

Organic matter cycling along geochemical, geomorphic and disturbance gradients in vegetation and soils of African tropical forests and cropland - Project TropSOC DATABASE_v1.0

2.4. Forest – Pu soil inventory

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Introduction

The dataset comprises two plot and sample identifiers, followed 3 variables describing $^{239+240}\text{Pu}$ characteristics and detection limits at the plot level in each of TropSOC's forest plots. The last 3 variables provide bulk densities of mineral, organic and litter layers sampled specifically for the $^{239+240}\text{Pu}$ assessment. Values below the detection limit or missing bulk density data are indicated by -9999.

Data structure

No.	Variable	Explanation	Unit
1	plotID	unique identifier of each plot and point where data were collected	-
1	sampleID	unique identifier of any soil or vegetation sample taken in the field	-
3	mean_Pu	total activity of the fallout radionuclides ^{239}Pu and ^{240}Pu in one kg sample material reported on ashed mass basis. 1 Bq corresponds to one radioactive decay per second; if the $^{239+240}\text{Pu}$ activity is below the detection limit of 0.01 Bq kg ⁻¹ the value is set to -9999	Bq kg ⁻¹
4	sd_Pu	standard deviation of total $^{239+240}\text{Pu}$ activity measurements based on at least three aliquot measurements	Bq kg ⁻¹
5	detec_limit	flag indicating if $^{239+240}\text{Pu}$ activity is below (flag = 1) or above (flag = 0) the detection limit of 0.01 Bq kg ⁻¹	-
6	BD_m_soil	bulk density of mineral soil layer	g cm ⁻³
7	BD_O	bulk density of organic soil layer	g cm ⁻³
8	BD_L	bulk density of organic litter layer	g cm ⁻³

Methods

To estimate effective soil redistribution since the 1960s, measurements of fallout radionuclides $^{239+240}\text{Pu}$ were used following Calitri et al. (2019) and Ketterer et al. (2004). Mineral soil sampling for all sites was carried out using a manual soil corer (Ø 6.8 cm and 120 cm length). Before analyses, undisturbed soil cores of known sample volume were weighed for bulk density calculations after drying at 105°C. Sub-sampling of soil samples into smaller, representative portions was carried out on homogenised sample material using a sample splitter (Sample Splitter RT 6.5, Retsch, Germany). Then, 30 g of milled sample was dry-ashed for at least 8 hours at 600 °C to remove organic matter. Subsequently, the samples were spiked using 30 pg (c. 0.0044 Bq) of a ^{242}Pu tracer solution (NIST 4334).

Sample leaching was carried out by heating 16 M nitric acid (HNO_3) overnight at 80 °C, subsequently filtering the solution and adjusted the concentration of HNO_3 to 8 M. All present plutonium species were adjusted to the Pu (IV) oxidation state by first adding an acidified $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ solution (2 mg ml^{-1} of the leached solution) and subsequently adding a sodium nitrite (NaNO_2) solution (20 mg ml^{-1} of the leached solution). Afterwards, samples were heated at 75 °C for two hours. Tetravalent Pu was separated from the leached solution using a Pu-selective TEVA resin (2 mg of TEVA per millilitre of leached solution). Following the occasional, two-hour long agitation, the resin was collected in a pipette tip equipped with a glass wool plug. This disposable column was first rinsed with 2 M aqueous HNO_3 to remove unretained matrix elements (e.g. uranium (U)), then it was rinsed with 8 M HCl to elute thorium (Th) and finally it was rinsed again with 2 M aqueous HNO_3 (rinse volume = 1 ml per 30 mg of TEVA). Plutonium was eluted using 0.05 M aqueous ammonium oxalate. Finally, activities of $^{239+240}\text{Pu}$ were measured using mass spectrometry (Thermo X Series II quadrupole ICP-MS, located at Northern Arizona University). The ICP-MS instrument is equipped with an APEX HF high-efficiency sample introduction system. Upon analysis, masses of ^{239}Pu and ^{240}Pu present in the samples were converted into the summed activity of $^{239+240}\text{Pu}$, in Becquerel per kg. Data quality was evaluated through the analysis of blanks (soils or rocks devoid of Pu), duplicates and control samples of known $^{239+240}\text{Pu}$ activities (Standard Reference material 4350b – River sediment for radioactivity measurements from NIST). Detection limit for $^{239+240}\text{Pu}$ activity was assessed at 0.01 Bq kg^{-1} corresponding to 5 Bq m^{-2} . For a scientific interpretation of these results see Wilken et al. (2021).

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