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

3.3. Cropland – $^{239+240}$Pu soil inventory

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Doetterl S., Asifiwe R.K., Baert G., Bamba F., Bauters M., Boeckx P., Bukombe B., Cadisch G., Cizungu L.N., Cooper M., Hoyt A., Kabaseke C., Kalbitz K., Kidinda L., Maier A., Mainka M., Mayrock J., Muhindo D., Mujinya B.B., Mukotanyi, S.M., Nabahungu L., Reichenbach M., Rewald B., Six J., Stegmann A., Summerauer L., Unseld R., Vanlauwe B., Van Oost K., Verheyen K. Vogel C., Wilken F., Fiener P. Organic matter cycling along geochemical, geomorphic and disturbance gradients in forests and cropland of the African Tropics - Project TropSOC Database Version 1.0. *Earth System Science Data* XXX, DOI XXX, 2021.

Wilken F., Fiener P., Ketterer M., Meusburger K., Muhindo D.I., Van Oost K., Doetterl S. Assessing soil erosion of forest and cropland sites in wet tropical Africa using $^{239+240}$Pu fallout radionuclides. *Soil Discussion (pre-print).* https://doi.org/10.5194/soil-2020-95., 2021.

Introduction

The dataset comprises two plot and sample identifiers, followed 3 variables describing $^{239+240}$Pu characteristics and detection limits at the catchment level for TropSOC's cropland catchments. The last variable provide bulk densities of the mineral soil layer sampled specifically for the $^{239+240}$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.	-
2	sampleID	unique identifier of any soil or vegetation sample taken in the field	-
3	mean_pu	mean $^{239+240}$Pu activity in one kg sample material reported on ashed mass basis, 1 Bq corresponds to one radioactive decay per second; if the $^{239+240}$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 mean $^{239+240}$Pu activity	Bq kg^{-1}
5	detec_limit	flag indicating if $^{239+240}$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}

Methods

To estimate effective soil redistribution since the 1960s, measurements of fallout radionuclides $^{239+240}$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 $FeSO_4 \cdot 7H_2O$ 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 (i.e. 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 $^{239+240}Pu$ 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 $^{239+240}Pu$, 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 $^{239+240}Pu$ activities (Standard Reference material 4350b – River sediment for radioactivity measurements from NIST). Detection limit for $^{239+240}Pu$ 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).

Acknowledgment

TropSOC was funded via the Emmy-Noether-Program of the German Research Foundation (project ID 387472333).

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