

## PENGEMBANGAN METODE ANALISIS METIL MERKURI BERDASARKAN PRINSIP ANALISIS KIMIA HIJAU MENGGUNAKAN EKSTRAKSI PADAT-CAIR TANDEM DENGAN UPLC-ICPMS

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### INTISARI

Merkuri (Hg) merupakan unsur yang aktif secara kimia dan biologis serta merupakan salah satu senyawa polutan yang paling beracun dan berbahaya di lingkungan. Merkuri memiliki efek berbahaya pada kesehatan manusia bahkan pada konsentrasi yang sangat rendah. Konsentrasi metil merkuri (MeHg) yang sangat bervariasi pada organisme sehingga membutuhkan metode pengukuran yang sensitif dan selektif dalam memisahkan merkuri organik (khususnya MeHg) dan merkuri anorganik (inHg).

Penelitian ini bertujuan untuk mengembangkan metode analisis metil merkuri berdasarkan prinsip analisis kimia hijau menggunakan ekstraksi padat-cair yang dikombinasikan dengan *Ultra performance Liquid Chromatography Inductively Couple Plasma Mass Spectrometer* (UPLC-ICPMS). Ekstraksi padat-cair dilakukan dengan pelarut 0,5% (b/v) L-sistein dalam 2% (v/v) HNO<sub>3</sub> sebanyak 1 mL dengan berat sampel 50 mg atau rasio berat sampel: berat L-sistein = 1:100. Penggunaan elusi gradien pada UPLC selama 300 detik dengan fasa gerak 0,5% L-sistein dalam 2% HNO<sub>3</sub> (A) dan metanol 100% (B) dapat memisahkan MeHg dan inHg secara spesifik pada waktu retensi yang berbeda. Keberadaan ion logam lain (Li, Be, Mg, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Sr, Mo, Cd, Sb, Tl dan Pb) dalam matriks sampel ataupun dalam kurva standar tidak mempengaruhi linearitas kurva MeHg dan inHg. *Limit of Detection* (LoD) MeHg sebesar 0,0009 ng/kg dan *Limit of Quantification* (LoQ) 0,003 ng/kg, sedangkan LoD inHg sebesar 0,001 ng/kg dan LoQ 0,004 ng/kg. Akurasi persentase *recovery* MeHg dari *certified reference material* (CRM) DORM-4 sebesar 94,57% dengan pengulangan 10 kali. Metode ini dapat digunakan dalam pengukuran MeHg dan inHg pada sampel ikan hiu, ikan tuna dan beras.

Evaluasi prinsip kimia hijau dilakukan dengan metode *Green Analytical Procedure Index* (GAPI), *Complementary Green Analytical Procedure Index* (ComplexGAPI), *Analytical Greenness Calculator* (AGREE) dan *Analytical Greenness Metric for Sample Preparation* (AGREEprep). Hasil evaluasi menunjukkan metode ini memiliki kelebihan dalam penggunaan sampel yang sedikit (50 mg), pelarut yang tidak mudah terbakar (0,5% L-sistein dalam 2% HNO<sub>3</sub>), volume pelarut yang kecil (1 mL) sehingga dihasilkan limbah dengan volume kecil (<1 mL). Adanya pengembangan metode penentuan MeHg ini dapat memberikan solusi yang relatif lebih mudah, cepat, akurat dan efisien serta sedikit menghasilkan limbah.

**Kata kunci:** Metil merkuri, UPLC-ICPMS, elusi gradien, *green analytical chemistry*

## GREEN ANALYTICAL CHEMISTRY METHOD DEVELOPMENT OF METHYL MERCURY WITH SOLID-LIQUID EXTRACTION COUPLED UPLC-ICPMS

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### ABSTRACT

Mercury (Hg) is a chemically and biologically active element and is one of the most toxic and hazardous pollutant compounds in the environment. Mercury has harmful effects on human health even at very low concentrations. The concentration of methyl mercury (MeHg) varies greatly in organisms, requiring a sensitive and selective measurement method to separate organic mercury (especially MeHg) and inorganic mercury (inHg).

This study aims to develop a methyl mercury analysis method based on the principle of green analytical chemistry using solid-liquid extraction combined with Ultra Performance Liquid Chromatography Inductively Couple Plasma Mass Spectrometer (UPLC-ICPMS). Solid-liquid extraction was carried out using a solvent of 0.5% (w/v) L-cysteine in 2% (v/v) HNO<sub>3</sub> as much as 1 mL with a sample weight of 50 mg or a sample weight ratio: L-cysteine weight = 1:100. The use of gradient elution on UPLC for 300 seconds with a mobile phase of 0.5% L-cysteine in 2% HNO<sub>3</sub> (A) and 100% methanol (B) can separate MeHg and inHg specifically at different retention times. The presence of other metal ions (Li, Be, Mg, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Sr, Mo, Cd, Sb, Tl and Pb) in the sample matrix or in the standard curve does not affect the linearity of the MeHg and inHg curves. The Limit of Detection (LoD) of MeHg was 0.0009 ng/kg and the Limit of Quantification (LoQ) was 0.003 ng/kg, while the LoD of inHg was 0.001 ng/kg and the LoQ was 0.004 ng/kg. The accuracy of MeHg recovery percentage from certified reference material (CRM) DORM-4 was 94.57% with 10 repetitions. This method can be used in measuring MeHg and inHg in shark, tuna, and rice samples.

Evaluation of green chemistry principles was carried out using the Green Analytical Procedure Index (GAPI), Complementary Green Analytical Procedure Index (ComplexGAPI), Analytical Greenness Calculator (AGREE) and Analytical Greenness Metric for Sample Preparation (AGREEprep) methods. The evaluation results showed that this method has advantages in using a small sample (50 mg), non-flammable solvents (0.5% L-cysteine in 2% HNO<sub>3</sub>), small solvent volume (1 mL) so that waste is produced with a small volume (<1 mL), The development of this MeHg determination method can provide a solution that is relatively easier, faster, more accurate and efficient and produces little waste.

**Keywords:** Methyl mercury, UPLC-ICPMS, gradient elution, green analytical chemistry