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# Simultaneous Determinations of 20 kinds of common drugs and pesticides in human blood by GPC-GC-MS/MS

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## Introduction

On-line gel permeation chromatography-gas chromatography/mass spectrometry (GPC-GC-MS) is a unique technique to cleanup sample that reduce the time of sample preparation. GPC can efficiently separates fats, protein and pigments from samples, due to this advantage, on-line GPC is widely used for pesticide analysis. Meanwhile, compared to widely used GC-MS, GC-MS/MS techniques provide much better selectivity thus significantly lower detection limits. In this work, a new method was developed for rapid determination of 20 common drugs and pesticides in human blood by GPC-GC-MS/MS. The modified QuEChERS method was used for sample preparation.

## Experimental

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/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.

### Sample pretreament

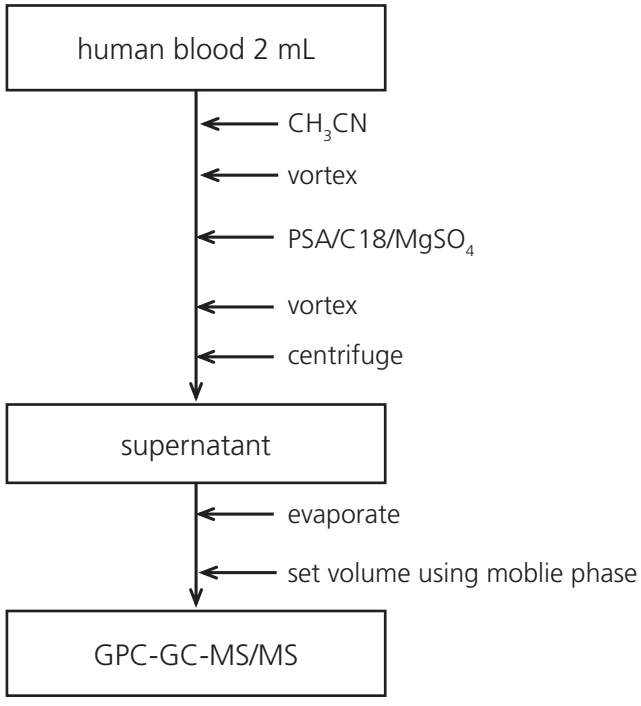


Figure 1 Schematic flow diagram of the sample preparation

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## Instrument

**GPC**

| | |
|---|---|
| Mobile phase | : acetone/cyclohexane (3/7, v/v) |
| Flow rate | : 0.1mL/min |
| Column | : Shodex CLNpak EV-200 (2 mmI.D. x 150 mmL.) |
| Oven temperature | : 40 °C |
| Injection volume | : 10 µL |

**GCMS-TQ8030**

| | |
|---|---|
| Column | : deactivated silica tubing [0.53 mm(ID) x 5 m(L)]<br>+pre-column Rtx-5ms [0.25 mm(ID) x 5 m(L)]<br>Rtx-5ms [0.25mm(ID) x 30 m(L), Thickness: 0.25 µm] |
| Injector | : PTV |
| Injector time program | : 120 °C(4.5min)-(80 °C/min)-280 °C(33.7 min) |
| Oven temperature program | : 82 °C(5min)-(8 °C/min)-300 °C(7.75 min) |
| Linear velocity | : 48.8 cm/sec |
| Ion Source temperature | : 210 °C |
| Interface temperature | : 300 °C |

# Results

For all of analytes, recoveries in the acceptable range of 70~120% and repeatability (relative standard deviations, RSD)≤5% (n=3) were achieved for matrices at spiking levels of 0.01 µg/mL. The limitis of detection were 0.03~4.4 µg/L.

The method is simple, rapid and characterized with acceptable sensitivity and accuracy to meet the requirements for the analysis of common drugs and pesticides in the human blood.

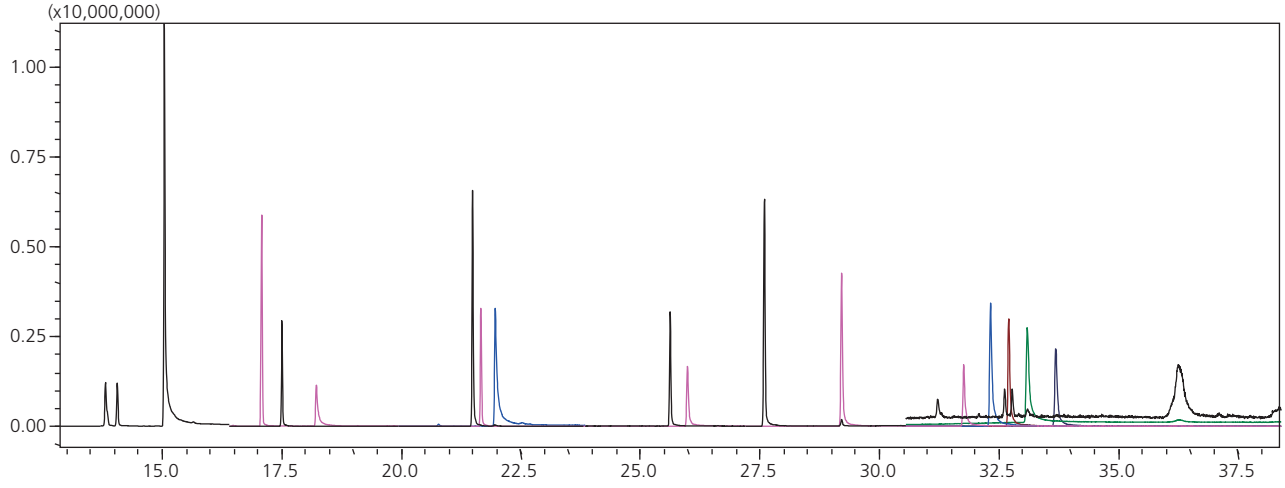


Figure 2 MRM chromatograms of standard mixture

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Table 1 Results of method validation for drugs and pesticides
(Concentration range: 5-100 µg/L, LODs: S/N≥3, LOQs: S/N≥10, RSDs: n=3)

| No. | Compound Name | $t_R$ (min) | Correlation Coefficient* | LOD (µg/L) | LOQ (µg/L) | 0.01 µg/mL | |
|---|---|---|---|---|---|---|---|
| | | | | | | Recovery (%) | RSD (%) |
| 1 | Dichlorvos | 10.795 | 0.9993 | 0.103 | 0.345 | 72.9 | 2.99 |
| 2 | Methamidophos | 11.800 | 0.9994 | 0.023 | 0.076 | 85.3 | 3.58 |
| 3 | Barbital | 15.210 | 0.9994 | 0.018 | 0.058 | 72.4 | 1.72 |
| 4 | Sulfotep | 17.580 | 0.9995 | 0.011 | 0.037 | 110.7 | 2.27 |
| 5 | Dimethoate | 18.310 | 0.9993 | 0.400 | 1.333 | 103.7 | 3.10 |
| 6 | Malathion | 21.555 | 0.9997 | 0.005 | 0.016 | 82.7 | 2.52 |
| 7 | Chlorpyrifos | 21.715 | 0.9996 | 0.010 | 0.033 | 85.7 | 3.57 |
| 8 | Phenobarbital | 22.000 | 0.9995 | 0.353 | 1.177 | 79.6 | 3.25 |
| 9 | Parathion | 22.180 | 0.9993 | 0.003 | 0.009 | 92.3 | 3.17 |
| 10 | Triazophos | 25.675 | 0.9994 | 0.046 | 0.155 | 87.7 | 1.32 |
| 11 | Zopiclone deg. | 26.025 | 0.9993 | 0.189 | 0.631 | 83.5 | 1.28 |
| 12 | Diazepam | 27.635 | 0.9992 | 0.007 | 0.022 | 98.3 | 1.55 |
| 13 | Midazolam | 29.250 | 0.9994 | 0.048 | 0.160 | 87.1 | 2.01 |
| 14 | Zolpidem | 31.225 | 0.9993 | 1.298 | 4.325 | 99.3 | 1.01 |
| 15 | Clonazepam | 31.795 | 0.9995 | 0.432 | 1.440 | 110.0 | 1.57 |
| 16 | Estazolam | 32.335 | 0.9994 | 0.092 | 0.305 | 103.7 | 1.37 |
| 17 | Clozapine | 32.400 | 0.9991 | 0.050 | 0.167 | 100.6 | 3.12 |
| 18 | Alprazolam | 32.730 | 0.9993 | 0.028 | 0.095 | 103.3 | 1.48 |
| 19 | Zolpidem | 33.095 | 0.9995 | 1.027 | 3.425 | 87.3 | 1.75 |
| 20 | Triazolam | 33.700 | 0.9992 | 0.027 | 0.091 | 81.3 | 2.56 |

# Conclusion

A very quick, easy, effective, reliable method in human blood based on modified QuEChERS method was developed using GPC-GCMS-TQ8030. 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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