

Benzoyl Isothiocyanate Modified Surface of Silica Gel as Extraction Material for Adsorpting Steroid Hormones in Water

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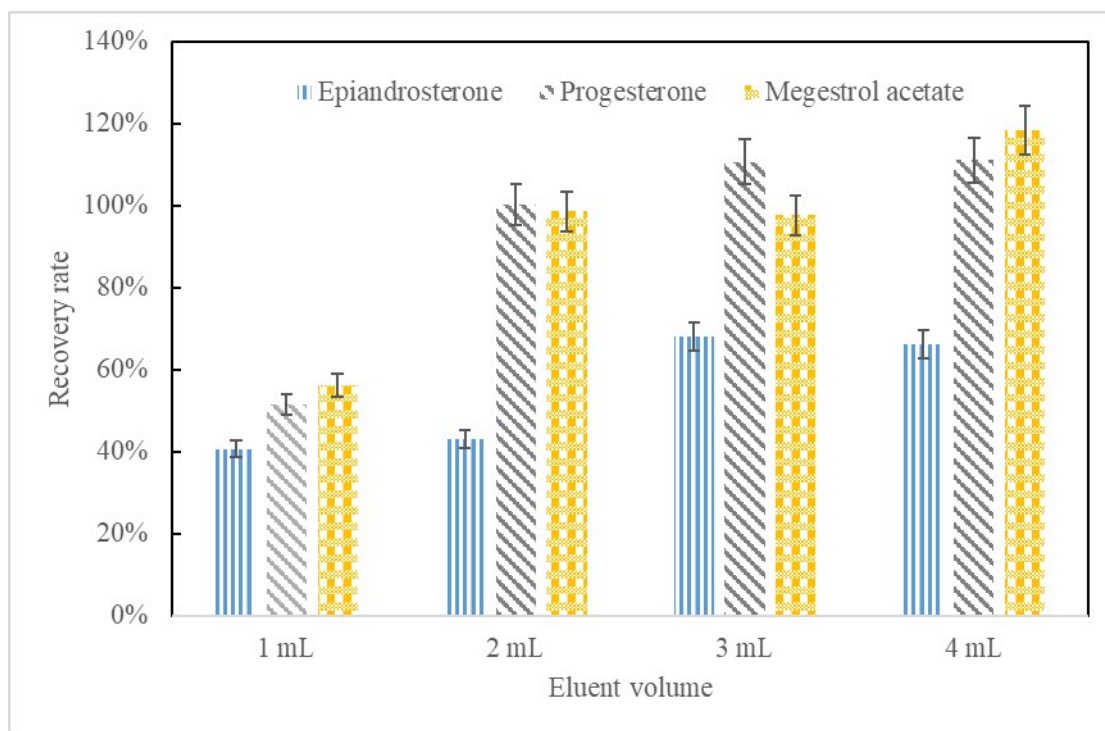


Fig. S1. Recovery rate of the modified silica gel for extraction of steroid hormones with different volume of eluent.

TABLE S1 The gradient elution program for HPLC-MS/MS

Gradient time/min	Mobile phase A/%	Mobile phase B/%
1.00	50	50
7.00	50	50
15.00	0	100
17.00	0	100
17.10	70	30
21.00	Stop	

Mobile phase A: 0.1% formic acid in water; Mobile phase B: acetonitrile

TABLE S2 Qualitative, quantitative ion and mass spectrometric parameters for steroid hormone drugs

Steroid hormones	Ionization mode	Precursor ion (m/z)	Product ions (m/z)	Q1 Pre (V)	CE	Q3 Pre (V)	Retention time (min)
Epiandrosterone	ESI+	291.0	273.0	30.0	11.0	17.0	7.728
			255.0	12.0	17.0	25.0	
Progesterone	ESI+	315.2	97.2	11.0	23.0	16.0	10.737
			109.0	15.0	27.0	17.0	
Megestrol acetate	ESI+	385.1	267.2	26.0	19.0	27.0	11.011
			325.2	13.0	16.0	21.0	

CE: capillary electrophoresis

TABLE S3 Recovery (R_r (%) \pm SD(%), n=3) of 3 steroid hormones in the 5 cyclic extractions by the modified silica gel.

Steroid hormones	Cycle index				
	1st	2nd	3rd	4th	5th
Epiandrosterone	58.7 \pm 4.3	56.0 \pm 2.5	57.9 \pm 3.4	55.5 \pm 0.9	54.7 \pm 1.3
Progesterone	93.8 \pm 2.5	88.7 \pm 3.3	82.9 \pm 4.9	81.1 \pm 4.7	85.9 \pm 6.5
Megestrol acetate	105.1 \pm 5.5	82.0 \pm 3.2	83.8 \pm 2.2	81.8 \pm 4.0	82.1 \pm 5.5

TABLE S4 Comparison of the presented extraction method with other published methods towards steroid hormones.

Methods	LOD/ $\mu\text{g}\cdot\text{L}^{-1}$	Sample volume/mL	Compounds	$R_r/\%$	Matrices	Ref.
SPE- GCxGC-TOF MS	0.007-0.114	5	25	70.0-123	Water	1
SPE- HPLC	0.25	15	1	97.4-99.2	Beef	2
SPDE-LC-MS/MS	0.5-3.4	1000	15	75.6-101	Water	3
SPE-MEKC	1.15-1.59	10	3	93.5-104	Water	4
MSPE-GC-T/MS	4-8	250	3	94.6-110	Water	5
SPE-HPLC-MS/MS	0.02-0.13	5	3	54.7-112	Water	This study

REFERENCES

1. R. Stepan, P. Cuhra and S. Barsova, *Food Addit Contam A*, 2008, **25**, 557-565.
2. Y. R. Gong, Y. L. Niu, X. H. Gong, M. H. Ma, X. W. Ren, W. H. Zhu, R. M. Luo and B. L. Gong, *J Sep Sci*, 2015, **38**, 1254-1261.
3. L. Sun, W. Yong, X. G. Chu and J. M. Lin, *J Chromatogr A*, 2009, **1216**, 5416-5423.
4. F. K. Liu, *J Chromatogr A*, 2008, **1215**, 194-202.
5. R. A. Perez, B. Albero, J. L. Tadeo, E. Molero and C. Sanchez-Brunete, *Chromatographia*, 2014, **77**, 837-843.