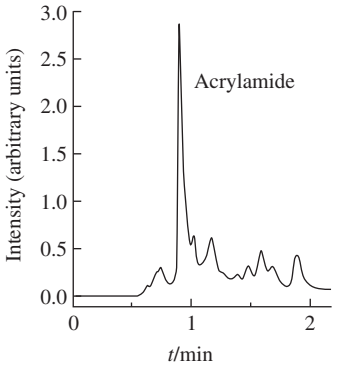

Figure 2 Chromatogram of sample with acetonitrile as extracting solvent (acetonitrile as eluent in SPE).

The standard solution of acrylamide (acetonitrile as a solvent) was scanned by a UV-VIS spectrometer, and the maximum absorption peak was at 202 nm. Therefore, the wavelength of Tunable

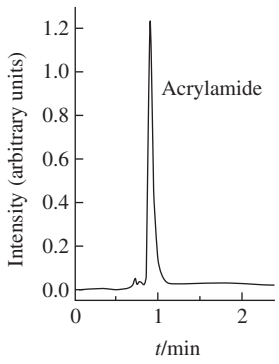

Figure 1 Chromatogram of sample with distilled water as extracting solvent (acetonitrile as eluent in SPE).

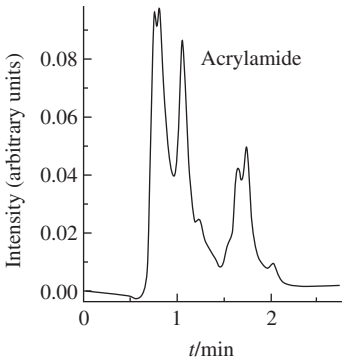

Figure 3 Chromatogram of sample with distilled water as extracting solvent (distilled water as eluent in SPE).

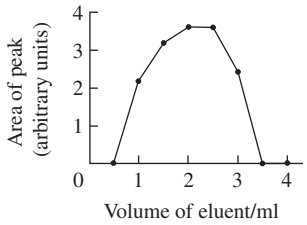

Figure 4 Correlation between volume of eluent and eluting peak area.

UV detector (TUV) was set to 202 nm. The mobile phase was the mixture of acetonitrile and water, which was selected from various ratios (10:90, 7:93, 6:94 and 5:95, v/v), and the best separation effect was achieved at 6:94 ratio. The flow rate of mobile phase was 0.15 ml min^{-1}.

The calibration curve of acrylamide shows good linearity in the range of 0.1–10 μg cm^{-3} with a correlation coefficient of 0.9999. As shown in Table 1, the average recovery is 98.65%. The detection limit is 10 ng cm^{-3} ($S/N = 3$), and relative standard deviation (RSD) is 2.11% (Table 1). It was found that the contents of acrylamide in mainstream are 4.753, 6.889 and 7.991 μg per cigarette in flue-cured cigarette of three main trademarks of Hongta Group.

In conclusion, a new simple and precise method for the determination of acrylamide in tobacco mainstream smoke was developed and the pre-treatment process was optimized. The target compound can be extracted and purified easily, and detected without derivatization in a very short time (6 min). The method is very suitable for batch determination of acrylamide in cigarette smoke.

Table 1 The average recovery and RSD of acrylamide.

Added/ ng cm^{-3}	Testing time/min	Average recovery (%)	RSD (%)
100	6	99.23	2.11
1000	6	99.51	2.53
10000	6	97.21	3.01
Average		98.65	2.55

References

1 P. C. Augas, M. J. Fitzhenry, G. Giannikopoulos and P. Varelis, *Anal. Bioanal. Chem*., 2006, **385**, 1526.
2 J. Rosen and K.-E. Hellenas, *Analyst*, 2002, **127**, 880.
3 L. A. Mucci, H.-O. Adami and A. Wolk, *Int. J. Cancer*, 2005, **1**, 169.
4 V. Faca, M. Coram, D. Phanstiel, V. Glukhova, Q. Zhang, M. Fitagibbon, M. Mclntosh and S. Hanash, *J. Proteome Res*., 2006, **5**, 2009.
5 T. D. Wilde, B. D. Meulenaer, F. Mestdagh, Y. Govaert, W. Ooghe, S. Fraselle, K. Demeulemeester, C. V. Peteghem, A. Calus, J.-M. Degroodt and R. Verhe, *J. Agric. Food Chem*., 2006, **54**, 2199.
6 E. Tareke, P. Rydberg, P. Carlsson, S. Eriksson and M. Tornqvist, *J. Agric. Food Chem*., 2002, **50**, 4998.
7 J. G. Hogervorst, L. J. Schouten, E. J. Konings, P. A. Brandt and R. A. Goldbohm, *Cancer Epidemiol. Biomarkers Prev*., 2007, **16**, 2304.
8 E. Wirfalt, B. Paulsson, M. Tornqvist, A. Axmon and L. Hagmar, *Eur. J. Clin. Nutr*., 2008, **62**, 314.
9 T. Bjellaas, P. T. Olesen, H. Frandsen, M. Haugen, L.-H. Stolen, J.-E. Paulsen, J. Alexander, E. Lundanes and G. Becher, *Toxicol. Sci*., 2007, **98**, 110.
10 A. Becalski, B. P.-Y. Lau, D. Lewis and S. W. Seaman, *J. Agric. Food Chem*., 2003, **51**, 802.
11 D. S. Mottram, B. L. Wedzicha and A. T. Dodson, *Nature*, 2002, **419**, 448.
12 V. A. Yaylayan, A. Wnorowski and C. P. Locas, *J. Agric. Food Chem*., 2003, **51**, 1753.
13 K. Mastovska and S. J. Lehotay, *J. Agric. Food Chem*., 2006, **54**, 7001.
14 C. T. Kim, E.-S. Hwang and H. J. Lee, *Food Chem*., 2007, **101**, 401.

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