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Electronic supplementary information for

# A cost-effective method applicable to the speciation analysis of lead in various seafood based on short-column ion chromatography-inductively coupled plasma mass spectrometry

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## 1. Chemicals, materials and apparatus used in the experiment

The standards of inorganic lead ($Pb^{2+}$) was purchased from National Institute of Metrology, China. The standards of trimethyl lead chloride (TML) and triethyl lead chloride (TEL) were purchased from Dr. Ehren-storfer Company (Germany). The analytical grade of $HNO_3$ was purchased from Sigma-Aldrich, Chian (Shanghai, China). The analytical grade of sodium ethylenediamine tetraacetate (EDTA) and tetrabutylammonium hydroxide (TBAH) were purchased from Macklin Company, China (Shanghai, China). The water used in the experiment is Milli-Q water (18.2 MΩ·cm), which was prepared with Millipore pure system. Strong cation exchange guard column (Zorbax 300SCX, 4.6×12.5 mm) was purchased from Agilent Technology (Shanghai, China).

The IC-ICP-MS system, which couples an Agilent 7800 inductively coupled plasma mass spectrometry (ICP-MS, Agilent of USA) with a Dionex ICS-900 ion chromatography (ThermoFisher Scientific, China), was used in this study.

**Table S1:** Optimal parameters of short-column IC-ICP-MS system

| Parameter | Value |
|---|---|
| **IC system** | |
| IC separation column | Two consecutive strong cation guard column (Zorbax 300SCX, 4.6×12.5 mm) |
| IC eluent | 1.0 mM EDTA-0.25 mM TBAH (pH 3.0) |
| IC elution mode | iso-elution |
| Sample injection volume | 10 µL |
| Flow rate of IC eluent | 1.0 mL/min |
| Temperature of column | 30 ºC |
| **ICP-MS system** | |
| ICP-MS RF power | 1350 W |
| ICP-MS cool gas flow rate | 15 L/min |
| ICP-MS auxiliary gas flow rate | 1.0 L/min |
| ICP-MS nebulizer gas flow rate | 0.80 L/min |
| ICP-MS makeup gas flow rate | 0.30 L/min |
| Monitored isotope (m/z) | $^{208}$Pb |

**Table S2:** The analytical performance of the developed short-column IC-ICP-MS method

| Pb species | Equation | Coefficient (R) | Linear range | LOD |
|---|---|---|---|---|
| $Pb^{2+}$ | Y=67711×C (ng/mL)+35.0 | 0.9998 | 0.1-10 ng/mL | 5.0 pg/mL |
| TML | Y=58872×C (ng/mL)+1206 | 0.9998 | 0.1-10 ng/mL | 6.0 pg/mL |
| TEL | Y=56878×C (ng/mL) -564 | 0.9999 | 0.1-10 ng/mL | 6.0 pg/mL |

**Table S3:** Effect of the extracting solvent volume and extraction mode on lead extraction efficiency in shellfish (*Morulauva*) sample

| Extracting solvent volume | 0.10 g of dried *Morulauva* sample | |
|---|---|---|
| | Extraction mode | Extraction efficiency of Pb |
| 8 mL | 4.0 mL for previous soaking and first extraction, 4.0 mL for second extraction | 77 % |
| 10 mL | 5.0 mL for previous soaking and first extraction, 5.0 mL for second extraction | 87 % |
| 12 mL | 6.0 mL for previous soaking and first extraction, 6.0 mL for second extraction | 94 % |
| | 7.0 mL for previous soaking and first extraction, 5.0 mL for second extraction | 93 % |
| 14 mL | 7.0 mL for previous soaking and first extraction, 7.0 mL for second extraction | 95 % |
| | 8.0 mL for previous soaking and first extraction, 6.0 mL for second extraction | 94 % |

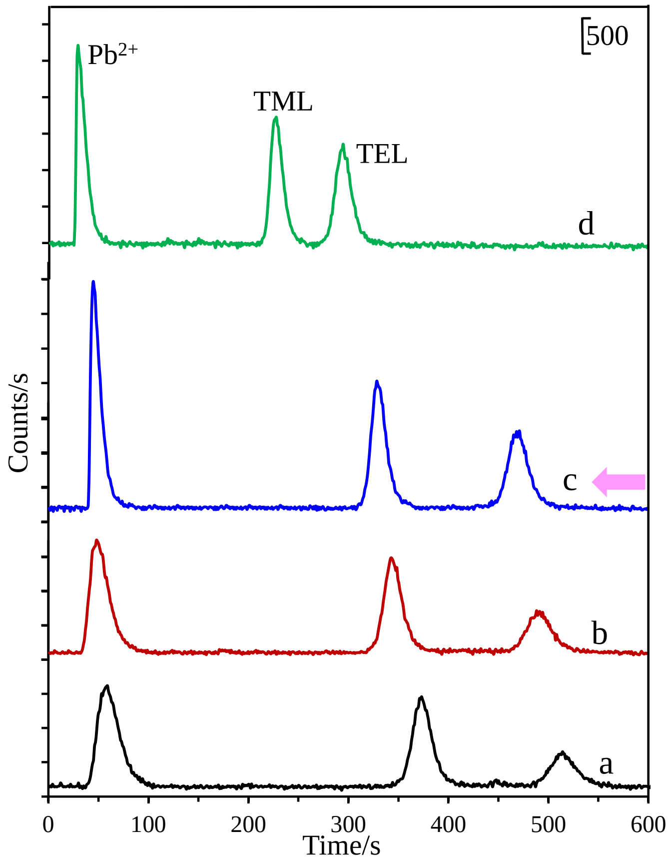

**Figure S1:** Effect of EDTA concentration in the mobile phase on the IC separation of Pb$^{2+}$, TML and TEL after they were pre-chelated with 0.3 mM of EDTA. The concentrations of Pb$^{2+}$, TML and TEL are all 1.0 ng/mL, and the IC-ICP-MS chromatogram was obtained under the conditions listed in Table S1 except for EDTA concentration in the mobile phase. a: 0.25 mM; b: 0.5 mM; c: 1.0 mM; d: 2.0 mM.

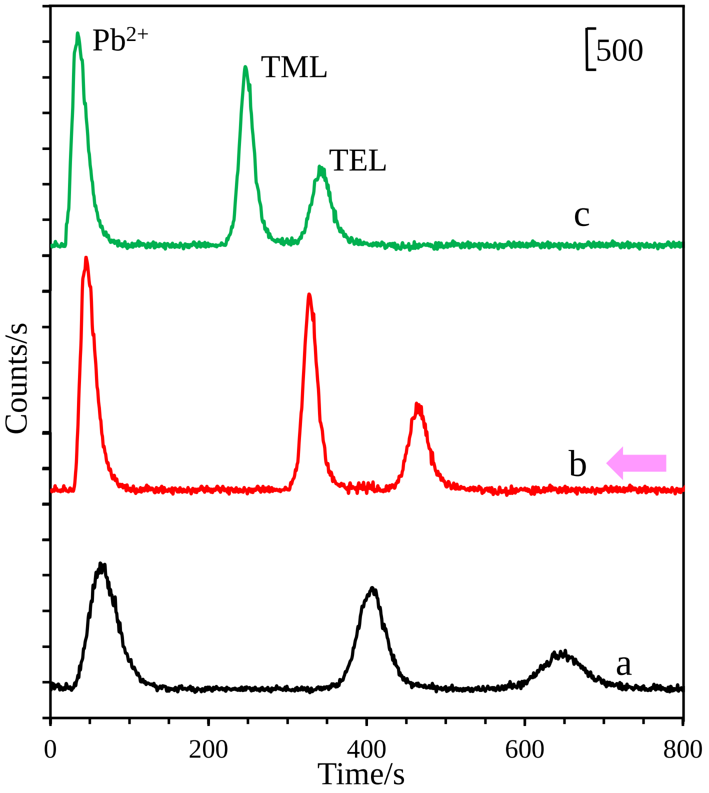

**Figure S2**: Effect of TABH concentration in the mobile phase on the IC separation of $Pb^{2+}$, TML and TEL after they were pre-chelated with 0.3 mM of EDTA. The concentrations of $Pb^{2+}$, TML and TEL are all 1.0 ng/mL, and the IC-ICP-MS chromatogram was obtained under the conditions listed in Table S1 except for TABH concentration in the mobile phase. a: 0.10 mM; b: 0.25 mM; c: 0.50 mM.

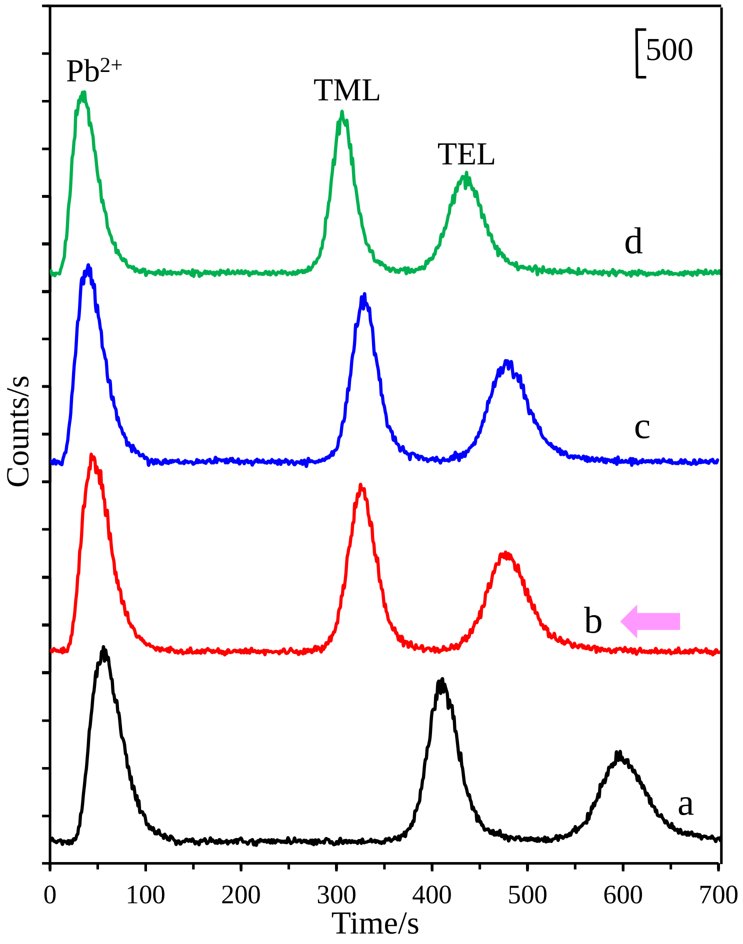

**Figure S3:** Effect of flow rate of the mobile phase on the IC separation of Pb$^{2+}$, TML and TEL after they were pre-chelated with 0.3 mM of EDTA. The concentrations of Pb$^{2+}$, TML and TEL are all 1.0 ng/mL, and the IC-ICP-MS chromatogram was obtained under the conditions listed in Table S1 except for the flow rate of the mobile phase. a: 0.90 mL/min; b: 1.00 mL/min; c: 1.10 mL/min; d: 1.20 mL/min.

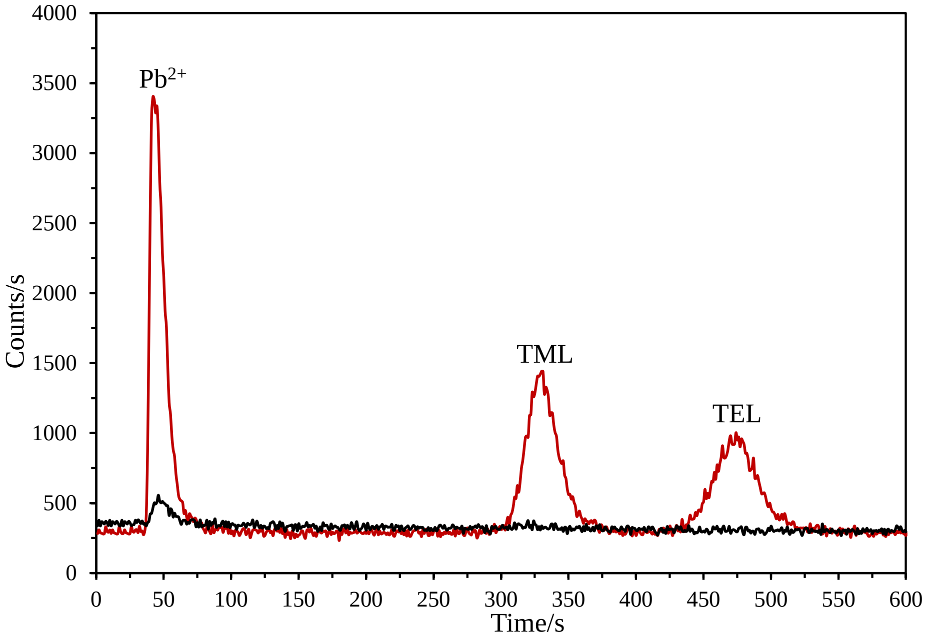

**Figure S4:** The IC-ICP-MS chromatograms for determining 1.0 ng/mL of $Pb^{2+}$, TML and TEL standards under the optimal conditions listed in Table S1 after they were pre-chelated with 0.3 mM of EDTA. Black line is the reagent blank.

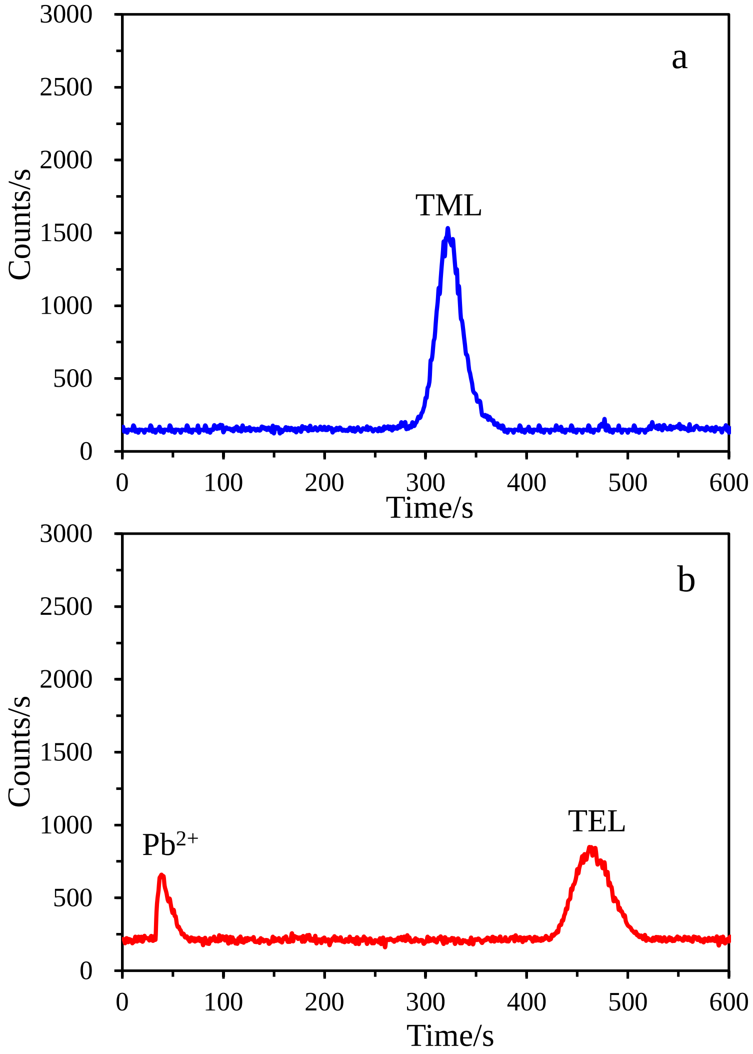

**Figure S5:** The IC-ICP-MS chromatograms of TML standard (a) and TEL standard (b) after they were pretreated with pre-soaking microwave-assisted extraction using 100 mM of $HNO_3$ as extracting solvents. Data was obtained under the optimal conditions listed in Table S1, after they were pre-chelated with 0.3 mM of EDTA. The concentrations of TML and TEL are all 1.0 ng/mL.

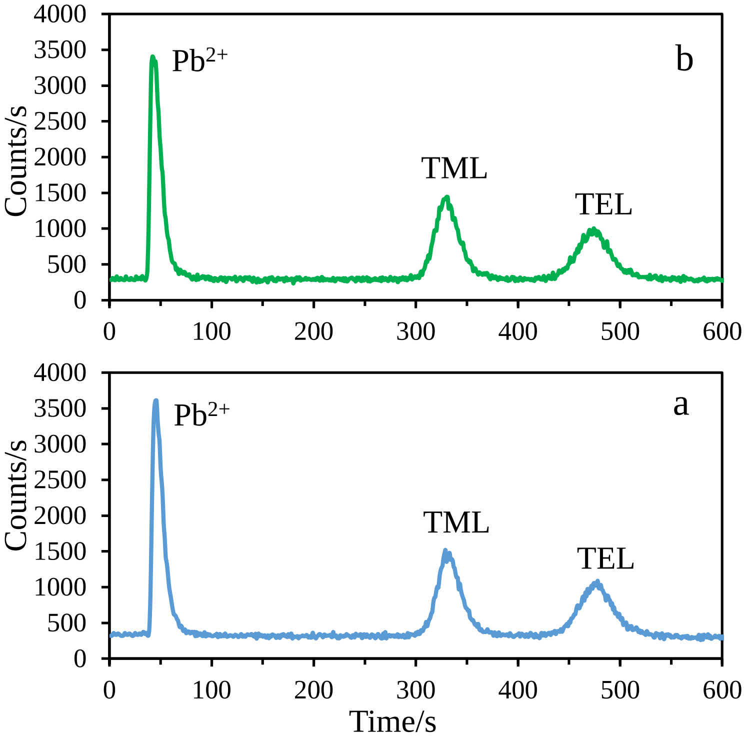

**Figure S6:** The IC-ICP-MS chromatograms of the mixed standard of lead species obtained under the optimal conditions listed in Table S1, after they were pre-chelated with 0.3 mM of EDTA. The concentrations of each lead species are all 1.0 ng/mL. (a): Original mixed standard; (b) The mixed standard pretreated with pre-soaking microwave-assisted extraction.