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ACCELERATOR MASS SPECTROMETRY DATING OF MEADOWCROFT ROCKSHELTER MAIZE

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ABSTRACT. The Meadowcroft Rockshelter in southwestern Pennsylvania is best known for its pre-Clovis occupation. Potentially important for later times is the recovery of maize macrobotanical remains from higher strata dating as early as the 4th century BC based on radiometric radiocarbon (^{14}C) dates on wood charcoal. These remains have been considered to be potentially as old as the earliest microbotanical evidence for maize in Michigan, New York and Québec recovered from directly dated charred cooking residues adhering to pottery. The results of accelerator mass spectrometry (AMS) dating 17 samples from maize specimens from all Meadowcroft strata producing maize, indicate that the specimens originated from historical use of the shelter, most likely after AD 1800. These results further emphasize the need to obtain direct dates on maize macrobotanical remains recovered from early contexts prior to the development and common use of AMS dating.

KEYWORDS: AMS dating, chronology, maize, paleobotany.

INTRODUCTION

The histories of the spread of maize (*Zea mays* ssp. *mays*) north and south of central Mexico where it evolved from an annual teosinte (*Zea mays* ssp. *parviglumis*) 9000–7000 years ago (Matsuoka et al. 2002), its adaptations to wide ranges of climatic and edaphic conditions, the timings of its adoptions by far-flung Native American societies, and the impacts of its adoption, if any, on regional subsistence-settlement systems remain important topics of research for archaeologists, geneticists, and paleoethnobotanists (e.g., Staller et al. 2006; Bonavia 2013; Grobman 2013; Blake 2015; Pearsall 2019). While major strides have been made in the past few decades in building knowledge on each of these topics through a variety of analytical methods and techniques, the crop's histories remain far from settled in many regions. One such region is temperate northeastern North America (hereafter Northeast), one of the last regions where maize was adopted, but where it became the main crop of agricultural systems after AD 1000–1300 (Hart and Lovis 2013). Resolving the timing of the crop's adoption is necessary to anchor maize's histories in this region and has been a long-standing focus of research that is yet to be resolved (e.g., Emerson et al. 2020; Dotzel 2021; Simon et al. 2021; Stewart 2021). Current microbotanical evidence from Michigan (Schultz site; Albert et al. 2018), New York (Vinette site; Hart et al. 2007a), and Québec (Place-Royale site; Gates St-Piere and Thompson 2015) (Figure 1), and potentially southern New England (Dotzel 2021), in the form of phytoliths and starch recovered from accelerator mass spectrometry (AMS)-dated cooking residues adhering to pottery sherd interior surfaces indicates use by at least cal. 300 BC. However, the macrobotanical evidence, which until recently was largely in line with this date for the greater Northeast, is in a state of flux.

Early applications of accelerator mass spectrometry (AMS) to directly radiocarbon (^{14}C) date macrobotanical remains in the Northeast showed that maize recovered from early contexts at

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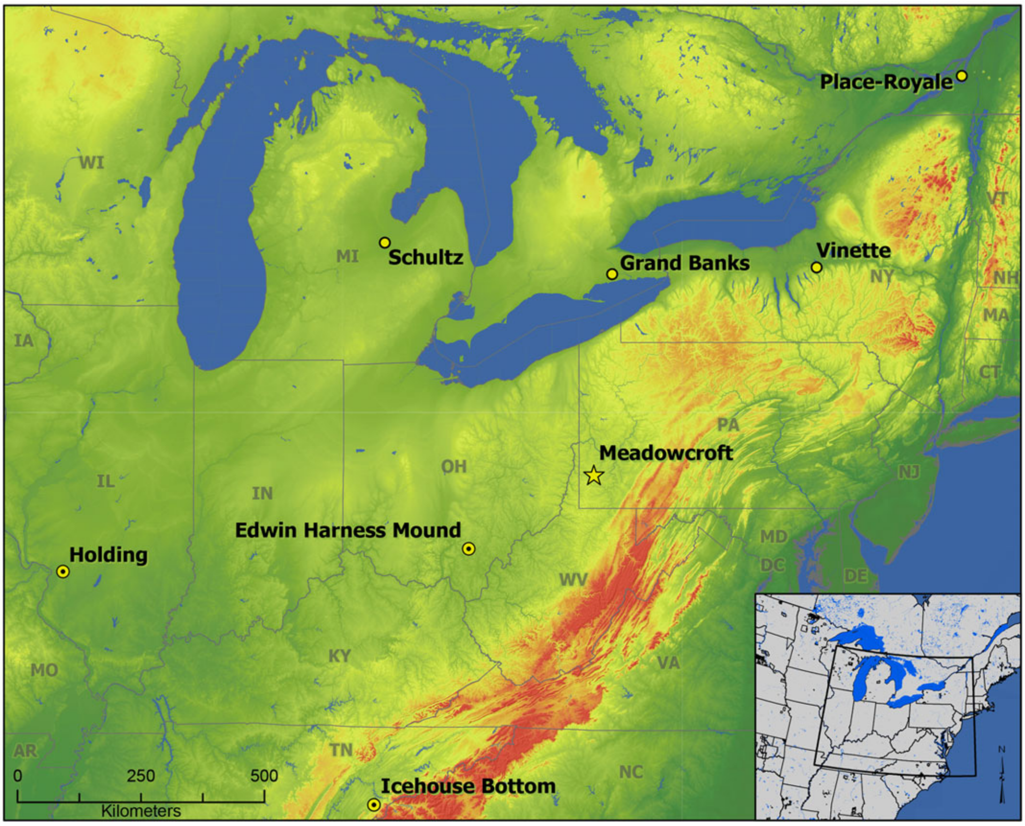

Figure 1 Locations of sites mentioned in the text. Circles with dots are sites with recently discredited evidence.

several sites dated much later than the contexts suggested (e.g., Conard et al. 1984; Murphy 1989). On the other hand, AMS dates on material identified as maize from several sites seemingly confirmed an early presence for the crop (Figure 1). Two sites in particular produced dates that have anchored macrobotanical evidence for early maize (Smith 2017): Holding in Illinois (2077 ± 70 BP, cal 2σ 355 BC–AD 116, median 90 BC; 2017 ± 50 BP, cal 2σ 93 BC−AD 69, median 9 BC; Riley et al. 1994) and Edwin Harness Mound in Ohio (1730 ± 60 BP, cal 2σ AD 136–423, median AD 303; Ford 1987) along with Icehouse Bottom to the south in Tennessee (1730 ± 85 BP, cal 2σ AD 129–537, median AD 329 and 1720 ± 105 BP, cal 2σ AD 84−569, median AD 338; Chapman and Crites 1987) (Figure 1). Unlike current practice, the samples used to generate these dates were not subjected to stable carbon isotope ratio measurement. Rather, a mean $\delta^{13}C$ value ($^{13}C/^{12}C$ ratio) for C_4-pathway plants was used in the ^{14}C age calculations to account for carbon isotope fractionation effects (Taylor and Bar-Yosef 2014). Archaeological maize in the Northeast produces $\delta^{13}C$ values (~ –15.1 to –7.4‰) that are substantially less negative than those of C_3-pathway plants (~ –28.6 to –23.3‰) (Hart et al. 2007b). There are C_4-pathway plants native to the Northeast such as purslane (*Portulaca oleracea*, Tankersley et al. 2016). However, the large structures of maize (kernels, cob fragments) make it unlikely that macrobotanical remains from C_4-pathways plants native to the region it would be mistaken for maize. While today $\delta^{13}C$ values for fractionation calculations are generally obtained

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online in the accelerator mass spectrometer on prepared graphite, $\delta^{13}C$ values obtained through isotope-ratio mass spectrometry (IRMS) can be used to confirm identifications of maize macrobotanical remains when those identifications are not confident (e.g., Simon et al. 2021).

Recently, Simon (2017) obtained $\delta^{13}C$ measurements on the originally dated Holding site samples, which indicated they were C_3-pathway plant remains rather than maize. Subsequently Simon and colleagues (2021) obtained AMS dates and $\delta^{13}C$ measurements on macrobotanical remains identified as maize from Edwin Harness Mound and Icehouse Bottom—the originally dated samples were no longer extant. They found that some remains are not maize based on $\delta^{13}C$ values, while others were confirmed as maize but dated much later in time than the original samples. While these results did not prove the originally dated samples were not maize, they did raise that possibility. At present, then, the earliest, as-yet unquestioned, directly AMS-dated macrobotanical sample identified as maize in the Northeast is from the Grand Banks site in southern Ontario (1570 ± 90 BP, cal 2σ AD 258–650, median AD 491; Crawford et al. 1997) (Figure 1). There is now a chronological gap between the micro- and macrobotanical evidence for maize of over 800 years.

Maize macrobotanical remains potentially older than those from Grand Banks have been recovered from various sites in the Northeast but have yet to be directly dated and subjected to IRMS (McConaughy 2008; Hart and Lovis 2013; Stewart 2021). Obtaining direct dates on these remains coupled with IRMS $\delta^{13}C$ measurements to confirm identifications is needed to help clarify the histories of maize in the region.

Caves and rockshelters, two categories of archaeological site relatively rare in the Northeast, provide excellent conditions for preservation of charred and desiccated maize macrobotanical remains and have provided key evidence for early maize in Mexico, Mesoamerica, and the American Southwest (e.g., Piperno and Flannery 2001; Merrill et al. 2009; da Fonseca et al. 2015; Kennett et al. 2017; Swarts et al. 2017; Torres-Rodríguez 2018). Most prominent of such sites in the Northeast is the stratified Meadowcroft Rockshelter located on Cross Creek, an east-west-flowing tributary of the Ohio River, southwest of Pittsburgh, Pennsylvania near the West Virginia boarder (Figure 1; Adovasio et al. 1978; Adovasio 2010). Excavated primarily in 1973–1979 and sporadically thereafter, the site is best known for its pre-Clovis component (e.g., Haynes 2015; Carr 2018; Williams and Madson 2020). However, potentially important for maize history is a series of charred and desiccated cobs and cob fragments recovered from later strata as reported in Adovasio and Johnson (1981). These strata were defined chronologically with radiometric ^{14}C dates on wood charcoal, and as was common practice at the time, these were used to assign dates to the maize (Table 1). ^{14}C dates with median calibrated dates of 403 BC and 349 BC from the earliest stratum to yield maize (Stratum IV) are in-line with the earliest dates for maize microbotanical remains in the Northeast. These finds have been cited as potential evidence for early maize in the Northeast, but the need for direct dates to confirm their early age has been noted often (e.g., Crawford et al. 2006; McConaughy 2008; Hart and Lovis 2013); it is now accepted practice to directly AMS date crop remains because there is no necessary chronological relationship between spatially associated wood charcoal and the crop remains (Blake 2006). Here, we report direct AMS dates on a series of 17 samples of maize cobs/cob fragments recovered from Meadowcroft. The results emphasize the need to directly AMS date macrobotanical remains of maize and other crops recovered and reported prior to the development and common use of AMS dating.

Table 1 Maize cob data. Associated radiocarbon dates from Adovasio and Johnson (1981). Rows, grain thickness, cupule width, charring, and notes from Cutler and Blake (1977) as published in Adovasio and Johnson (1981).

Catalog no.	Sample no.	Stratum	Depth in stratum	Associated ^{14}C age(s) BP	Median cal date	Charred?	Rows	Grain thickness (mm)	Cupule width (mm)	Notes
FS-160-12	–	XI-IX mix	Mix	post 685±80 pre 175±50	AD 1314 AD 1782	No	12	3.4	8.1	
FS-130-10	2	Lower XI	Mix	post 685±80 pre 175±50	AD 1314 AD 1782	No	8	4.2	12.8	
FS-130-10	Not sampled	Lower XI	Mix	post 685±80 pre 175±50	AD 1314 AD 1782	No	12	3.7	7.0	
FS-130-10	1	Lower XI	Mix	post 685±80 pre 175±50	AD 1314 AD 1782	No	12–14	3.4	9.2	
FS-130-10	Not sampled	Lower XI	Mix	post 685±80 pre 175±50	AD 1314 AD 1782	No	14	–	9.0	Same as 12–14 row cob above.
FS-130-26	2	Lower XI	Mix	post 685±80 pre 175±50	AD 1314 AD 1782	No	16	4.0	8.0	Cupules compressed. Row number is higher than usual for Indian corn from eastern U.S. Could this be post 1800?
FS-130-26	1	Lower XI	Mix	post 685±80 pre 175±50	AD 1314 AD 1782	No	12	3.6	8.2	Recent?
FS-750	–	XI	0–10	post 685±80 pre 175±50	AD 1314 AD 1782	No	8	3.4	11.3	
FS-183-3	–	IX	0–10	685±80	AD 1314	No	14	3.9	8.8	Cupules compressed. Modern?
FS-1881-1	–	IX	0–10	685±80	AD 1314	Yes	12	3.8	6.2	Cupules open.
FS-269-8	1	VII	0–10	1290±60 925±65	AD 735 AD 1118	No	10	3.6?	7.6	

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Table 1 (*Continued*)

Catalog no.	Sample no.	Stratum	Depth in stratum	Associated ^{14}C age(s) BP	Median cal date	Charred?	Rows	Grain thickness (mm)	Cupule width (mm)	Notes
FS-269-8	2	VII	0–10	1290±60 925±65	AD 735 AD 1118	No	12	4.0	10.0	
FS-750-5	3	V	0–10	2155±65 2075±125	196 BC 94 BC	No*	12	3.2	5.5	Glumes probably long.
FS-750-5	2.2	V	0–10	2155±65 2075±125	196 BC 94 BC	No	12	3.1	6.5	
FS-750-5	2.1	V	0–10	2155±65 2075±125	196 BC 94 BC	Yes	12?	2.8	9.0	
FS-790-5	1.1	V	0–10	2155±65 2075±125	196 BC 94 BC	Yes	14?	3.0	5.0	
FS-790-3	–	V	0–10	2155±65 2075±125	196 BC 94 BC	Yes	10	3.3	7.3	Rows slightly twisted
FS-811-3	–	IV	10–20	2325±75 2290±90	403 BC 349 BC	Yes	16	2.7	3.8	Probably popcorn—very small, some rows only partially developed

*Cutler and Blake (1977) indicated this cob fragment was carbonized. However, only two of the samples from this provenience in the collection are carbonized and this fragment is not. The cupule width matches Cutler and Blake's measurement as well as their statement on glumes.

THE MEADOWCROFT MAIZE REMAINS

Adovasio and Johnson (1981) reported maize macrobotanical remains in the form of charred and desiccated cobs and cob fragments from the upper strata (IV–XI) of the Meadowcroft Rockshelter (Table 1). Identification of the maize was done by Cutler and Blake (1977) at the Missouri Botanical Garden as reported in an unpublished manuscript and summarized by Adovasio and Johnson (1981). The earliest of these was a small, charred cob fragment identified as probably 16-row popcorn, from Stratum IV associated with ^{14}C dates on wood charcoal of 2355 ± 75 BP and 2290 ± 90 BP. Stratum VI yielded 10-, 12-, and 14-row cob fragments associated with wood charcoal dates of 2155 ± 65 BP and 2075 ± 125 BP. Cob fragments representing 10- and 12-row maize were recovered from Stratum VII with associated wood charcoal dates of 1290 ± 60 BP and 925 ± 65 BP. Botanical remains from Stratum IX included 12- and 14-row maize associated with a date on wood charcoal of 685 ± 80 BP. Cobs and cob fragments from Stratum XI, from 8-, 12-, and 14-rowed maize were reported as dating later than 685 ± 80 BP and earlier than 175 ± 50 BP. In their unpublished report on the maize, Cutler and Blake (1977: 1) related that the maize from Meadowcroft was "surprisingly large and vigorous, the cobs firm and thickened." They indicated that three of the cobs/cob fragments, one from Stratum IX and two from Stratum XI, were possibly modern, post-1800, and recent, respectively (Table 1). This suggested the possibility of some mixing of earlier and later deposits within these strata. They attributed most of the remaining cob fragments in these strata to their prehistoric Midwest 12-Row maize category, with a few ascribed to the prehistoric Eastern 8-Row category.

METHODS AND MATERIALS

The Meadowcroft Rockshelter maize remains are curated at the Senator John Heinz History Center in Pittsburgh along with the rest of the site's collection. All maize remains are identified by catalog number, wrapped in aluminum foil, and stored in capped plastic vials. The specimens were placed on loan to the New York State Museum (NYSM) where photography and sampling were completed. For catalog numbers with fragments from multiple cobs, cupule width measurements were used to correlate them with Cutler and Blake's inventory (Table 1). Images and data for the sampled specimens are presented in Supplement 1.

Small samples of 18 cobs and cob fragments were taken under low magnification with a solvent-cleaned scalpel or razor blade. Samples from fragments of different cobs that had been assigned the same Meadowcroft catalog number were given sample numbers to distinguish them in Tables 1 and 2 and Supplement 1. Any adhering sediment was scraped off the area sampled prior to cutting. The samples were weighed, wrapped in aluminum foil, placed in labeled plastic bags, and shipped to the W. M. Keck Carbon Cycle Accelerator Mass Spectrometry Laboratory (KCCAMS) at the University of California-Irvine for isotope-ratio measurement and AMS dating. At KCCAMS, all samples were subjected to the standard acid-base-acid (1N HCl and 1N NaOH, 75°C) pretreatment. Details on sample pretreatment, combustion, graphite reduction, and AMS analysis are available on the KCCAMS website (https://sites.uci.edu/keckams/facilities/). Corrections for isotopic fractionation were performed with δ^{13}C values obtained on prepared graphite using the AMS spectrometer. A Thermo Finnigan Delta Plus stable isotope-ratio mass spectrometer (IRMS) with Gas Bench input was used at KCCAMS to measure δ^{13}C values to a precision of <0.1‰ relative to standards traceable to PDB. The ^{14}C ages were

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Table 2 Meadowcroft maize cob samples AMS dating results. Asterisks in the $\delta^{13}C$ (‰) column indicate the samples were too small to provide additional material for IRMS measurement.

Catalog (sample) no.	UCIAMS no.	$\delta^{13}C$ (‰)	^{14}C age (BP)	Cal. 95.4% (AD)
FS-811.3 (1)	248506	−10.6	100±15	1694–1726 (26.7) 1811–1917 (68.7)
FS-811.3 (2)	248507	−10.5	110±15	1693–1727 (24.4), 1810–1919 (71.0)
FS-790.5 (1.1)	248508	−10.4	115±15	1691–1728 (23.4), 1809–1921 (72.0)
FS-790.5 (1.2)	248509	−8.9	120±15	1687–1730 (23.1), 1806–1925 (72.4)
FS-790.5 (2.1)	248510	−8.4	75±15	1696–1725 (30.1), 1811–1839 (29.1), 1877–1915 (36.3)
FS-790.5 (2.2)	248511	−10.8	125±20	1683–1736 (24.6), 1802–1936 (70.8)
FS-790.5 (3.1)	248512	−9.4	90±15	1695–1725 (28.9). 1811–1862 (31.5), 1868–1917 (35.0)
FS-790.3	248513	−9.4	115±15	1691–1728 (23.4), 1809-1921 (72.0)
FS-183.3	248514	−8.4	110±15	1693–1727 (24.4), 1810–1919 (71.0)
FS-1811.1	Sample too small to analyze			
FS-269.8 (1)	248515	−8.9	115±15	1691–1728 (23.4), 1809–1921 (72.0)
FS-269.8 (2)	248516	*	115±15	1691–1728 (23.4), 1809–1921 (72.0)
FS-750	248517	−9.8	120±15	1687–1730 (23.1), 1806–1925 (72.4)
FS-130.10 (1)	248518	*	200±15	1656–1884 (26.8), 1736–1804 (61.3), 1930–. . . (7.3)
FS-130.10 (2)	248519	−9.1	105±15	1694–1726 (25.5), 1811–1918 (70.0)
FS-160.12	248520	−9.2	135±20	1677–1743 (25.8), 1751–1765 (3.7), 1799–1942 (66.0)
FS-130.26 (1)	248521	−9.5	75±15	1695–1725 (28.7), 1811–1862 (31.4), 1867–1917 (35.4)
FS-130.26 (2)	248522	−9.6	85±15	1695–1725 (29.7), 1811–1855 (30.4), 1869–1871 (0.4), 1876–1917 (35.0)

calibrated in OxCal v. 4.4.4 (Bronk Ramsey 2009) using the IntCal20 Northern Hemisphere terrestrial ^{14}C calibration curve (Reimer et al. 2020).

RESULTS

AMS ^{14}C ages and calibrated dates and δ^{13}C values are presented in Table 2. The sample from specimen FS-1811.1 did not yield enough carbon after pretreatment for analysis, and samples FS-269.8 (2) and FS-130.10 (1) were too small to provide enough material for IRMS measurement. Given the results of the remaining samples, providing additional material of these specimens for assay was unwarranted. While there is no doubt based on Cutler and Blake's analysis and their physical appearance that the specimens are maize cobs/cob fragments (Supplement 1), the δ^{13}C values ranging from –8.4 to –10.8 confirm their identifications as maize (Table 2).

All dates are historical and remarkably consistent given that the samples were recovered from four separate strata. There is no record and no visual evidence of the maize remains being treated with consolidants or adhesives. The application of most of the commonly used consolidants and adhesives would result in older, not younger, ages than anticipated (Crann and Grant 2019). Three of the organic consolidants and glues analyzed by Crann and Grant (rabbit skin glue, technical gelatine, and wheat starch) produced modern ages.

The technical gelatine analyzed by Crann and Grant was manufactured in 1980, close in time to the Cutler and Blake's analysis. Using a fraction modern carbon (FMC) value for Meadowcroft specimen FS-811.3 (1) of 0.9875, the FMC of 0.752 for an expected ^{14}C age of 2290 BP, and the FMC value for technical gelatine of 1.103 with the mass balance equation (0.9875–0.752)/(1.1013–0.752)*100 indicates that 67.42% of the ^{14}C in the specimen would need to have been contributed by the technical gelatine to result in an offset of 2190 ^{14}C years. This suggests a heavy application that would be visible and prevent the smears of carbon that occurred when handling of the cob fragment for sampling. All consolidants and adhesives tested by Cran and Grant (2019:1062) have δ^{13}C values more negative than archaeological maize in the Northeast ranging from –36.0 to –15.8‰ (median = –27.5). That the Meadowcroft maize δ^{13}C values are well within the range for archaeological maize in the Northeast also suggests the absence of treatment with consolidants or adhesives. Furthermore, if any of the organic materials analyzed by Crann and Grant had been applied to the Meadowcroft maize specimens, they would have been removed by the base step of the standard acid-base-acid treatment applied to the samples at KCCAMS and have no effect on the analytical results.

The AMS dates, disprove an early presence for maize at the Meadowcroft Rockshelter and go beyond Cutler's and Blake's (1977) suggestion that three of the specimens date after AD 1800. The calibrated dates are multimodal; the largest probabilities fall within the nineteenth to early twentieth centuries, with probabilities generally <30% falling in the late seventeenth to early eighteenth centuries. When modeled as an OxCal uniform Phase, the 95.4% Date estimate is *1717–1743* (9.3%), *1828–1960* (86.2%). The OxCal runfile for the model is provided in Supplement 2 and full results of the model are presented in Supplement 3. Clearly originating from historical use of the site, the maize remains have no relationship to the long history of Native American occupations of Meadowcroft. Their presence in strata associated with occupations dating as early as the 4th century BC is evidently the result of bioturbation or other disturbance. It should be noted that extreme care was taken during

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Table 3 Examples of potentially early maize macrobotanical remains in the Northeast based on radiocarbon dates on wood charcoal.

Archaeological site	Location	Associated ^{14}C age (BP)	Cal. 95.4% range	Source
Shohola Flats	Delaware River valley, northeastern Pennsylvania	3150±70 to 2810±150	1600–1590 BC (0.05) 1543–1222 BC (94.9) 1421–750 BC (93.6) 685–667 BC (0.5) 636–588 BC (1.2) 579–572 BC (0.1)	Stewart (2021)
Thorp	Upper Ohio River valley, southwestern Pennsylvania	1900±60	39–11 BC (2.8) AD 2–250 (91.8) AD 296–309 (1.0)	McConaughy (2008)
Deposit Airport I	Upper Delaware River valley, southeastern New York	1850±40	AD 81–99 (2.7) AD 110–255 (86.5) 286–324 (6.2)	Stewart (2021)
Childers	Upper Ohio River valley, West Virginia	1610±90	AD 252–291 (5.4) AD 319–640 (90.1)	Wymer (1992)

the multiyear excavations at Meadowcroft Rockshelter to note the presence of bioturbation or other disturbances; however, it is apparent that disturbances in the area which produced the maize remains were missed. It should also be stressed that the same strata which yielded the maize remains evidenced no disturbance elsewhere on the site.

DISCUSSION AND CONCLUSIONS

The timings of the adoption of maize are ongoing research topics throughout the Western Hemisphere. In the northeastern North America, two lines of evidence have been used to determine when maize becomes archaeologically visible: microbotanical remains recovered from directly dated food residues adhering to pottery and directly dated macrobotanical remains. Until recently these two lines of evidence were generally in agreement for the region as a whole with early directly dated microbotanical evidence in the eastern Great Lakes and St. Lawrence River Valley and early directly dated macrobotanical evidence from the riverine interior. The early evidence from the riverine interior was recently discredited, leaving the earliest directly dated macrobotanical evidence from the Great Lakes region in southern Ontario, some 800 years later than the earliest directly dated microbotanical evidence. The Meadowcroft maize had the potential to bridge that gap, but it joins a growing list of macrobotanical remains once thought to represent early use of maize in the Northeast that have been shown to date much later in time or to have been misidentified as maize (e.g., Murphy 1989: 348; Conard et al. 1984; Simon 2014, 2017; Simon et al. 2021).

At the time the specimens were recovered, the extent of potential biological disturbance was underestimated. Apparently, the maize specimens were transported downward from a higher level and the extent of the bioturbation was not perceived by the excavators. There were no reasons at the time of the Meadowcroft maize recovery to doubt the maize remains' stratigraphic sequence, association with ^{14}C dates, or Cutler and Blake's assignments of the majority of cobs to their prehistoric morphotypes. The Meadowcroft results further emphasize the need for AMS dates and $\delta^{13}C$ IRMS measures on purported maize macrobotanical remains recovered from contexts in the Northeast that are potentially earlier than Grand Banks such as those listed in Table 3.

The gap between the earliest direct dates on maize micro-and macrobotanical remains in the Northeast continues. This situation is not untypical; maize microbotanical remains pre-date macrobotanical remains in several areas of the Americas where environmental conditions do not favor macrobotanical preservation (e.g., Pohl et al. 2007; Lombardo et al. 2020). Ultimately the two lines of evidence need to be reconciled as suggested by Dotzel (2021). This will require additional laboratory and actualistic experimentation to determine under what conditions and contexts maize micro- (Crowther 2012; Raviele 2011) and macrobotanical (e.g., King 1987; Dezendorf 2013; Whyte 2019) remains preserve in the archaeological record.

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SUPPLEMENTARY MATERIAL

To view supplementary material for this article, please visit https://doi.org/10.1017/RDC.2022.18

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