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Title: Determination of nitroalkanes in mainstream cigarette smoke by heart-cutting multidimensional gas chromatography system coupled with mass spectrometry detection

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2 gas chromatography system coupled with mass spectrometry detection

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#### 12 13 14 Highlights

15 A GC-GC/MS method was developed and validated for the determination of 6  
16 nitroalkanes in mainstream cigarette smoke.

17 The home-made double oven GC-GC/MS equipment used could realize  
18 cryo-concentration at the inlet of the second column.

19 The GC-GC/MS method exhibited higher sensitivity and lower noise than GC/MS.

20 The method required only a simple vapor phase collection step before injection.

21 The GC-GC/MS method avoided the false positive/negative results that appeared in  
22 GC/MS.

#### 23 24 ABSTRACT

25 In this paper, heart-cutting two-dimensional GC/MS (GC-GC/MS) method in  
26 combination with a simple sample collection procedure was developed for the  
27 determination of 6 nitroalkanes in mainstream cigarette smoke. The method could  
28 remove large amounts of impurities on-line in the first polar column by heart-cuts and  
29 separate from the left interferences in a second mid-polar column. And the target  
30 compounds could be focused at the inlet of the second column by cryo-concentration.  
31 Compared to conventional GC/MS, GC-GC/MS achieved a lower noise level and  
32 sensitivity at least an order of magnitude higher. Furthermore, the GC-GC/MS method  
33 could avoid the false negative and false positive results that appeared in the compared  
34 conventional GC/MS analysis. By trapping the vapor phase of 20 cigarettes smoke,  
35 the LODs and LOQs of the nitroalkanes were 1.3 to 9.8 and 4.3 to 32.6 ng/cigarette,  
36 respectively, and all linear correlation efficiencies were larger than 0.999. The  
37 validation results also indicate that the method has high accuracy (spiked recoveries  
38 between 84% and 102%) and good repeatability (RSD between 7.2% and 9.4%). The  
39 developed method was applied to analyze 1 Kentucky reference cigarette (3R4F) and  
40 10 Chinese commercial brands of cigarettes. The research results indicated that  
41 nitromethane, nitroethane, 2-nitropropane and 1-nitro-n-pentane were detected in  
42 mainstream cigarette smoke, but 1-nitro-n-butane and 2-nitropropane, which were  
43 reported by one previous study, were not detected in all cigarette samples.

44 *Keywords:* Heart-cut; Two-dimensional GC; Cold trap; Mainstream smoke;

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