

**RADIOCARBON DATING OF VEGETATION HORIZONS,
'ILLUSTRATED BY AN EXAMPLE FROM THE HOLOCENE COASTAL PLAIN
IN THE NORTHERN NETHERLANDS**

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ABSTRACT

Schoute, J. F. Th., J. W. Griede, W. G. Mook & W. Roeleveld 1981 Radiocarbon dating of vegetation horizons, illustrated by an example from the Holocene coastal plain in the northern Netherlands. *In*: A. J. van Loon (ed.): Quaternary geology: a farewell to A. J. Wiggers - Geol. Mijnbouw 60: 453-459.

Radiocarbon dating of vegetation horizons (fossil A₀/A₁-soil horizons occurring within the younger Holocene marine deposits) is subject to contamination by old carbon which forms part of the sedimentary matrix. Samples of recent mud from several locations in The Netherlands give apparent radiocarbon ages of up to 5900 years BP, demonstrating that a significant ageing effect may occur. Notably the age of the organic residue, left after alkali pretreatment of the sample of vegetation horizons, is affected by this contamination. A substantial part of the total amount of the organic matter in these vegetation horizons consists of humic substances. Since the larger part of these humic substances has actually been formed *in situ* by humification of fresh organic material, it is to be expected that radiocarbon dating of humic substances (dissolved in the alkaline extracts) gives a better indication of the true age of the vegetation horizons than the dates from the (apparently too old) residues. This hypothesis is compared to geological evidence in the coastal plains of the northern Netherlands and of Suriname.

INTRODUCTION

The present-day coastal plain of the northern Netherlands (Fig. 1, inset) developed in the course of the Holocene mainly as the result of tidal deposition during a gradually slowing sea-level rise. ROELEVELD (1974) and GRIEDE (1978) depicted the Holocene geological and palaeogeographical history of the area on the basis of extensive field investigations. Their studies revealed the occurrence of alternating periods of relatively strong and reduced marine activity; these periods were considered as transgressive and regressive intervals respectively (Table I). During transgressive intervals the marine depositional realm extended inland, whereas during regressive intervals marine influences decreased. During the earlier part of the sequence, until about 3000 C-14 years BP, regressive intervals resulted in the formation of peat beds, intercalated in the clastic tidal sequence. From about 3000 BP onward, however, regressive intervals are registered by the occurrence of non-erosive unconformities within the marine deposits, which result from periods of non-deposition. It is believed that the decrease in the rate of sea-level rise is responsible for this changing registration of regressive ten-

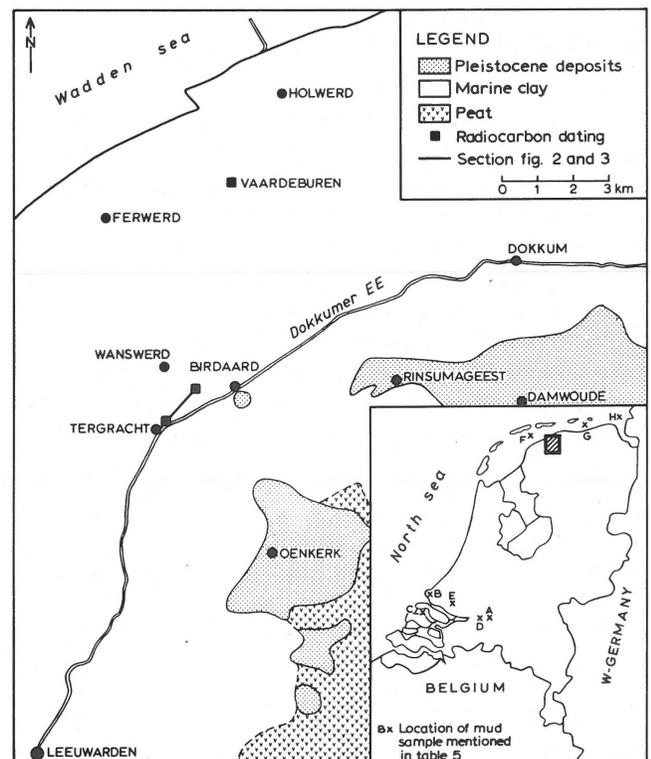


Fig. 1
Map showing sample locations

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Table I
Transgressive/regressive chronology of the Subboreal and Subatlantic in the northern Netherlands.

AGE	CONV. 14 C YRS. B.P.	TRANSGRESSIVE / REGRESSIVE PERIODS
SUBATLANTIC		DUNKERQUE III B
	750	HOLLAND IX
	950	DUNKERQUE III A
	1150	HOLLAND VIII
	1350	DUNKERQUE II
	1650	HOLLAND VII
	2000	DUNKERQUE I B
	2500	HOLLAND VI
	2650	DUNKERQUE I A
	2900	HOLLAND V
	3000	DUNKERQUE O
	3200	HOLLAND IV B
	3225	CALAIS IV B
	3400	HOLLAND IV A
SUBBOREAL	3700	HOLLAND IV A
	3900	CALAIS IV A
	4150	HOLLAND III
	4300	CALAIS III
4550		
5000		

dencies in time.

The non-erosive unconformities occurring within the younger Holocene deposits are frequently associated with so-called vegetation horizons. These vegetation horizons are in general dark brown or black bands which consist of a clayey matrix with a certain concentration of organic matter. The organic matter is composed of finely dispersed minute particles of black organic material and of humus components. Currently SCHOUTE (in prep.) is studying in detail (with the aid of micromorphological techniques) the composition, origin and palaeoecological implications of vegetation horizons from the Schildmeer and Birdaard areas in the northern

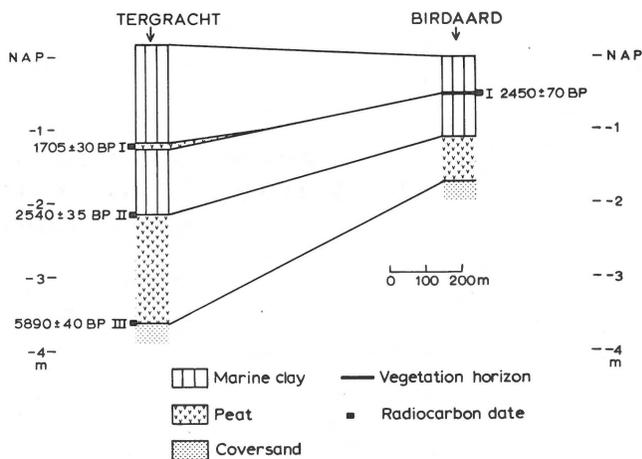


Fig. 2
Section Tergracht-Birdaard (location see figure 1)

coastal district (Fig. 1). Generally speaking these vegetation horizons can be considered to represent fossil A_0/A_1 -soil horizons which developed on top of the underlying marine sediments during periods of non-deposition; they were supposedly formed in a semi-terrestrial environment. At places these vegetation horizons may pass laterally into peaty, or occasionally gyttja-like horizons (WILDVANG, 1938: 'Blauer Strahl'; MÜLLER, 1955, 1958: 'Fossiler A_h -Horizont', 'Humusdwog'; ROEVELD, 1974; SCHOUTE, in prep.).

STATEMENT OF THE PROBLEM

In connection with the study of the Holocene evolution of the north-eastern part of the province of Friesland (GRIEDE, 1978) radiocarbon dates were obtained from two sections, near Tergracht and Birdaard, respectively (Fig. 2, for location see Fig. 1). The Tergracht and Birdaard sections, which are only 825 m apart, show strong lithostratigraphical similarities. In both sections the Pleistocene subsoil is covered by a distinct (lower) peat bed, which in its turn is covered by clastic marine deposits of 1-2 m in thickness. At Tergracht a minor peat bed of 5-6 cm in thickness is found intercalated in the marine deposits, whereas at Birdaard a vegetation horizon occurs within the marine deposits. Correlation of the Birdaard vegetation horizon with the Tergracht peat horizon (which occurs

Table II

Conventional radiocarbon ages (years B.P.) of vegetation horizons (VH) and peat (P). Column 3 gives the percentage of carbon of the original sample. Following the routine acid treatment, alkaline extractions were carried out with x% NaOH solution at y °C during z hours. The alkaline extracts were subsequently precipitated by acidification.

Sample (Fig. 2)	VH/P	%C	Treatment			Age	Lab.nr. GrN-
			x	y	z		
Birdaard I	VH	0.9	—	—	—	2450 ± 70	7645
Tergracht I	P	> 20	0.5	20	24	1705 ± 30	7560
Tergracht II	P	> 20	0.5	20	24	2540 ± 35	7561
Tergracht III	P	> 20	0.5	20	24	5890 ± 40	7562

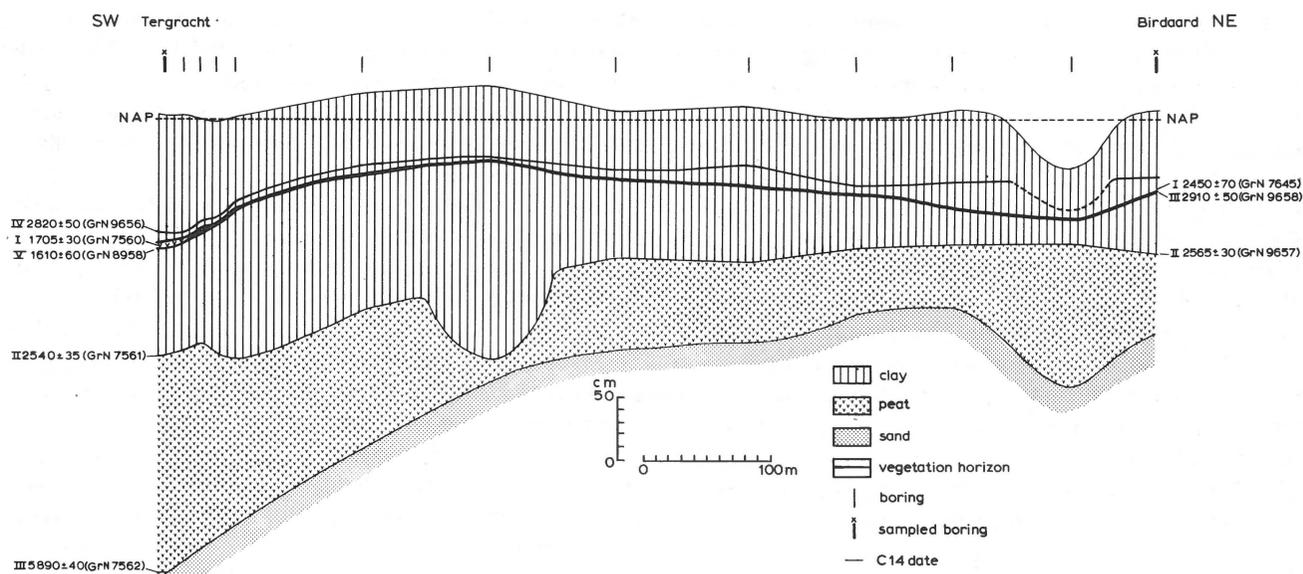


Fig. 3
The Tergracht-Birdaard section (location see figure 1)

in a depression) appears to be likely. However, the radiocarbon dates obtained from these respective horizons (GRIEDE, 1978) contradict such correlation: the peat horizon at Tergracht (I) was dated at 1705 ± 30 BP, whereas the vegetation horizon at Birdaard (I) yielded a result of 2450 ± 70 BP (see Table II).

GRIEDE (1978) accepted both the radiocarbon dates of Tergracht I and Birdaard I as correct and, consequently, assumed that the seemingly simple stratigraphic correlation of the Tergracht and Birdaard sections was erroneous. As a matter of fact, Griede had good reason to accept the Tergracht I and Birdaard I dates. In the transgressive-regressive chronology of the northern Netherlands, which is based on many C-14 datings of peat layers and vegetation horizons from all over the area (ROELEVELD, 1974; GRIEDE, 1978), both ages fit well. The date of 2450 ± 70 BP (Birdaard I) indicates the onset of the Dunkerque-IB transgressive interval (compare the date of 2540 ± 35 at the top of the lower peat at Tergracht II), the date of 1705 ± 30 BP (Tergracht I) is concordant with the onset of the Dunkerque II transgressive interval (Table I, Fig. 2). It should be noted in this respect that, according to the explanation given by ROELEVELD (1974), the development of vegetation horizons (or minor peat layers) in the upper Holocene sequence is caused by the influence of increasing wetness, i.e. results from the onset of a transgression. In this view radiocarbon dates of such horizons are primarily indicative of the starting date of a new transgression and give only a *terminus ante quem* for the preceding regressive interval. SCHOUTE (in prep.) re-examined the situation in the Birdaard-Tergracht area in relation to his detailed study of vegetation horizons. He was able to demonstrate that the lithostratigraphic correlation of the Tergracht I peat horizon and the Birdaard I vegetation horizon was actually correct; moreover, it became obvious that in both the Tergracht and Birdaard sections a

(second) vague, vegetation horizon did occur above the radiocarbon dated levels (Fig. 3).

Schoute's observations necessitated a more thorough analysis of the discrepancy between lithostratigraphical and radiocarbon correlations between the Tergracht and Birdaard sections, since a simple false lithostratigraphic correlation was out of the question. Theoretically, it is not impossible to find a diachrony of many centuries for a single lithostratigraphic horizon over some horizontal distance. However, in the present case, the sections under consideration are less than 1 km apart. Considering the chronological regularity observed in the occurrence of transgressive and regressive intervals in the northern coastal district, it seems unnecessary to explain the considerable age differences between the lithostratigraphically equivalent Tergracht I and Birdaard I horizons in terms of local diachrony. Hence, the conclusion is unavoidable that at least one of the pair of radiocarbon dates Tergracht I and Birdaard I is false. In the following sections this conclusion will be discussed further.

ANALYSIS OF THE PROBLEM

Assuming, on the basis of the arguments stated above, that at least one of the C-14 datings Tergracht I and Birdaard I is not correct, the probability of the available C-14 dates has been reviewed. It was concluded, on the basis of the following arguments, that the dates of Tergracht I and II are the most likely, and hence that the age of Birdaard I has to be rejected.

- (1) The dates Tergracht I, II and III are mutually consistent.
- (2) The pollenanalytical data of the boring Tergracht (VAN MUYEN, 1978) do not contradict the C-14 dating results Tergracht I and II.
- (3) The morphological situation in the area suggests that the

upper clastic bed has been deposited during the Dunkerque III transgressive period, due to its rather strong morphological expression (e.g. gully levees, backswamps) which is not seen (in the northern coastal district) in deposits of the Dunkerque II period. This implies that in the Birdaard-Tergracht area the sediments of the Dunkerque II period are to be found on a lower level, viz. in between the two vegetation horizons (Fig. 3), which is in accordance with the date Tergracht I.

(4) 25 years of experience with radiocarbon dating of peat beds occurring within the coastal Holocene sequence in The Netherlands and adjacent countries has shown such dates to be generally reliable as far as reproducibility and internal consistency is concerned; C-14 dates on vegetation horizons, of which only a limited number has been carried out so far, on the other hand, seem to be less consistent and to show greater variability (cf. BENZLER & GEYH, 1969; ROELEVELD, 1974; GRIEDE, 1978).

In order to test this idea a new series of four radiocarbon dates was performed on the sections under consideration (Table III, Fig. 3). This series includes a second determination of the suspected vegetation horizon at Birdaard (Birdaard III) as well as a number of additional dates to strengthen the chronologic framework as a whole (Birdaard II, Tergracht IV and Tergracht V).

INTERPRETATION OF THE RADIOCARBON DATES

Two dates are now available of the (lower) vegetation horizon at Birdaard, which are mutually inconsistent: 2450 ± 70 BP (Birdaard I) and 2910 ± 50 BP (Birdaard III). The sample Birdaard III has been pretreated in order to remove possible contamination by young humus components originating from higher levels (alkaline extracts). Details of the treatment are given in tables II and III. The first and fourth alkaline extract of the Birdaard III sample have been dated in order to judge

Table III

Conventional radiocarbon ages (years B.P.) of vegetation horizons (VH) and peat (P). Column 3 gives the percentage of carbon of the original sample. Following the routine acid treatment, alkaline extractions were carried out with x% NaOH solution at y°C during z hours. The alkaline extracts were subsequently precipitated by acidification.

Sample (Fig. 3)	VH/P	%C	Treatment			Age	Lab.nr. GrN-
			x	y	z		
Tergracht IV	VH	0.8	0.25	20	24	2820 ± 50	9656
Tergracht V	P	> 20	0.5	25	3	1610 ± 60	8958
Birdaard II	P	> 20	0.5	20	24	2565 ± 30	9657
Birdaard III (residu)	VH	0.2	5	80	4×48	2910 ± 50	9658
1st extr.		0.2	5	80	48	2090 ± 50	9744
4th extr.*		0.15	5	80	48	2210 ± 80	9745

* 2nd and 3rd alkaline extractions were performed similarly to 1st extraction (each 48 hours); both removed an amount of carbon of 0.2% of the original sample.

whether any possible young contaminating substance was left. These datings resulted in ages of 2090 ± 50 BP and 2210 ± 80 BP, respectively, which are considerably younger than the date on the residue of 2910 ± 50 BP. Since the original date for the Birdaard vegetation horizon (Birdaard I), 2450 ± 70 BP, had been determined without sample pretreatment, the difference in age of c. 500 years between the samples Birdaard I and III might be explained by assuming that Birdaard I was contaminated by younger humus components. However, the other evidence at hand indicates that the dates Birdaard I and III are both unreliable. The top of the lower peat in the Tergracht and Birdaard sections yielded mutually consistent ages of 2540 ± 35 BP and 2565 ± 30 BP, respectively, and this is in favour of their credibility. If it is accepted on this basis that the date of 2565 ± 30 BP for the top of the lower peat at Birdaard is correct, then datings of the overlying vegetation horizon of 2450 ± 70 BP and 2910 ± 50 BP are obviously highly improbable.

This conclusion is supported by the fact that the intercalated peat horizon at Tergracht, which is the lateral equivalent of the (lower) Birdaard vegetation horizon, yielded the much younger age of 1705 ± 30 BP; this date presents no problems in relation to the age of the top of the underlying lower peat. A redating, this time of the basal part only, of the intercalated peat horizon at Tergracht gave a comparable result of 1610 ± 60 BP (Tergracht V). On the other hand, the dating of the upper vegetation horizon (Fig. 3), where it is situated above the peat horizon, resulted in the quite inconsistent value of 2820 ± 50 BP (Tergracht IV). This last result strengthens the opinion that the radiocarbon dates from peat in the Tergracht-Birdaard sections are mutually consistent, whereas the dates from vegetation horizons deviate, being considerably too old.

SOURCES OF ERROR IN DATING LOW-CARBON SAMPLES FROM ORIGINALLY MARINE DEPOSITS

The phenomenon of ageing of radiocarbon determinations by

contamination of 'old' carbon-containing material has been described frequently (e.g. BENZLER & GEYH, 1969; MENKE, 1969; GEYH, 1971; DICKSON ET AL., 1978; BOWEN, 1978; LOWE & WALKER, 1980; SUTHERLAND, 1980). The following sources of error might induce ageing of datings of vegetation horizons.

(1) Part of the organic matter dated consists of allochthonous material that was (re)deposited together with the mineral sediment: reworked 'old' organic matter. This material may be composed of very small particles of clastic organic matter (plant and animal remains). The lower peat and possibly soils that developed on the Pleistocene substrate are considered, in this particular situation, as the most probable source of such allochthonous organic matter. Indeed, in clayey deposits organic matter occurs rather commonly. For alluvial sediments in The Netherlands a close relationship between clay content and the content of organic matter has been determined (WIGGERS, 1950, 1960; HISSINK, 1954), as the settling rate of clay and particulate organic material in water is about the same (DE HAAN, 1977).

(2) Another source of error might be the presence of humus components strongly absorbed to surfaces and particularly to/in interlaminar layers of clay minerals (SCHNITZER & KHAN, 1972; BABEL, 1975; KONONOVA, 1975; SCHNITZER, 1978). Humus components can thus be transported and deposited together with clay particles.

(3) In some cases apparent ageing is caused by (old) groundwater, in literature generally described as the 'hard-water effect' (MÜNNICH & VOGEL, 1959; GEYH, 1971). This, however, does not apply to organic matter of terrestrial origin as in our case. Also in very shallow pools apparent ageing by relatively old groundwater carbon is absent, due to the fast equilibration of the dissolved inorganic carbon with atmospheric CO₂.

The vegetation horizons considered have low to very low contents of organic carbon (c. 0.8-3% C). Particularly in

sediments and soils of these relatively low carbon contents a slight contamination with ancient carbon can produce a serious error (GEYH, 1971; BOWEN, 1978; SUTHERLAND, 1980).

The ageing effect of 'old' carbon can be quantified by considering the mixing equation:

$$a^{14}_{\text{meas}} = a^{14}_{\text{true}} - x(a^{14}_{\text{true}} - a^{14}_{\text{cont}}) \quad (1)$$

where a^{14} refers to the C-14 activity (in %) and the subscripts 'meas', 'true' and 'cont' to the measured, the true and the contaminants activity, respectively. By inserting the ages instead of activities by using

$$T = -8033 \ln a^{14}/100 \quad (2)$$

the contamination equation is:

$$T_{\text{true}} - T_{\text{meas}} = -8033 \ln \left(1 + \frac{x}{1-x} (1 - e^{(T_{\text{meas}} - T_{\text{cont}})/8033}) \right) \quad (3)$$

Table IV presents a few numerical examples of the ageing effect.

From research carried out on samples of recent mud in the Isotope Physics Laboratory at Groningen it appears that organic matter transported by and with the mud can have a considerable age. From a few locations recently deposited mud was collected. After removal of the carbonate fraction by hydrochloric acid treatment, the organic fraction was dated after combustion of the whole sample or of the alkali extracted fraction. The fractions dated and the results are given in table V. The location numbers refer to figure 1.

HYPOTHESIS

It was concluded above that old carbon, which forms part of the sedimentary matrix material of vegetation horizons, may well cause a considerable increase of the apparent radiocarbon ages of such vegetation horizons. This conclusion suggests that the age difference between the alkaline extracts on the one hand (2090 ± 50 and 2210 ± 80 BP) and the organic residue on the other (2910 ± 50 BP) of the Birdaard veget-

Table IV
The ageing effect of various fractional amounts (x) of contamination of C14 age T_{cont} on a measured age T_{meas} . The effect is calculated from eq. 3 and presented as $T_{\text{meas}} - T_{\text{true}}$.

T_{true} (years B.P.)	$T_{\text{cont}} = 2000$ B.P.			4000 B.P.			6000 B.P.		
	x = 10%	25%	50%	10%	25%	50%	10%	25%	50%
0	190	570	1600	340	990	2660	460	1300	3400
1000	100	310	890	270	790	2180	400	1150	3060
2000	—	—	—	190	570	1600	340	990	2660
3000				100	310	890	270	790	2180
4000				—	—	—	190	570	1600
5000							100	310	890
6000							—	—	—

Table V
Conventional radiocarbon dates (years B.P.) of the organic fraction of recently sedimented mud at various locations in The Netherlands. The location numbers refer to figure 1 (inset). The carbonate fraction was removed by acid treatment. The alkaline extract was obtained by treatment with 3% NaOH solution for 24 hours at 20 °C.

location	nr	Lab. nr. GrN-	Total organic carbon	alkaline extract
Dordtse Biesbosch	A	6569		3415 ± 75
Europoort	B	7045	3220 ± 90	
Grevelingen Dordtse	C	7046	4670 ± 150	
Biesbosch Rotterdam	D	7047	720 ± 110	
Harbour	E	7048	4350 ± 100	
Friesland	F	6754	5910 ± 90	
Groningen	G	6797	3320 ± 140	
East Friesland (Germ.)	H	6850	1475 ± 65	

ation horizon may be explained in a different manner than usual. The usual explanation of such a difference in age, as was mentioned above, is to regard the residue age as the true one and the younger age of the alkaline extract as being caused by contamination of younger infiltrated humus components. However, if it is accepted that in vegetation horizons significant contamination by older material occurs, it is conceivable that notably the age of the organic residue, which is the older one, is affected by this contamination (Table IV). Thus it is probable that *the date of the alkaline extract gives a better indication of the true age of the formation of the vegetation horizon.*

Indeed, in vegetation horizons developed in a semi-terrestrial environment the larger part of the humic substances (and these are leached during the process of alkaline extraction) are actually formed *in situ* by humification of fresh organic materials (chemical and biological degradation of plant and animal residues and synthetic activities of micro-organisms). The humic substances³ formed in this manner are considered to be the typical organic soil constituents. They constitute a distinctive and integral part of the soil (SCHNITZER & KHAN, 1972;

³ Organic matter can be grouped into (1) non-humic substances and (2) humic substances. Non-humic substances exhibit still recognizable physical and chemical characteristics (e.g. amino acids, carbohydrates, fats, waxes, resins, etc.). Most of these compounds are relatively easily attacked by micro-organisms in the soil and have a relatively short life span. Humic substances are usually separated from the other humus components and divided into different fractions by their characteristic solubilities and dispersibilities in alkali and acid. They have no specific physical and chemical characteristics and are brown to black substances of low and high molecular weight, formed by secondary synthesis reactions. They are more stable than the starting material (Schnitzer & Khan, 1972; Flaig et al., 1975; Kononova, 1975; Schnitzer, 1978; Stevenson, 1979).

Table VI
Radiocarbon dates at Burnside, Suriname.

Sample	Age (conv. C-14 years B.P.)	Material	Lab. nr. GrN-
Burnside I	2140 ± 50	veg. hor.	7828
Burnside I	1440 ± 70	1st alkaline extract	7828 E1
Burnside I	1430 ± 70	2nd alkaline extract	7828 E2
Burnside I	1380 ± 70	3rd alkaline extract	7828 E3

FLAIG ET AL., 1975; KONONOVA, 1975; SCHNITZER, 1978; STEVENSON, 1979). Considering the amounts of organics extracted by our alkali treatment (Table III), a substantial part of the total amount of the organic matter in these vegetation horizons consists of humic substances. Therefore, it is to be expected that dating the humic substances from these horizons would yield reliable results.

However, the extraction of humic substances should be carried out by reproducible methods. Furthermore, one must be aware of the risk of contamination by young humic substances and young roots, as vegetation horizons usually are situated close to the present surface.

DISCUSSION

According to the hypothesis stated above, the alkaline extract of the organic content will in many cases consist of humic substances which were formed almost exclusively during the actual formation of vegetation horizons and hence yield reliable dating results. The dates of the alkaline extracts of the sample Birdaard III (2090 ± 70 and 2210 ± 80 BP) agree better with the stratigraphic setting and the expected age of 1800 BP than the date of the more resistant residual organic fraction (2910 ± 50 BP) (Table III) and this is in favour of the view put forward above.

A comparable case can be mentioned from the dating results of a vegetation horizon at Burnside, Suriname (Table VI), described by ROELEVELD & VAN LOON (1979). These authors explain the dates of the alkaline extracts as an indication of a young contamination in the sample. By incomplete removal the age obtained for the residual material of Burnside I, 2140 ± 50 BP could still be too low. However, the dates on the alkaline extracts of Burnside I (around 1400 BP) are in much better agreement with the original age estimate of the authors: 1000-1300 BP or younger.

GRIEDE (1978) mentions a date of 2485 ± 65 BP for a vegetation horizon at Vaardeburen (Fig. 1), much older than the expected age of 1600 BP. Although in this case no separate datings were made on the different organic fractions, it seems plausible that here too contamination by older sedimentary organic matter did occur.

Concluding, it can be stated that the reliability of radiocarbon dates on vegetation horizons is, at least in certain cases,

open to doubt. This may have induced erroneous chronostratigraphical interpretations. However, it is hard to estimate to what extent the phenomenon of contamination with 'old' carbon affected the ages of the various samples.

The large differences between expected ages and determined ages in the samples Birdaard I, Birdaard III, Burnside and Vaardeburen suggest that the effect may be quite considerable. On the other hand, in several of these cases incorrect geological interpretations and correlations may also partly be responsible for the discrepancies observed.

Finally, another possibility of obtaining suitable material for radiocarbon determinations of vegetation horizons and similar phenomena can be suggested; viz. to date only separate autochthonous components of the macroremains (seeds, fruits, leaves, stems, twigs) of the sample. The procedure may present a few difficulties through which the application is hindered. Firstly, it must be decided whether part of the macroremains is autochthonous. A second, more serious objection is the collecting of sufficient datable material.

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