^{14}C ACTIVITY IN DIFFERENT SECTIONS AND CHEMICAL FRACTIONS OF OAK TREE RINGS, AD 1938–1981

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ABSTRACT. The ^{14}C activity in rings from an oak tree grown in a suburb of Uppsala, Sweden has been studied for the period, AD 1938 to 1981. We compare the results with the atmospheric carbon dioxide records from Abisko, northern Sweden, where local or regional contamination from fossil-fuel combustion can be disregarded. We assess the influence from different chemical pretreatment procedures in use and compare HCl-NaOH-HCl treatment with cellulose extraction. We split each ring into two samples corresponding to early (spring) and late wood. A more refined partitioning has been applied to the years 1963 and 1964.

INTRODUCTION

The aims of the present investigation were twofold. By utilizing the small requirements imposed on sample material by the AMS technique, we intended to make a refined spatial study of the ^{14}C activity in sections of tree rings in a Swedish oak around the bomb peak maximum in the atmosphere. We also conducted a careful investigation of chemical pretreatment to determine the best method of extracting the fraction reflecting the contemporary atmospheric activity. We chose four time intervals in order to establish: 1) whether any local industrial effect can be attributed to the growing site; 2) to what extent radial transportation in the tree affects the activity in different chemical fractions; 3) a comparison of the activity in the different wood fractions with that in the atmosphere around the maximum ^{14}C bomb effect to elucidate the carbon uptake in the oak.

SAMPLE PRETREATMENT ACCORDING TO THE LITERATURE

In 1954, de Vries and Barendsen introduced sample pretreatment, using HCl, NaOH and HCl with water washes between the three steps to remove humic acids. This pretreatment also extracts resin from the wood. Although varying from laboratory to laboratory, such a pretreatment is often called an AAA (acid-alkali-acid) treatment. Many laboratories have used organic solvents to extract resinous components. In such cases it is very important to remove the solvents. The normal procedure is to avoid evaporation to dryness before the solvents are removed by long boiling with water. Jansen (1973) studied the transfer of carbon from solvents to samples.

In radiocarbon literature, "wood cellulose" denotes a variety of products such as holocellulose, hemicelluloses, alpha-cellulose or even a mixture of cellulose and lignin. Often cellulose is extracted. According to a recipe by Olson and Broecker (1958), lignin has also been used for studies of the ^{14}C activity in wood.

Wilson, Gumbley and Speddin (1963) demonstrated that resin from a tree ring could yield a much higher activity than cellulose from the same ring, which meant that the extract, resin, was radially mobile within the tree. Whether these nutrients are ever used is an unsolved question. Nutrients stored in the roots of oaks are probably used before leaves form to produce some early wood. Jansen (1970) extracted resin with ethyl acetate for several days and obtained activity differences

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of up to about 5% of the standard between the resin and the product lignin plus wood, and could not find any reason for a difference in activity between lignin and cellulose from the same ring.

At the Ninth International Radiocarbon Conference in 1976, three different contributions (Cain 1979; Long *et al.* 1979; Olsson 1979) to recent tree-ring research were discussed, all with results indicating some percent increase of the ^{14}C activity of the extract compared with the cellulose for rings from the pre-bomb era. These rings were still sapwood when the bomb peaks in the summers of 1963 through 1965 resulted in excess atmospheric ^{14}C activity of about 100%. Cain and Suess (1976) and Cain (1979) chemically treated oak samples by acetone extraction (2 h), NaOH treatment (2%, boiling) rinsing three times with distilled water and final acidification. Some samples were converted to cellulose. Cain reported that the oak incorporates later carbon when sapwood is transformed to heartwood, and that the later carbon is removed only when the sample is bleached to pure cellulose. Cain (1978) claimed that incomplete removal of carbonaceous material (principally lignin) may lead to erroneous results.

Long *et al.* (1979) investigated heartwood rings of *Pinus longaeva*, which were still sapwood at the time of the pronounced bomb peaks. Although they used 4% HCl at 80° to 90°C overnight, 1% NaOH at 80° to 90°C overnight, HCl treatment and wash, they did not succeed in removing all of the extractives with high ^{14}C activity. An extraction by a benzene-ethanol mixture followed by a holocellulose extraction yielded a sample with the expected activity.

Olsson (1979) and Olsson, Klasson and Abd-El-Mageed (1972) presented results from sapwood of *Pinus sylvestris* (L.) using repeated NaOH treatments with thorough water washes between each step throughout the procedure. The tree was felled in 1969 and the investigated rings derived from 1945–1950. After one night, some bomb material was still left, but the final product, insoluble in NaOH, yielded an activity in good agreement with that of the cellulose and extracted wood processed by wood chemists. Olsson, El-Gammal and Göksu (1969) had earlier detected strongly increased activity in extract from wood deriving from 1935–1941. Olsson (1980) presented results obtained by a wood chemist on heartwood and sapwood of *Pinus aristata* submitted by C. W. Ferguson. The lignin content was slightly less than 30% of the wood in both cases, but the total extractives amounted to about 17 and 6%, respectively. The chemical composition of the extractives proved to be very different for heartwood and sapwood. A similar analysis of all components was made for *Pinus sylvestris*, for which the ^{14}C peak was detected for wood from 1935–1941 (Olsson, El-Gammal & Göksu 1969). Olsson (1980) demonstrated that a first NaOH treatment, lasting 30 min (1% at 80°C), removed the extractives to a very limited extent. The samples were then thoroughly washed with water before the next NaOH treatment. As a typical case for the Uppsala Laboratory, the wash was repeated four times, to "almost neutral." Both second and third NaOH treatments lasted overnight, but the yield and ^{14}C results indicate that the third extraction was not necessary. In the following research, the number of NaOH extractions (with a thorough wash between) was limited to two. The yield, if any, of the second NaOH extraction was extremely low at the present investigation.

Glad and Nydal (1982) studied the efficiency of a very short NaOH treatment using the heartwood of *Pinus sylvestris*. They removed hardly any of the extractives with a 5-min NaOH treatment.

Tans, de Jong and Mook (1978) applied an AAA treatment with the NaOH treatment for 24 h at 80°C (4%), and stressed that it was followed by a thorough washing with demineralized water. They also prepared alpha-cellulose and compared the results with those from the whole wood, wood treated with 4% HCl at 80°C for 24 h and various components of the resin. Because they used oak wood from 1962 and 1963 in a tree with sapwood-to-heartwood transition in 1961–1964,

they could study the activity increase during the two years and any peculiarities at the transition. These authors also published a value for the excess ^{14}C after AAA treatment of wood from 1971, which is significantly higher than after cellulose extraction (512.5 ± 3.6‰ and 496.4 ± 2.8‰). Similar differences were obtained for 1962 and 1963. An explanation could be "that the AAA treatment attacks early wood more than it does late wood (Tans, de Jong & Mook 1978: 235)." These authors offer an alternative explanation, that lignin remains after the AAA treatment, and it formed later in the year than cellulose.

Dai and Fan (1986) studied the excess ^{14}C at different latitudes (68°N, 47°N and 27°N) using tree rings from 1962 to 1967. The samples were said to have been repeatedly treated according to routine HCl-NaOH-HCl procedure. No further details were given. The northernmost sample (white spruce) from the Mackenzie Delta, Canada, yielded a Δ value for 1962 far above that for atmospheric CO_2 obtained for Svalbard and Abisko at 78° and 68°N (Olsson & Klasson 1970). The 1966 value also appears unexpectedly high in comparison with our findings. Pending results of reliability tests of the total experimental procedure, and especially detailed descriptions of the sample handling before the combustion, their few results must be treated with care.

Grootes *et al.* (1989) studied the increase of the excess at 47°N (Washington) using the equal increments of a Sitka spruce for each of the years, 1962 to 1964, and even tried to ascribe each sample to the proper growth period by taking the expected varying radial growth rate into consideration. The values for 1962 are almost the same from early May to the end of September. Values steadily increase for 1963 and are almost the same for all samples from 1964 as in August–September 1963. Thus, the peak seen in atmospheric CO_2 results from northern latitudes is not detectable in the Sitka spruce. The Δ values are also significantly lower than those from comparable sites (same latitude). Different explanations are thoroughly discussed and the conclusion reached is that CO_2 from biospheric decay must have strongly affected ^{14}C activity.

The yields of the chemical treatments in the present investigation were determined only on a semiquantitative basis. However, it is clear that early wood yielded no or almost no NaOH-soluble fraction. When this small fraction could be recovered, it was less than about 3% of the total wood. The late wood yielded a soluble fraction for all rings but less than about 10%.

Because lignin is formed later than the cellulose (Fritts 1976: 67, 100), the cellulose should have a lower activity than the insoluble fraction when the activity is rising. If, however, resin is used for the formation of cells, a memory effect will influence the ^{14}C content. All the extractives from late wood are more ^{14}C active from 1951 to 1957 than the other fractions, but the insoluble fractions recovered from 1953 and 1954 have lower ^{14}C activities than the cellulose and are the two lowest of the present 97 Δ^{14}C values.

SAMPLE MATERIAL

We used an 80-year-old oak (*Quercus robur*) from a suburb of Uppsala (60°0′N, 17°38′E) cut in 1981. The widths of the tree rings ranged from 1 to 10 mm, where the early (spring) wood shows a fairly constant value of 0.5 to 1 mm, whereas the width of the late wood part shows a larger variation. The sapwood consisted of 13 to 14 rings. The transition to the heartwood was thus not observed in the same ring around the log.

We obtained the Δ^{14}C atmospheric CO_2 record for air collected at Abisko (68°20.5′N, 18°49.3′E) (Fig.1) in northern Sweden, and in parts published earlier (Olsson & Karlén 1965; Stenberg & Olsson 1967; Olsson & Klasson 1970). Results from Abisko are missing for most of the year 1965. The atmospheric values from Svalbard (78°N), however, indicate a maximum of about 810‰ as a realistic value for 1965.
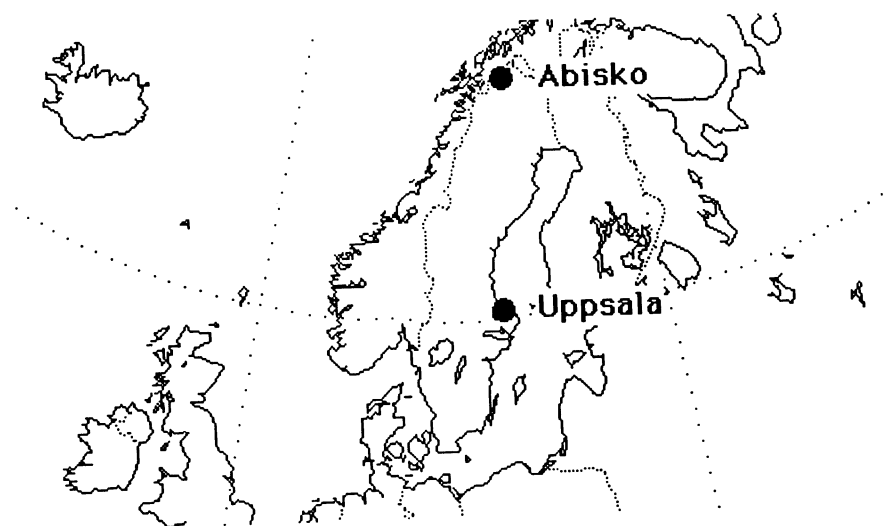
Fig. 1. Map of Sweden showing the sampling sites at Uppsala and Abisko

CHEMICAL PRETREATMENT IN THE PRESENT INVESTIGATION

For this study, we followed the standard chemical pretreatment technique used in the conventional radiocarbon laboratory in Uppsala for many years. Figure 2 shows a flow diagram of the different steps where we define the three fractions, INS, SOL and CELL, relevant to the discussion of the results. We carefully performed washes with distilled water between the different steps. The solution in the bleaching step consisted of 8 g $NaClO_2$, 11 ml 1 M HCl in 3 dl H_2O.

We pretreated between 200 and 600 mg of early wood and 500 mg to 2.5 g of late wood. The average yields for the fractions, INS, SOL and CELL, were 43%, 1%, 22% and 51%, 3%, 34% for the early and the late wood, respectively. A notable feature was the relatively small SOL fractions for the early wood, which made this fraction measurable on only 9 out of the 16 tree rings studied. A finer division was made for the rings from 1963 and 1964, where the early wood was separated and the late wood split into three equal-sized sections. We selected this wood from a radial section a few degrees (~3 cm) apart from that used for all other measurements. A duplicate sample from this section was also prepared for the early wood from 1965.

The organic material obtained in the pretreatment was combusted with CuO at 800°C for *ca.* 10 min. Conversion of CO_2 to graphite was thereafter conducted with iron and hydrogen, according to a modified procedure (Hut, Östlund & van der Borg 1986).

MEASUREMENTS

We made the measurements at the Svedberg Laboratory in Uppsala where we used the EN-tandem for the $\Delta^{14}C$ determination (Possnert 1990). We normalized the results to the old NBS oxalic-acid standard and made routine internal quality checks with material of known activity. In total, 97 samples were measured. An absolute accuracy of 1% was obtained with a beam time of about 40 min per sample, including the standard. The mass fractionation, $\delta^{13}C$, was measured independently with a Varian Mat mass spectrometer.
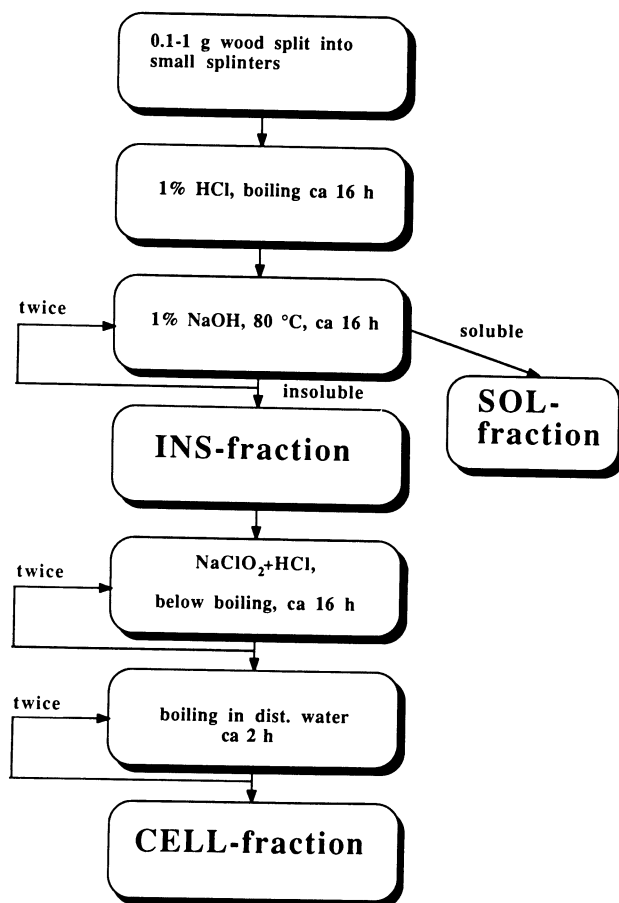
Fig. 2. Flow diagram of the pretreatment chemistry procedure used

RESULTS

The tree rings represent four different time periods (I—1938–1939; II—1951–1956; III—1962–1967; and IV—1980–1981) and three different chemical fractions (INS, SOL and CELL) for early and late wood for each year, respectively.

The measured $\delta^{13}C$ values were almost the same for all samples with a slightly lower value for the SOL fraction, $-25.9 \pm 0.7\%o$ (late wood, n = 15) vs. PDB, compared with $-24.2 \pm 1.2\%o$ (CELL late, n = 15), $-24.7 \pm 0.8\%o$ (INS late, n = 16), $-24.1 \pm 0.6\%o$ (CELL early, n = 5) and $-24.5 \pm 1.2\%o$ (INS early, n = 16). The SOL fraction yield for the early wood was unfortunately too low to permit any stable isotope measurements.

Figures 3 A–C and 4 and Table 1 show all the $\Delta^{14}C$ results. Generally the SOL fraction clearly deviates from the INS and CELL fractions as expected *a priori*, whereas a smaller deviation exists between the INS and CELL parts.

The SOL fraction exhibits systematic behavior in the different periods. We obtained about 40%o lower values than for the INS and CELL fractions for 1938–1939. The opposite is observed for the period, 1951–1956, when the values are up to 80%o higher compared with CELL and INS, except for the two early wood values, which are close to the CELL fraction. For the bomb-peak maximum, all SOL values are below the corresponding CELL results. It is striking that the SOL
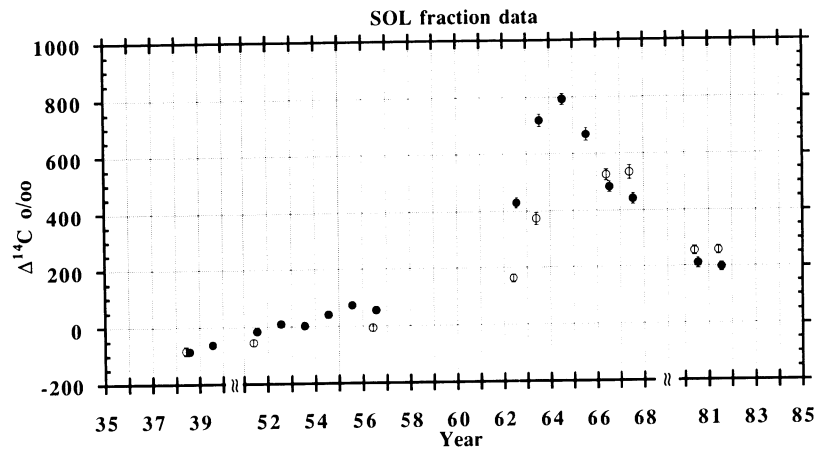
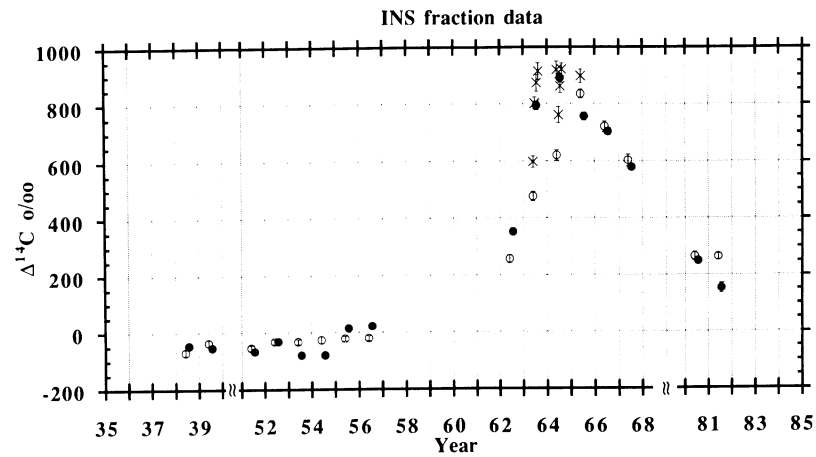Fig. 3A. Δ^{14}C results for the different chemical fractions; ○ — early wood and ● — late wood; SOL fraction

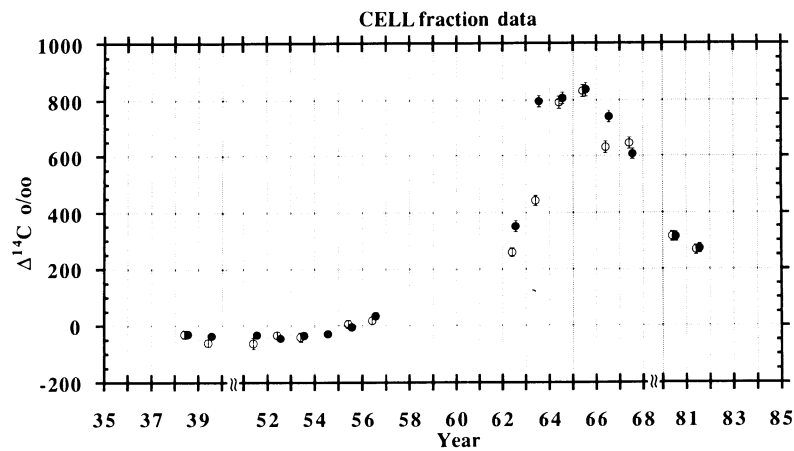

Fig. 3B. INS fraction; x — refined partitioning, see text

Fig. 3C. CELL fraction

TABLE 1. $\Delta^{14}C$ and $\delta^{13}C$ Results for all Tree Rings and all Fractions Studied

Lab. no.	Year	Sample	$\delta^{13}C‰$	$\Delta^{14}C‰$	±
Ua-2137	1938	EW*, SOL		−82	16
Ua-2138	1951	EW, SOL		−54	12
Ua-2139	1956	EW, SOL		−5	12
Ua-2140	1962	EW, SOL		162	13
Ua-2141	1963	EW, SOL		373	21
Ua-2142	1966	EW, SOL		526	19
Ua-2143	1967	EW, SOL		534	24
Ua-2144	1980	EW, SOL		255	15
Ua-2145	1981	EW, SOL		257	15
Ua-2146	1938	EW, INS	−26.0	−68	11
Ua-2147	1939	EW, INS	−24.6	−35	11
Ua-2148	1951	EW, INS	−23.9	−52	9
Ua-2149	1952	EW, INS	−25.2	−31	8
Ua-2150	1953	EW, INS	−23.3	−32	8
Ua-2151	1954	EW, INS	−24.8	−25	12
Ua-2152	1955	EW, INS	−25.5	−21	8
Ua-2153	1956	EW, INS	−23.3	−20	8
Ua-2154	1962	EW, INS	−24.5	258	13
Ua-2155	1963	EW, INS	−26.6	476	17
Ua-2156	1964	EW, INS	−23.4	621	18
Ua-2157	1965	EW, INS	−23.8	835	15
Ua-2158	1966	EW, INS	−23.5	721	18
Ua-2159	1967	EW, INS	−23.4	603	21
Ua-2160	1980	EW, INS	−24.2	265	15
Ua-2161	1981	EW, INS	−26.9	265	10
Ua-2162	1938	EW, cell	−25.0	−30	15
Ua-2163	1939	EW, cell	−24.6	−60	12
Ua-2164	1951	EW, cell	−23.4	−61	19
Ua-2165	1952	EW, cell	−24.1	−33	12
Ua-2166	1953	EW, cell	−23.7	−40	16
Ua-2167	1955	EW, cell		6	14
Ua-2168	1956	EW, cell		19	12
Ua-2169	1962	EW, cell		259	15
Ua-2170	1963	EW, cell		443	18
Ua-2171	1964	EW, cell		792	22
Ua-2172	1965	EW, cell		832	21
Ua-2173	1966	EW, cell		631	20
Ua-2174	1967	EW, cell		646	20
Ua-2175	1980	EW, cell		315	17
Ua-2176	1981	EW, cell		267	16
Ua-2177	1938	LW**, SOL	−26.0	−84	12
Ua-2178	1939	LW, SOL	−24.8	−60	11
Ua-2179	1951	LW, SOL	−26.7	−15	11
Ua-2180	1952	LW, SOL	−26.0	10	11
Ua-2181	1953	LW, SOL	−25.8	3	11
Ua-2182	1954	LW, SOL	−26.7	41	11
Ua-2183	1955	LW, SOL	−24.6	73	12
Ua-2184	1956	LW, SOL		55	11
Ua-2185	1962	LW, SOL	−26.8	429	18

TABLE 1. (Continued)

| Lab. no.	Year	Sample	$\delta^{13}C‰$	$\Delta^{14}C‰$	±
Ua-2186	1963	LW, SOL	−25.5	719	22
Ua-2187	1964	LW, SOL	−25.1	794	18
Ua-2188	1965	LW, SOL	−25.9	669	21
Ua-2189	1966	LW, SOL	−25.8	483	18
Ua-2190	1967	LW, SOL	−26.0	441	18
Ua-2191	1980	LW, SOL	−26.6	211	18
Ua-2192	1981	LW, SOL	−25.8	198	16
Ua-2193	1938	LW, INS	−25.0	−45	8
Ua-2194	1939	LW, INS	−23.4	−53	8
Ua-2195	1951	LW, INS	−26.0	−65	8
Ua-2196	1952	LW, INS	−23.9	−31	8
Ua-2197	1953	LW, INS	−24.5	−78	8
Ua-2198	1954	LW, INS	−24.2	−78	8
Ua-2199	1955	LW, INS	−23.4	14	8
Ua-2200	1956	LW, INS	−25.1	22	8
Ua-2201	1962	LW, INS	−25.4	352	12
Ua-2202	1963	LW, INS	−24.4	794	15
Ua-2203	1964	LW, INS	−25.1	890	15
Ua-2204	1965	LW, INS	−25.9	756	13
Ua-2205	1966	LW, INS	−24.3	704	12
Ua-2206	1967	LW, INS	−24.6	578	12
Ua-2207	1980	LW, INS	−24.9	249	11
Ua-2208	1981	LW, INS	−25.3	152	17
Ua-2209	1938	LW, cell	−25.0	−29	9
Ua-2210	1939	LW, cell	−25.4	−35	8
Ua-2211	1951	LW, cell	−25.1	−31	8
Ua-2212	1952	LW, cell	−25.0	−44	11
Ua-2213	1953	LW, cell	−24.1	−34	7
Ua-2214	1954	LW, cell		−28	10
Ua-2215	1955	LW, cell	−22.3	−4	12
Ua-2216	1956	LW, cell	−23.7	35	12
Ua-2217	1962	LW, cell	−25.7	351	19
Ua-2218	1963	LW, cell	−23.3	795	19
Ua-2219	1964	LW, cell	−21.8	807	20
Ua-2220	1965	LW, cell	−23.7	837	23
Ua-2221	1966	LW, cell	−23.9	741	20
Ua-2222	1967	LW, cell	−22.9	608	18
Ua-2223	1980	LW, cell	−24.9	314	17
Ua-2224	1981	LW, cell	−25.3	272	16
Ua-2225	1963	EW, INS	−26.0	598	20
Ua-2226	1963	LW, INS 1	−23.8	802	21
Ua-2227	1963	LW, INS 2	−23.9	873	29
Ua-2228	1963	LW, INS 3	−24.7	914	29
Ua-2229	1964	EW, INS	−24.4	919	30
Ua-2230	1964	LW, INS 1	−24.0	762	28
Ua-2231	1964	LW, INS 2	−23.0	862	23
Ua-2232	1964	LW, INS 3	−23.8	920	22
Ua-2233	1965	EW, INS	−24.3	897	23

*EW = early wood
**LW = late wood
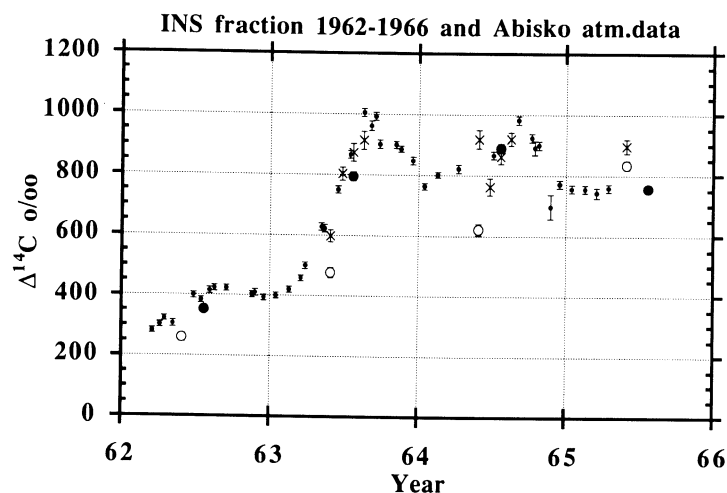
Fig. 4. Δ^{14}C results for the INS fraction; O — early wood, ● — late wood; and x — refined partitioning, see text; • — Abisko atmospheric activity record for 1962–1966

from the early wood is lower than that for the late wood when the atmospheric activity shows an increase, whereas the opposite is true for the decrease. It is tempting to interpret this as a memory effect caused by nutrients in the roots used for the early wood, because part of the wood forms before the buds form. This suggestion is supported by measurements on two sap samples from birch trees from 1985 and 1986, which indicate a slightly higher activity than the contemporaneous atmosphere. A third sap sample from 1988 agreed, however, with the atmosphere, which shows the complexity in a tree's metabolism.

The INS fractions behave similarly to the CELL fractions, except for the late wood in 1953 and 1954, when the values are statistically lower than for the corresponding early INS and CELL samples. Of special interest is the detailed division of the years 1963 and 1964. This refined partitioning followed by the preparation of the INS fraction made it possible to follow activity changes during the summers of these two years (Fig. 4). In the case of the early wood, two independent measurements from the two slightly different radial sections can be compared. The slight difference between the two preparations can be explained by and illustrates the problem of properly separating only the early wood, not including a small percentage from the neighboring late wood sections, since the rings are thin and the cell structure gradually changes. Some wood rays perpendicular to, and small stepwise radial displacements of, the rings must also be considered in this context. Good agreement is generally achieved with the atmospheric data, although some of the INS values are significantly lower.

Finally, the CELL fraction shows an excellent concordance with the atmospheric record (Fig. 5), although the value for the late wood integrates the summer activity. The low activity for the early wood in 1962 and 1963 is of special interest, however.

DISCUSSION

Our primary criterion for judging the quality of our pretreatment chemistry was the agreement obtained between the different tree fractions and the atmospheric activity. It is obvious from the present investigation that the CELL fraction seems to be the most appropriate choice in the procedure, although the agreement for a thoroughly pretreated INS fraction including high temperatures, long preparation time and careful washing, is almost the same as for the CELL fraction. Thus, the AAA treatment normally used in Uppsala seems to remove the resin satisfactorily, even though the SOL extracts have moved radially. One alternative explanation,
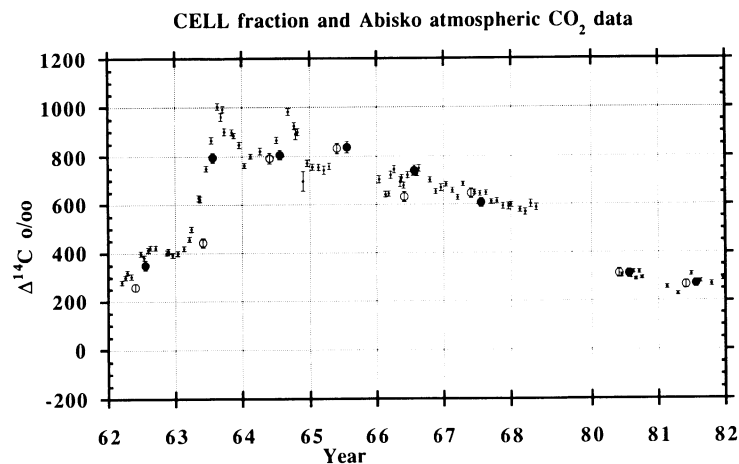
CELL fraction and Abisko atmospheric CO$_2$ data

Fig. 5. Δ^{14}C results for the CELL fraction o — early wood; ● — late wood; • — Abisko atmospheric activity record

besides the problem of cutting the tree rings accurately, for the somewhat different results for the almost duplicate samples during the years 1963–1965 (Fig. 4) may be that the samples from the section used for the partitioning were cut and pretreated seven years later. The wood was then considerably drier, possibly changing the chemical composition. The INS fraction naturally contains some compounds that will be removed during the CELL preparation, when the color is changed and ~50% of the material is dissolved. The origin of these compounds is not well known at present.

As a measure of the past atmospheric activity in central Sweden, the data from Abisko in northern Sweden, were chosen. Although the distance from Uppsala is about 1000 km north, the same activity seems to be valid for the air in the Uppsala region. This has clearly been demonstrated by comparison of air data as well as activity in plant material from the Smara site, 25 km east of Uppsala, from the year 1979 until now (Olsson, ms. in preparation). A local industrial effect in the tree-ring data set cannot be ignored, however, because the oak has grown in a suburb of the town. For the years, 1938–1939, this effect is <2–4%, if the Δ^{14}C values obtained (INS and CELL) are compared with similar data from other researchers. Surprisingly, this local industrial effect is, in view of the increasing combustion of fossil fuels in the Uppsala area in recent decades, still true for the data obtained for 1980 and 1981. Slightly lower activity (~2%) compared with the con-temporary atmosphere has been obtained in plant material from 1984 from the downtown area, where the traffic is heavy. Two tomato plants grown in 1968 in buckets on the balcony outside the ^{14}C laboratory in the center of Uppsala and two leaf samples from Järlåsa also indicate a local industrial effect of 1–2 ± 1%, compared with atmospheric CO$_2$ from Abisko and Nordkapp (Nydal & Løvseth 1983). If an industrial effect is present, it should be more pronounced during the late autumn, winter and early spring (September to mid-May), when heating is needed, and thus, of minor influence during the growing period of the oak. The air masses over Sweden are also changed rapidly (hours to days) by the west wind from the Atlantic.

Except for the early wood, which is formed during a few weeks at the end of May, it is difficult to assign a specific growing time for the late wood, because many external factors, local, as well as regional and temporal/climatological are influential. The total growing period is normally from June to the end of August. We see no clear evidence for any delay between the atmosphere and the CELL and INS fraction activity on a time scale of weeks, as earlier reported by Grootes *et al.* (1989). We observe a memory effect in the early wood during the years, 1962–1963, with signifi-cantly lower activity than in the atmosphere.

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