Analytical and Bioanalytical Chemistry

Electronic Supplementary Material

Total reflection X-ray fluorescence spectrometry for trace determination of iron and some additional elements in biological samples

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Table S1 Optimized sample pretreatment parameters for TXRF and/or GFAAS measurement

|                      | Bone marrow-derived macrophages | Liver cells | Liver tissues |
|----------------------|---------------------------------|-------------|---------------|
|                      |                                 |             | Mouse         |
|                      |                                 |             | Beef SRM 1577c|

### A) Sample digestion

|                      | 1 mL HNO₃ | 1 mL HNO₃ | 4 mL HNO₃ |
|----------------------|-----------|-----------|-----------|
| Digestion time and temperature | 15 sec at RT | 15 sec at RT | 19 min at 40°C |
| Sample amount        | 1.6 – 3.3 µg | 15·10³ - 1,900 ·10³ cells | 2.7 - 27.6 mg |

### B) TXRF measurement

1. Calibration with internal standard
   - 10 µL of Ga (100 mg/L)
   - 10 µL of Ti (100 mg/L)
2. Homogenization of internal standard
   - 1 min
3. Applied digest volume
   - 10 µL
4. Drying of sample carrier
   - at 60 °C
5. Measurement live time
   - 500 s

### C) GFAAS measurement

1. Calibration with external Fe standards in the concentration ranges … in µg L⁻¹
   - 20 – 100
   - HC: 20-100
     - 50-160
     - 70-200
     - KC: 30-200
     - HSC: 5-55
     - LSEC: 7-40
     - 40-170
   - KC/HSC/LSEC: no dilution
   - 10-55
     - 20-100
     - 50-200
     - 30 - 200
   - 21 / 11 / 6 / 3 / no dilution
2. Sample dilution factors
   - 2
   - HC: 11 / 14 / no dilution
     - KC/HSC/LSEC: no dilution
3. Applied sample amount
   - GFAAS: 20 µL
**Table S2** Temperature program for Fe determination by GFAAS

| Step      | Temperature [°C] | Ramp [°C s\(^{-1}\) | Hold [s] |
|-----------|------------------|----------------------|----------|
| Drying    | 70               | 4                    | 20       |
| Drying    | 90               | 3                    | 20       |
| Drying    | 110              | 5                    | 20       |
| Drying    | 125              | 5                    | 20       |
| Pyrolysis | 350              | 50                   | 20       |
| Pyrolysis | 950              | 300                  | 10       |
| Auto zero | 950              | 0                    | 6        |
| Atomization | 2300         | 1000                 | 6        |
| Cleaning  | 2450             | 500                  | 4        |