WILDERVANK-ZANDPLAS III: AN INVESTIGATION USING NATURAL ISOTOPES

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ABSTRACT: A depression near Wildervank (province of Groningen, the Netherlands) has been investigated using natural isotopes. In particular, the gyttja section has been dated in great detail. Reservoir corrections were determined, based on $^{13}C/^{14}C/^{18}O$ contents of modern material from nearby sites.

KEYWORDS: Natural isotopes, gyttja, siderite, dating, Allerød, Younger Dryas.

1. INTRODUCTION

The elongated depression Zandplas III, west of Wildervank in the province of Groningen, filled with gyttja, peat, sand and loam has been described by Cleveringa et al. (1994) and Groenendijk (1997). The lithological scheme, divided in units, is shown in figure 1. Palynological investigations suggested that unit 6 has been formed during the Allerød, unit 4 during Preboreal and Boreal, and units 1 and 2 either during the Younger Dryas or around the transition Bølling/Allerød. The most likely date for the lenses of calcareous gyttja (unit 3) seemed to be Younger Dryas.

Geologically, the depression is likely to be part of a valley system. The eolic sand in the subsoil of the depression had not been displaced by running water, however. Based on both pollen analysis and lithology, Cleveringa et al. (1994) concluded that the depression had been filled with undeep stagnating water sometime during the Late Glacial, as indicated a.o. by seeds and pollen of bog bean (*Menyanthes trifoliata* L.). Apparently a waterflow from a system under pressure welled up in the depression. Low temperatures during the Younger Dryas would have caused calciumcarbonate from this water to precipitate.

The deposit has recently been investigated using the natural isotopes ^{13}C, ^{14}C and ^{18}O. The radio-active isotope ^{14}C was used for dating, the stable isotopes ^{13}C and ^{18}O were used for traces purposes, giving information on the origin of the carbon.

2. ^{14}C-DATING THE DEPOSIT

Radiocarbon dates were obtained from peat, gyttja carbonate and seeds. The results are shown in table 1. All ^{14}C dates are reported in BP, which means this result includes a correction for isotopic fractionation to a standard value of $^{13}\delta = -25‰$ (Mook & Streurman, 1983). This is also the case for the reported carbonate measurements. This means that if there is a reservoir age, this still has to be subtracted from the reported radiocarbon age (Mook, 2000).

The peat layer from depths 96–149 cm (*Hypnaceae* peat, unit 4, see fig. 1) yields a consistent set of ^{14}C dates, all around 11,000 BP. This peat has been dated using standard ^{14}C procedures, including physical and chemical pre-treatment. From the 'calcareous gyttja' between depths of 150–158 cm (unit 3), organic matter was collected for dating. By further analysis (discussed below), we later determined that this 'calcareous gyttja' is mainly Fe-carbonate (siderite). For this part, the samples were split in two fractions by sieving according to size: a coarse fraction (>180 μm) and a fine fraction (<180 μm). The measured ^{14}C dates fluctuate as a function of depth, the youngest being 9160±80 BP (GrN-26730) and the oldest 11,180±40 BP (GrN-24304). The organic material is apparently not homogeneous, as is corroborated by additional measurements discussed below. In general, the fine fraction is much younger; this fraction probably represents material, intruded from younger sediments above the gyttja. Below the 'calcareous gyttja', the ^{14}C dates become more stable, a little older than 11,000 BP with identical (within error) dates for the coarse — and fine — (GrN-24111 and 24305, resp.) fraction.

The measured ^{14}C results are also shown in figure 2. In figure 2A, the ^{14}C results for all fractions measured are plotted as a function of depth. The error bars plotted correspond to 1σ. Figure 2B shows the $^{13}\delta$ value of the organic material dated (coarse fraction only, i.e. >180 μm size) and the carbonate content as a function of depth. It is clear that not all results can be correct. Given the dated material and the pretreatment the *Hypnaceae* peat of unit 4 must have been formed during the later Allerød or around the transition Allerød-Younger Dryas. And
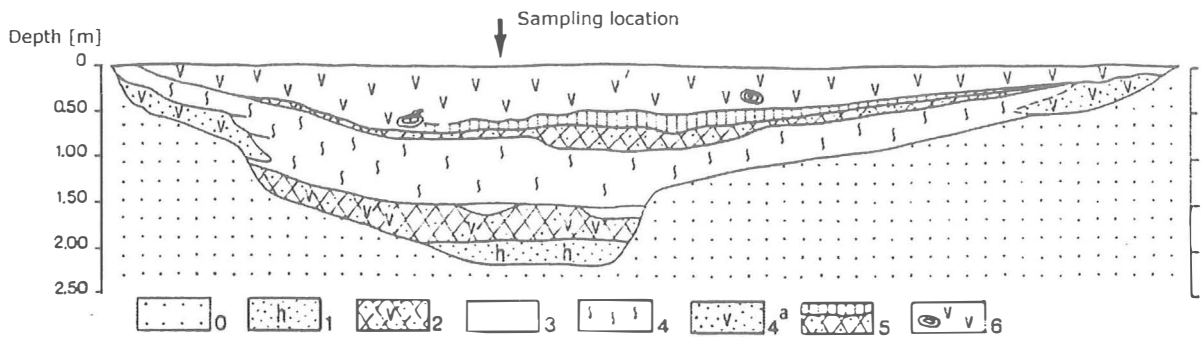
Fig. 1. Lithology for depression Zandplas III near Wildervank, the Netherlands. Lithological units are numbered as follows: 0. Eolic sand; 1. Sand with humus; 2. Sand gyttja with peat; 3. 'Calcareous gyttja' (in fact organic layer containing siderite, see text); 4. *Hypnaceae* peat; 4a. Sandy peat; 5. Sandy gyttja with loam at the top; 6. Peat.

Table 1. Wildervank-Zandplas III. Radiocarbon measurements performed in Groningen. GrN = conventional; GrA = AMS.

Depth (cm NAP)	Unit (fig. 1)	Lab No.	Material	Fraction*	Measured ^{14}C age (BP)	Error (1ó)	$^{13}\delta$ (‰)	%C**	Ash (%)	Adjusted ^{14}C age
Hypnaceae *peat (unit 4)*										
96–97	4	GrN-24110	peat		10710	110	-28.12	46	9	
119–120	4	GrN-24301	peat		10920	80	-29.32	49	8	
134–135	4	GrN-23934	peat		11260	60	-29.32	44	23	
134–135	4	GrN-24007	peat	humics	10830	80	-28.68			
148–149	4	GrN-24914	peat		11010	110	-31.07	56	0	
149–150	4	GrN-23921	peat		9850	60	-31.44	53	4	
149–150	4	GrN-24008	peat	humics	9740	60	-30.78			
'Calcareous gyttja' (unit 3)										
150–151	3	GrA-17319	seeds		11395	45	-24.78			11395
150–151	3	GrN-24307	organics	>180 µm	10830	230	-32.52	35	29	9920
151–153	3	GrN-24303	organics	>180 µm	10220	60	-30.84	18	58	9450
151–153	3	GrN-26730	organics	<180 µm	9160	80	-29.45	21	42	8510
151–153	*3*	*GrA-10320*	*carbonate*		*10940*	*60*	*-3.58*			*10340*
151–155	3	GrN-23792	organics	fulvics	8960	110	-26.80	2.5	89.9	
153–155	3	GrN-24304	organics	>180 µm	11180	40	-30.91	27	44	10410
153–155	3	GrN-23936	organics	<180 µm	10190	60	-29.32	22	45	9560
153–155	*3*	*GrN-23713*	*carbonate*		*10440*	*80*	*-3.48*			*9840*
Average	3		organics		10630					9940
values	3		*carbonate*		*10740*					*10090*
155–156	3	GrN-23748	organics	>180 µm	11050	60	-29.98	22	55	10360
155–156	3	GrN-24005	organics	<180 µm	10340	80	-29.85	25	44	9660
155–156	*3*	*GrN-23715*	*carbonate*		*10600*	*210*	*-4.41*			*9910*
155–159	3	GrN-23790	organics	fulvics	8370	100	-26.60	2.4	84.2	
156–157	3	GrN-24302	organics	>180 µm	11230	110	-30.17	23	49	10520
156–157	3	GrN-24004	organics	<180 µm	10560	70	-29.56	22	44	9910
156–157	*3*	*GrA-10315*	*carbonate*		*10700*	*60*	*-4.13*			*10100*
156–158	3	GrN-23789	organics	humics	10080	100	-30.00	30	40	
157–158	3	GrN-23716	organics	>180 µm	10850	70	-29.91	17	64	10160
157–158	3	GrN-24003	organics	<180 µm	10280	60	-29.36	12	50	9640
157–158	*3*	*GrA-11239*	*carbonate*		*10780*	*60*	*-5.56*			*9960*
Sandy peat (units 2 and 1)										
158–159	2	GrN-24306	organics	>180 µm	10910	210	-30.08	16	69	10150
158–159	2	GrA-11244	organics	<180 µm	10390	60	-28.72	5	89	10300
174–175	2	GrN-24111	organics	>180 µm	11310	140	-28.02	52	8***	11310
174–175	2	GrN-24305	organics	<180 µm	11210	220	-27.43	20	54***	11210

* For peat, the datable fraction is humine.
** After chemical treatment.
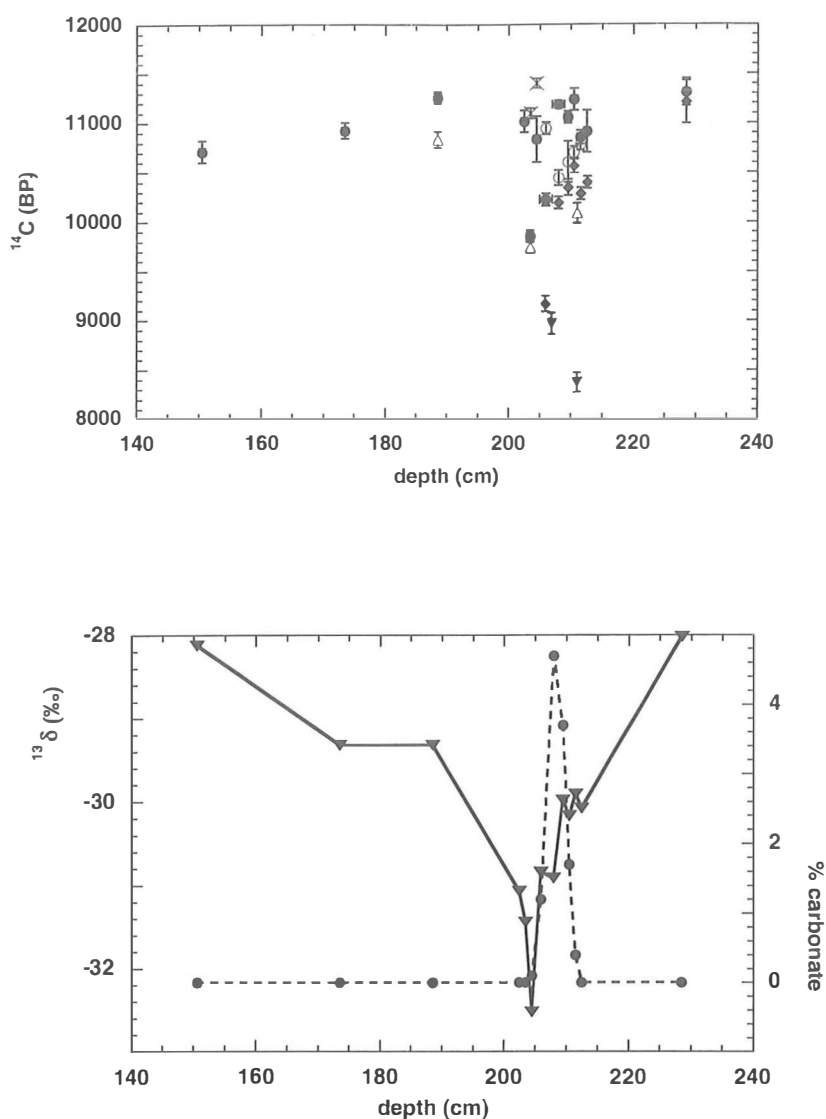*** Most sand removed after treatment.

Fig. 2. Results for isotopic measurement of the Wildervank depression as a function of depth. (A) ^{14}C dates (in BP 1s errors) for various fractions. (•) Coarse fraction (>180 μm); (♦) fine fraction (<180 mm); (Δ) humic fraction; (▼) fulvic fraction; (O) carbonate; (×) *Menyanthes* seeds. (B) ^{13}δ (in ‰) for organic material (triangles, solid line, left axis) and carbonate content (circles, dashed line, right axis) as a function of depth. The carbonate content is given in % of the wet weight (the dry weight is about a factor of 7 higher).

that means that a number of dates from material in the layer of 'calcareous gyttja' (unit 3) are incorrect. Two possible causes for the aberrant results will be discussed.

3. CAUSE 1: RESERVOIR EFFECTS

The 'calcareous gyttja' part of the sediment (unit 3) has been investigated in detail. In water/carbonate systems the natural isotopes to be considered are ^{13}C, ^{14}C and ^{18}O (Mook, 2000). They have, in general,

values as indicated schematically in table 2. Soil lime can be dissolved by infiltrating water : $CO_2 + H_2O + CaCO_3 \rightarrow Ca^{2+} + 2HCO_3^-$. In *running water*, the ^{13}δ value for bicarbonate (HCO_3^-) is around -12‰. The dissolved CO_2, in equilibrium with the HCO_3^-, has a ^{13}δ value of around -20‰. Plants that perform uptake of this CO_2 (submerged plants) then will have ^{13}δ = -38 ‰ since the isotopic fractionation for this process is 18‰. The ^{18}δ values for the oxygen isotope range between -7 and -8‰ for water, and from -5,5 to -6.5‰ for carbonate. The activity ratio (Mook & Van der Plicht, 1999) for the isotope ^{14}C, ^{14}a,

Table 2. Natural carbon and oxygen isotopes for running and stagnant water: schematic overview. The δ values are reported with respect to their international standards: $^{13}\delta$ = PDB; $^{13}\delta$ carbonate = PDB; $^{18}\delta$ water = SMOW (Mook, 2000).

	Running water	Stagnant water
Bicarbonate HCO_3	$^{13}\delta = -12‰$	$^{13}\delta \approx 0‰$
Dissolved CO_2	$^{13}\delta = -20‰$	$^{13}\delta \approx -4‰$
Submerged plants	$^{13}\delta = -38‰$	$^{13}\delta = -22‰$
Water	$^{18}\delta = -7.5‰$	$^{18}\delta = +0‰$
Carbonate	$^{18}\delta = -6.5‰$ $^{13}\delta = -10‰$	$^{18}\delta = 1.0‰$ $^{13}\delta = +2‰$
Recent ^{14}C activity (carbonate and organic)	$^{14}a = 50 \rightarrow 85\%$ $\Leftarrow$	$^{14}a \approx 100\%$ $\Rightarrow$

ranges between 50 and 85%. The value of 50% corresponds to deep water (long residence time); the value of 85% to shallow water (short residence time). Indeed, the average value for water in the Netherlands is 85%. For *stagnant water*, the $^{13}\delta$ values for HCO_3^-, dissolved CO_2 and plants are about 12 ‰ higher as compared to those for running water. The $^{18}\delta$ value is 0 to +1 ‰, and the activity ratio $^{14}a \approx$ 100%. Note that these activity numbers are corrected for the nuclear bomb-peak. Nuclear explosions enhanced the ^{14}C content of the atmosphere in the 1960s. Since the atmospheric ^{14}C concentration has been measured we can correct for this 'bomb-effect' (Meijer et al., 1994).

The numbers in the two columns (running and stagnant) are extreme cases, the boundary between the two can be crossed, giving rise to situations with mixed numbers.

The standard activity ratio for ^{14}C, ^{14}a, is 100%, which corresponds to the oxalic acid standard and 1950 AD (Mook & Van der Plicht, 1999). For non-atmospheric (non-terrestrial) carbon reservoirs, recent activity ratios can be ^{14}a <100%. This causes contemporaneous events to have different ^{14}C ages. For example, dissolved carbonate with a recent activity of 85% (due to the carbon-mix of recent and fossil origin) has an apparent age of 1300 years; submerged plants feeding from this carbon therefore have an apparent age ('reservoir effect') of 1300 ^{14}C years with respect to contemporaneous atmospheric plants (Olsson, 1983). This reservoir effect is even observed in food chains. When a considerable part of the diet consists of food with apparent ages (such as fish),

dated animal or human remains (bones) show reservoir effects (Lanting & Van der Plicht, 1996b). Examples of other studies on ^{14}C in submerged plants and dating complications are performed by Pazdur et al. (1999) and Krajcar Bronic et al. (1992).

When applied to the measurements for the Wildervank deposit, we can conclude the following based on stable isotope measurements. We analysed the stable isotopes of the carbonate from the 'calcareous gyttja' section at 153–155 and 155–156 cm depth. The $^{13}\delta$ values are -6.67 and -6.66‰, and the $^{18}\delta$ values -0.37 and +0.21‰, respectively. This means that this probably corresponds to a situation not too different from the 'stagnant water' situation (table 2), because especially the measured $^{18}\delta$ value of the carbonate is significant for an equilibrium condition.

The reservoir age for the carbonate can therefore not be large, perhaps a few centuries. We measured ^{14}C ages at four depths, with only small differences (except GrN-23713). The averaged ^{14}C age for the carbonate of the 'calcareous gyttja' is about 10,700 BP (see table 1). This corresponds very well with the ^{14}C age for the Younger Dryas, with or without small reservoir age correction.

Even without allowing for a few centuries of apparent age, the ^{14}C age for the carbonate in the 'calcareous gyttja' would be *younger* than that of the peat layer above, which has ^{14}C ages around 11,000 BP (ignoring the date for peat next to the 'calcareous gyttja'). This in itself is a rather unique observation. Usually the carbonate shows older ages than the contemporaneous organic matter.

4. CAUSE 2: MOBILE ORGANIC MATERIAL

Based on their solubilities, the organic compounds are subdivided in fulvic acids (soluble in alkaline and in acids) and humic acids (soluble in alkaline, precipitating in acids). A residual fraction (humine) is insoluble in acids and alkaline. These organic molecules, originating from decomposing organic matter, are relatively resistant against further degradation. The fulvic acids represent a very instable phase of humic components, which is mobile in acid environments and move relatively quickly through a profile. Since in general there is net water transport downwards (or lateral, or even upwelling), this means that they show a younger age than the other fractions at the same depth. The fulvic fraction corresponds to Boreal peat formation.

Organic ^{14}C samples, such as peat, are chemically pretreated with acid/alkali/acid (AAA). The first step removes carbonates and fulvic acids; the second step removes humic acids; the final acid step removes any CO_2 adsorbed during the previous step (Mook & Streurman, 1983). For the Wildervank *Hypnaceae*

peat section (unit 4: 96-150 cm), the alkali extract corresponds with humic acid and shows ^{14}C ages slightly older or close to those of the corresponding fully pretreated peat (GrN-24007/23934; GrN-24008/ 23921; see table 1 and fig. 2A). This is indeed what one would expect.

For the 'calcareous gyttja' section (150-158 cm), the first acid bath extract corresponding with the fulvic acids is considerably younger than the corresponding fully pretreated peat (GrN-23792, 23790; see table 1 and fig. 2A). This is the most mobile fraction, which must have flowed in from the peat section above. This part of the system is 'open', also for the humic acids (GrN-23789).

Two dated seeds (GrA-17317, 17319) are truly *in situ* material. It became possible to date such small samples after the arrival of AMS (Van der Plicht et al., 2000). All other organic materials (such as the fulvic and humic acids) are secondary deposits in the gyttja section, or at least partly. This is based on the ages being younger than the peat layer above.

Both seeds are from *Menyanthes trifoliata* L. This plant obtains its ^{14}C from the atmosphere through the photosynthesis process. Thus, the seeds represent atmospheric/terrestrial ^{14}C values. This is confirmed by the results of their [13]δ measurements ($\approx$ -25 ‰).

The *Menyanthes* seeds were selected just above and just in the 'calcareous gyttja' section (see table 1 and fig. 2A). They yield ^{14}C ages of 11.090 and 11.395 BP, respectively.

5. THE CORRECTED ^{14}C AGES OF WILDERVANK-ZANDPLAS III

The right column of table 1 shows our estimates of the 'real' [14]C ages. By real ^{14}C ages we mean that the measured ^{14}C ages (also in table 1) are adjusted by subtracting a reservoir age. Submerged waterplant remains are presumably the origin of the organic material from the gyttja.

Information on reservoir effects was obtained from a few selected analogous sites in the Netherlands (provinces of Groningen and Drenthe). We collected fresh water mussels and associated waterplants. The carbonate of the mussels has been analysed for [13]δ, [18]δ and ^{14}C; the waterplants were analysed for [13]δ and [14]C. The samples were all recent material, so that the combined analysis provides the reservoir ages for [14]C. The results of our measurements are summarized in figure 3A (mussels) and B (waterplants; carbonate). The ^{14}C measurements (horizontal axis) are reported as activities ([14]a in %, where 100% corresponds with the standard). In the right-bottom area of the figures are the stagnant waters; in the left-top area those for running water. Note that the ^{14}C activities are corrected for the nuclear bomb effect.

From the carbonate measurements (fig. 3A), we obtained the relation [18]δ/^{14}C; transferred to the waterplants (fig. 3B), we could than establish the relation [13]δ (plants)/[14]C. This set of measured combined isotope ratios gives information used for the reservoir adjustment (right column in table 1), as will be described below. The [18]δ values range between c. 0 ‰ (stagnant water) and c. -6 ‰ (running water). For example, a reservoir age of 1300 years corresponds with a recent activity of 85%; this in turn corresponds with a [13]δ value for the submerged plant of about -37‰ (see fig. 3:B). For a ^{14}C activity of 100% we take a [13]δ value of -28‰ (known value for peat). Thus, we have a line from ([14]a,[13]δ) = (85%, -37‰) to (100%, -28‰), from which we can determine the adjustment to a measured ^{14}C age, based on its measured [13]δ value. This is valid for gyttja, provided they consist of remains of submerged waterplants. However, the *Hypnaceae* peat of unit 4 consists of the remains of mosses that took their CO_2 from the atmosphere, so that *Hypnaceae* peat cannot have reservoir effects (even if their [13]δ values are more negative than -28 ‰).

For gyttja, we take the line ([14]a, [13]δ) = (85%, -37‰) to (100%, -22‰). The adjusted ^{14}C sequence (right column in table 1) is corrected for the reservoir effect this way.

Illustrative is also the dating of a gyttja sample (B) at the adjacent location Wildervank, Zandplas II (Groenendijk, 1997: pp. 137–139). The ^{14}C date is 12,060±80 BP (GrN-18057), the [13]δ value is -29.80‰. The reservoir adjustment yields an adjusted ^{14}C age of 11,380 BP, confirming the palynological determination (Allerød).

Concerning the chronologies, we have to consider two timescales: the ^{14}C timescale, and the calibrated (historical) timescale. The ^{14}C timescale is expressed in BP (Before Present); the calibrated timescale in calBP, i.e. calendar years relative to 1950 AD. Thus, calBP = 1950 – AD (Van der Plicht, 2000). Because of the variations in the natural ^{14}C concentration in the atmosphere, there is no simple relationship between the BP and calBP timescales. This relationship can be established by calibration, i.e. measuring samples by both ^{14}C and another, independent and preferably absolute method. A calibration curve based on dendrochronologically dated wood is now available for the Holocene (Stuiver et al., 1998). For older times, the ^{14}C timescale can be compared with records like varved sediments and U-dated corals. The relevant part of the calibration data for the Wildervank depression is shown in figure 4. We show data from the calibration curve Intcal98 (Stuiver et al., 1998) and a varved sediment from Lake Suigetsu, Japan (Kitagawa & Van der Plicht, 2000). The errors plotted for both records are 1σ uncertainties.

The Younger Dryas is dated between c. 11,550–12,900 calBP (using the ice core records) and
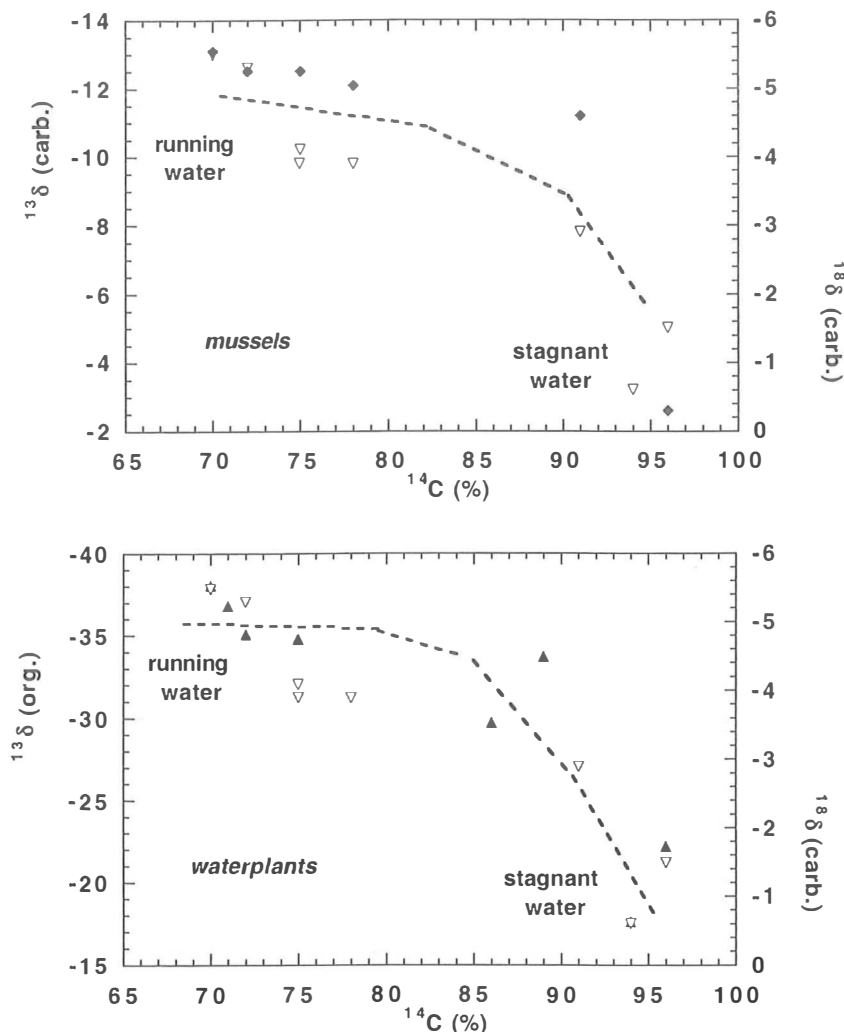
Fig. 3. $^{13}\delta$, $^{18}\delta$ and ^{14}C for carbonate from mussels (A), and for organic matter from submerged plants (B). The samples are representative for recent material. A: (◆) $^{13}\delta$ carbonate (left vertical axis); (∇) $^{18}\delta$ carbonate (right vertical axis). B: (▲) $^{13}\delta$ plants (left vertical axis); (∇) $^{18}\delta$ carbonate (right vertical axis). The dashed lines are indicative, meant for clarification only. The ^{14}C activities are corrected for the nuclear bomb effect.

10,150–10.800 BP (^{14}C); the beginning of Allerød c. 14,000 calBP and ca. 11,800 BP (Lanting & Van der Plicht 1996a). Note that the duration of the Younger Dryas, 1350 years, is much shorter in ^{14}C years (see fig. 4) due to a large 'plateau' in the calibration curve.

6. THE TRUE NATURE OF THE 'CALCAREOUS GYTTJA'

Usually, carbonate from organic deposits shows reservoir effects (apparent ages) of max. 1300 years. This corresponds with an original ^{14}C activity well below 100% (85%). For the Wildervank depression, however, the peat above the 'calcareous gyttja' (see fig. 1) has a ^{14}C age of c. 11,000 BP. The carbonate is significantly younger (a maximum ^{14}C age of 10,700 BP). The in situ *Menyanthes* seeds have a ^{14}C age corresponding with the peat (Allerød). It appears, therefore, that the 'calcareous gyttja' was formed later than the above lying peat. During its deposition in existing sediment some organic material and the dated *Menyanthes* seed were imbedded, which explains the ^{14}C ages of these materials. The 'calcareous gyttja' did not precipitate on top of existing sediment, as should be expected for true calcareous gyttja, deposited from upwelling water during a cold period.

This conclusion was corroborated later by a chemical analysis of the material, which resulted in a high Fe_2O_3 content, and in a very low CaO content
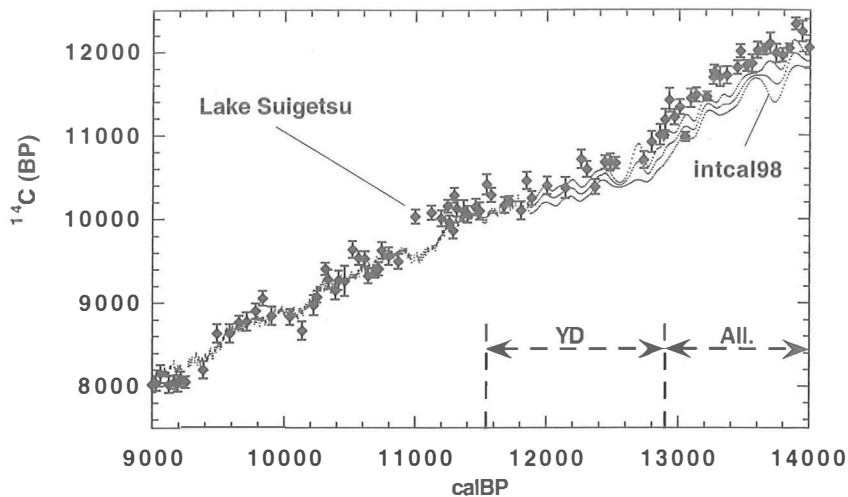
Fig. 4. Relevant part of the ¹⁴C calibration curve. Vertical: ¹⁴C age (in BP); horizontal: calibrated age relative to 1950 AD (expressed in calBP). The recommended calibration curve Intcal98 (Stuiver et al., 1998) is shown (dotted line; 1σ error envelope), together with data from ¹⁴C measurements for the Lake Suigetsu varved sediment (Kitagawa & Van der Plicht, 2000). The Younger Dryas and Allerød periods are indicated.

Table 3. Composition analysis of a siderite sample (numbers in %). L.O.I.: Loss on ignition.

Compound	Concentration (%)
Fe_2O_3	10.49
MnO	0.14
P_2O_5	0.06
CaO	0.45
TiO_2	0.02
K_2O	0.01
Al_2O_3	0.12
Na_2O	0.06
SiO_2	47.20
L.O.I.	39.3

(see table 3). Apparently, the mineral form is ferro-carbonate or siderite. Indeed, the carbonate had a bright yellow appearance when freshly cut, but quickly turned grey of black after exposure. The carbonate must have been transported through the peat layer — or from a lateral direction. The origin was apparently not calcium bicarbonate dissolved in groundwater, but decaying organic material in combination with a Fe bicarbonate solution. Siderite can only be formed when a peat layer exists — enabling leaching of Fe from the sediment.

The bottom part of the Zandplas III section (unit 2: >158 cm) yields a peat date of 11,270 BP (average value for GrN-24111 and 24305; see table 1). It is not clear, however, whether in this sandy peat reservoir corrections should be applied. Because of

the very small amount of organic material, only acid treatment was applied so that the possibility of contamination cannot be excluded. Hence, the ages reported should be considered minimum ages.

7. CONCLUSION

The 'calcareous gyttja' (unit 3) in the sediments of the Wildervank-Zandplas III depression appears to be a secondary deposit of siderite. The ¹⁴C age of a *Menyanthes* seed in this siderite does not date its deposition, but the forming of the sediments in which the siderite was deposited. Carbonate in this siderite layer has an average, reservoir-adjusted ¹⁴C age of 10,330 BP and is therefore younger than the superimposed peat of unit 4, which was formed around the Allerød/Younger Dryas transition. Unit 2, the sandy peat in which top the siderite had been deposited, was most likely formed during mid/late Allerød.

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