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## A SUMMARY OF RADIOCARBON MEASUREMENTS OF FLUVIAL AND COLLUVIAL DEPOSITS IN CATCHMENTS OF SOUTH-CENTRAL ONTARIO, CANADA

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**ABSTRACT.** Fluvial and colluvial deposits of Late Holocene age in South-Central Ontario catchments have provided few <sup>14</sup>C dates, most by conventional methods registering century-old ages. Other young deposits, dated by conventional and accelerator mass spectrometry radiocarbon (AMS <sup>14</sup>C), have yielded bomb-affected post-1950 ages over variable time limits. Attempts to date the base of Ah and lower-in-section soil horizons, in Early to Late Holocene stream terrace deposits, have yielded atomic bomb effects. Comparing bomb contamination in Late Holocene fluvial deposits, using both conventional and AMS methods, identifies a mix of bomb-affected beds juxtaposed with dated beds, the latter yielding ages with narrow standard deviations. Colluvial deposits overlying key glacial sections in the Rouge Catchment, while rare, yield bracketed AMS ages for an Ahbk horizon that refines weathering times relative to previously obtained conventional <sup>14</sup>C dates. Bomb-affected sediment appears variably distributed within floodplain soils and in the ground soil of a colluvial section. Mass wasted deposits, with AMS <sup>14</sup>C ages spread over the last few centuries, appear related to Little Ice Age (LIA) changes in climate, corroborated by pollen records. Further, these AMS-<sup>14</sup>C dated beds calibrate weathering of secondary Fe-Al oxihydroxides over the first half a millennium of weathering time.

**KEYWORDS:** bomb <sup>14</sup>C, <sup>14</sup>C age, colluvial deposits, floodplain, Little Ice Age.

### INTRODUCTION

Previous work on Late Holocene fluvial terraces and colluvial deposits in the Rouge catchments (Rouge River and Little Rouge Creek) in southern Ontario (Figures 1A, 1B) have, from the outset, focused on lithostratigraphy (Karrow 1967; Mahaney et al. 2014), weathering and soil morphogenesis (Mahaney and Sanmugadas 1986; Mahaney and Terasmae 1988; Mahaney and Hancock 1993a). Later, the focus shifted to landforms and water quantity (Weninger and McAndrews 1989; TRCA 2007a, 2007b). Occasionally, terrace and slope soil stratigraphy benefitted from relative dating (RD) methods using soluble Fe and Al phases, with these estimates partly built upon radiocarbon-age controls and plant-soil interactions (Mahaney et al. 2016). However, Fe and Al extraction databases used to elucidate paleoenvironmental reconstructions from fluvial, beach (Sauer et al. 2008) and lacustrine deposits (Mahaney et al. 2001), and some of the uncertainty as to the exact composition of particular extractions has been previously discussed by Mahaney et al. (2016). What is crucial with RD methods applied to young soil weathering is the start-up ages assigned to minute concentrations of Fe and Al oxihydroxides, where age changes are at the level of 0.01 percent, and changes occur over a few centuries. Conventional <sup>14</sup>C and AMS <sup>14</sup>C measurements are compared to start-up ages of the weathering process, similar to earlier approaches (Lichter 1998; Ewing and Nater 2002).

Several papers have focused on the dating of fluvial terraces (Phillips et al. 1998; Guzmán et al. 2013) and moraines (Zreda and Shanahan 2000; Carcaillet et al. 2013), using OSL and cosmogenic dating methods such as <sup>10</sup>Be, <sup>21</sup>Ne, and <sup>36</sup>Cl. These types of studies have not been applied to the Holocene geochronology of Southern Ontario. OSL dating has not proved

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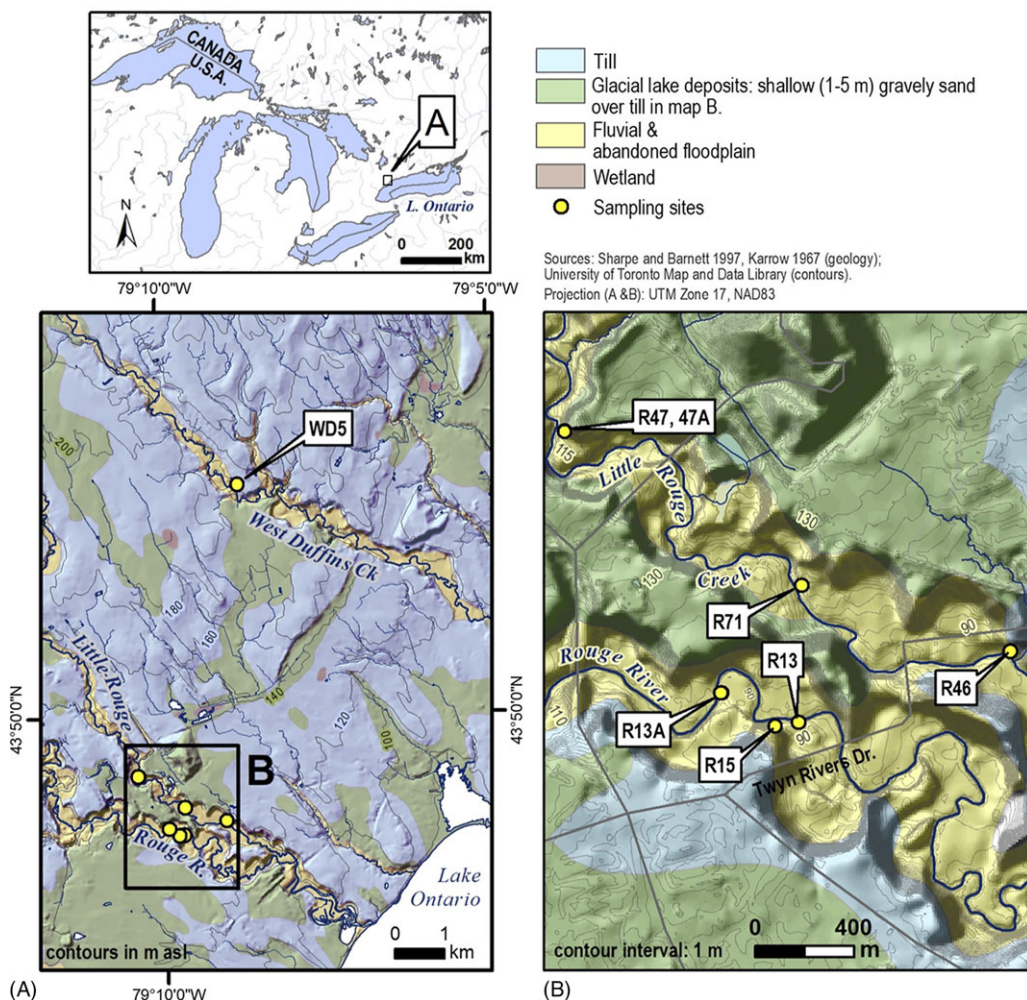


Figure 1 (A) Surface geology and location of sampling sites in the Rouge River/Little Rouge Creek and West Duffins Creek catchments; (B) a more detailed view of Rouge/Little Rouge sites with topography shown by one-meter contours. Topographic controls taken from Sharpe et al. (1999).

practical in central Southern Ontario catchments because of zeroing problems and high concentrations of carbonate (Galina Høtt, personal communication 1992). However, OSL has been used (Lovis et al. 2012) to date nearby Lake Michigan sand dunes with reasonable results. With the quartz-diminished sediment in moraine, terrace and floodplain sediment, cosmogenic dating has not been applied to unraveling the Holocene geochronology of Southern Ontario. While moraines with interstadial sediments have occasionally yielded minor organics for  $^{14}\text{C}$  dating (Karrow 1967), post-Lake Iroquois sediment terrace/floodplain sequences have had to rely on relative-age dating (RD) by fluvial terrace soil stratigraphy, Fe-Al extractions (Mahaney et al. 2014) and young, near modern, conventional  $^{14}\text{C}$  ages of Ahb horizons in both floodplain and colluvial deposits.

For these young deposits, initial weathering trends, slope instability and time of overbank sedimentation can be tentatively tied to inferred changes of climate, such as the LIA or simply

decadal changes in weather/stream discharge flooding. Recent climate change is sometimes inferred from pollen records (McAndrews 1981; McAndrews and Boyko-Diakonow 1989), and dates in terrace and colluvial sections alone, rather than from compositional studies of sections resulting in an incomplete understanding of the paleoenvironment. Moreover, sedimentary investigations in the Rouge and West Duffins catchments (Figure 1A), as in other drainages, suffer from a lack of organic materials suitable for radiocarbon dating. This makes Fe and Al extracts of prime importance in the attempt to unravel leaching histories and paleoenvironmental reconstructions. Here, we compare previous conventional  $^{14}\text{C}$  and recent AMS dating of floodplain and colluvial sections to refine age control of the first few centuries of sediment weathering and startup of Fe-Al extraction estimates. Here, we report on new AMS  $^{14}\text{C}$  measurements intended to address this question and remaining bomb carbon-14 concentrations that produce post-1950 ages.

### REGIONAL GEOLOGY AND STUDY AREA

Surface runoff in the Rouge and West Duffins basins (Figures 1 A, B) of central Southern Ontario drains across thick till/lacustrine/deltaic-encrusted sediment draped over Ordovician shales of the Whitby Formation (Liberty 1955, 1964). Within both catchments, bedrock outcrops are rare, found only in the R46 section (stratigraphy in Mahaney 2015, and in Mahaney and Hancock 1993a; site location on Figure 1B), in a prominent outcrop near Twyn Rivers Drive on Little Rouge Creek, located three km north of Lake Ontario, and 1 km north of the confluence with Rouge River. All surface till/lacustrine sections date from the Laurentide Ice Sheet (LIS) and all soil materials are sourced from these sediments.

Climatic data for the area highlights a mean annual temperature (MAT) of  $\sim 7^\circ\text{C}$ , with extremes of  $-34^\circ\text{C}$  and  $40^\circ\text{C}$ . A mean annual precipitation (MAP) of 860 mm, coupled with a mean annual actual evapotranspiration (AE) of 530 mm (Brown et al. 1968; Phillips and McCulloch 1972), yields a mean annual water surplus of 330 mm. Even with soil moisture reserves shrinking there is available soil moisture for leaching. Throughput of soil moisture over the 11-kyr post-Lake Iroquois time-period may only be estimated from solution of calcite in soil epipedons, with secondary precipitation of carbonate in Ck horizons known to increase with time (Mahaney et al. 2016). Thus, the thickness of secondary carbonate in Ck horizons increases with age of the pedon. Exceptions to this occur in profiles in landslide debris, discussed by Mahaney (2015), where wetting depth is reduced to a few centimeters, in response to the presence of a unique hydrophilic fern (*Adiantum pedantum*) that produces a unique soil bioclimate greatly reducing the throughput of soil water. Slow leaching increases the ratio of Si:Al to 2:1, and coupled with plentiful Ca, thereby initiates genesis of Ca-smectite, confirmed by XRD. Thus, overall the region produces a soil leaching environment that is capable of moving bomb contaminants and selective Fe/Al extracts in micropores usually associated with root channels, at present and in the past. Of all extracts (pyrophosphate Fe-Al, acid ammonium oxalate Fe-Al, and Na-dithionite Fe-Al, Parfitt and Childs 1988), only  $\text{Fe}_o$  (using acid ammonium oxalate) is of concern, as it is partly soluble, so any concentration reported will be minimal. Such leaching effects do not appear to affect radiocarbon dating results, as both methods produce ages with narrow standard deviations of decadal plus or minus range. Of all other oxihydroxides,  $\text{Fe}_p$ ,  $\text{Fe}_d$ , and  $\text{Al}_d$  offer the best prospects for RD age control, as discussed later in the paper.

Later meteorological data, released by the Ontario Ministry of the Environment (1984), covers a broad area bordering the north shore of Lake Ontario, providing mean figures, as follows:

mean annual precipitation (MAP)—900 mm; mean actual evaporation (AE)—550 mm; with runoff ~250–300 mm, and snowfall ~100–150 mm. In less than two decades the MAP increased 40 mm, AE 20 mm. Considering these values as approximate, and coupled with slightly elevated MAP and AE relative to previously recorded climatic data noted above, such increases of MAP and AE possibly reflect either increased urban warming or global warming. Despite a declining mean water surplus soil water remains sufficient to maintain soils at or beyond field capacity most of the year. Soils in these sections, with fine sandy to silt loam textures, normally yield about ~5% water content (unpublished data from 78 soil profiles studied in both Rouge catchments). Still more recent MAT for the 20-yr period 1999–2019 is MAT at 8.6°C with MAP at 1020 mm for Pickering, Ontario which is on the east boundary of the Rouge River (see-<https://en.climate-data.org/north-america/canada/ontario/pickering-4854/#temperature-graph>), highlight continuing increases.

Over the fifty-year period from the 1970s to the present, MAT has risen 1.6°C, which is close to the 1.3°C rise in worldwide temperature over nearly the same period documented by the Paris Agreement—(IPCC 2021). Along with this rise in MAT, MAP is seen to rise by 120 mm/yr.

## **MATERIALS AND METHODS**

Soil descriptions mainly follow guidelines established by the NSSC (1995), Soil Survey Staff (1975), and Birkeland (1999). The Ck horizon designation (k=carbonate) originates from the U.S. Soil Taxonomy. Minor departures include the Cu horizon (unweathered parent material) from Hodgson (1976) and the “h” identifier of the A horizon (h= humus), which follows the CSSC (1998) and is used when color is darker than 10YR 3/1. B horizons, when carrying the “w,” replace the former color B group (Soil Survey Staff 1975; NSSC 1995). In the Canadian soil classification weathered B horizons are designated “m” for modified. Soil color assessment based on Oyama and Takehara’s (1970) soil color chips, essentially the same as the Munsell System (Macbeth 1992). Site selection was based on previous stratigraphic assessment (Mahaney and Sanmugadas 1986; Mahaney and Hancock 1993b; Mahaney et al. 2016), and on new sites intended to put an RD age fix on common 3 m terraces (R71). RD terrace age in both the Rouge and West Duffins Creek catchments is determined by terrace height above stream flow, using the amended relationship of Bufe et al. (2017) where time of incision ( $\Delta t_i$ ) = depth of incision (Di)/incision rate (Evi). Time of incision = 11 ka, average depth of incision = 35 m/incision rate average = 318 cm/1 kyr. While this approximates an RD age of 1ka for the 3 m terrace (R71), age estimates of older surfaces become complicated by early release of permafrost, declining catchment area, variable discharge with lowering of Lake Iroquois, and changes of climate with a hemlock infestation changing surface runoff as outlined by Mc Andrews (1981), McAndrews et al. (1989), and Mahaney et al. (2016). In addition, there is no way without <sup>14</sup>C datable strata in terrace sections to account for Evb (beveling time; Bufe et al. 2017) following Evi (incision) to adjust height above stream flow. The best available estimate of age is height of terrace above stream flow.

Approximately 500 g samples were collected from horizons at each site to allow for particle size, clay mineral, soil chemistry and geochemical analyses. The samples were air dried, pretreated for particle size analysis where necessary, and treated with 30% H<sub>2</sub>O<sub>2</sub> to partially remove organic contaminants. Also, when necessary, samples were later wet sieved to separate sand fractions, the <63 μm fraction (silt plus clay) subjected to particle size analysis by hydrometer (Day 1965). RD Fe-Al extraction methods are outlined and described in Dormaar and Lutwick (1983), Parfitt and Childs (1988), and Mahaney et al. (2016).



For  $^{14}\text{C}$  dating, soil samples were air dried, subsamples intended for lab analysis were treated similarly for both conventional and AMS dating. All samples had macroscopic foreign material/roots removed under  $10\times$  magnification, followed by acid leaching to remove carbonates, and lastly washed with distilled water. Given the young age of all samples, NaOH treatment was considered not necessary and acid-cleaned dried soil organic matter (SOM) was submitted for  $^{14}\text{C}$  dating by first conventional, and later, AMS  $^{14}\text{C}$  methods. Conventional  $^{14}\text{C}$  was carried out at the Brock University Radiocarbon Laboratory operated by Howard Melville from 1970 until its decommissioning in 2008. AMS  $^{14}\text{C}$  for sample analysis reported here comes from the Chrono 14 laboratory at Queens University Belfast, Belfast, U.K. AMS  $^{14}\text{C}$  ages reported here as fraction modern corrected using  $^{13}\text{C}$  measured using AMS  $^{13}\text{C}$ , following Stuiver and Polach (1977).

Atomic bomb-contaminated samples producing post-1950 AMS  $^{14}\text{C}$  ages were analyzed using the Levin Int. Cal curves from the OxCal 4.1.1 system and the bomb curves at QUB in Northern Ireland—CALIBomb. Hence, the concentrations of post-1950 AMS  $^{14}\text{C}$  relevant in this case are measured at the start-up time of atomic testing (1950), and a return to near-zero contamination at some future indeterminate time up to 2020. The IntCal and QUB bomb curves post-1950 bomb contamination ages in the mid curve sections of Figures 4B and 5B were derived from atmospheric and tree ring tests only (in Austria and Germany), with no peat/soil sampling.

CALIBomb recent changes summarize several ways of reporting  $^{14}\text{C}$  activity levels relative to a standard now in use, but no convention has been established for reporting nuclear weapons testing (post-bomb)  $^{14}\text{C}$  levels in samples. The use of fraction modern (Fm) has been replaced with a new symbol— $\text{F}^{14}\text{C}$ —to prevent confusion since some previous samples may or may not have been fractionation corrected (Reimer et al. 2004, 2020; Calib.org.) (personal correspondence, AJT Jul 2023).  $\text{F}^{14}\text{C}$  registers 1.0 (1950), with values  $<1.0$  pre-1950;  $>1.0$  post-bomb dates.

## RESULTS

### R13 Floodplain Section

The floodplain in the Rouge Valley (including both the Rouge River and Little Rouge Creek drainages; Figures 1A, B) has been subjected to mass wasting (earth slips), and deposition of dislodged trees along the waterways, as shown in Figure 2. Such bank erosion, compounded by ice blockage in spring and overbank flooding occasionally in summer, cover a large extent of the floodplain, producing a network of alluvial channels, in which aborted soil profile development is revealed by successions of Ahk/Cuk/Ahb/Coxbk/Cubk horizons in meter-deep profiles. These are survivors of short (decadal to-century-long) weathering events. As indicated in Figure 3, such soil successions in sandy-silty sediment often show slight increases of clay with depth, but with detrital, not pedogenic patterns only. Conventional  $^{14}\text{C}$  dating, prior to AMS  $^{14}\text{C}$  coming on stream, provided evidence for soil development over decades (less than a century), documenting recent stages of abortive soil development (i.e. Ahbk horizons) caused by episodic ice blockage flooding. These thin, young soils, with occasional slight clay increases with depth, show no pedogenic downward movement as supported by field test or mean phi calculations. Further XRD analyses of the clay fraction, previously indicated by Mahaney et al. (2016), show only detrital patterns of primary and secondary minerals, with calcite showing minor dissolution in Ah relative to Cubk horizons, where it increases. Overbank flooding is illustrated by increased calcite in the Ahk surface soil horizon in R13. Soil



Figure 2 Rouge Floodplain at Site R13A about ~400 m from site R13. For the location of R13, sampled in 1974, see Figure 1B.

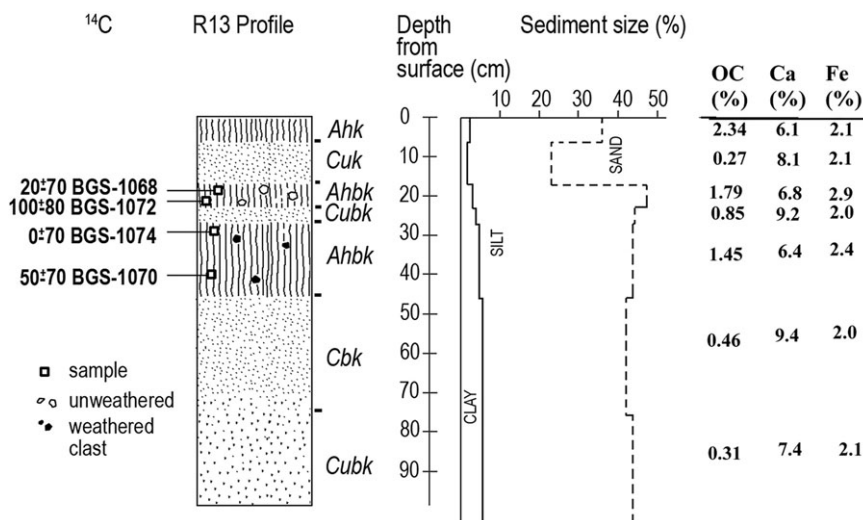


Figure 3 The R13 profile with conventional-derived <sup>14</sup>C dates and particle size (clay=solid line; silt=dashed line-silt+clay-100=sand). OC=organic carbon; Ca=total Ca; Fe=total Fe.

chemistry, down-section in R13, yields minor fluctuations of pH, conductivity, organic carbon, nitrogen, total Ca and Fe that represent abortive soil development over, at most, a century duration. Very slight adjustments of pH are fine tuned to organic carbon/N and conductivity that highlight initial soil development disrupted by recurring overbank sedimentation.

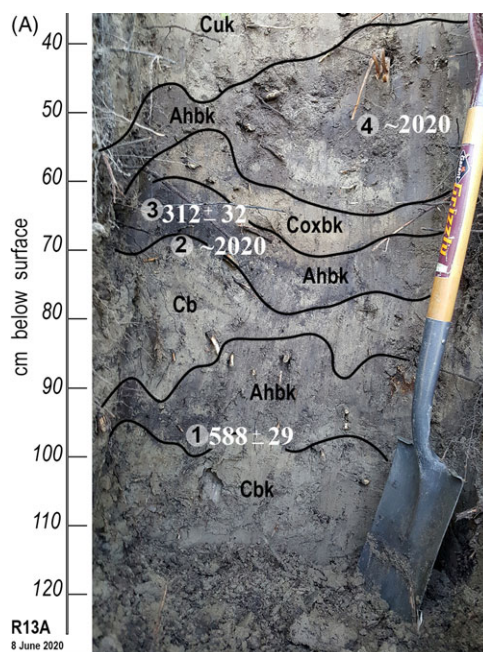


Figure 4 (A) R13A profile with convoluted horizons relative to R13 and new AMS  $^{14}\text{C}$  ages. Positive AMS  $^{14}\text{C}$  ages for samples (1) and (3) show median probability little different than raw conventional ages.

### R13A Floodplain Section

Recent excavation of R13A (Figure 4A), at the same relative elevation (2 m) above the river (Figure 1B) and position as R13 (Figure 3), reveals a similar succession of overbank sediment (Cuk), atop a buried group of Ahbk/Cubk horizons extending down to stream level with a fully formed Entisol/Regosol (Ahbk/Cbk) in place. The surface Cu horizon shows no evidence of weathering and little incursion of root growth, suggesting a decade-long (or less) age. Despite the up to 1-m depth in the two profiles—R13 and R13A—close correlation of horizons is not possible given random floodplain erosional events at the two sites. However, stacking of multiple sequences of Ah horizons, separating thin flood deposits (Cu horizons) in both profiles, overlie a common, lower, better-developed buried soil with an Ah(k)/Cbk/Cuk profile. Similar to this approximation of the two closely spaced sections, the mineralogy and chemistry in R13A is a close copy to R13.

AMS  $^{14}\text{C}$  measurements gave ages to within a range of several centuries, which is consistent with the release of secondary Fe-Al oxides and hydroxides reported by Mahaney et al. (2016). We expect most of the age discrepancy between conventional and AMS dates are due to the different sample sizes, for AMS, smaller samples are used and the conventional ages were just “bulk dates”. However, modern samples showing bomb  $^{14}\text{C}$  effects (samples 2 and 4, Figure 4A) are located close to samples (1 and 3) with positive dates, the latter with narrow standard deviations ( $\pm 29$ – $32$  yr). This proximity (to within  $\sim 5$  cm) of contaminated and uncontaminated samples suggests an almost random chance that young Ahb horizons may deliver AMS  $^{14}\text{C}$  ages which contain bomb  $^{14}\text{C}$  in some cases, but not others. This suggests a

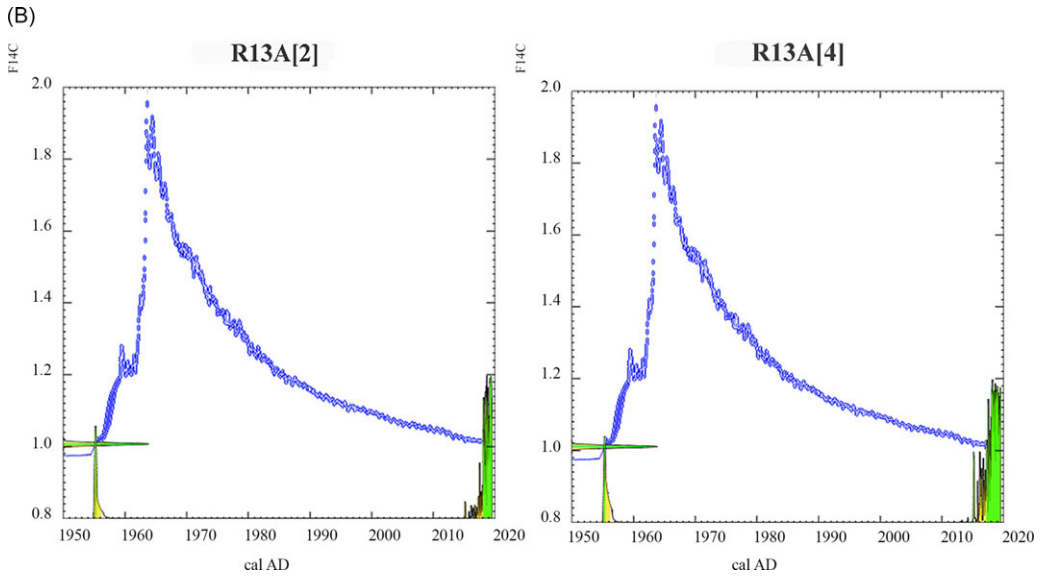


Figure 4 (B) calculation of the post-1950 ages using bomb- $^{14}\text{C}$  calibration curves of Hua et al. (2013) for samples (2) and (4) in Figure 4A.

better strategy would be a detailed separation of different organic components of the soil horizons, perhaps by stepped combustion (McGeehin et al. 2001).

Two samples, numbered 2 and 4 in section R13A, yielded post-bomb results at 70 cm and 50 cm depths, respectively. However, sample 3 in the section, unaffected by bomb effects, dates to  $312 \pm 32$  yr BP (calibrated to 301–464 cal yr BP) and is within the upper buried Ahb horizon complex of an older but immature soil, complete with convoluted micro beds that appear disturbed by interconnected thin lenses of overbank sediment from minor incursions of flooding in recent times. The lower uncontaminated sample (1) is within the lowest Ahbk horizon complex, part of which dates to  $588 \pm 29$  yr BP (calibrated to 537–647 cal yr BP). The difference between raw and calibrated ages for samples 1 and 3, varies between a decade for sample 1 and a century for sample 3. We take the older dates as representative of the age of the deposits, where no bomb  $^{14}\text{C}$  is evident.

### R15 and R71 Terrace Sections

Surface Ah horizons at a high (14–15 m) terrace (R15) and a low (3–4 m) terrace site, (R71) (Figure 1B for location), respectively, were selected to test levels of bomb contamination in the bottom 1-cm of each surface soil horizon (Figure 5A). The Ah horizon in R15 on the older high terrace (<11 ka) (Mahaney and Sanmugadas 1986; Mahaney et al. 2016) is ~15 cm thick, similar to the Ah horizon in R71 (not previously published) on the younger 3-m terrace (~1.0 ka). Both sites, previously dated by RD methods (R15 in Mahaney et al. 2016 and R71), show close agreement in Ah horizon thickness. This suggests that both surface horizons, despite their disparate ages, reach near maturity in a matter of a few centuries, at least with a steady-state input-output of organic carbon and nitrogen achieved. Previous work attests to the ~1.0–1.5 kyr value of organic matter in local Ah horizons to reach equilibrium where input equals



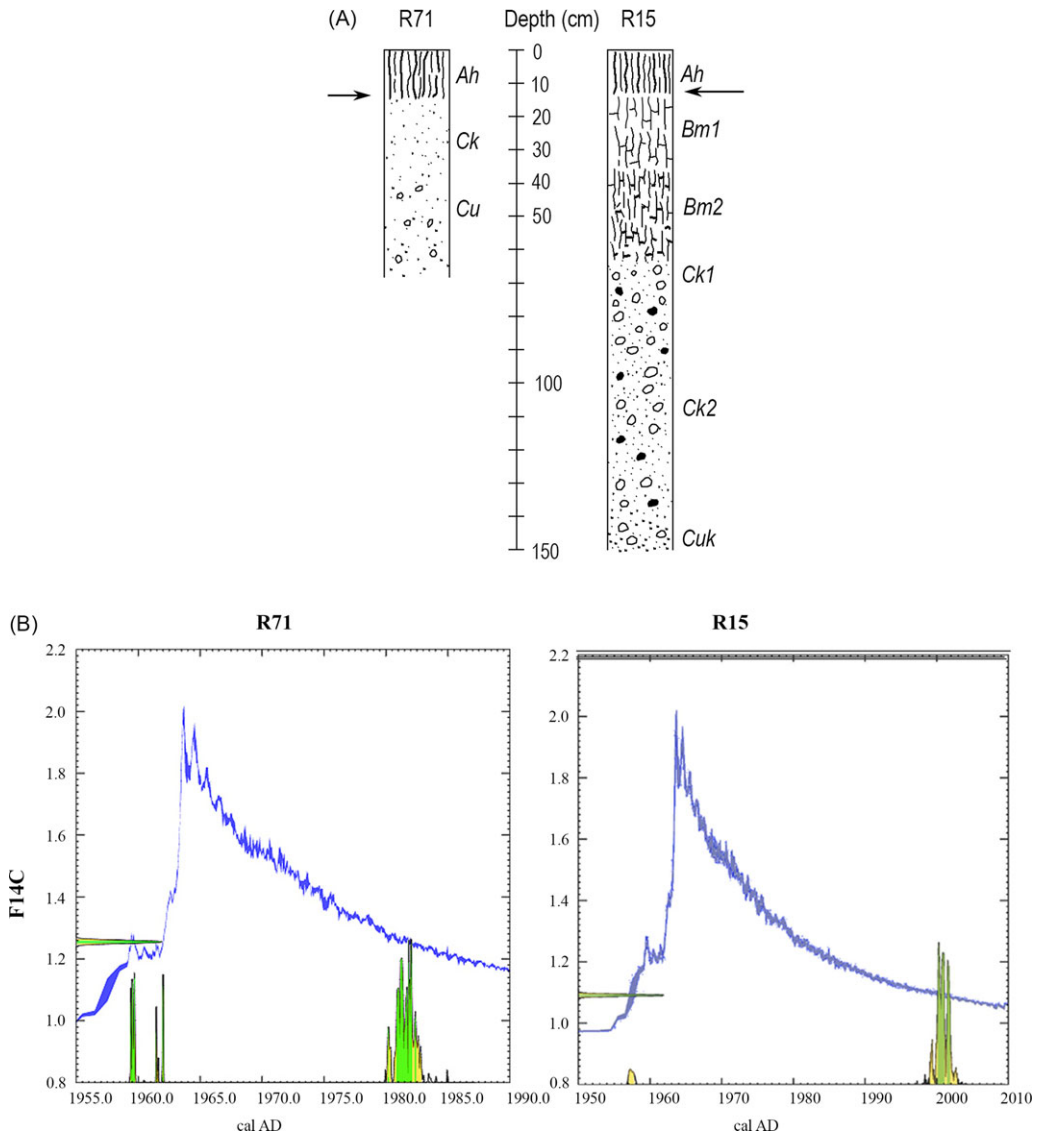


Figure 5 (A) Sections R71 (Late Holocene) and R15 (Early Holocene) in the Rouge/Little Rouge catchment. Arrows indicate sample locations; (B) calculation of the post-1950 ages using bomb- $^{14}\text{C}$  calibration curves of Hua et al. (2022) for sections R71 and R15 in Figure 5A.

output (Mahaney and Sanmugasdas 1986; Mahaney et al. 2016). However, both the basal Ah horizons R15 and R71 (see Table 1) show post-bomb radiocarbon dates.

#### R47 Colluvial Succession

The R47 and R47A successions (Figures 6–8) are pedostratigraphic complexes, which comprise a paleosol and ground soil formed in fine pebbly colluvium in the upper part of the R47 section in the Little Rouge Creek catchment (Figure 1B). The paleosols in R47 and R47A (from about 30-cm depth and below), overtopped with colluvium in which a thin ground soil formed, is

Table 1 Details of conventional and AMS radiocarbon ages in Late Holocene soils of the Rouge Catchment, South Central Ontario

Site	Sample	Depth (cm)	Material	Location (elevation)	Figure	<sup>14</sup> C age	Calibrated age (CE, 1σ, 68.3%)	Calibrated age (CE, 2σ, 95.4%)	F <sup>14</sup> C <sup>a</sup>	±	Lab number
R13	Ahbk (1)	0	Soil	Rouge R, floodplain (2 m)	3	20 ± 70	1675–1916	1675–1942	—	—	BGS 1068
	Ahbk (1)	20	Soil	Rouge R, floodplain (2 m)	3	100 ± 80	1685–1928	1665–1950	—	—	BGS 1072
	Ahbk (2)	30	Soil	Rouge R, floodplain (2 m)	3	0 ± 70	1695–1916	1679–1941	—	—	BGS 1074
	Ahbk (2)	40	Soil	Rouge R, floodplain (2 m)	3	50 ± 70	1694–1917	1674–1943	—	—	BGS 1076
R13A	Ahbk (4)	50	Soil	Rouge R., floodplain (2 m)	4	Post-1950	1955 or 2016–2019		1.0107	0.0032	UBA 43436
	Ahbk (3)	60	Soil	Rouge R, floodplain (2 m)	4	312 ± 32	1516–1641	1486–1649	0.9619	0.0038	UBA 43435
	Ahbk (2)	70	Soil	Rouge R, floodplain (2 m)	4	Post-1950	1954–1955 or 2018–2019		1.0071	0.0031	UBA 43434
	Ahbk (1)	90	Soil	Rouge R, floodplain (2 m)	4	588 ± 29	1320–1403	1303–1413	0.9294	0.0034	UBA 43433
R47	Ah (1)	5	Soil	L. Rouge, terrace ~30 (m)	6	(–4000) <sup>1</sup>	1963 or 1967–1968		1.645 <sup>2</sup>	0.014 <sup>2</sup>	BGS 1231

(Continued)

Table 1 (Continued)

Site	Sample	Depth (cm)	Material	Location (elevation)	Figure	<sup>14</sup> C age	Calibrated age (CE, 1σ, 68.3%)	Calibrated age (CE, 2σ, 95.4%)	F <sup>14</sup> C <sup>a</sup>	±	Lab number
	Ahb (2)	35	Soil	L. Rouge, terrace ~30 (m)	6	200 ± 80	1641–1950	1515–1950	—	—	BGS 1230
R47A	2Ahbk(1)	60	Paleosol	L. Rouge, terrace ~30 (m)	6	304 ± 28	1521–1644	1495–1652	0.9629	0.0034	UBA 43338
	2Ahbk (2)	68	Paleosol	L. Rouge, terrace ~30 (m)	6	413 ± 27	1442–1482	1433–1619	0.9499	0.0032	UBA 43432
R15	Basal Ah	10	Soil	Rouge R., terrace (14–15 m)	5	Post-1950	1957 or 2000–2001		1.0914	0.0031	UBA 41065
R71	Basal Ah	10	Soil	L. Rouge, terrace (3 m)	5	Post-1950	1959–1962 or 1980–1982		1.2549	0.0045	UBA 41061
WD5	Ahb	45	Soil	W. Duffins (terrace 3 m)	9	600 ± 70	1303–1405	1280–1433	—	—	BGS 2627

Notes: Finite ages (R13, R13A horizons 1 and 3, R47-Ahb, R47B and WD5) were calculated using Oxcal and the IntCal20 calibration curve (Reimer et al. 2020; Bronk Ramsey et al. 2023); post-bomb values were calculated using Calib.org/CALIBomb (Reimer and Reimer 2024 CALIBomb [WWW program] at <http://calib.org> accessed 2023-05-03). For post-bomb values, the calibration results for 1σ and 2σ were the same for the values given.

<sup>a</sup>Value reported by the laboratory.

<sup>1</sup>Value quoted in original date report.

<sup>2</sup>Estimated from Calib.org/CALIBomb, error based on similar measurements by BGS.

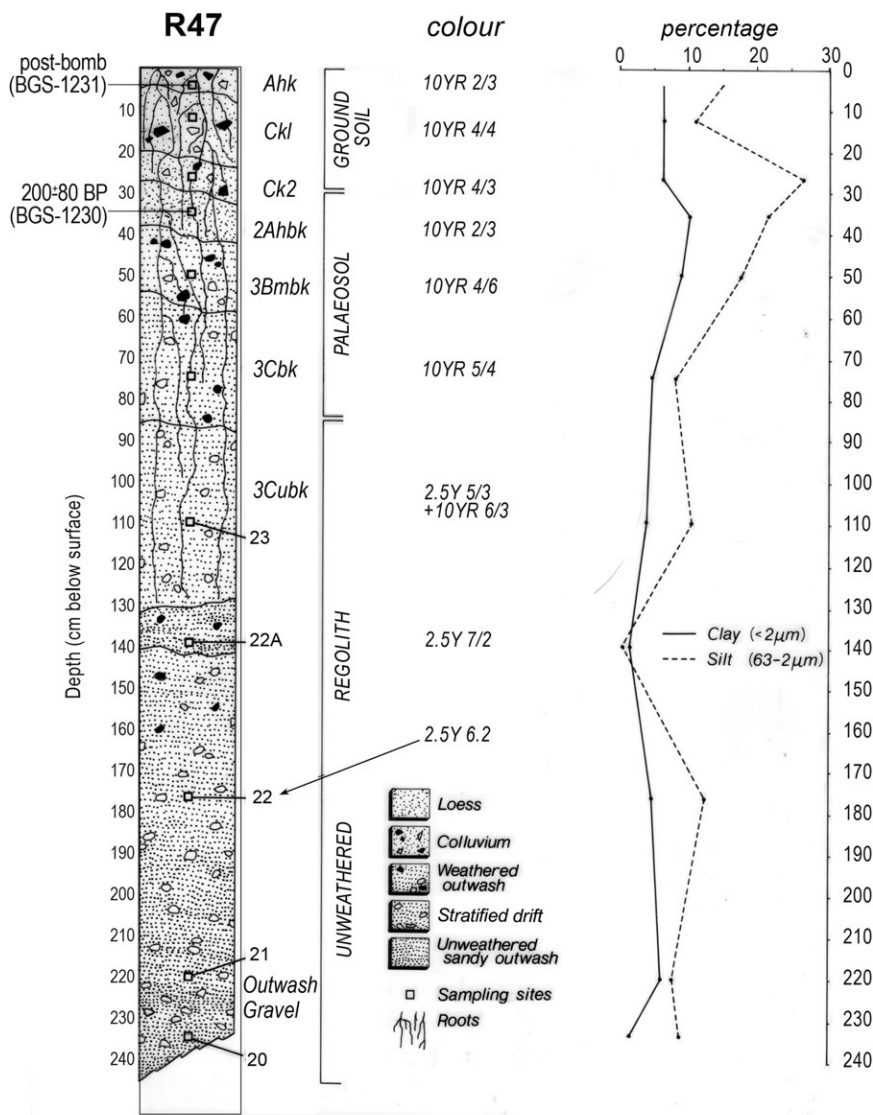


Figure 6 R47 section in colluvium, mixed outwash, and Halton moraine sediment in the upper part of section R47 (Mahaney and Hancock 1993a, 1993b).

variably dated by both conventional and AMS  $^{14}\text{C}$ . Previous (1990) dating of the mid-section of the 2Ahbk horizon in the R47 paleosol yielded an uncalibrated  $^{14}\text{C}$  age of  $200 \pm 80$  (BGS-1230); (Mahaney and Hancock 1993b) age (Figure 6). A later resampling (2020) of the R47A profile by AMS  $^{14}\text{C}$  (see below, Figures 7 and 8) yielded uncalibrated ages of  $304 \pm 28$  BP for the upper  $\sim 2$ cm and  $413 \pm 27$  yr BP for the lower  $\sim 2$ cm of the buried 2Ahbk horizon. Calibrated ages for these two horizon positions yield ages of 295–415 cal yr BP (upper) and 331–517 cal yr BP (lower). The mid-section (5 cm) of the Ahk horizon in the ground soil, gives a post-bomb value of  $F^{14}\text{C}$  of  $\sim 1.645$ , only consistent with formation in the 1960s. The paleosol, formed in the colluvial beds of the section, with its 2Ahbk/3Bmbk/3Cbk/3Cubk profile





Figure 7 R47A section showing the upper part of the R47 Section with moraine, outwash and colluvial deposits resampled in 2020—view to the south along Little Rouge Creek.

(Figure 6)—the combined ground-paleosol, approximately 120-cm thick—correlates with the thickness of postglacial Late Holocene paleosols in other catchments of central Southern Ontario (Mahaney et al. 2016). The one-century spread (for both raw and calibrated ages) for development of the 2A<sub>h</sub>b<sub>k</sub> horizon is probably a minimum given that the uppermost part of this horizon was likely eroded (truncated) when it was over-topped by colluvial emplacement—the deposit in which the ground soil formed. Particle size trends through the succession (Figure 6) follow unremarkable variations from sampling site 20 in the upper till body of R47 at 240-cm depth, becoming less silty in the sand beds upward in section, grading into higher silt content further up. The highest silt values are in the 2A<sub>h</sub>b<sub>k</sub>, which may be air-inflused sediment. The argument for pedogenesis in this young buried pedon rests more on slow build-up of organic carbon without demonstrable removal of carbonate and/or increase in clay.

### R47A Colluvial Succession

These two, near-parallel soil/paleosol sections—R47 and R47A—exhibit two distinct differences in profile horization: firstly, the presence of fresh sediment (C<sub>uk</sub>) between the surface soil and the 2A<sub>h</sub>b<sub>k</sub> of the paleosol in the R47A profile (Figure 8); secondly, a 15–20-cm difference in the thickness of the ground soil (30–35 cm in R47 and 50–60 cm in R47A). We did not carry out a full excavation of the paleosol in R47A, because we sought only to achieve AMS <sup>14</sup>C dating of the buried 2A<sub>h</sub>b<sub>k</sub> horizon, the lower horizons already having been fully analyzed in Mahaney and Hancock (1993b).

### West Duffins Creek—Floodplain and Terraces

West Duffins Creek (Figure 1A), like the neighboring Rouge catchments, winds its way southward from the Interlobate Moraine (formed by combined action of the Simcoe and

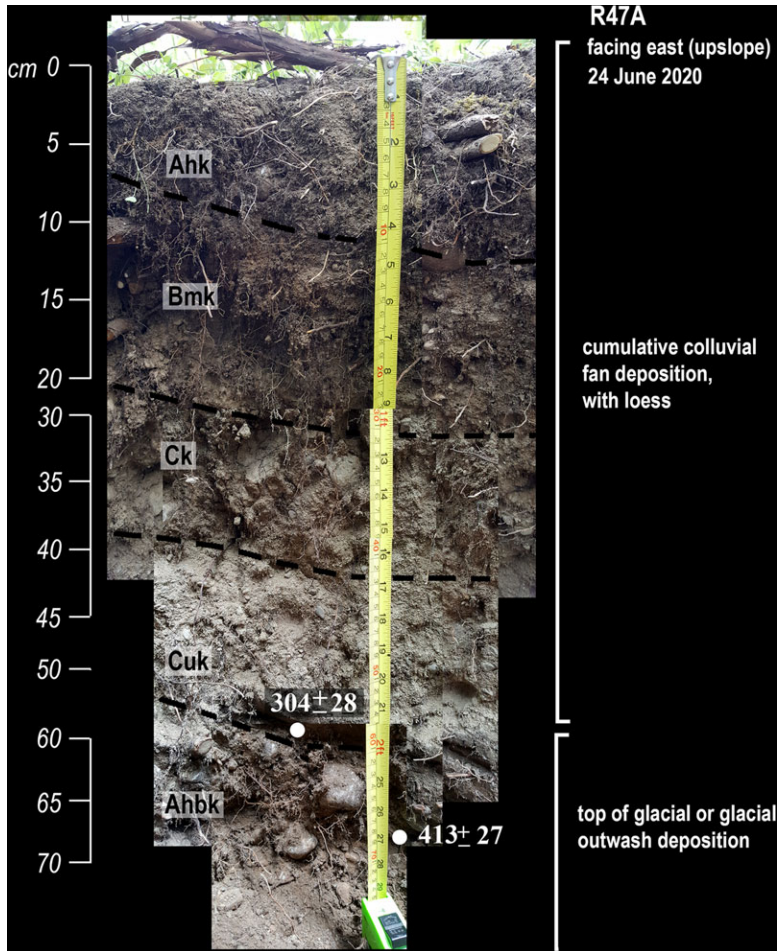


Figure 8 R47A profile with new AMS  $^{14}\text{C}$  from upper and lower beds in the Ahbk horizon providing ca. 110 yr for its establishment prior to burial by colluvium. Burial 304 yr BP in the mid 17th century places displacement within the mid-LIA. Positive AMS  $^{14}\text{C}$  ages for the upper and lower beds of the Ahbk horizon show median probability ages little different than raw conventional ages.

Ontario lobes of the LIS) into Lake Ontario, exposing similar successions with variable incisions of Wisconsin-age till, moraine, and deltaic sediment. The southern reaches of the basin show the greatest range of floodplain and older terraces (Figure 1A). These deposits, ranging from recent, fresh ice-dammed deposits, to older terraces with mature Inceptisols formed in alluvium, lie on top of unweathered, thick, deltaic sediment of Scarborough age, as outlined in the Southern Ontario glacial succession of Terasmae and Dreimanis (1976), Mahaney and Hancock (1993a, 1993b) and Sharpe et al. (1999). As in the nearby Rouge catchments, the soil deposit-weathering trends in the W. Duffins Basin includes overbank ice-dammed sediment that overtop floodplain successions of Ahb/Cubk/Ahbk/Cbkox/Cbk/Cubk profiles. On higher terraces, epipedons (Ah-Bw/m horizons) are clearly differentiated, free of dissolved carbonate, and occur in overlying variations of Cox/Ck/Cuk substrate horizons, similar to profiles R71 and R15 in the Rouge catchments. Unfortunately,  $^{14}\text{C}$  datable materials are rarely found within these soil stratigraphic successions (excluding WD5 discussed below),

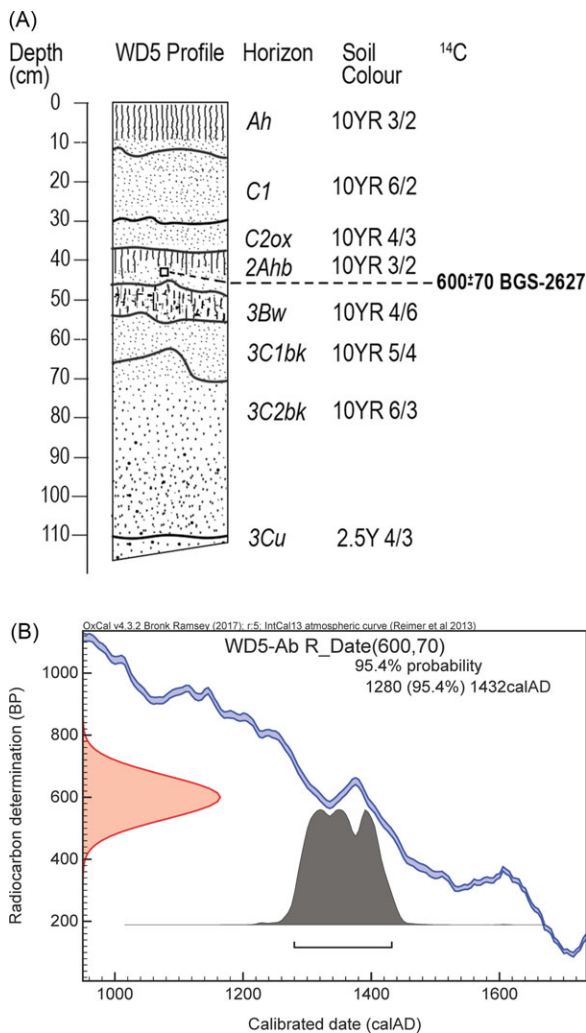


Figure 9 (A) WD5 profile from the adjacent West Duffin Creek Catchment, located in a floodplain terrace ~3 m height, with an Ahbk horizon <sup>14</sup>C dated to 600 ±70 yr BP by conventional means; (B) Oxcal v.4.4.4 plot of the age with a 95.4% probability marking the calendar age between 1280 and 1433 AD placing it within the Medieval Warm Period.

the only dating methods being limited to soil-profile expression, acid dissolution of carbonate during expanding thickness of epipedons with age, and extractable forms of Fe and Al (Mahaney et al. 2016). The two <sup>14</sup>C dates attempted both gave post-bomb results.

### WD5 Section

East of the Rouge, in the West Duffins Creek floodplain, a buried soil (site WD5, Figure 1A) with ~70 cm thickness, includes a thin, 8-cm-thick Bw horizon (Figure 9A), which is the first of its kind in all of the floodplain sites investigated since 1973. The conventional age of its 2Ahb horizon is 600 ±70 yr BP (BGS-2627), which calibrates to between AD 1280 and AD 1433 (Figure 9B), placing the sediment to the beginning of a “Little Ice Age” (LIA) in Ontario at AD 1300 discussed by Finkelstein and Davis (2006), even though this age range is more commonly

associated with the Medieval Warm Period ~750–1350 AD (Crowley 2000; Goosse et al. 2006). More commonly, the age bracket for the Little Ice Age ranges from ~1450–~1850 AD (Crowley 2000), although this varies among authors. Because the buried soil epipedon was truncated during deposition by overlying beds, its age is a minimum, reinforcing the argument that an enduring floodplain was stable long enough for an Inceptisol to develop in its distal and older sector, which was later buried ~700–900 yr ago. With the floodplain in both drainages taking up to 60% of the valley width, its age must extend at least to a few millennia. Details of particle size and mineralogy are similar to those for R13 reported here (Figure 3), with downward trends indicating a detrital bed succession. This is the first discovery of a Bw horizon in floodplain sediments in any of the prominent successions of South-Central Ontario, and presumably with a minimum conventional age of  $600 \pm 70$  yr BP.

## DISCUSSION

Reconstruction of the postglacial history of catchments in central Southern Ontario is hampered by lack of radiocarbon-datable sediment with the exception of variable centuries-old floodplain and colluvial sections with Ahb or Ahbk soil horizons, the latter group being rare in the Rouge catchment. As indicated here, and summarized in Table 1, dating buried floodplain Ahb or Ahbk horizons is a hit and miss proposition. As often as not, one accurately dated bed is located within centimeters of, and potentially offset by, sediment carrying bomb imprints and post-1950 ages. It may be that bomb contamination comes from surface runoff and soil water throughput with contaminated organic carbon following root systems or micro channels within the floodplain matrix material. Given closeness to ground water, some contamination may ingress the floodplain from below, although the database for R13A argues against this possibility. Using IntCal, the  $F^{14}C$  value is known at the start of contamination (at about AD 1950) and at termination (AD 2020) or less, but the IntCal analysis provides no atomic bomb contamination values between these two timelines. AMS  $^{14}C$  improves on older conventional dates by providing  $F^{14}C$  at start-up and near present-day residual concentrations of contamination, if above 1.0  $F^{14}C$ . As well, for samples not subject to atomic bomb contamination, ages for Ahb and Ahbk horizons stretching centuries back into the Holocene provide higher resolution baseline dates, which is a vast improvement over previous conventional timelines and avoids listing such samples as “modern.”

### AMS $^{14}C$ Post-1950 Calibration

Concentration of AMS  $^{14}C$  contamination reported here is solvable only at the start-up of nuclear atmospheric testing at 1950, and the post-1950 cal AMS  $^{14}C$  return over several decades to ~1980–2020 levels, measured at ~1.0  $F^{14}C$  by AMS (see Figures 4B, 5B). These measurements show a relatively small peak in the 1950s, presumably from atmospheric tests by the US and Russia, followed by airborne testing at sites in Europe, with termination at 1980–2020 as shown on Figures 4B and 5B. Hence, many researchers, therefore, simply rate samples “modern.” Choosing between these two solutions—start up and termination—entails a consideration of fossil fuel and industrial combustion that releases  $^{14}C$  as free  $CO_2$ , which adds to or may dilute bomb  $^{14}C$ , or both. These contaminants are not uniform across hemispheres, with bomb effects higher in the Arctic, lower in the mid-latitudes, where they are offset by industrial emissions, and lower still in the Southern Hemisphere.

Because small amounts of measured contamination still reside in the terrace samples, and nil in the floodplain, this suggests different leaching mechanisms at work in the two landforms.



Surface runoff, soil water movement, and groundwater flux are the most likely leaching systems. It may be that with only two of these systems—runoff and soil water profile throughput—operating in the terraces, and all three operating in the floodplain, groundwater flux working in tandem with overbank flooding is the most effective system for reworking contaminants, as demonstrated with the range of contamination noted above. For particle binding to be responsible for retention of bomb contaminants, the only likely clay mineral present in all sections is illite or possibly illite-smectite, all dependent upon broken bonds and interlayer molecular water not fully filled with  $K^+$  or other ions. Broken bonds can take up water (Brindley and Brown 1980) and most likely bomb contamination.

### AMS $^{14}C$ Pre-1950 Ages

AMS  $^{14}C$  on uncontaminated samples demonstrates there can be more time for Ah and Ahb horizon development prior to burial and irrespective of depth. Above ~70 cm, some samples yield post-1950 ages. The tentative approximate 70-cm depth is based on depth in the floodplain below which contamination appears to cease. The AMS  $^{14}C$  pre-1950 ages for samples, are consistent with the results of Fe/Al extractions outlined earlier by Mahaney et al. (2016), which show coupled Ahb/Coxb horizons requiring more than a century for weathering to register. This is especially the case with  $Fe_d$  (Na-dithionite Fe), which is used as a relative time proxy for weathering of total Fe ( $Fe_t$ ) into secondary hydroxide/oxide form (Blume and Schwertmann 1969; Dormaar and Lutwick 1983; Parfitt and Childs 1988; Birkeland et al. 1989; Schwertmann and Taylor 1989). Despite the high carbonate composition of most sediment in local catchments (~80% carbonate and 20% silicate, on average), there is sufficient Fe-Al in the silicate group to provide a reasonable relative age assessment, even for sediment weathering over the last few centuries. Beyond the  $Fe_d/Fe_t$  ratio as a relative time estimate,  $Fe_o$  (acid ammonium oxalate) extractions taken from the floodplain soils provide, with arithmetic adjustment, an initial estimate of ferrihydrite ( $Fe_{10}O_{13} \cdot 9H_2O$ ), a major weathering product and one of the secondary oxihydroxides in soils. The concentration of  $Fe_o$  multiplied by 1.7 (Parfitt and Childs 1988) equals the concentration of ferrihydrite. Since ferrihydrite is partly soluble, its increase with time is not linear, but linked with its two common oxihydroxides (hematite and goethite) =  $Fe_d$ , which by itself is a measure of relative time. The quotient  $Fe_d/Fe_t$  yields the slow release of oxihydroxides relative to total Fe.

To test  $Fe_d/Fe_t$  as an RD age measurement, calibrated by AMS  $^{14}C$  in the first few centuries, data in Figures 10A and 10B illustrate initial age plots rising slowly in progression and in alignment with stream incision estimates arrived at earlier, with a cap of 11 ka correlated with recession of the Ontario Lobe of the LIS and onset of Glacial Lake Iroquois. Even with wide ranges of  $Fe_d/Fe_t$  quotients, indicated by box volumes (Figure 10A), major valley incisions producing terrace systems, the older and middle terrace groups are clearly differentiated from the floodplain group.

The study presented here is limited by the measurement of only bulk soils. This seriously limits the interpretation of the results and emphasizes the need for a systematic approach using specific temperature extractions (e.g., McGeehin et al. 2001) or perhaps selection of specific organic compounds (e.g., Casanova et al. 2021).

### CONCLUSIONS

Previous conventional  $^{14}C$  age determinations of floodplain, terrace, and colluvial soils in the Rouge catchments yielded very young ages with large standard deviations, often with

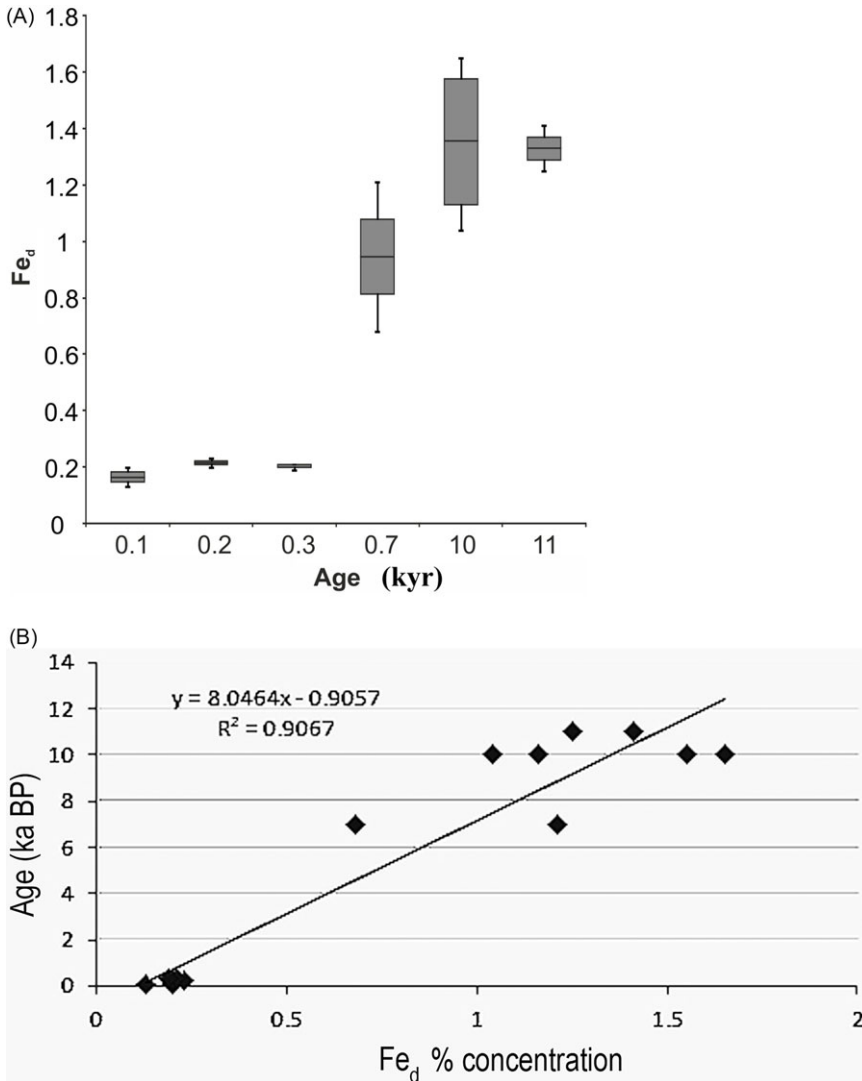


Figure 10 (A) Correlation with box plots of  $Fe_d$  and projected valley incision ages for terraces in the Rouge basin. The floodplain sites (R13, 13A) were staggered from 0.1 to 0.3  $\times 10^3$   $^{14}C$  YBP (Mahaney and Terasmae 1988; Mahaney et al. 2016). Ages of older sites are based on relative age indices as follows: R46 = 6–7 ka (Mahaney 2015); R61, R62, and R60 = <11ka, relative height of 15–17 m above the existing stream level in Little Rouge Creek (Mahaney et al. 2014b). Given the young ages for the pedostratigraphic units in the floodplains, all horizons are included in the analysis. For the older profiles—R46–R62—only the epipedons (Ah + Bw[t]) horizons are included in the analysis as these are the most weathered beds (horizons) in each profile (with modifications from Mahaney et al. 2016); (B) positive regression of  $Fe_d$  illustrates growth of Fe oxihydroxides over time (from Mahaney et al. 2016).

post-1950 ages far into the future. Using the AMS  $^{14}C$  ages obtained and reported here, we attempted to improve these age estimations to better assess soil successions in both floodplains, on the one hand (R13, R13A), and Ah horizons in higher terrace soils (R71, R15), coupled with colluvial (R47, R47A) sets, on the other. The floodplain soils, possibly containing reworked organic carbon yield both signatures of bomb  $^{14}C$  and AMS  $^{14}C$  ages at depths tentatively down to ~70 cm. These older ages provide more time to explain variable concentration levels of

Fe-Al extractions, which were used as RD age indicators in previous studies (Mahaney and Sanmugasdas 1986; Mahaney et al. 2016). The new AMS ages are consistent with the time required to produce  $Fe_d$  age values ( $Fe_d=Na$ -dithionite extractions) obtained in more robust, that is, thicker buried soils in R13 and close correlatives in R13A.

Variations of organic carbon, particle size and/or section aspect may influence sample contamination, with root position and sediment microfractures controlling movement of contaminants. Perhaps the most important outcome of this work is the tighter age control on the younger stages of weathering and soil morphogenesis in two of the major catchments of South-Central Ontario. However, it is clear that a more detailed study should be undertaken. With better control over the first five-to-six hundred years of soil morphogenesis, it is now possible to estimate, with some reliability, the variable concentrations of specific secondary Fe and Al oxides and hydroxides, at start-up in the initial soil weathering phase. Because the calcite-silicate ratio is uniform within the parent materials in the catchments, soil morphogenetic changes—particle size, pH, organic carbon/N—in older pedons can be more tightly constrained within the established relative age proxy.

## ACKNOWLEDGMENTS

This work was funded by Quaternary Surveys, Toronto, by minor research grants to WCM from York University and from past grants from NSERC (Natural Sciences and Engineering Research Council of Canada). WCM is indebted to students in Methods of Sediment and Soil Analysis (Geography 3510), Fall 2013 and 2015, for stimulating discussions of the data contained herein. We thank Paula Reimer and Stephen Hoper (Chrono 14, QUB) for monitoring the samples through the dating process and for assistance with the calibration curves. We also appreciate critical comments from four anonymous reviewers and gratefully thank the editor, Tim Jull, for his technical and editorial assistance.

## COMPETING INTEREST DECLARATION

This is to confirm we have no competing interests to declare.

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