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[14]C-Analysis of calcite coatings in open fractures from the Klipperås study site, Southern Sweden

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FROM THE KLIPPERÅS STUDY SITE, SOUTHERN SWEDEN

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ABSTRACT

Carbonate samples from open fractures in crystalline rock from the Klipperås study site have been analysed for their ^{14}C contents using accelerator mass spectrometry. This technique makes it possible to analyse very small carbonate samples (c. 1 mg C). The analyses show low but varying contents of ^{14}C. However, contamination by CO_2 have taken place affecting small samples more than others. Attempts have been made to quantify the contamination and thus evaluate the analyses of the fracture samples.

The obtained low ^{14}C values can be due to:

1) An effective retention of ^{14}C by sorption/fractionation forcing ^{14}C onto the calcite surfaces in the near-surface zone which means that the ^{14}C contribution to the deeper levels is diminished or 2) the penetration depth of surface groundwater is very shallow. The former is suggested as more probable based on evaluations of the hydrochemical conditions (Smellie et al 1987) and the fracture mineral studies (Tullborg 1986).

CONTENTS

1. INTRODUCTION

^{14}C-dating has been successful regarding determination of the age of precipitation of e.g. post-glacial carbonate minerals. However, it is suspected that ^{14}C (present in the HCO_3) within the groundwater can be sorbed on carbonate minerals coating the fracture walls. Thus, ^{14}C analyses of fracture coating calcites will probably shed some light on the water/mineral interactions along the groundwater flow paths and to what depth modern carbonate has been transported by the groundwater. Although there are quite a lot of difficulties to interprete the result of a ^{14}C-analysis from a sample of fracture filling carbonate, due to a complex process of leaching/precipitation and sorption, a significant value of ^{14}C in a fracture filling is very informative concerning the influence of modern recharge water.

The accelerator mass-spectrometry technique makes it possible to analyse very small samples. This is important when analysing fracture minerals from drillcores where the amount of calcite is very limited.

The Klipperås study site, (investigated within the SKB Program for radwaste disposal) was considered suitable for this study because:

- It is a carefully investigated area which is penetrated by 14 core-drillings (down to 250 m depth or more).
- The fracture mineralogy has been previously studied (Tullborg, 1986) and the results point to a relatively extensive downward penetration of surface groundwater.
- A relatively high content of ^{14}C (72.5+/-2.3 pMC) in calcite from a crush zone at 100 m vertical depth was measured in a pilot study (Tullborg 1986b).

2. BRIEF DESCRIPTION OF THE KLIPPERÅS STUDY SITE

As a result of SKB's studies, the Klipperås area, situated 45 km west of Kalmar, has been carefully investigated considering
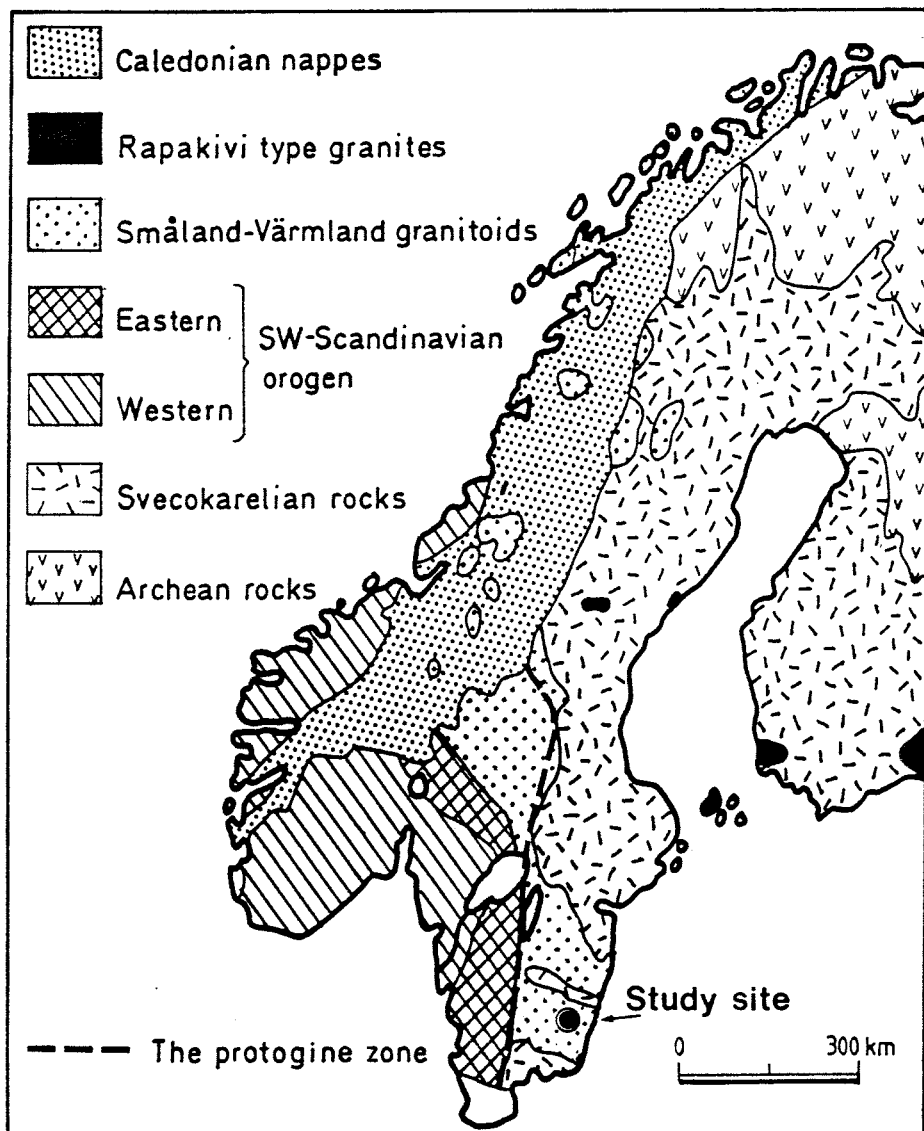
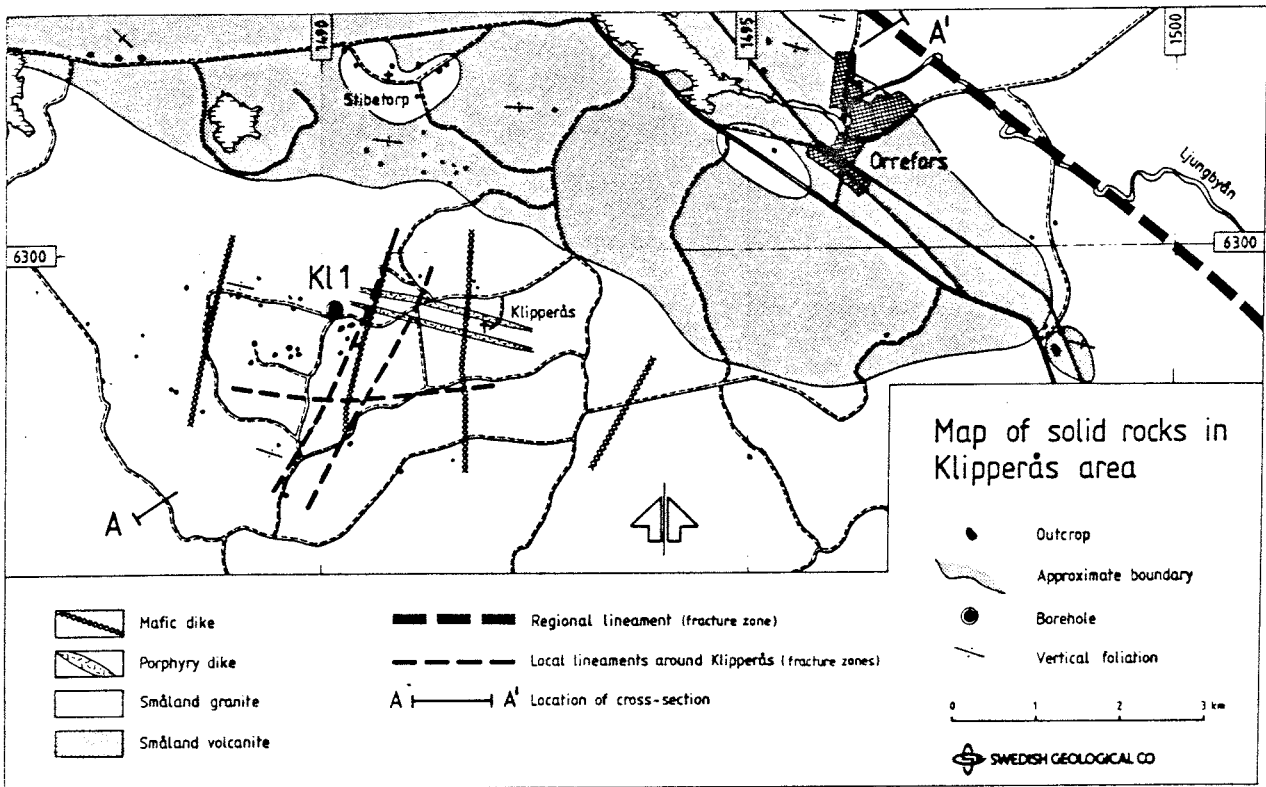Figure 1. Location of the Klipperås study site.

Figure 2A. Map of solid rocks in the Klipperås study site and
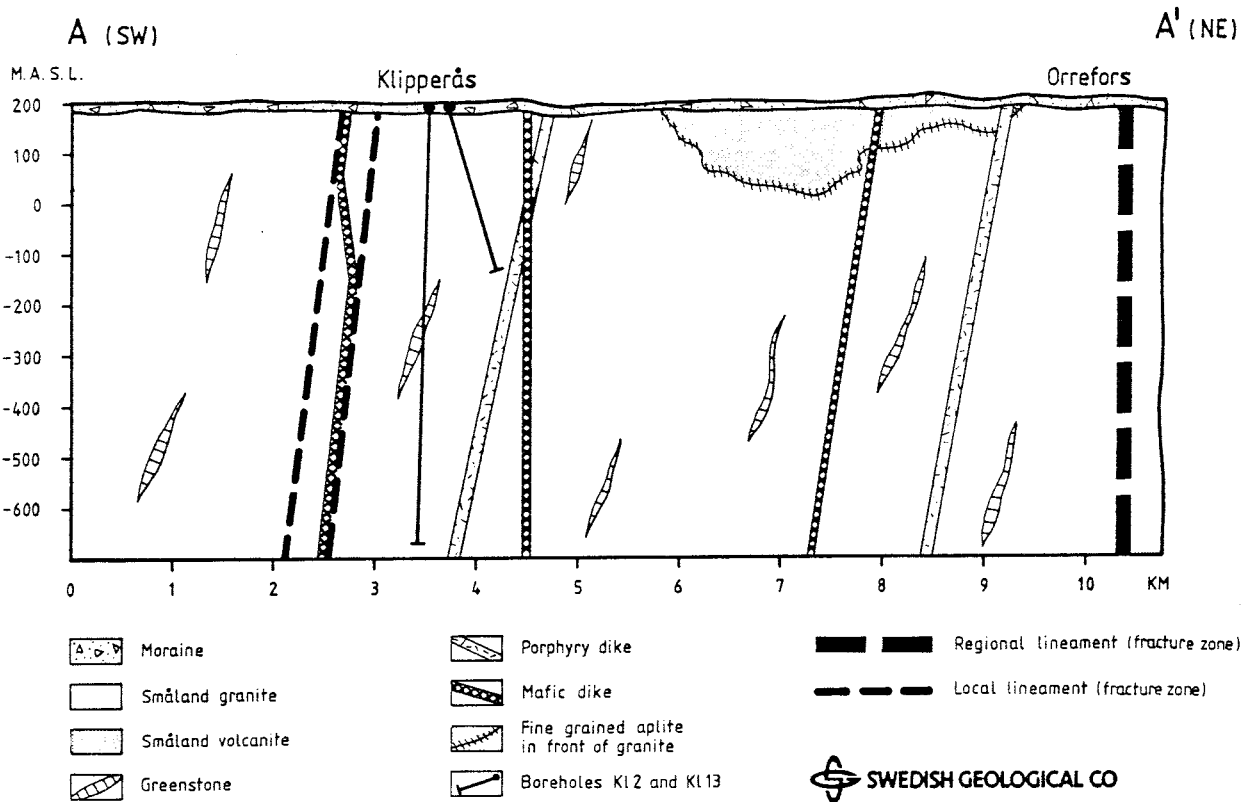surroundings (Olkiewicz & Stejskal 1986).

Figure 2B. Schematic vertical cross section of the Klipperås
area (Olkiewicz & Stejskal, 1986).

geology (Olkiewicz & Stejskal, 1986) and hydrogeology (Gentz-
schein, 1986). The location of the study site is shown in figure
1.

Geology

Geologically, the area is situated within the Svecofennian belt
which, at the test site, is represented by serorogenic granites
(Småland-Värmland granites) and acid volcanics (Småland por-
phyries. The topography of the area is flat with an almost com-
plete till cover which makes bedrock mapping extremely difficult.
However, based on the drilling programme as well as geophysical
interpretations (Stenberg & Sehlstedt, 1986) a geological map of
the study site has been constructed (Fig.2) (Olkiewicz & Stej-
skal, 1986). A grey-red, medium-grained and homogenous granite,
usually called "Växjö granite", dominates the study site. In
parts a weak E-W-trending foliation can be seen in the granite.

In addition to the granite and acid volcanics, different types of
metabasites are found within the drillcores. Also several dike
generations have been identified; N-S and NE-SW-trending Svecon-
orwegian dolerite dikes as well as presumably older composite
dikes of porphyry and metabasite mostly trending c. E-W.

Tectonically, the study site at Klipperås is characterized by
several distinct zones, some of which coincides with the more or
less vertical dikes of dolerite (Fig.3). Most of the lineaments
indicated by geophysical and hydrogeological measurements are
vertical to subvertical, although exceptions occur (e.g. the
horizontal zone (H1) at c. 785 metres depth as well as zone 4
which dips 65° to SE).

Hydrogeology

Hydrogeological investigations including water balance studies,
groundwater table registrations and water injection tests were
reported by Gentzschein (1986). The water injection tests showed
relatively high values of conductivity in the uppermost parts of
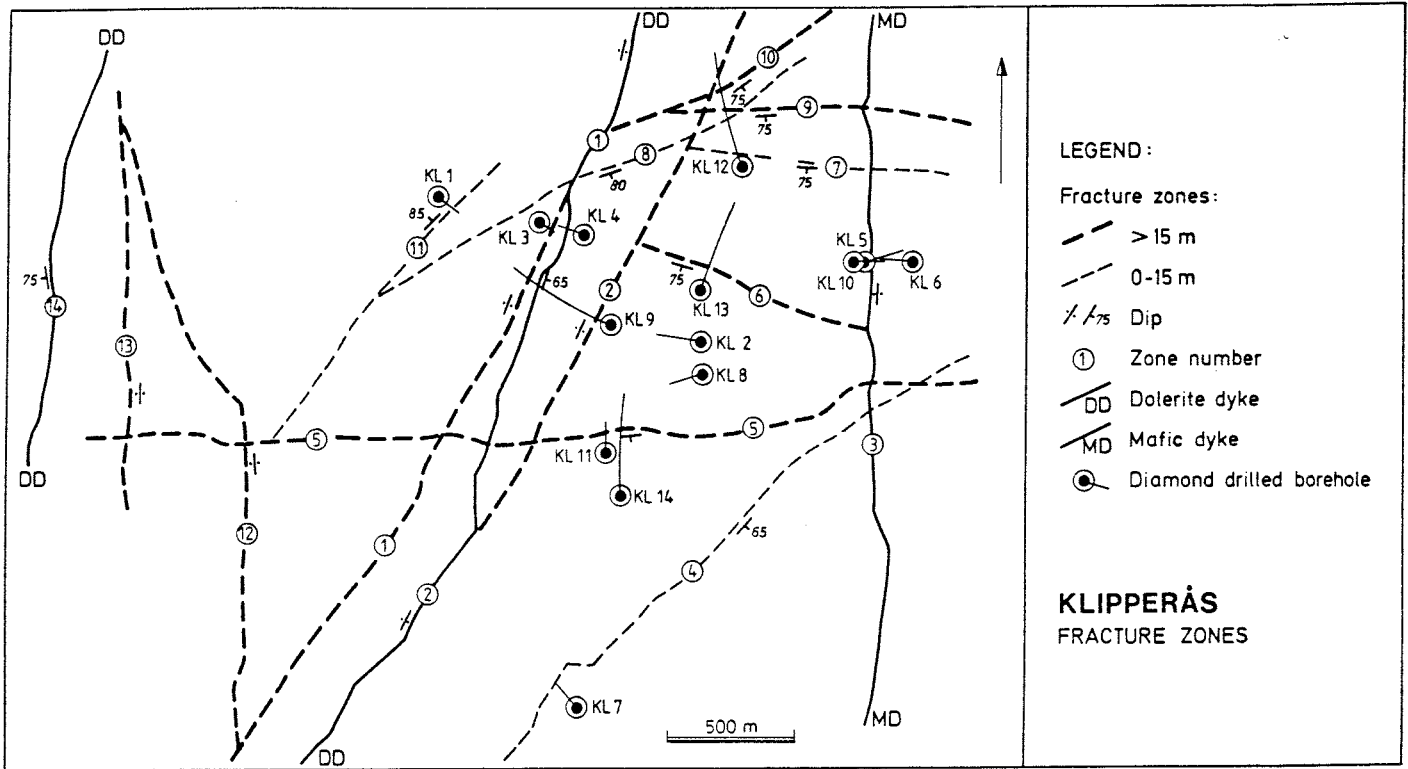the boreholes (10^{-9}m/s to 10^{-5}m/s). Also at depth, high conduc-

Figure 3. Location of the fracture zones and mafic dykes within
the Klipperås study site (Olkiewicz & Stejskal, 1986).

tivity values were recorded, but below 500 m values of 10^{-11}m/s to 10^{-9}m/s dominate. The flat topography of the area implies a low hydraulic gradient. This was also confirmed by piezometric measurements in the boreholes. With the exception of one section, the pressure differences was lower than 5 m water column. The water injection tests as well as an isotope investigation of fracture filling calcites (Tullborg, 1986) point to generally lower hydraulic conductivities in the mafic rock types than within the granite.

Water chemistry

The hydrochemical conditions have been evaluated by Smellie et al. (1987). According to that study, representative water samples were obtained at three levels, all from different drillholes. Sampled depths are Kl 2:329m, Kl 1:406-564m and KL 9:696m. Two groundwater types were found; groundwaters of near-surface origin (Ca-HCO$_3$-type; Kl 2:329m and Kl 9:696m) and of intermediate origin (characterized by decreased contents of Ca, Mg and HCO$_3$ in comparision with the near-surface waters; Kl 1:406-564m). No saline waters were detected. Reducing conditions as well as a stable pH between 7.6 and 8.3 were common for all the samples. Calculations of saturation indices for carbonate minerals show that most carbonate minerals were close to equilibrium.

As the groundwater of near-surface water were found as deep as 600 m vertical depth (696 m in drillhole Kl 9) it indicates that: "much of the groundwater collected during sampling has originated from groundwater reservoirs at substanstially higher levels than those depth actually sampled, and their presence at greater depth is due to downward hydraulic gradients along active fracture zones, many of which have been shown through drilling to be mainly sub-vertical in orientation through the bedrock" (Smellie et al., 1987).

^{14}C-analyses of two of the groundwater samples which are lacking tritium have been carried out. One of the samples represents the intermediate groundwater type (Kl 1:406) whilst the other represents the near-surface water type (Kl 9:696). The analyses gave

2.91 and 2.15 pmC (28 375 y and 30 795 y) respectively (table 1).
This is considered to be very low in groundwaters of near-surface
type. However, the low ^{14}C-content in the groundwater could be a
result of several processes like dissolution and precipitation of
fracture filling calcites as well as sorption of ^{14}C on the
carbonate coating the fracture walls along the water flow paths.

Table 1. ^{14}C-analyses of groundwater samples fram Klipperås
(Smellie et al., 1987)

Sample	pmC
Kl 1:406 m	2.91
Kl 9:696 m	2.15

3. SAMPLING

The intention has been to sample fracture coating calcites from
open, water conducting fractures at different depths within
fracture zones and single fractures. Also samples from the
sections sampled for groundwater have been included in the study
(Kl 1: 436.25, Kl 1 435.3 and Kl 9: 705.6). Samples along the
drillcore Kl 7 have been collected in order to complement the
already analysed single sample from the fracture zone penetrated
by the same drillcore (at 125 m core length). Samples have also
been taken from drillcores Kl 1 and Kl 12.

The hydraulic conductivity (not measured in drillcore Kl 7) as
well as the frequency of calcite and/or rust coated fractures
versus depth for each drillhole are shown in figures 4 to 7.
As can be seen in the figures, there is a decrease in the fre-
quency of calcite coatings close to the surface generally
corresponding to an enrichment of Fe-oxyhydroxide. This means
that oxidising water with low pH percolates to depths varying
from ten to one-hundred metres in the area.

Thin sections have been made of the samples collected. The
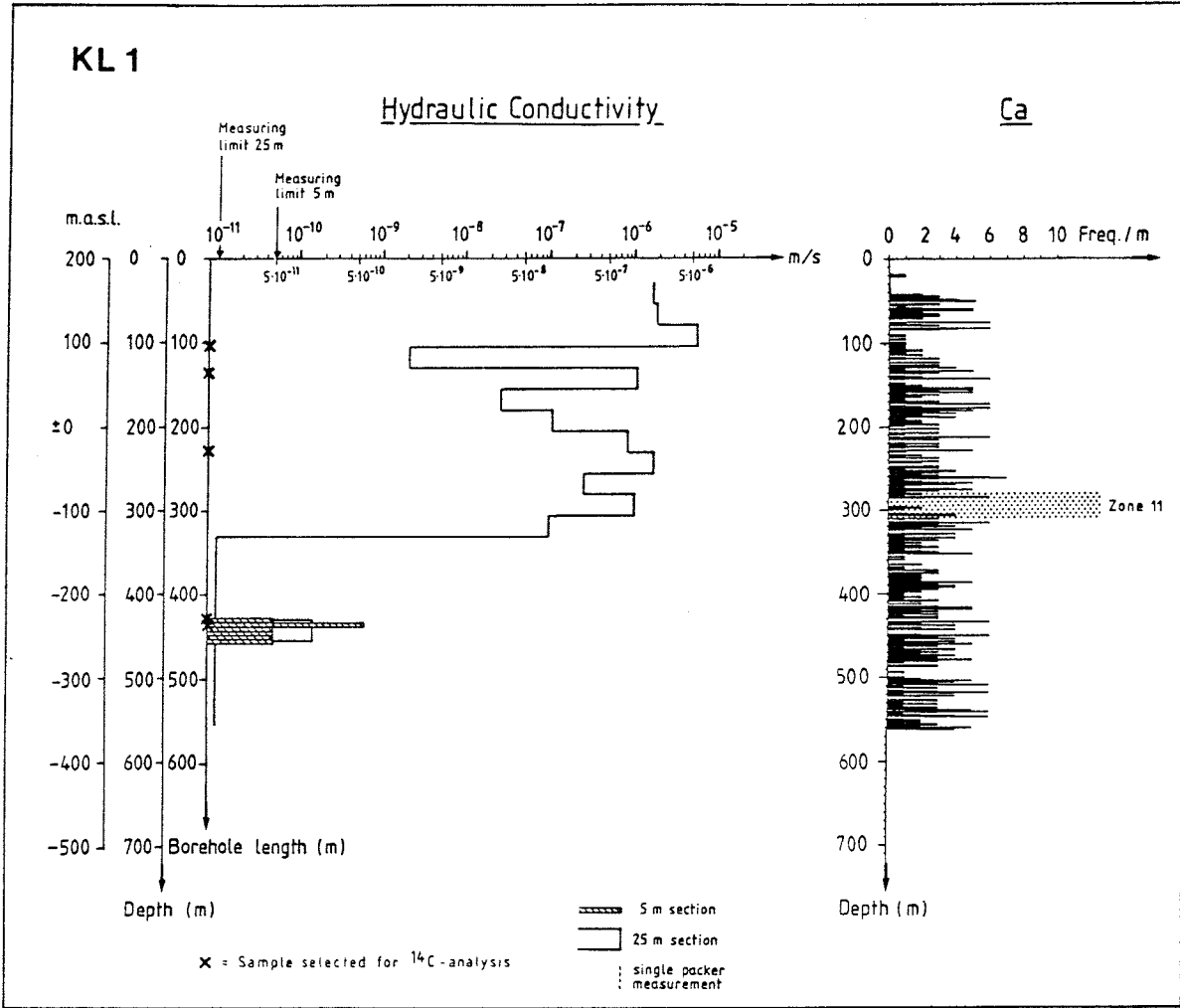
Figure 4.

Hydraulic conductivity and frequency of calcite coated fractures (Ca) in the drillcore KL 1. Sample selected for ^{14}C-analysis is marked with X.
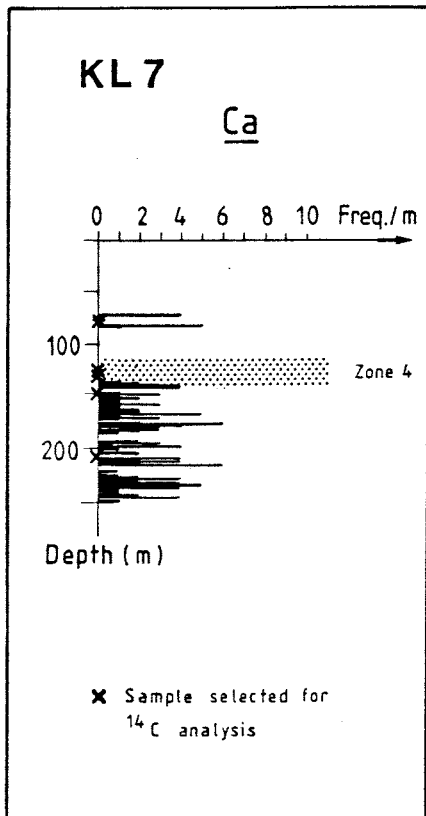
Figure 5.

Frequency of calcite coated fractures (Ca) in drillcore Kl 7. Hydraulic conductivity measurements have not been carried out in this drillhole. Location of sample selected for ^{14}C-analysis is marked with X.

Figure 6.   Hydraulic conductivity as well as frequency of
calcite (Ca) and Fe-oxyhydroxide (Fe) in drillcore
Kl 9. Location of sample selected for ^{14}C-analysis is
marked with X.

Figure 7. Hydraulic conductivity as well as frequency of calcite (Ca) and Fe-oxyhydroxide (Fe) coated fractures in drillcore Kl 12. Location of sample selected for ^{14}C-analysis is marked with X.

calcite considered for ^{14}C-analysis, within this project, have to be the very last mineralisation within a fracture and should show low frequency of twinning. Fina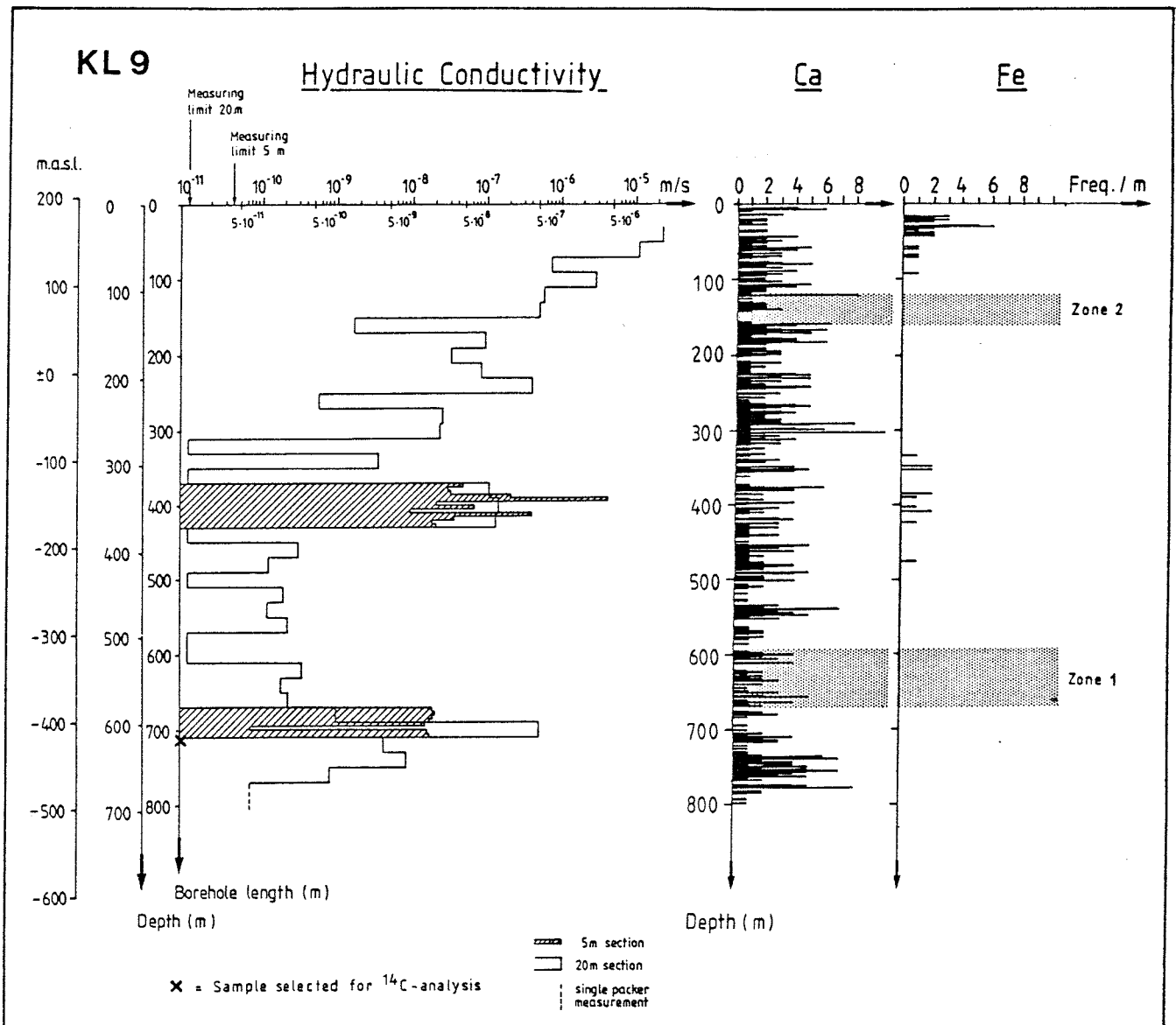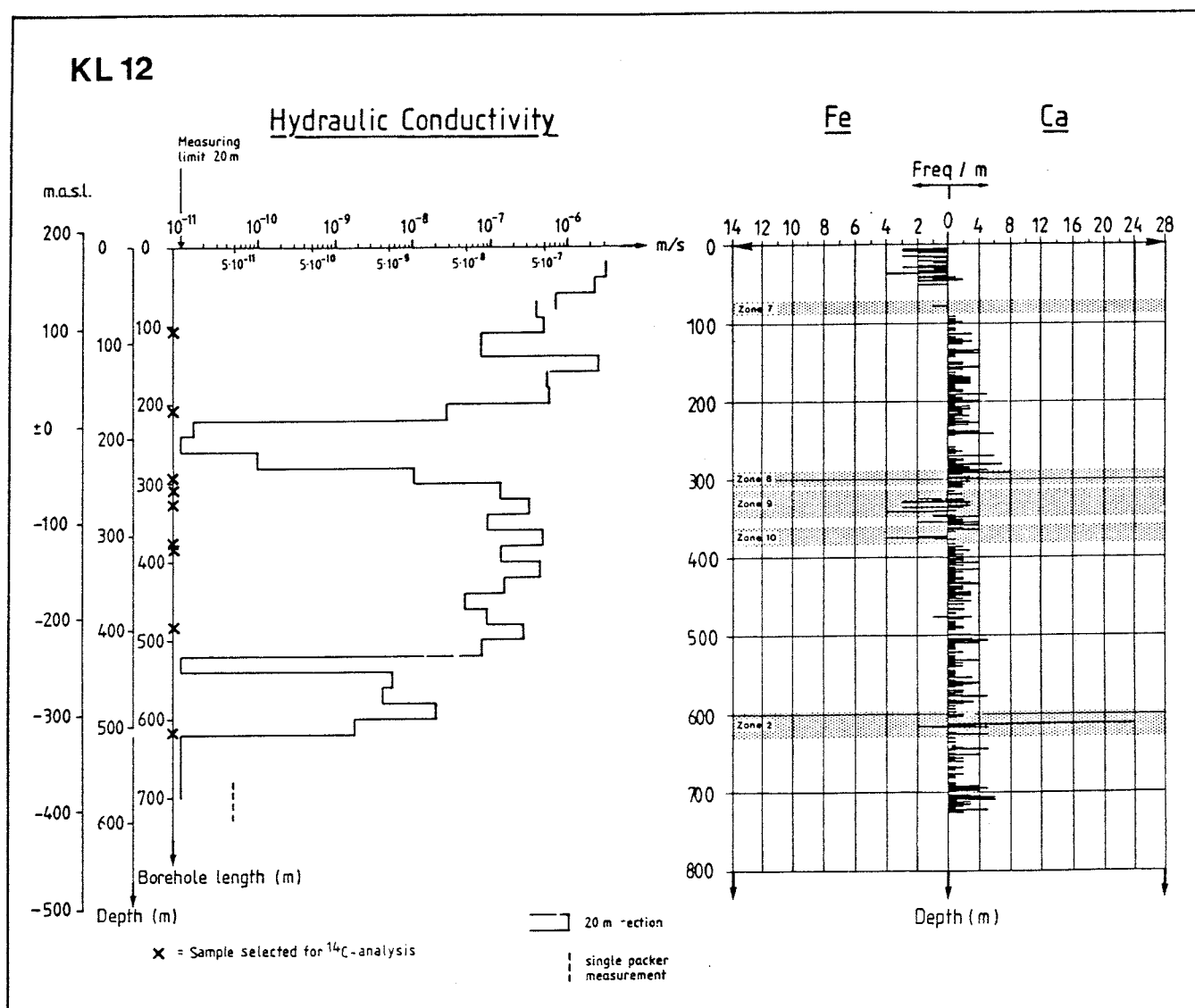lly, 23 sample were selected for ^{14}C-analyses. The sample preparation was carried out at the Tandem Accelerator Laboratory, University of Uppsala.

4. SAMPLE PREPARATION AND ANALYSES

No extraordinary precautions were made neither in the storage of the samples nor in the pretreatment procedure compared with the normal laboratory carbonate treatment. The material was submitted to the laboratory as cores of the rock with the open fissure surfaces untreated. The calcite was visually not easily distinguished from the other minerals. Each surface of interest was however mechanically scraped of with a stainless steel tool and the white powder thus obtained was weighed. This mixture of calcite and other minerals was leached (after a maximum exposure to the atmosphere of two hours) in 0.5 M HCL in order to release the carbon from the carbonates as CO_2 gas. The gas was cleaned from water by passing a tripple cold trap at 80°C and finally freezed out in a container kept at liquid nitrogen temperature. A reference volume was used to measure the total amount of carbon obtained (labeled carbon in table 2 and 3) in this pretreatment.

Before the accelerator measurements of the ^{14}C content, a conversion of the CO_2 gas to solid graphite for the ion source was performed. A Fe-cathalytic reaction at 800°C with the presence of hydrogen was exploited in this step. The determination of the ^{14}C concentration was finally done by employing the Uppsala EN-tandem accelerator as an ultra sensitive mass spectrometer.

Each sample was sequentially measured and normalised to the international oxalic acid standard from NBS. No δ^{13}C investigations were carried out and hence no corrections for the natural mass fractionation performed. This effect on the other hand is in the order of 0.05 pM and thus neglectable compared with the measuring accuracy and other uncertainties.

5. RESULTS AND DISCUSSION

The results of the ^{14}C analyses are shown in table 2. As can be seen the analysed samples show varying but generally very low ^{14}C contents, except for Kl 7:125 (analysed in an earlier pilot-study). This single sample differs from the other fracture samples in that it consists of more fine-grained, sandy material from an, probably recently reactivated, fracture zone. This sample is therefore more easily contaminated with atmospheric CO_2. This result is not included in the diagrams and figures of ^{14}C in the fracture calcites although it is included in table 2 and in the discussion in the text. Figure 8 shows modern C (μg) plotted versus weight of pure C in the samples. According to the diagram, zero sized samples still have 5 and 10 μg modern carbon. This contamination can either be attributed to adsorption on the sample surface during storage or introduced during the chemical treatment chemistry. The latter is more likely since the pre-treatment contaminations to first order are independent of the sample size.

In order to get more information about the extent of the contamination a sealed fracture, from the Finnsjön study site, containing hydrothermal Al-silicate minerals coprecipitated with calcite was selected for analysis. Microscopy of the reference fracture sealing shows 1) a complex prehnite-calcite coating with a prismatic texture of the calcite 2) a thin zeolite-calcite sealing with a granular texture of the calcite. The thin zeolite-calcite sealing transsect the older prehnite-calcite sealed fracture.

X-ray diffractometry (XRD) of the two sealings confirm the occurance of prehnite and showed that the zeolite minerals present in the thin sealing in the centre of the fracture are laumontite and analcime. Furthermore analyses of stable isotopes of the two calcite generations fits well with the earlier described intervall for hydrothermal calcites in Finnsjön according to Tullborg & Larson (1982). The values obtained are: 1) for the calcite in the prehnite sealing $\delta^{18}O = -18.1$ o/oo and $\delta^{13}C = -4.2$ o/oo 2) for the calcite in the zeolite sealing $\delta^{18}O = -15.2$ o/oo and $\delta^{13}C = -4.6$ o/oo.

Table 2. ^{14}C analyses of fracture calcites from Klipperås

Sample	modern C (μg)	pmC	Weight (pure C) mg
Kl 1:100.4	15.504	1.36 ± 0.09	1.14
Kl 1:140.4 A	39.064	0.76 ± 0.09	5.14
Kl 1:233.4	43.977	1.37 ± 0.10	3.21
Kl 1:435.3	16.906	0.79 ± 0.07	2.14
Kl 1:436.25	17.728	2.77 ± 0.17	0.17
Kl 7:80.3	31.088	1.16 ± 0.11	2.68
Kl 7:125*	116	72.5 ± 2.3	0.16
Kl 7:143.0	11.776	0.92 ± 0.10	1.28
Kl 9:705.6	23.736	2.76 ± 0.18	0.86
Kl 12:102.9	15.633	0.81 ± 0.11	1.93
Kl 12:203.8-9 A	32.250	2.15 ± 0.16	1.50
Kl 12:203.8-9 B	7.552	4.72 ± 0.51	0.16
Kl 12:297.45	7.911	2.93 ± 0.21	0.27
Kl 12:302	13.161	1.23 ± 0.13	1.07
Kl 12:321.1	9.983	9.33 ± 0.57	0.107
Kl 12:377.2	16.849	0.83 ± 0.09	2.03
Kl 12:378.1	11.100	0.74 ± 0.09	1.50
Kl 12:478.0	18.785	0.65 ± 0.08	2.89
Kl 12:616.9 A	20.862	1.22 ± 0.14	1.71
Kl 12:616.9 B	16.638	1.41 ± 0.13	1.18

A and B represent different calcite generation within one single fracture

* = result earlier reported by Tullborg (1986b).

Table 3. ^{14}C-analyses of reference sample from a sealed fracture within the Finnsjön study site

Sample	pmC	Weight (pure C) mg
Fi 6:351' A	0.44 ± 0.04	7.7
Fi 6:351' B	3.81 ± 0.43	0.16
-"- -"-	2.82 ± 0.32	0.48
-"- -"-	1.95 ± 0.16	0.80
-"- -"-	1.48 ± 0.13	8.3

A = sample containing granular calcite
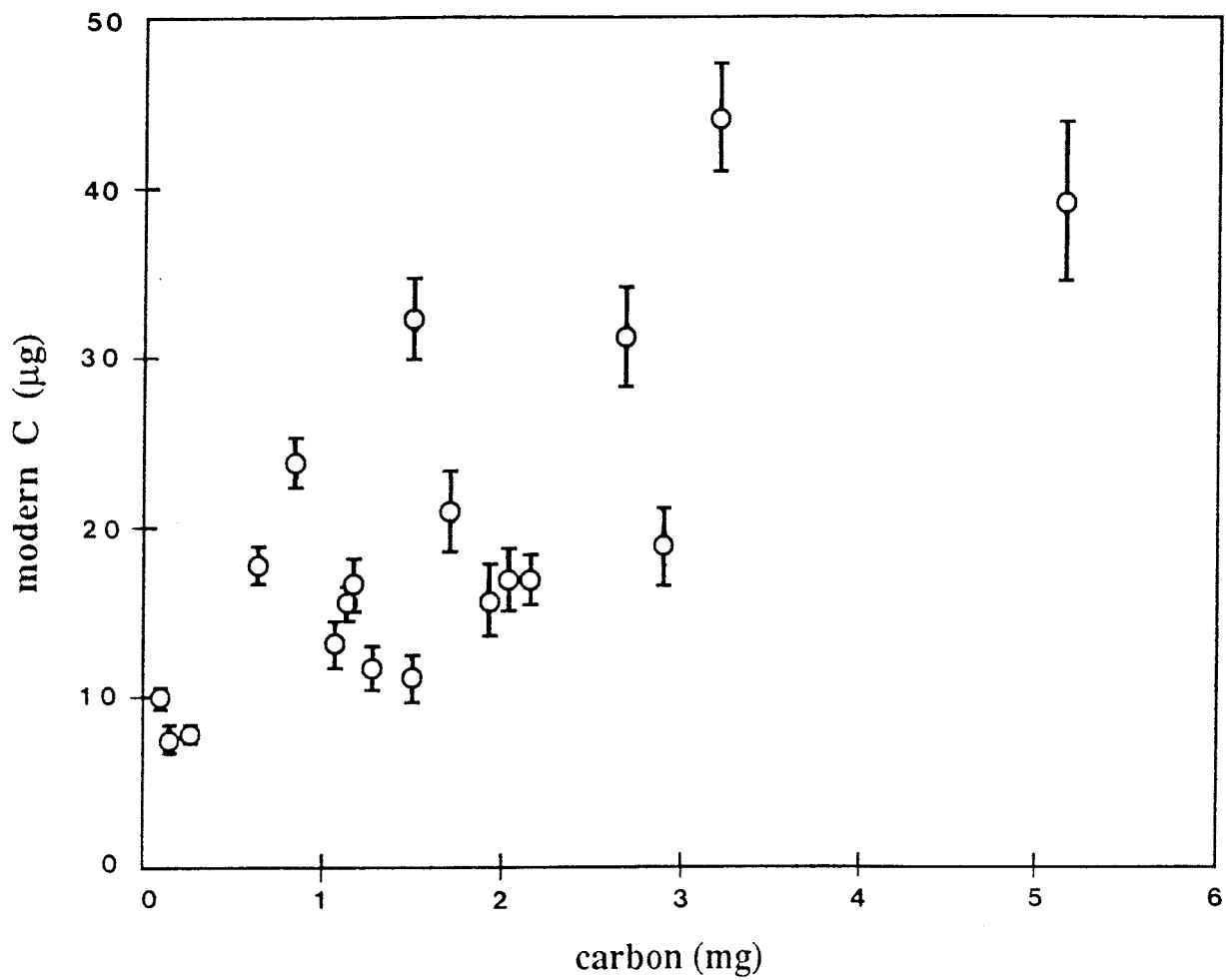B = sample containing prismatic calcite

Figure 8. ^{14}C in fracture calcites (modern C, μg) plotted versus weight of sample (pure C in mg).

The above described features all point to a hydrothermal origin
of the two calcite generations in this fracture which means that
the fracture sealings should be too old to preserve any ^{14}C from
the time of precipitation. One sample from the granular calcite
in the middle of the sealing was analysed as well as four samples
from the prismatic calcite (0.16 mg, 0.48 mg, 0.80 mg and 8.3
mg). The results are shown in table 3. Figure 9 shows pmC for
calcites from open fractures (Klipperås) as well as the reference
samples from a sealed fracture (Finnsjön) plotted versus weight
of pure C in sample. The line is just a guide for the eye, but
the slope for the smaller samples corresponds roughly to a
constant 10 μg modern carbon contamination. The result of the
reference sample analyses (which fit well into the trend) streng-
then that contamination obviously takes place during the sample
preparation. However, the largest sample (8.3 mg) showed a value
of 1.48 pmC. This can be due to additional contamination taking
place during the storage of the drillcores. It should be pointed
out that the reference sample has been stored in 8 years before
analysis, compared to storage time of three years for the Klipp-
erås samples. The divergence between the granular and prismatic
reference sample (7.7 and 8.3 mg respectively) can be due to
their different textures and thus different active surfaces
exposed for CO_2 upptake.

As can be seen in figure 9 a small number of fracture samples
from Klipperås plot above the "background" line (although with a
low significance). If a constant value of 7±2 μg modern carbon is
subtracted from the measured values figure 9 will change to the
appearance shown in figure 10. This background subtraction seems
to be consistent since the slope disappear. One conclusion from
this background subtraction procedure is, that for samples
containing small amounts of calcite (less than 0.5 mg carbon)
accurate measurement is dependent on a well reproducable chemis-
try giving exactly the same contribution of modern carbon from
sample to sample.

The samples plotted above the background line are; Kl 12:321.1 m
and Kl 12:203.8-9 m A as well as the samples from the sections
sampled for groundwater; Kl 1:436.25 m and Kl 9:705.6 m. Concern-
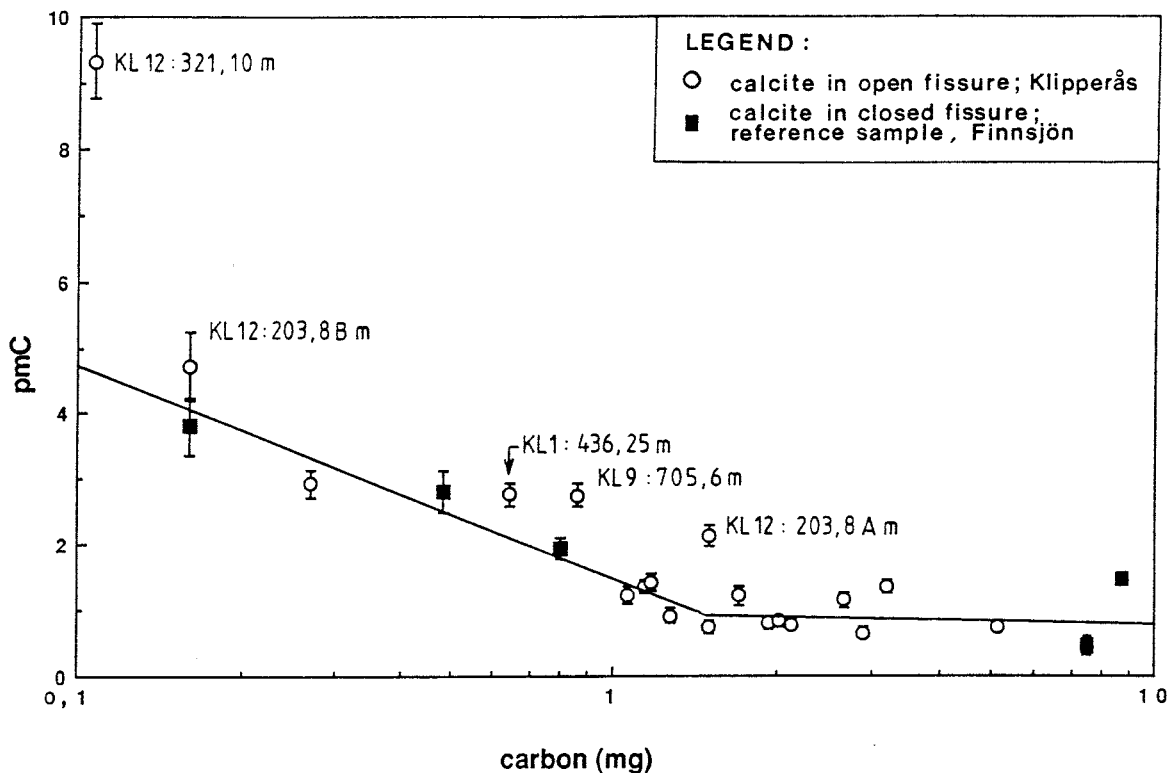
Figure 9. ^{14}C in fracture calcites and reference samples (pmC) plotted versus weight of sample (pure C, mg).
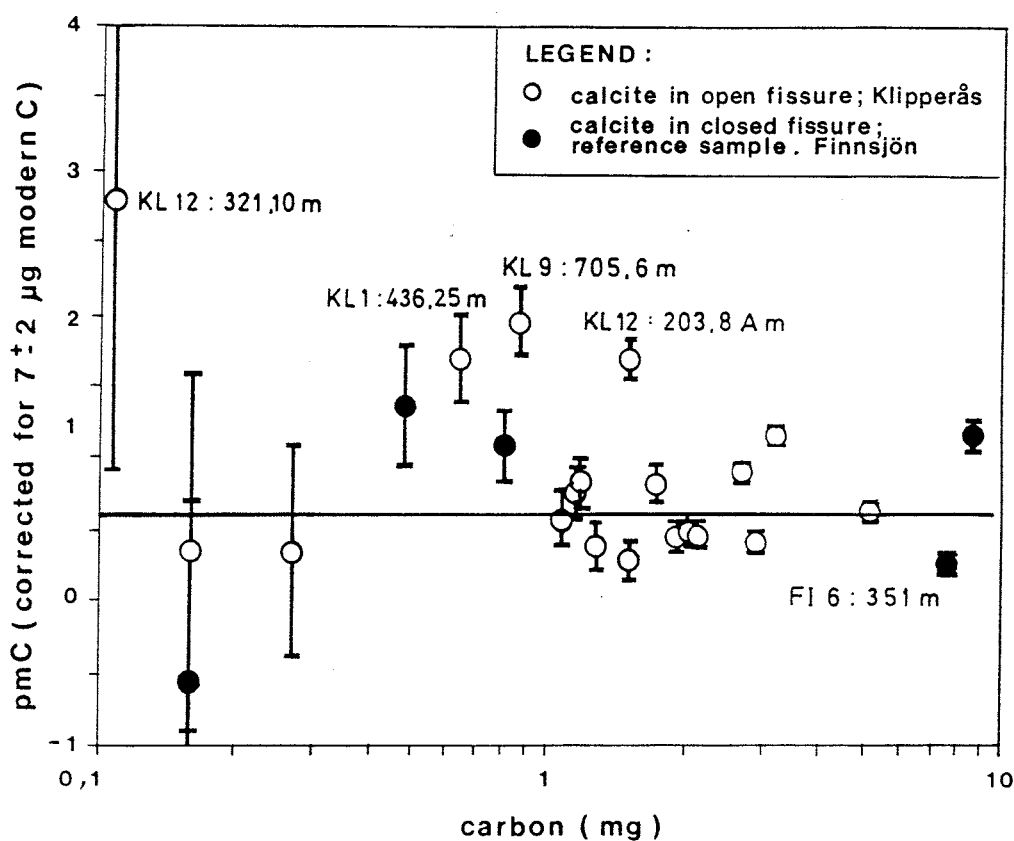

Figure 10. ^{14}C in fracture samples corrected for contamination of 7±2 μg carbon plotted versus carbon content in the sample.

ing the two samples from drillcore Kl 12 it is noticed that both of them are situated in hydraulically conductive zones and the sample (Kl 12:321.1 m) showing the highest pmC (9.3) is situated within a fracture zone. The samples corresponding to the sections sampled for groundwater showed ^{14}C contents of approximately the same order as the water sampled in the fractures. Due to fractionation processes (cf. Faure, 1979) a higher ^{14}C content would have been expected in the calcite than in the water. However, it is reasonable to assume that the ^{14}C content in the fracture calcites is inhomogenous (due to zoning) and as it has not been possible to analyse seperate layers in the calcite crystals, the recorded ^{14}C contents represent average values which should be lower than the values of surface layers.

Conclusively, the fracture samples showing ^{14}C contents higher than the background values (although not always significant) are the samples where it is reasonable to detect a ^{14}C signal, based on geological and hydrogeological information from the Klipperås area.

6. CONCLUSION

Accelerator mass spectrometry analyses of ^{14}C make it possible to analyse fracture calcites from drillcore material, i.e. very small samples can be analysed (c. 1 mg C). The ^{14}C analyses of fracture calcites from Klipperås yielded low but varying values of ^{14}C. Suggesting that the ^{14}C values above the "background line" have some significance, the following geological interpretations are possible:

1) An extensive groundwater circulation takes place facilitating downward percolation of ^{14}C loaded HCO_3-water in the bedrock. However the ^{14}C content in the groundwater decreases rapidly due to dissolution of "dead" calcite in the near surface leaching zone and due to sorption of ^{14}C on restite calcites in the transition zone between calcite dissolution/precipitation (probably 50 to 150 m depth, or even deeper in fracture zones). This could be the explanation for the very high value in Kl 7:125

(72.5 pmC; Tullborg (1986b))and the low but somewhat increased values in Kl 12:321 m (fracture zone) and Kl 12:203.8-9 m.

2) An extensive circulation of near surface groundwater takes place affecting the upper 100 to 200 m whereas stagnant conditions prevails at depth, i.e. two separate aquifers. This could also explain the high ^{14}C content in the crush zone in Kl 7:125 m and the lower values recorded in a potential "mixing zone" between the two aquifers (Kl 12:203.8-9 m and Kl 12:321.1 m).

At the present state of knowledge about the Klipperås area, we would suggest alternative 1 as the more probable. This means that an extensive sorption of ^{14}C takes place on calcite surfaces in the near surface calcite coated fractures which result in a rapid decrease in ^{14}C content in the water and thus in the fracture calcites deeper down. Sorption of ^{14}C on calcite surfaces has been shown by laboratory experiments (Allard et al., 1980).

It is possible that the effect of ^{14}C sorption on carbonate surfaces is underestimated e.g. when estimating residence time of groundwater. It is suggested that future ^{14}C studies of fracture carbonates will concentrate on the near surface zone in order to clarify the ^{14}C contribution to deeper levels in the bedrock.

Another conclusion is that significant contamination obviously takes place during storage of the drillcores as well as sample preparation. For future analyses of ^{14}C in fracture calcites attempts should be made to eliminate or decrease the risks of contamination.

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