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Simultaneous Determinations of 17 abused drugs and organophosphorus pesticides in human blood by GPC-GCMS

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Introduction

On-line gel permeation chromatography-gas chromatography/mass spectrometry (GPC-GCMS) is a unique technical to cleanup sample that reduce the time of sample preparation. GPC can efficiently separates fats, protein and pigments from samples, due to this advantage, GPC-GCMS was widely used to pesticides analysis. In this work, a new method was developed for rapid determinations of 17 abused drugs and organophosphorus pesticides in the human blood by GPC-GCMS. The modified QuEChERS method was used for sample preparation. The human blood samples were extracted with acetonitrile, then was purified by PSA, C18 and $MgSO_4$ to remove most of the fats, protein and pigments in samples, then after on-line GPC-GC/MS analysis which further removed macromolecular interference material, such as protein and cholesterol, the background interference brought about by the complex matrix in samples was effectively reduced. For all of analytes, recoveries in the acceptable range of 70~115% and repeatability (relative standard deviations, RSD)≤5% (n=3) were achieved for both matrices at spiking levels of 0.05 and 0.5 μg/mL. The limitis of detection were 0.001~0.012 mg/L. The method is simple, rapid and characterized with acceptable sensitivity and accuracy to meet the requirements for the analysis of abused drugs and organophosphorus pesticides in the human blood.

Experimental

Sample pretreament

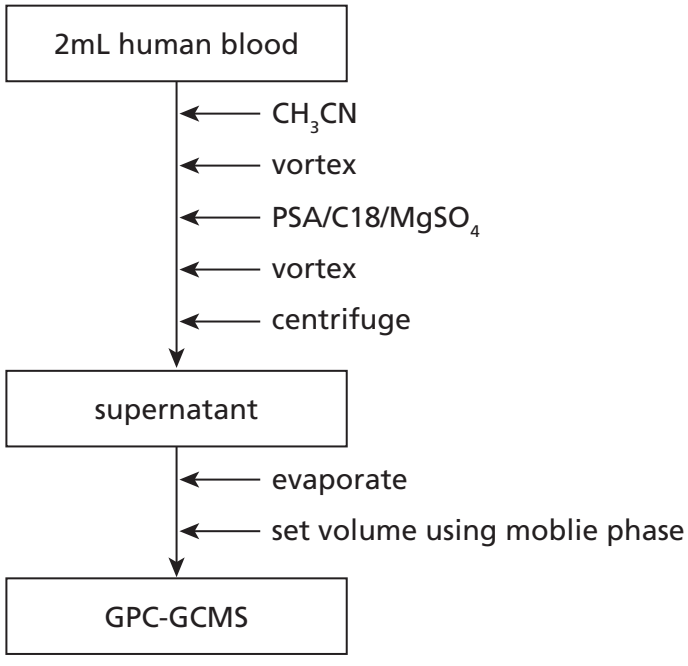

Figure 1 Schematic flow diagram of the sample preparation

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Instrument

Analytical Conditions

GPC	
Mobile phase	: Acetone/Cyclohexane = 3/7 (v/v)
Flow rate	: 0.1mL/min
Column	: Shodex CLNpak EV-200 (2mm (ID)x150mm)
Oven temperature	: 40 °C
Injection Volume	: 20 µL

GCMS	
Column	: deactivated silica tubing [0.53mm (ID)x5m (L)] +pre-column Rtx-5ms [0.25mm (ID)x5m (L)] Rtx-5ms [0.25mm (ID)x30m (L), Thickness 0.25µm]
Injector	: PTV
Injector time program	: 120 °C (5min)-(100 °C/min)-250 °C (35.7min)
Oven temperature program	: 82 °C (5min)-(8 °C/min)-300 °C (9.75min)
Linear velocity	: 48.8cm/s
Ion Source temperature	: 210 °C
Interface temperature	: 280 °C

Figure 2 Shimadzu GPC-GCMS

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Results

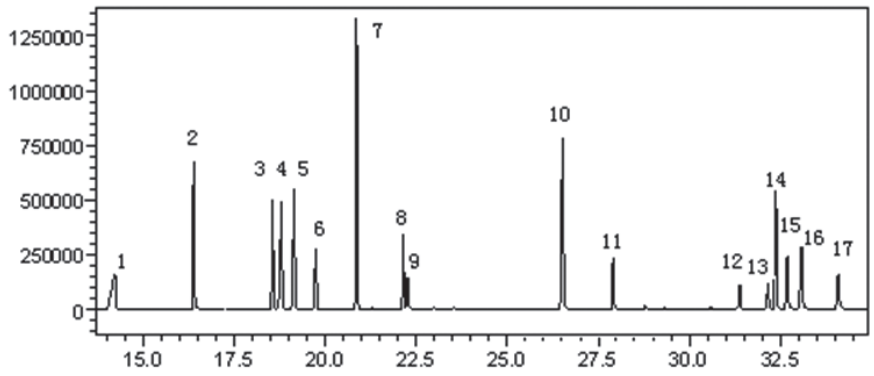

Figure 3 Chromatograms of SIM from mix standards

Table 1 Retention times, linear equation, correlation coefficients, limitis of detection (LODs, S/N≥3), limitis of quantification (LOQs, S/N≥10), the averager recoveries and the relative standard deviations (RSDs, n=3) for drugs and pesticides

No.	Compound Name	t_R (min)	Correlation Coefficient*	LOD (mg/L)	LOQ (mg/L)	0.05 μg/mL		0.5 μg/mL	
						Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
1	Acephate	14.200	0.9995	0.004	0.012	85.3	3.58	91.0	3.30
2	Benzocaine	16.417	0.9996	0.004	0.012	72.4	1.72	75.3	3.34
3	Dimethoate	18.592	0.9993	0.002	0.006	110.7	2.27	99.0	4.04
4	Amobarbital	18.817	0.9992	0.002	0.006	103.7	3.10	97.3	1.57
5	Pentobarbital	19.167	0.9995	0.003	0.009	82.7	2.52	89.0	3.37
6	Meprobamate	19.758	0.9993	0.002	0.006	71.4	3.57	76.7	2.00
7	Lidocaine	20.883	0.9992	0.002	0.006	77.3	3.25	80.0	3.30
8	Parathion	22.175	0.9993	0.003	0.009	87.7	1.32	89.3	1.71
9	Phenobarbital	22.267	0.9987	0.007	0.021	78.0	1.28	79.0	2.53
10	Carbamazepine	26.533	0.9994	0.003	0.009	98.3	1.55	97.7	3.25
11	Diazepam	27.933	0.9996	0.002	0.006	86.0	2.01	89.0	2.25
12	Nitrazepam	31.400	0.9976	0.012	0.036	99.0	1.01	99.3	3.24
13	Clonazepam	32.175	0.9985	0.011	0.033	110.0	1.57	102.0	2.94
14	Clozapine	32.400	0.9991	0.001	0.003	111.7	1.37	104.3	2.41
15	Estazolam	32.725	0.9992	0.001	0.003	103.3	1.48	103.7	2.01
16	Alprazolam	33.133	0.9996	0.003	0.009	87.3	1.75	92.3	1.65
17	Triazolam	34.175	0.9996	0.006	0.018	81.3	2.56	84.7	2.97

* Concentration range: 50μg/L~1000μg/L

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Conclusion

A very quick, easy, effective, reliable method in human blood based on modified QuEChERS method was developed using GPC-GCMS. The performance of the method was very satisfactory with results meeting validation criteria. The method has been successfully applied for determination of human blood samples and ostensibly has further application opportunities, e.g. biological samples.

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