

Radiocarbon

1976

ANTWERP UNIVERSITY RADIOCARBON DATES I

R VANHOORNE and W VAN DONGEN

Department of General Botany, State University Centre, Antwerp,
Groenenborgerlaan 171, B-2020 Antwerp, Belgium

INTRODUCTION

The radiocarbon dating laboratory was set up during 1972 and 1973 with financial support of the FKFO (Fonds voor Kollektief Fundamenteel Onderzoek) Belgium, to enable us to place Upper Pleistocene and Holocene sediments in their correct stratigraphic position. Dating of samples began in the middle of 1974.

Our work is concerned with measuring ^{14}C activity in the form of benzene, with a liquid scintillation counter (ICN Tracerlab Manual Coruflo) which is specially protected against cosmic rays by lead casing. 3ml of benzene is introduced into the counting vial (low potassium glass) plus 1ml scintillation mixture of PPO (10g/l) and POPOP (0.1g/l) in toluene.

The sample counting time varies between 1900 and 3000 minutes, with print-outs at 100 minute intervals. Before and after sample counting, a background sample is counted (3ml of spectroscopic benzene and 1ml scintillation mixture). A typical background value is 2.149 cpm/gC benzene. Each week the counting efficiency of the apparatus is tested, using a ^{14}C hot standard. The modern standard used is oxalic acid, prepared by wet oxidation. Its 0.95 activity is 8.298 cpm/gC. $\delta^{13}\text{C}$ values have not been measured. In the near future we intend to use the ANU-Sucrose radiocarbon standard as working standard, where fractionation effects are negligible (Polach & Krueger, 1972; H-121). Its activity is 13.385 cpm/gC ($\pm 160\%$ modern).

Dates are reported in years BP, rounded off to the nearest 10 years, and are based on a half-life of 5568 ± 30 years. One standard deviation includes error of measurement (Poisson-error) and error of chemistry as defined by Polach (1972, H-92). These are calculated by a test of reproducibility. Age determinations numbered 1-118 have a standard deviation in which both errors of measurement and chemistry are included. Dates from No. 119 on only include the error of measurement. When samples are diluted, the percentage original benzene is stated.

Samples are pretreated by successively boiling in NaOH N/10 and HCl 2N and then washed and dried. Oxidation of the samples is carried out in a combustion bomb, according to the technique described by Barker, Burleigh & Meeks (1969) and Burleigh (1972, B1)(efficiency: $\pm 95\%$).

The conversion to acetylene takes place in a lithium-reactor (efficiency: $\pm 90\%$) and trimerization into benzene is activated by a catalyst (KC-Perlkator, Kalichemie, Hannover) as described by Scharpenseel and Pietig (1969) and Kölle (1972). Using this catalyst we obtain yields of ca 85% (wrt C_2H_2).

To check our results, we synthesized 3ml of benzene from the same stump of fossil wood 10 times. By counting the activity of each of the samples and comparing these with the modern standard we obtained the following age determinations, shown in Table 1.

TABLE 1
Age determinations of 10 samples from the same stump of fossil wood

Activity cpm/gC sample	Date: year BP	$x-\bar{x}$	$(x-\bar{x})^2$
3.242 \pm 0.038	7551 \pm 102	171	29,241
3.226 \pm 0.034	7591 \pm 82	211	44,521
3.446 \pm 0.039	7062 \pm 78	318	101,124
3.340 \pm 0.039	7310 \pm 100	70	4900
3.252 \pm 0.040	7527 \pm 103	147	21,609
3.300 \pm 0.039	7411 \pm 101	31	961
3.348 \pm 0.039	7293 \pm 80	87	7569
3.379 \pm 0.039	7217 \pm 80	163	26,569
3.304 \pm 0.041	7400 \pm 122	20	400
3.286 \pm 0.039	7443 \pm 82	63	3969

$$\text{Mean value: } \bar{x} = \frac{\sum x}{n} = 7380 \text{ yr} \quad \Sigma(x-\bar{x})^2 = 240,863$$

Activity cpm/gC modern standard = 8.298 \pm 0.040

$$\text{one standard deviation: } s = \frac{\Sigma(x-\bar{x})^2}{n-1} = 164 \text{ yr}$$

$$\begin{aligned} \text{error}_{\text{measured}}^2 &= \text{error}_{\text{chemistry}}^2 + \text{error}_{\text{counting}}^2 \\ \text{error}_{\text{chemistry}} &= \sqrt{\text{error}_{\text{measured}}^2 - \text{error}_{\text{counting}}^2} = \\ &= \sqrt{164^2 - 100^2} = 129 \text{ yr} \end{aligned}$$

The standard deviation on an individual measurement calculated from the 10 determinations given in Table 1 yielded a value of 164 yr, well above the average standard deviation of ± 100 yr due to counting statistics alone as mentioned above. This discrepancy can only be explained if a not unimportant error of chemistry contributes to the total error of the date. This contribution was calculated to be 129 yr. After repeating our procedure, we found an error of pipetting (when filling a counting vial) of this order of magnitude.

We then weighed the quantity of benzene in the counting vial, so that errors of this kind may reasonably be ignored. In our later results

we take only the counting error into consideration. Table 2 compares dates from our laboratory and those obtained in other laboratories on the same sample.

TABLE 2
Cross check samples

Lab no.	Date: year BP	Check sample	Date: year BP
Antw-101	1400 ± 180	SRR-125	1361 ± 55
		GaK-3702	1390 ± 100
Antw-100	2120 ± 120	GrN-7036	1860 ± 45
Antw-109	2370 ± 140	IRPA-139	2140 ± 93
Antw-98	3880 ± 110	SRR-6	3940 ± 80
		BM-331	3770 ± 85
		UB-66	3970 ± 70
		A-569	4290 ± 90
		NPL-5	4310 ± 90
Antw-104	4390 ± 140	UCLA-739	4280 ± 80
		LJ-1488	4370 ± 50
		BM-248	4160 ± 110
		BM-203	4150 ± 110
		GrN-5884	5140 ± 45
Antw-97	5560 ± 140		
	5150 ± 140		
Antw-94	11,350 ± 180	GrN-6045	11,950 ± 65
Antw-1	12,060 ± 160	GrN-6073	12,010 ± 65
Antw-96	30,000 ± 3320	GrN-6046	30,180 ± 270

ACKNOWLEDGMENTS

We wish to thank W G Mook of the Natuurkundig Lab, Rijksuniversiteit, Groningen, Netherlands for sending us check samples and his comment on our results. Also the Scottish Universities Research and Reactor Centre, Glasgow, Scotland, is acknowledged for its check samples. We also thank the staff of the Research Laboratory of the British Museum, London, for their generosity and valuable information and our colleague K Van Camp for his precious help in statistics.

SAMPLE DESCRIPTIONS

GEOLOGIC SAMPLES

A. Belgium

ANTW-95. Zedelgem 9, Snellegem

12,070 ± 198

10,120 BC

Accumulation of stalks at 330cm below surface in sand intercalated between 2 peat beds (51° 09' 53" N, 3° 06' 37" E). Coll Aug 1974 by R Vanhoorne. *Comment*: date is consistent with late glacial age, inferred from pollenologic analysis.

Sijsele series

Wood and peat at base of Maldegem layer (51° 12' 33" N, 3° 20' 25" E). Coll 1974 by R Vanhoorne and C Verbruggen.

ANTW-83. Moerkerke 17 **11,560 ± 170**
9610 BC
Peat, 150 to 160cm below surface overlain by sand.

ANTW-127. Flo-Sij-13264/L2 **11,490 ± 180**
9540 BC
Wood in peat layer at 240cm below surface.

General Comment: palynologic study shows late glacial vegetation with *Betula* dominant. Date fits well with scheme proposed by R Vanhoorne and C Verbruggen (1975, in press). Dilution: 77% sample.

Gistel 26 series, Roksem, Belgium

(51° 10' 19" N, 3° 02' 27" E). Coll Aug 1974 by R Vanhoorne.

ANTW-92. Gistel 26, 280-285 **12,120 ± 140**
10,170 BC
Wood from peat layer at 280 to 285cm below surface. *Comment:* date corresponds with late glacial age of peat indicated by palynologic study.

ANTW-146. Gistel 26, 273-275 **10,800 ± 140**
8850 BC
Mosses washed from peat layer at 273 to 275cm below surface. *Comment:* date confirms late glacial age, revealed by pollen spectrum at 273 to 275cm. *Betula:* 23.7%; *Pinus:* 0.5%; *Salix:* 2.5%; *Hippophae:* 1.5%; herbs: 71.8%.

ANTW-153. Gistel 26, 401-405 **11,950 ± 199**
10,000 BC
Sample at 401 to 405cm below surface of peat layer 10cm thick. *Comment:* sample diluted 75%. Date supports late glacial age, indicated by pollen spectrum at 405cm. *Betula:* 27%; *Pinus:* 1.2%; *Salix:* 7%; *Juniperus:* 0.6%; herbs: 64.2%.

General Comment: little difference between pollen spectra of the 2 peat layers. Arboreal pollen is highest at 280 to 281cm. No detailed subdivision of late glacial at Gistel can be made on palynologic grounds.

ANTW-93. Houtave 3, Jabbeke **11,430 ± 170**
9480 BC
Wood from peat layer at 320 to 325cm below surface (51° 10' 46" N, 3° 06' 32" E). Coll Aug 1974 by R Vanhoorne. *Comment:* date is consistent with late glacial age indicated by stratigraphic position.

Houtave 4 series, Jabbeke

(51° 11' 19" N, 3° 06' 50" E). Coll Feb 1974 by R Vanhoorne.

ANTW-133. Houtave 4, 130-135 **10,480 ± 140**
8530 BC

Sample from 130 to 135cm below surface from upper part of peat bed 10cm thick. *Comment*: date agrees with late glacial age, indicated by pollen diagram: *Betula* increasing from 18.5% to 77.3% from bottom to top, percentages of *Salix* and *Pinus* remaining low without noticeable change. Percentages of non arboreal pollen change from 21.6 to 74.1%.

ANTW-138. Houtave 4, 135-140 **10,370 ± 140**
8420 BC

Sample from 135 to 140cm below surface from lower part of peat bed 10cm thick. *Comment*: date supports a late glacial age, revealed by pollen diagram, where sum of arboreal pollen is less than 17% of total pollen. *Betula* dominates *Salix* and *Pinus*.

ANTW-132. Houtave 4, tree roots **3560 ± 114**
1610 BC

Tree roots from sand interbedded between 2 peat layers. *Comment*: dilution: 57% sample. Remnants of Sub-Boreal wood, penetrating through upper peat layer.

ANTW-137. Houtave 4, 270-275 **11,400 ± 140**
9450 BC

Sample from 270 to 275cm below surface from upper part of peat layer 10cm thick. *Comment*: date is consistent with late glacial age, indicated by pollen diagram. Percentages of arboreal pollen vary between 32 and 8%. *Betula* dominates *Salix* and *Alnus*.

ANTW-128. Houtave 4, 275-280 **11,380 ± 180**
9430 BC

Sample from 275 to 280cm below surface from lower part of peat layer 10cm thick. *Comment*: dilution: 83% sample. Date agrees with late glacial age of peat indicated by pollen diagram. Percentages of arboreal pollen vary between 28 and 35%. *Alnus* dominates *Betula*, *Pinus* and *Salix*.

General Comment: dates of upper and lower parts of each peat bed are very similar. The arboreal pollen belongs mainly to *Betula*. The percentages of *Pinus*, *Salix* and *Alnus* are low except in ANTW-128.

Stekene series

Peat from Maldegem layer (51° 13' 05" N, 4° 00' 40" E). Coll 1974 by C Verbruggen.

ANTW-131. Zeg-Ni-144-1/L10 **10,510 ± 160**
8560 BC

Sample from 210cm below surface. *Comment* (CV): palynologic study indicates late glacial vegetation with *Betula*, *Salix*, and 5% *Pinus*. Based on our present knowledge of evolution of late glacial vegetation in N part of sandy Flanders, this date should be ca 1000 yr too young. Dilution: 83% sample.

- 10,860 ± 140**
8910 BC
- ANTW-134. Zeg-Ni-144-1/L8**
Sample from 300cm below surface. *Comment* (CV): palynologic study reveals open vegetation with *Salix* and *Betula*. Stratigraphic position and vegetation point to early late glacial period, probably 1000 to 1500 yr older than radiocarbon date.
- Leffinge series**
Wood from base of Holland peat (51° 08' 40" N, 2° 52' 13" E). Coll 1974 by C Verbruggen.
- 4630 ± 140**
2680 BC
- ANTW-102. Leffinge, Sample 1**
Wood from alder stump, 180cm below surface.
- 5190 ± 140**
3240 BC
- ANTW-105. Leffinge, Sample 2**
Wood from bog oak trunk at base of Holland peat. Sample from 200cm below surface.
General Comment (CV): pollen analysis confirms Atlantic age. The 2 dates, ANTW-105 and -102, fit perfectly as former indicates end of Atlantic mixed oak forest and the latter, initiation of peat growth.
- Steenkerke series**
Samples from peat layer 250 to 350cm and 550 to 600cm below surface (51° 04' 12" N, 2° 41' 34" E). Coll 1974 by R Paepe and C Baeteman.
- 4150 ± 115**
2200 BC
- ANTW-123. Boring 407, Brugge-Calais Hwy, 300**
Peat from 300cm below surface. *Comment*: date agrees well with stratigraphic position of peat, which corresponds to Holland peat.
- 5830 ± 115**
3880 BC
- ANTW-136. Boring 407, Brugge-Calais Hwy, 600**
Peat from 600cm below surface. *Comment*: date corresponds with stratigraphic position of peat, included in Calais layer.
- >37,430**
>35,480 BC
- ANTW-139. Handboring 27, Leie canal, Eeklo**
Peat from layer at depth 610cm (51° 11' 37" N, 3° 31' 24" E). *Comment*: date agrees with age, indicated by stratigraphic position of peat.
- 1360 ± 120**
AD 590
- ANTW-112. Wamp 2, Arendonk**
Wood in peat layer, at least 1m deep. Sample from 90 to 100cm below surface (5° 01' 03" E, 51° 16' 30" N). Coll 1975 by L Beyens.

- ANTW-113. Ipenrooi 6** **370 ± 160**
AD 1580
Sphagnum peat from 60 to 70cm below surface. Peat layer was 110cm thick (51° 29' 10" N, 4° 45' 04" E). Coll 1975 by L Beyens. *Comment* (LB): rapid growth of peat is supposedly ca 1.6mm/yr.
- ANTW-115. Wortel 2** **1910 ± 140**
AD 40
 Peat from layer 6m thick. Sample from 50 to 60cm below surface (51° 23' 52" N, 4° 47' 41" E). Coll 1975 by L Beyens. *Comment* (LB): date agrees well with sequence pub by IRPA (R, 1975, v 17, p 1).
- ANTW-116. Kasterlee 5** **7490 ± 200**
5540 BC
 Peat from layer 100cm thick. Sample from 50 to 60cm below surface. Layer rested on glauconitic tertiary sands (51° 40' 58" N, 4° 55' 41" E). Coll 1974 by L Beyens.
- Lille 3 series**
 Wood and peat from peat layer 100cm thick (51° 13' 57" N, 4° 52' 30" E). Coll 1975 by L Beyens.
- ANTW-119. Lille 3, wood** **6520 ± 110**
4570 BC
 Wood from 75 to 80cm below surface. Dilution: 83% sample.
- ANTW-120. Lille 3, peat** **9360 ± 250**
7410 BC
 Peat from 75 to 80cm below surface. Dilution: 42% sample.
- ANTW-122. Lille 4** **6020 ± 140**
4070 BC
 Peat from layer 90cm thick. Sample from 70 to 90cm below surface (51° 11' 34" N, 4° 52' 30" E). Coll 1975 by L Beyens. *Comment*: dilution: 66% sample.
- ANTW-125. Wuustwezel 1** **4040 ± 140**
2090 BC
 Peat from layer 100cm thick. Sample from 40 to 60cm below surface (51° 24' 34" N, 4° 38' 04" E). Coll 1975 by L Beyens. *Comment*: dilution: 43% sample.
- ANTW-129. Wuustwezel 2** **4560 ± 315**
2610 BC
 Peat from layer 40 to 120cm below surface. Sample from 80 to 100cm (51° 23' 31" N, 4° 37' 00" E). Coll 1975 by L Beyens.
- Paal 1 series**
 Peat from layer 0 to 100cm below surface. Samples taken every 10cm between 25 and 95cm (51° 03' 20" N, 5° 09' 54" E). Coll 1975 by L Beyens.

- ANTW-140. Paal 1, 85-95** **10,250 ± 180**
8300 BC
 Sample from 85 to 95cm below surface. *Comment:* dilution: 60%
 sample.
- ANTW-141. Paal 1, 75-85** **10,980 ± 160**
9030 BC
 Sample from 75 to 85cm below surface. *Comment:* dilution: 85%
 sample.
- ANTW-142. Paal 1, 65-75** **8750 ± 140**
6800 BC
 Sample from 65 to 75 cm below surface. *Comment:* dilution: 83%
 sample.
- ANTW-151. Paal 1, 55-65** **9640 ± 120**
7690 BC
 Sample from 55 to 65cm below surface.
- ANTW-149. Paal 1, 45-55** **7870 ± 120**
5920 BC
 Sample from 45 to 55cm below surface.
- ANTW-150. Paal 1, 35-45** **4690 ± 120**
2740 BC
 Sample from 35 to 45cm below surface. *Comment:* dilution: 83%
 sample.
- ANTW-143. Paal 1, 25-35** **2130 ± 140**
180 BC
 Sample from 25 to 35cm below surface. *Comment:* dilution: 47%
 sample.

B. Scotland

- ANTW-91. Cairngorm Estate, Sample 1** **7380 ± 150**
5430 BC
 Wood from a mature pine stump recovered from eroded blanket bog 120cm thick; alt, + 640m (57° 08' N, 3° 40' W). Coll 1974 by D K Ferguson and M De Keersmaecker. *Comment* (DKF): although found well above present tree limit, 450 to 500m on N-facing slopes, growth appears to have been fairly dense, up to 10 trees with stem diams varying from 10 to 35cm/200m².
- ANTW-107. Cairngorm Estate, Sample 2** **4170 ± 140**
2220 BC
 Wood from mature pine stump embedded in peat layer 160cm thick at depth 93 to 105cm below surface; alt, + 530m (57° 09' N, 3° 39' W). Coll 1974 by D K & H H Ferguson. *Comment* (D & H F): represents period of relative dryness which permitted recolonization of peat bog. Pine tree was at least 140 yr old when it died.

C. France

Fère-en-Tardenois 12 series

Peat layer (49° 13' N, 3° 31' E). Coll 1974 by R Vanhoorne.

ANTW-126. Fère 12, 35-40 **5430 ± 115**
3480 BC

Sample from 35 to 40cm below surface from peat layer 140cm thick. *Comment:* ¹⁴C date indicates end of Atlantic period. However, pollen assemblage, containing no elm and only little oak and lime, is characteristic of Sub-Boreal period. Considering beginning and end of Sub-Boreal, presumed pollen age is 500 to 3000 yr older than radiometric date.

ANTW-121. Fère 12, 60-65 **8940 ± 140**
6990 BC

Sample from 60 to 65cm below surface from peat layer 140cm thick. *Comment:* pollen stratigraphy reveals perhaps the Piottino oscillation, which, according to Zoller (1960), extended from 10,050 to 9650. The ¹⁴C dating is about 1000 yr younger.

ANTW-117. Fère 12, 80-85 **9580 ± 160**
7630 BC

Sample from 80 to 85cm below surface from peat layer 140cm thick. *Comment:* pollenflora can indicate Pre-Boreal age.

ANTW-114. Fère 12, 110-115 **10,900 ± 330**
8950 BC

Sample from 110 to 115cm below surface from peat layer 140cm thick. *Comment:* ¹⁴C age, pointing to Late Dryas, can fit with the palynoflora.

ANTW-106. Fère 12, 120-140 **11,630 ± 180**
9680 BC

Sample from 120 to 140cm below surface from peat layer 140cm thick. *Comment:* no pollen spectrum is available.

Fère-en-Tardenois 1 series

Peat layer (49° 13' N, 3° 31' E). Coll Nov 1974 by R Vanhoorne. This place, lying in the same valley as the previous site, is ca 50m from Fère 12.

ANTW-108. Fère 1, Sample 1 **1910 ± 110**
AD 40

Sample from 90 to 100cm below surface from peat layer 100cm thick. *Comment:* pollen spectrum, with small percentages of beech, suggests onset of Sub-Atlantic period, considered to have begun 800 to 400 yr earlier than ¹⁴C date.

ANTW-135. Fère 1, Sample 2 **1210 ± 110**
AD 740

Sample from 45 to 60cm below surface from peat layer 100cm. *Comment:* pollen assemblage contains beech and hornbeam in only small quantities.

REFERENCES

- Barker, Harold, Burleigh, Richard, and Meeks, Nigel, 1969, New method for the combustion of samples for radiocarbon dating: *Nature*, v 221, no. 5175, p 49-50.
- Burleigh, Richard, 1972, Bomb combustion of radiocarbon samples: 8th internatl conf on radiocarbon dating, *Proc, New Zealand*, v 1, sess B, p B1-B10.
- Kölle, W, 1972, Die Benzolsynthese als Präparationschritt in der Flüssigkeits-Szintillationspektrometrie: *GIT Fachzeitschrift für das Laboratorium*, Heft 12, Jg 16, p 1411-1419, Dec 1972.
- Mook, W G, 1974, Absoluut dateren met ^{14}C : inleiding ^{14}C symposium, Groningen, p 1-27, Jan 1974.
- Polach, H, 1972, Cross checking of NBS-oxalic acid and secondary laboratory radiocarbon dating standards: 8th internatl conf on radiocarbon dating, *Proc, New Zealand*, v 2, sess H, p H92-H120.
- Polach, H and Krueger, H A, 1972, Isotopic fractionation of NBS oxalic acid and ANU-Sucrose radiocarbon dating standards: 8th internatl conf on radiocarbon dating, *Proc, New Zealand*, v 2, sess H, p H121-H128.
- Scharpenseel, H W and Pietig, F, 1969, Einfache Boden und Wasserdatierung durch Messung der ^{14}C oder Tritium-konzentration: *Geoderma*, v 2, no. 4, p 273-289.
- Vanhoorne, R and Verbruggen, C, 1975, Problèmes de subdivision du Tardiglaciaire dans la région sablonneuse du Nord de la Flandre en Belgique: *Pollen et spores* (in press).
- Zoller, H, 1960, Pollenanalytische Untersuchungen zur Vegetationsgeschichte der in-subrischen Schweiz: *Denkschr Schweiz Naturf Gesell*, B LXXXIII, Abh 2, p 80.