

*Supplementary information*

**Simultaneous Analysis of Residual Prohibited Doping Substances  
in Foods using Gas Chromatography–Tandem Mass Spectrometry**

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**Table S1** Suppliers of standard compound

| Compound                                                                                               | Supplier           | Location                  |
|--------------------------------------------------------------------------------------------------------|--------------------|---------------------------|
| 1-Androstenediol                                                                                       | NMI                | West Lindfield, Australia |
| 1-Androstenedione                                                                                      | NMI                | West Lindfield, Australia |
| 19-Norandrosterone                                                                                     | NMI                | West Lindfield, Australia |
| 19-Noretiocholanolone                                                                                  | NMI                | West Lindfield, Australia |
| 1-Testosterone                                                                                         | Steraloids         | Newport, RI, USA          |
| 5 $\alpha$ -Androst-1-ene-3 $\alpha$ -ol-17-one (1-Testosterone-M)                                     | NMI                | West Lindfield, Australia |
| 3-OH-4-methoxy-tamoxifen                                                                               | NMI                | West Lindfield, Australia |
| 3 $\alpha$ -OH-tibolone                                                                                | TRC                | Toronto, Canada           |
| 3 $\beta$ -OH-tibolone                                                                                 | TRC                | Toronto, Canada           |
| 4-OH-cyclofenil                                                                                        | NMI                | West Lindfield, Australia |
| 4-OH-testosterone                                                                                      | NMI                | West Lindfield, Australia |
| 6-OH-bromantan                                                                                         | TRC                | Toronto, Canada           |
| 4-Androsten-3,6,17-trione (6-OXO)                                                                      | Steraloids         | Newport, RI, USA          |
| 4-Androsten-6 $\alpha$ -ol-3,17-dione (6-OXO-M)                                                        | Steraloids         | Newport, RI, USA          |
| Bolandioli                                                                                             | Steraloids         | Newport, RI, USA          |
| Bolasterone                                                                                            | The Upjohn Company | Hastings, MI, USA         |
| 7 $\alpha$ ,17 $\alpha$ -Dimethyl-5 $\beta$ -androstane-3 $\alpha$ ,17 $\beta$ -diol (Bolasterone-M)   | NMI                | West Lindfield, Australia |
| Boldenone                                                                                              | Sigma              | St. Luis, MO, USA         |
| 5 $\beta$ -Androst-1-en-17 $\beta$ -ol-3-one (Boldenone-M)                                             | NMI                | West Lindfield, Australia |
| Boldione                                                                                               | Steraloids         | Newport, RI, USA          |
| Calusterone                                                                                            | NMI                | West Lindfield, Australia |
| 7 $\beta$ ,17 $\alpha$ -Dimethyl-5 $\beta$ -androstanediol (Calusterone-M)                             | NMI                | West Lindfield, Australia |
| Carphedone                                                                                             | NMI                | West Lindfield, Australia |
| Nandrolone (19-nortestosterone)                                                                        | AKZO               | Cambridge, United Kingdom |
| 17 $\alpha$ -Methyl-5 $\beta$ -androstane-3 $\alpha$ ,17 $\beta$ -diol (Methyltestosterone-M2)         | NMI                | West Lindfield, Australia |
| Mibolerone                                                                                             | Steraloids         | Newport, RI, USA          |
| Morphine                                                                                               | Cerilliant         | Round Rock, TX, USA       |
| Norboletone                                                                                            | NMI                | West Lindfield, Australia |
| 13 $\beta$ ,17 $\alpha$ -Diethyl-5 $\alpha$ -gonane-3 $\alpha$ ,17 $\beta$ -diol (Norboletone-M alpha) | NMI                | West Lindfield, Australia |
| 13 $\beta$ ,17 $\alpha$ -Diethyl-5 $\beta$ -gonane-3 $\alpha$ ,17 $\beta$ -diol (Norboletone-M beta)   | NMI                | West Lindfield, Australia |
| Norclostebol                                                                                           | Steraloids         | Newport, RI, USA          |
| Norethandrolone                                                                                        | NMI                | West Lindfield, Australia |
| 17 $\alpha$ -Ethyl-5 $\beta$ -estrane-3 $\alpha$ -17 $\beta$ -diol (Norethandrolone-M1)                | NMI                | West Lindfield, Australia |
| 17 $\alpha$ -Hydroxyethyl-5 $\beta$ -estrane-3 $\alpha$ ,17 $\beta$ -diol (Norethandrolone-M2)         | NMI                | West Lindfield, Australia |
| Oxabolone                                                                                              | NMI                | West Lindfield, Australia |
| Oxymesterone                                                                                           | Radian             | Lafayette, IN, USA        |
| Clenbuterol                                                                                            | Sigma              | St. Luis, MO, USA         |
| 4-Chloro-3 $\alpha$ -hydroxy-androst-4-en-17-one (Clostebol-M)                                         | NMI                | West Lindfield, Australia |

**Table S1** (continued)

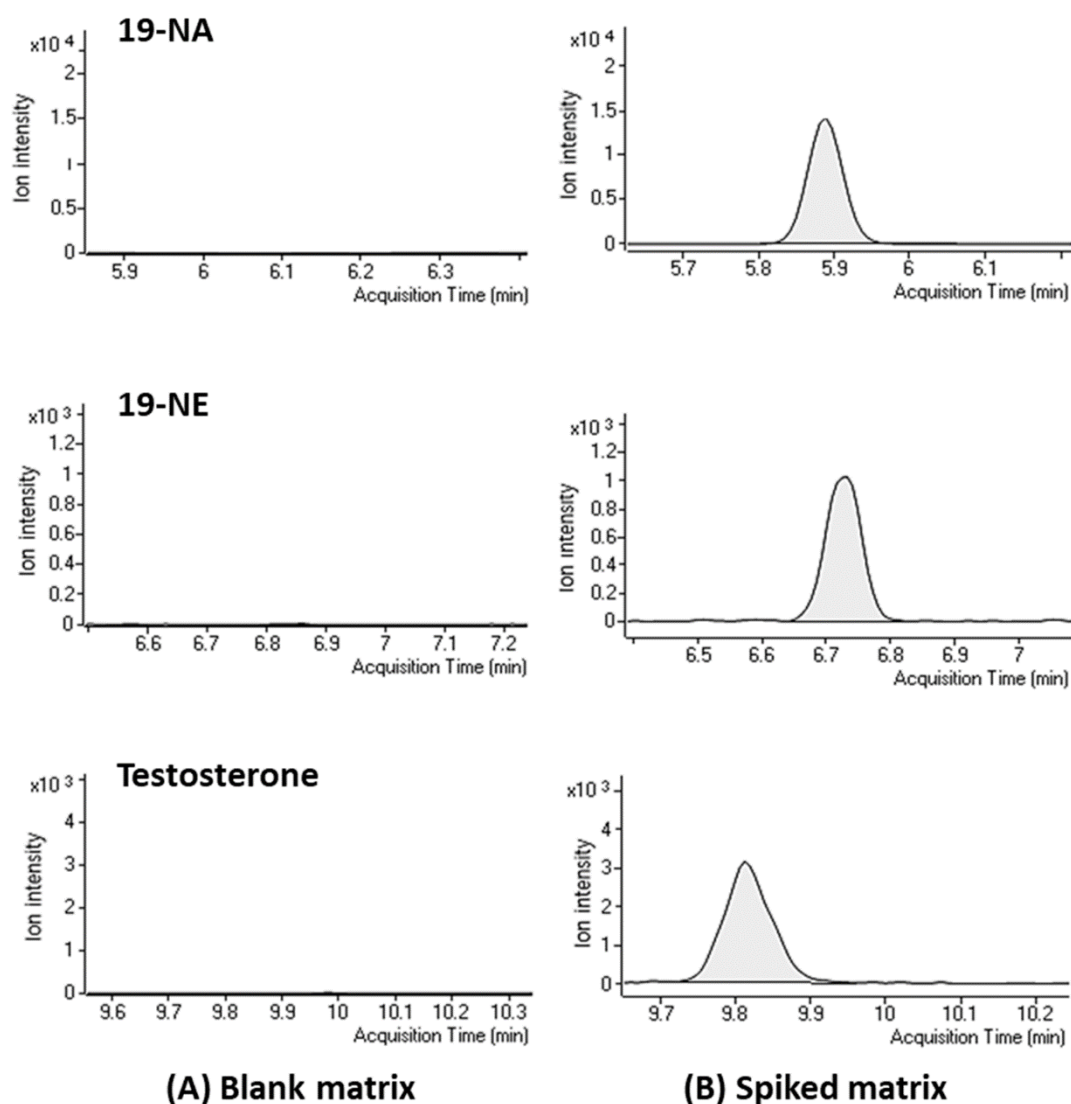
| Compound                                                                                                                 | Supplier              | Location                  |
|--------------------------------------------------------------------------------------------------------------------------|-----------------------|---------------------------|
| 2 $\alpha$ -Methyl-5 $\alpha$ -androstane-3 $\alpha$ -ol-17-one (Drostanolone-M)                                         | NMI                   | West Lindfield, Australia |
| Ethylestrenol                                                                                                            | Steraloids            | Newport, RI, USA          |
| Fluoxymesterone                                                                                                          | E.R.SQUIBB & SONS.INC | Brunswick, GA, USA        |
| 9 $\alpha$ -F-17 $\alpha$ -methyl-4-androsten-3 $\alpha$ ,6 $\beta$ ,11 $\beta$ ,17 $\beta$ -tetrol (Fluoxymesterone-M1) | NMI                   | West Lindfield, Australia |
| 9 $\alpha$ -Fluoro-17,17-dimethyl-18-norandrostane-4,13-diene-11 $\beta$ -ol-3-one (Fluoxymesterone-M2)                  | NMI                   | West Lindfield, Australia |
| 6 $\beta$ -OH-fluoxymesterone (Fluoxymesterone-M3)                                                                       | NMI                   | West Lindfield, Australia |
| Formestane                                                                                                               | Sigma                 | St. Luis, MO, USA         |
| bis-4-cyanophenyl-methanol (Letrozole-M)                                                                                 | NMI                   | West Lindfield, Australia |
| Mestanolone                                                                                                              | Steraloids            | Newport, RI, USA          |
| 1 $\alpha$ -Methyl-5 $\alpha$ -androstane-3 $\alpha$ -ol-17-one (Mesterolone-M1)                                         | NMI                   | West Lindfield, Australia |
| 1 $\alpha$ -Methyl-5 $\alpha$ -androstane-3 $\alpha$ ,17 $\beta$ -diol (Mesterolone-M2)                                  | NMI                   | West Lindfield, Australia |
| Metenolone                                                                                                               | Steraloids            | Newport, RI, USA          |
| 1-Methylene-5 $\alpha$ -androstane-3 $\alpha$ -ol-17-one (Metenolone-M)                                                  | NMI                   | West Lindfield, Australia |
| Epimetendiol (Methandienone-M1)                                                                                          | NMI                   | West Lindfield, Australia |
| 17 $\beta$ -hydroxymethyl-17 $\alpha$ -methyl-18-norandrost-1,4,13-triene-3-one (Methandienone-M9)                       | Seibersdorf           | Baden, Austria            |
| Methasterone                                                                                                             | Steraloids            | Newport, RI, USA          |
| 2 $\alpha$ ,17 $\alpha$ -dimethyl-5 $\alpha$ -androstane-3 $\alpha$ ,17 $\beta$ -diol (Methasterone-M)                   | WAADS                 | Cologne, Germany          |
| Methyl-1-testosterone                                                                                                    | NMI                   | West Lindfield, Australia |
| Methylclostebol                                                                                                          | TRC                   | Toronto, Canada           |
| Methyldienolone                                                                                                          | NMI                   | West Lindfield, Australia |
| Methylnortestosterone                                                                                                    | Steraloids            | Newport, RI, USA          |
| Methylstenebolone                                                                                                        | WAADS                 | Cologne, Germany          |
| 17 $\alpha$ -Methyl-5 $\alpha$ -androstane-3 $\alpha$ ,17 $\beta$ -diol (Methyltestosterone-M1)                          | NMI                   | West Lindfield, Australia |
| Salbutamol                                                                                                               | Astra                 | Alameda, CA, USA          |
| Stenbolone                                                                                                               | TRC                   | Toronto, Canada           |
| Testosterone                                                                                                             | NMI                   | West Lindfield, Australia |
| THC-COOH                                                                                                                 | Cerilliant            | Round Rock, TX, USA       |
| Zeranol                                                                                                                  | Wako                  | Osaka, Japan              |
| Zilpaterol                                                                                                               | TRC                   | Toronto, Canada           |
| 2 $\alpha$ -OH-methylethisterone (Danazol-M2)                                                                            | NMI                   | West Lindfield, Australia |
| 6 $\beta$ -OH-methandienone (Methandienone-M2)                                                                           | NMI                   | West Lindfield, Australia |
| a-Lenol ( $\alpha$ -Zearalenol)                                                                                          | Sigma                 | St. Luis, MO, USA         |
| b-Lenol ( $\beta$ -Zearalenol)                                                                                           | Sigma                 | St. Luis, MO, USA         |
| 4-chloro-18-nor-17 $\beta$ -hydroxymethyl,17 $\alpha$ -methyl-5 $\beta$ -androst-13-en-3 $\alpha$ -ol (DHCMT-M3)         | NMI                   | West Lindfield, Australia |
| Tetrahydronorethisterone                                                                                                 | Steraloids            | Newport, RI, USA          |
| Methyltestosterone                                                                                                       | SCHERING              | Kenilworth, NJ, USA       |
| d <sub>3</sub> -Testosterone                                                                                             | NMI                   | West Lindfield, Australia |
| d <sub>4</sub> -19-norandrosterone glucuronide                                                                           | NMI                   | West Lindfield, Australia |

**Table S2** Retention time and multiple reaction monitoring conditions for target substances

| Name                  | Retention time<br>(min) | Precursor ion<br>( <i>m/z</i> ) | Product ion<br>( <i>m/z</i> ) | Collision energy<br>(v) |
|-----------------------|-------------------------|---------------------------------|-------------------------------|-------------------------|
| 1-Androstenediol      | 8.89                    | 434                             | 405                           | 10                      |
| 1-Androstenedione     | 8.43                    | 415                             | 221                           | 20                      |
| 1-Testosterone        | 8.77                    | 432                             | 206                           | 10                      |
| 1-Testosterone-M      | 7.56                    | 432                             | 290                           | 10                      |
| 4-OH-testosterone     | 12.61                   | 520                             | 431                           | 20                      |
| Bolandiol             | 7.97                    | 420                             | 240                           | 5                       |
| Bolasterone           | 11.86                   | 315                             | 169                           | 20                      |
| Bolasterone-M         | 10.39                   | 374                             | 269                           | 10                      |
| Calusterone           | 11.98                   | 445                             | 355                           | 10                      |
| Calusterone-M         | 9.90                    | 269                             | 159                           | 20                      |
| Clostebol-M           | 10.44                   | 466                             | 431                           | 10                      |
| Drostanolone-M        | 7.92                    | 448                             | 343                           | 15                      |
| Ethylestrenol         | 5.93                    | 270                             | 241                           | 10                      |
| Fluoxymesterone       | 12.96                   | 552                             | 462                           | 10                      |
| Danazol-M2            | 13.09                   | 558                             | 193                           | 40                      |
| Methandienone-M2      | 12.59                   | 571                             | 337                           | 20                      |
| DHCMT-M3              | 11.56                   | 379                             | 343                           | 5                       |
| Norethisterone-M      | 8.52                    | 431                             | 193                           | 20                      |
| Fluoxymesterone-M1    | 12.58                   | 642                             | 447                           | 10                      |
| Fluoxymesterone-M2    | 8.72                    | 462                             | 208                           | 20                      |
| Fluoxymesterone-M3    | 13.35                   | 640                             | 605                           | 25                      |
| Mestanolone           | 11.01                   | 216                             | 159                           | 10                      |
| Mesterolone-M1        | 8.83                    | 433                             | 343                           | 5                       |
| Mesterolone-M2        | 9.18                    | 450                             | 255                           | 20                      |
| Methandienone-M1      | 6.14                    | 358                             | 301                           | 15                      |
| Methandienone-M9      | 9.47                    | 442                             | 236                           | 10                      |
| Metenolone            | 10.46                   | 446                             | 195                           | 20                      |
| Metenolone-M          | 8.42                    | 446                             | 431                           | 10                      |
| Methasterone          | 11.92                   | 462                             | 419                           | 10                      |
| Methasterone-M        | 9.50                    | 449                             | 269                           | 10                      |
| Methylclostebol       | 13.13                   | 480                             | 390                           | 10                      |
| Methyldienolone       | 11.59                   | 430                             | 285                           | 20                      |
| Methyl-1-testosterone | 10.42                   | 206                             | 165                           | 20                      |
| Methylnortesterone    | 10.77                   | 287                             | 207                           | 20                      |
| Methylstenebolone     | 11.44                   | 370                             | 220                           | 10                      |
| Methyltestosterone-M1 | 8.89                    | 435                             | 255                           | 15                      |
| Methyltestosterone-M2 | 9.18                    | 270                             | 213                           | 10                      |
| Mibolerone            | 11.69                   | 431                             | 341                           | 10                      |
| Norboletone           | 12.96                   | 460                             | 301                           | 10                      |

**Table S2** (continued)

| <b>Name</b>                        | <b>Retention time<br/>(min)</b> | <b>Precursor ion<br/>(<i>m/z</i>)</b> | <b>Product ion<br/>(<i>m/z</i>)</b> | <b>Collision energy<br/>(<i>v</i>)</b> |
|------------------------------------|---------------------------------|---------------------------------------|-------------------------------------|----------------------------------------|
| Norboletone-M alpha                | 11.67                           | 435                                   | 255                                 | 20                                     |
| Norboletone-M beta                 | 12.09                           | 435                                   | 255                                 | 20                                     |
| Norclostebol                       | 12.57                           | 452                                   | 321                                 | 20                                     |
| Norethandrolone                    | 12.50                           | 446                                   | 287                                 | 20                                     |
| Norethandrolone-M1                 | 10.47                           | 421                                   | 241                                 | 15                                     |
| Norethandrolone-M2                 | 12.60                           | 421                                   | 331                                 | 5                                      |
| Oxabolone                          | 12.22                           | 506                                   | 147                                 | 35                                     |
| Oxymesterone                       | 13.01                           | 534                                   | 301                                 | 40                                     |
| Stenbolone                         | 9.60                            | 446                                   | 208                                 | 15                                     |
| Boldenone                          | 9.57                            | 430                                   | 206                                 | 15                                     |
| Boldenone-M                        | 6.13                            | 194                                   | 163                                 | 20                                     |
| Boldione                           | 9.21                            | 428                                   | 206                                 | 15                                     |
| Testosterone                       | 9.83                            | 432                                   | 209                                 | 20                                     |
| 19-Norandrosterone                 | 5.89                            | 405                                   | 315                                 | 5                                      |
| 19-Noretiocholanolone              | 6.73                            | 405                                   | 225                                 | 15                                     |
| Nandrolone                         | 9.00                            | 418                                   | 194                                 | 18                                     |
| Clenbuterol                        | 2.55                            | 335                                   | 300                                 | 10                                     |
| 3 $\alpha$ -OH-tibolone            | 9.67                            | 443                                   | 193                                 | 30                                     |
| 3 $\beta$ -OH-tibolone             | 8.74                            | 443                                   | 193                                 | 30                                     |
| Zilpaterol                         | 4.31                            | 308                                   | 218                                 | 15                                     |
| Zeranol                            | 12.50                           | 433                                   | 295                                 | 25                                     |
| 4-OH-cyclofenil                    | 12.76                           | 422                                   | 367                                 | 20                                     |
| 6-OH-bromantan                     | 8.63                            | 395                                   | 133                                 | 15                                     |
| 6-OXO                              | 12.14                           | 516                                   | 411                                 | 15                                     |
| 6-OXO-M                            | 12.30                           | 518                                   | 319                                 | 30                                     |
| Carphedone                         | 2.28                            | 272                                   | 104                                 | 20                                     |
| Formestane                         | 12.51                           | 518                                   | 221                                 | 30                                     |
| Letrozole-M                        | 3.94                            | 291                                   | 160                                 | 10                                     |
| Morphine                           | 6.91                            | 429                                   | 287                                 | 20                                     |
| Salbutamol                         | 2.12                            | 369                                   | 191                                 | 20                                     |
| Tamoxifen-M                        | 13.22                           | 489                                   | 72                                  | 10                                     |
| THC-COOH                           | 11.80                           | 473                                   | 355                                 | 30                                     |
| $\alpha$ -Zearalenol               | 12.78                           | 446                                   | 317                                 | 25                                     |
| $\beta$ -Zearalenol                | 12.86                           | 446                                   | 317                                 | 25                                     |
| Methyltestosterone                 | 11.70                           | 446                                   | 301                                 | 20                                     |
| d <sub>3</sub> -Testosterone       | 9.80                            | 435                                   | 209                                 | 20                                     |
| d <sub>4</sub> -19-Norandrosterone | 5.86                            | 409                                   | 319                                 | 5                                      |



**Fig. S1** Representative chromatograms were obtained for endogenous steroids. (A) show that the prepared matrix can be used as a blank matrix for the endogenous standard spikes. (B) are the chromatograms after spiking the blank matrix with standards: 19-norandrosterone, 19-noretiocholanolone, and testosterone at concentrations of 15, 2, and 1  $\mu\text{g}/\text{kg}$ , respectively.

**Table S3** List of food items selected for analysis

| Matrix        | Item                                                                                                                                                                         |
|---------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Fatty liquid  | Hempseed oil (Canada), Lard oil (Japan)                                                                                                                                      |
| Fatty solid   | Pork (Sow, Barrow) (Korea, Mexico, Brazil), Chicken (Korea), Beef (Korea), Pig testicles (Korea), Poppy seed (China), Hempseed (Canada)                                      |
| Nonfat liquid | Fig juice (Korea), Barley tea (Korea), Oat drink (Canada), Milk (Korea)                                                                                                      |
| Nonfat solid  | Maize (Italy), Barley (Korea), Oats (Canada), Figs (Korea), Pomegranate (US), Cornmeal (China), Strong flour (US, Canada), Weak flour (US), Peach (Korea), Scallions (Korea) |

**Table S4** Validation results from the developed method

| Name                  | LOQ<br>(µg/kg) | Spiked Conc.<br>(µg/kg) | Precision<br>(%RSD) | Recovery<br>(%) | Matrix effect<br>(%) |
|-----------------------|----------------|-------------------------|---------------------|-----------------|----------------------|
| 1-Androstenediol      | 2.5            | 5                       | 7                   | 112             | -6                   |
| 1-Androstenedione     | 0.5            | 5                       | 10                  | 96              | +7                   |
| 1-Testosterone        | 2.5            | 5                       | 6                   | 114             | +1                   |
| 1-Testosterone-M      | 0.25           | 5                       | 9                   | 97              | -6                   |
| 4-OH-testosterone     | 2.5            | 5                       | 9                   | 112             | -26                  |
| Bolandirol            | 2.5            | 5                       | 3                   | 92              | -34                  |
| Bolasterone           | 1              | 5                       | 6                   | 90              | +6                   |
| Bolasterone-M         | 1              | 5                       | 12                  | 100             | +8                   |
| Calusterone           | 5              | 5                       | 9                   | 116             | -29                  |
| Calusterone-M         | 2.5            | 5                       | 10                  | 92              | -30                  |
| Clostebol-M           | 2.5            | 5                       | 9                   | 93              | -21                  |
| Drostanolone-M        | 0.5            | 5                       | 8                   | 95              | +5                   |
| Ethylestrenol         | 0.25           | 5                       | 12                  | 84              | -35                  |
| Fluoxymesterone       | 2.5            | 5                       | 14                  | 103             | +12                  |
| Danazol-M2            | 2.5            | 5                       | 12                  | 92              | +1                   |
| Methandienone-M2      | 2              | 2                       | 8                   | 94              | -38                  |
| DHCMT-M3              | 1              | 2                       | 13                  | 85              | -29                  |
| Norethisterone-M      | 0.25           | 5                       | 10                  | 97              | -19                  |
| Fluoxymesterone-M1    | 20             | 20                      | 16                  | 95              | +5                   |
| Fluoxymesterone-M2    | 0.25           | 5                       | 8                   | 103             | -6                   |
| Fluoxymesterone-M3    | 2.5            | 5                       | 5                   | 123             | +7                   |
| Mestanolone           | 2.5            | 5                       | 11                  | 87              | +11                  |
| Mesterolone-M1        | 2              | 5                       | 12                  | 96              | 0                    |
| Mesterolone-M2        | 2.5            | 5                       | 12                  | 92              | -16                  |
| Methandienone-M1      | 1              | 2                       | 3                   | 82              | -11                  |
| Methandienone-M9      | 5              | 5                       | 13                  | 104             | +5                   |
| Metenolone            | 2.5            | 5                       | 12                  | 100             | -9                   |
| Metenolone-M          | 0.5            | 5                       | 10                  | 101             | 0                    |
| Methasterone          | 1              | 5                       | 12                  | 111             | -12                  |
| Methasterone-M        | 0.5            | 5                       | 14                  | 107             | -24                  |
| Methylclostebol       | 2.5            | 5                       | 10                  | 112             | +20                  |
| Methyldienolone       | 5              | 5                       | 9                   | 106             | +1                   |
| Methyl-1-testosterone | 5              | 5                       | 10                  | 103             | +5                   |
| Methylnortesterone    | 5              | 5                       | 4                   | 106             | +8                   |
| Methylstenebolone     | 5              | 5                       | 10                  | 123             | -16                  |
| Methyltestosterone-M1 | 1              | 2                       | 10                  | 85              | 0                    |
| Methyltestosterone-M2 | 2              | 2                       | 12                  | 92              | -14                  |
| Mibolerone            | 2.5            | 5                       | 6                   | 91              | -13                  |
| Norboletone           | 2.5            | 5                       | 9                   | 120             | 0                    |

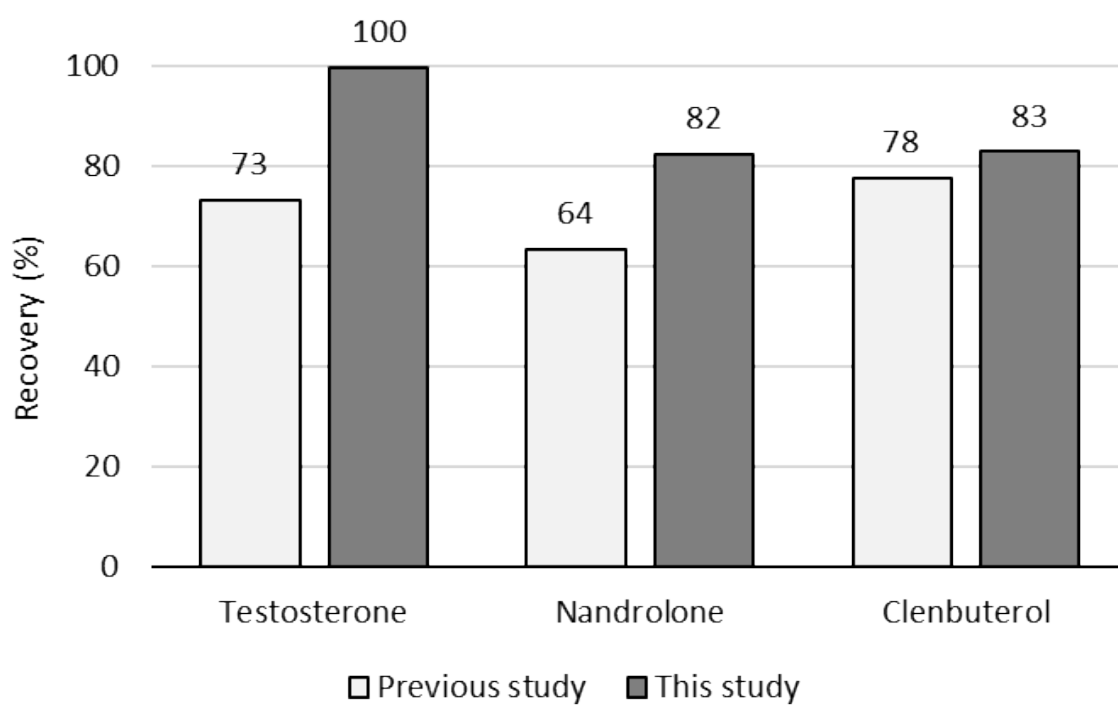
LOQ, limit of quantification



**Table S4** (Continued)

| Name                  | LOQ<br>(µg/kg) | Spiked Conc.<br>(µg/kg) | Precision<br>(%RSD) | Recovery<br>(%) | Matrix effect<br>(%) |
|-----------------------|----------------|-------------------------|---------------------|-----------------|----------------------|
| Norboletone-M alpha   | 0.25           | 5                       | 13                  | 98              | -33                  |
| Norboletone-M beta    | 2.5            | 5                       | 10                  | 93              | -32                  |
| Norclostebol          | 5              | 5                       | 6                   | 109             | -28                  |
| Norethandrolone       | 2.5            | 5                       | 14                  | 101             | +1                   |
| Norethandrolone-M1    | 0.5            | 5                       | 13                  | 92              | -12                  |
| Norethandrolone-M2    | 0.25           | 5                       | 6                   | 114             | +2                   |
| Oxabolone             | 2.5            | 5                       | 14                  | 112             | +1                   |
| Oxymesterone          | 5              | 5                       | 6                   | 95              | -14                  |
| Stenbolone            | 0.5            | 5                       | 10                  | 93              | 0                    |
| Boldenone             | 2.5            | 5                       | 5                   | 97              | -23                  |
| Boldenone-M           | 0.5            | 5                       | 9                   | 81              | -28                  |
| Boldione              | 5              | 5                       | 4                   | 88              | -26                  |
| Testosterone          | 0.1            | 1                       | 8                   | 100             | -3                   |
| 19-Norandrosterone    | 0.15           | 15                      | 7                   | 83              | -6                   |
| 19-Noretiocholanolone | 0.4            | 2                       | 10                  | 80              | -22                  |
| Nandrolone            | 1              | 1                       | 4                   | 82              | -27                  |
| Clenbuterol           | 0.01           | 1                       | 5                   | 83              | -2                   |
| 3α-OH-tibolone        | 0.2            | 2                       | 11                  | 95              | -18                  |
| 3β-OH-tibolone        | 0.2            | 2                       | 10                  | 87              | -14                  |
| Zilpaterol            | 0.4            | 2                       | 4                   | 97              | -16                  |
| Zeranol               | 1              | 2                       | 12                  | 86              | +9                   |
| 4-OH-cyclofenil       | 0.5            | 10                      | 7                   | 103             | -36                  |
| 6-OH-bromantan        | 5              | 10                      | 14                  | 102             | +9                   |
| 6-OXO                 | 5              | 10                      | 3                   | 105             | -18                  |
| 6-OXO-M               | 1              | 5                       | 5                   | 114             | -6                   |
| Carphedone            | 5              | 50                      | 7                   | 88              | -37                  |
| Formestane            | 2.5            | 50                      | 18                  | 113             | -8                   |
| Letrozole-M           | 0.5            | 10                      | 3                   | 96              | +11                  |
| Morphine              | 5              | 200                     | 13                  | 93              | -13                  |
| Salbutamol            | 5              | 200                     | 5                   | 82              | -31                  |
| Tamoxifen-M           | 10             | 10                      | 9                   | 89              | -37                  |
| THC-COOH              | 1.25           | 50                      | 6                   | 81              | -25                  |
| α-Zearalenol          | 2.5            | 5                       | 15                  | 117             | +2                   |
| β-Zearalenol          | 0.5            | 5                       | 8                   | 113             | +2                   |

LOQ, limit of quantification



**Fig. S2** Comparison of recoveries between this study and previous studies<sup>1-3</sup> in pork samples. The amounts of standards added for recovery were as follows: 1 µg/kg of testosterone, 2 µg/kg of nandrolone, and 5 µg/kg of clenbuterol in the previous studies; all three substances were 1 µg/kg in this study.

## References

- 1 E. Attalah, Y. S. Nasr, H. A. El-Gammal and F. A. Nour El-Dien, *Food Addit Contam Part A Chem Anal Control Expo Risk Assess*, 2016, **33**, 1545-1556.
- 2 C. L. Xu, C. F. Peng, K. Hao, Z. Y. Jin and X. G. Chu, *Anal Lett*, 2006, **39**, 555-568.
- 3 Z. H. Zhang, H. Yan, F. Y. Cui, H. Yun, X. H. Chang, J. H. Li, X. Liu, L. J. Yang and Q. R. Hu, *Food Anal Method*, 2016, **9**, 915-924.