

DEVELOPMENT OF SOIL STANDARD MATERIALS CONTAINING HAZARDOUS METALS FOR X-RAY FLUORESCENCE ANALYSIS

Yasuhiro SHIBATA, Junnosuke SUYAMA,
Masaru KITANO, and Toshihiro NAKAMURA
Department of Applied Chemistry, Meiji University

Standard material of soil containing hazardous metals i.e., Cr, As, Se, Cd, Hg, and Pb was developed for the X-ray fluorescence (XRF) analysis. Homogeneous standard soil was prepared by adding appropriate amount of aqueous standard solutions to the base soil and then drying and mixed with V-type mixing machine. Base soil powders ground to under 12.5 μm of modal particle size were Tachikawa loam, brown forest soil, and weathered granite soil containing 17.9, 9.43, and 3.49 mass% of Fe_2O_3 , respectively. Concentrations of Cr, As, Se, Cd, Hg, and Pb in soils were determined with metal furnace atomic absorption spectrometry of decomposed samples. For the XRF determination analytical lines were selected $\text{CrK}\alpha$, $\text{AsK}\alpha$, $\text{SeK}\alpha$, $\text{CdK}\alpha$, $\text{HgL}\alpha$ and $\text{PbL}\beta$ accompany with the corrections for overlapping of $\text{SeK}\beta$ to $\text{PbL}\beta$ and $\text{PbL}\alpha$ to $\text{AsK}\alpha$. Specimens for XRF analysis were prepared by powder briquette pressed in the 23mm ϕ Al ring with 300 kgf cm^{-2} and loose powder in 31mm ϕ of polyethylene cup covered with 6 μm polypropylene film. The durability of the standards was checked by intermittently XRF measuring, and was good endurance for 240 min irradiation of primary X-ray (50 kV, 80 mA). The calibration curves drawn with proposed standards were good linearity under 3000 mg kg^{-1} for Cr, As, Se, Cd, Pb, and 300 mg kg^{-1} for Hg. Calibration curves of 6 determinants in the 3 different base soils with two specimen mounting methods were remarkably differed with each other. Corrections with Compton scatter for $\text{AsK}\alpha$, $\text{SeK}\alpha$, $\text{CdK}\alpha$, $\text{HgL}\alpha$ and $\text{PbL}\beta$, and with background scatter for $\text{CrK}\alpha$ were effective and gave same slope of the calibration curves with different soil characters and mounting methods. The lower limits of detection were 0.5-6.4 mg kg^{-1} . The spike test for 5 determinants showed good recovery for gravel soil and pumice soil.