

INTISARI

Susu merupakan produk pangan yang lazim dikonsumsi, namun dapat terkontaminasi oleh logam-logam berat. Penelitian ini dilakukan untuk memvalidasi metode analisis kadmium (Cd), kromium (Cr), tembaga (Cu), mangan (Mn), dan nikel (Ni) dalam susu secara *inductively coupled plasma atomic emission spectroscopy* (ICP-AES). Metode ini memiliki keunggulan yakni cepat, sensitif, hanya memerlukan sampel sedikit untuk dianalisis, dan memiliki batas deteksi yang rendah.

Sampel susu didigesti dengan metode digesti basah menggunakan asam nitrat dan asam perklorat dengan berbanding 1:1 v/v, kemudian dianalisis menggunakan ICP-AES. Berbagai parameter validitas metode analisis diuji dan ditetapkan sesuai panduan *International Conference on Harmonization* (ICH).

Metode analisis Cd, Cr, Cu, Mn, dan Ni secara ICP-AES menghasilkan kurva kalibrasi yang linier pada panjang gelombang optimal masing-masing logam dengan nilai koefisien korelasi (r) > 0,997. Batas kuantifikasi logam Cd, Cr, Cu, Mn, dan Ni berturut-turut sebesar 0,0047 $\mu\text{g/mL}$; 0,0050 $\mu\text{g/mL}$; 0,0066 $\mu\text{g/mL}$; 0,0061 $\mu\text{g/mL}$; dan 0,0169 $\mu\text{g/mL}$. Metode ini memiliki presisi yang baik, dengan nilai simpangan baku relatif (RSD) Cd, Cr, Cu, Mn, dan Ni sebesar 3,18%; 4,17%; 3,05%; 2,93%; dan 4,47% pada uji keterulangan dan 5,28%; 5,06%; 3,67%; 3,67%; dan 11,17% pada uji presisi antara. Rerata persen perolehan kembali Cd, Cr, Cu, Mn, dan Ni sebesar 92,25%; 90,88%; 102,87%; 94,50%; dan 86,85% yang menggambarkan bahwa metode ini memiliki akurasi yang baik. Rerata kadar Cd, Cr, Cu, Mn, dan Ni yang terukur pada sampel susu masing-masing berkisar antara 0,2380-0,3374 mg/L; 0,1310-0,1933 mg/L; 0,1674-0,4877 mg/L; 0,1063-0,6682 mg/L; dan tidak terdeteksi-0,1396 mg/L.

KATA KUNCI : validasi metode analisis, susu, logam berat, ICP-AES

ABSTRACT

Milk is a daily consumed product that may be contaminated by toxic heavy metals. This study was conducted to validate the methods of analysis of cadmium (Cd), chromium (Cr), copper (Cu), manganese (Mn), and nickel (Ni) by inductively coupled plasma atomic emission spectroscopy (ICP-AES) in milk. This analytical method is rapid, sensitive, efficient, and has a very low detection limit.

Milk samples were digested by wet digestion method using 14 mL of nitric acid and perchloric acid (1:1), and then were analyzed using ICP-AES. Various parameters of analytical method validity were tested and determined by referring to the Guideline of International Conference on Harmonization (ICH).

The developed method of Cd, Cr, Cu, Mn, and Ni by ICP-AES had a linear calibration curves in each metal's optimal wavelength, with the correlation coefficients (r) > 0.997 . The limit of quantification of Cd, Cr, Cu, Mn, and Ni were found to be 0.0047 mg/mL; 0.0050 mg/mL; 0.0066 mg/mL; 0.0061 mg/mL; and 0.0169 mg/mL respectively. The analytical method was precise, with the relative standard deviation (RSD) of Cd, Cr, Cu, Mn, and Ni were found to be 3.18%; 4.17%; 3.05%; 2.93%; and 4.47% respectively for the interday precision and 5.28%; 5.06%; 3.67%; 3.67%; and 11.17% respectively for the intraday precision. The mean recovery percentage of Cd, Cr, Cu, Mn, and Ni were 92.25%; 90.88%; 102.87%; 94.50%; and 86.85% respectively, which shows the good accuracy of this method. This method was then applied to analyze Cd, Cr, Cu, Mn, and Ni in various samples of milk, with the concentration ranged from 0.2380 to 0.3374 mg/L; 0.1310 to 0.1933 mg/L; 0.1674 to 0.4877 mg/L; 0.1063 to 0.6682 mg/L; and not detected-0.1396 mg/L respectively.

Keywords : analytical method validation, milk, heavy metals, ICP-AES