

ATMOSPHERIC CO₂ CONTENT IN THE PAST DEDUCED FROM ICE-CORE ANALYSES

by

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ABSTRACT

Probably the only direct way to reconstruct historic atmospheric CO₂/air ratios is by measuring the air occluded in ice samples. For such studies, ice samples from very cold regions have an advantage in that the snow and ice probably never had been in contact with liquid water, which interacts with trapped atmospheric CO₂. However, even ice samples from very cold regions may have additional CO₂, which could have been enclosed during the formation of precipitation, adsorbed at the surface of snow and firn grains, or produced in the ice by chemical reactions on impurities. The gas in ice samples from Camp Century and Byrd station was extracted both by a vacuum-melting and by a mechanical crushing procedure and the CO₂ abundance measured by two different analytical methods. N₂/O₂/Ar ratios were also measured. The shift to lower CO₂ concentration in both cores at a certain depth found previously is confirmed. Changes in the atmospheric CO₂ concentration at the corresponding time, about the end of the last glaciation, is the probable explanation of our CO₂ results.

INTRODUCTION

The gas occluded in bubbles of old ice samples consists of air trapped during the process of ice formation. This led to attempts to reconstruct the history of the composition of the atmosphere by analysing the gas in ice samples of known age. Of special interest is the CO₂ content of the ancient atmosphere. Due to intensive land use and combustion of fossil fuel in the last century, the CO₂ content of the atmosphere has increased by about 20% to a present value of about 338 ppm (C D Keeling personal communication). The industrial CO₂ contribution is somewhat uncertain and natural variations in the amount of atmospheric CO₂ are unknown. Earlier measurements on ice samples (Berner and others 1980, Delmas and others 1980) suggest that toward the end of the last glaciation the CO₂ content of the atmosphere was significantly smaller than in the post-glacial period. We report new measurements with improved techniques on ice samples from ice cores from Antarctica and Greenland. Our aim is to obtain more exact values for CO₂ concentrations in different epochs.

More definite evidence regarding the suggested CO₂ increase toward the end of the last glaciation and more precise knowledge of the pre-industrial atmospheric CO₂ content are not only of scientific

interest but are also important to future energy policies.

ICE SAMPLES

In snow-covered areas, where the surface temperature never reaches the melting point of ice, ice is formed by sintering of dry snow. The sintering process leads to a densification of snow and firn and a decrease of the pore volume. The pore volume consists of channels which have multiple connections with each other. When the pore volume shrinks to about 10% of the total firn volume, the channels are pinched off and isolated bubbles are formed. If the air in the open pore space of firn is of atmospheric composition, we expect the air in newly formed bubbles to be of atmospheric composition also. Since gas diffusion through ice is very small, the composition of air occluded in these isolated bubbles essentially remains preserved. These considerations are only correct if no water is involved.

Ice samples from very cold areas, therefore, seem best suited for the study of ancient atmospheric composition.

We used, for our measurements, ice-core samples from Camp Century, Greenland (77°10'N, 61°08'W), Byrd station, Antarctica (80°01'S, 119°31'W), and North Central Greenland (74°37'N, 39°36'W). The mean annual air temperature in Camp Century is -24°C, but small ice layers are formed in warm summers, so that the influence of melt water on the CO₂ content cannot be ruled out. The core drilling, supervised by B L Hansen, reached bedrock at a depth of 1 387 m in 1966. The mean annual air temperature at Byrd station is -28.4°C, and the ice thickness is 2 164 m. The core drilling reached bedrock in 1968 (Ueda and Garfield 1969). Through the courtesy of Dr C C Langway, we obtained good quality samples of about 1 kg in weight from both cores for our measurements. We made measurements on 22 samples from the Camp Century core and 21 samples from the Byrd core, distributed over the entire depth. In North Central, Greenland, the core drilling was done with a drill constructed in Denmark as part of the Greenland Ice Sheet Program (Johnsen and others 1980). The mean annual air temperature is -31.7°C. The firn-ice transition is at a depth of 60 to 80 m. We made measurements on a sample from 103 m depth.

There are several ways in which CO₂ or, more generally, carbon can get into ice formed by sintering of dry snow. Some carbon, initially not located in the air bubbles, may later exchange and produce a small enrichment or depletion of CO₂ in the air bubbles.

TABLE I. ESTIMATED AMOUNT OF CARBON IN ICE

Source	Principal compounds	Estimated amount µg carbon kg ⁻¹ ice
Gases dissolved in supercooled droplets and in snow-flakes	CO ₂	100 (decreasing in a few days below 5)
CO ₂ adsorbed on surfaces of snow and firn grains	CO ₂	0.1
CO ₂ enclosed with air in isolated bubbles	CO ₂	16
Inorganic carbon dust, scavenged or deposited by dry fallout	CaCO ₃ , MgCO ₃	2
Organic carbon (pollen, tissue fragments)	-	0.04

SOURCES OF CARBON IN ICE

We now discuss estimates of the amount of carbon from different sources. The estimated values are presented in Table I.

The amount of CO₂ dissolved in snow probably depends on meteorological conditions during snow-fall. We measured, in cold dendritic snow, a CO₂ content corresponding to 150 µg carbon kg⁻¹ ice immediately after snow-fall and 35 µg carbon kg⁻¹ ice after one day of storage. These values include CO₂ from carbonates. On the basis of measurements of the inorganic carbon in firn samples from North Central, Greenland and from South Pole station (5 µg carbon kg⁻¹ ice (Schwander unpublished)) and subtracting the estimated contribution by carbonate, the CO₂ dissolved in old snow and firn is estimated to lie in the range of 0 to 5 µg carbon kg⁻¹ ice. The upper limit of this estimate is an order of magnitude lower than values published earlier (Stauffer and Berner 1978). The early values were probably too high because of contamination (Delmas and others 1980).

The amount of CO₂ adsorbed onto the surface of the firn grains has been estimated by Klinger and Ocampo (1979). A free surface of 5 m² kg⁻¹ corresponds to isolated firn grains with diameters of ~1mm. The free surface of the firn at the firn-ice transition is approximately 1 m² kg⁻¹. The amount of CO₂ adsorbed on this surface corresponds to 0.1 µg carbon kg⁻¹ ice.

Approximately 30 mm³ kg⁻¹ ice (at s.t.p.) of CO₂ is occluded in the air bubbles, corresponding to ~16 µg carbon kg⁻¹ ice for an atmospheric CO₂ concentration of 300 ppm. This is the most important source of carbon in ice from very cold areas.

The deposition of carbonaceous dust depends on the geographical location and the patterns of atmospheric circulation. Elemental concentrations for calcium and magnesium, measured by Cragin and others (1977), are given in Table II. The estimate in Table I for Byrd Holocene ice is made with the assumption that Ca and Mg are in the form of carbonates. The glacial-period values for Byrd and Camp Century cores are higher by a factor of ~2 and ~30, respectively.

TABLE II. AVERAGE CONCENTRATIONS OF THE ELEMENTS Ca AND Mg (CRAGIN AND OTHERS 1977)

	Camp Century (µg kg ⁻¹ ice)		Byrd station (µg kg ⁻¹ ice)	
	Ca	Mg	Ca	Mg
Holocene	5.1±3.4	2.6±1.3	3.4±1.7	2.4±1.8
Late Wisconsin	162±74	25±9	8.1±3.0	7.4±2.1

Fredskild and Wagner (1974) investigated pollen and tissue fragments in ice samples from Camp Century. Our estimate assumes that each pollen grain contributes an average of 10 ng carbon and that the tissues give the same amounts as all pollen grains. For Byrd station, no such measurements exist; however, the organic carbon content is expected to be smaller than that for Camp Century. The amounts of organic carbon and adsorbed CO₂ are, therefore, negligible. The CO₂ content in air bubbles can be considerably affected only by CO₂ dissolved in ice and by carbonates. Exchange between CO₂ in bubbles and CO₂ dissolved in ice occurs in Byrd core at depths below 800 m because the bubbles diffuse into the ice structure (Gow 1968). The possibility of exchanges between the different forms of inorganic carbon is discussed later, together with our results.

EXPERIMENTAL METHODS

The extraction method determines whether the total inorganic carbon or only one form of carbon will be extracted. Vacuum melting under acidic conditions permits the extraction of all carbonates as CO₂. Extraction methods which open air bubbles mechanically can extract the CO₂ only in air bubbles. We used both a vacuum-melting method and a mechanical dry-extraction method. The methods will be discussed in detail by Stauffer and others (in press) and Zumbunn (unpublished). We give here only a short description of the two methods.

In the melting method, roughly 300 g of ice is melted in an evacuated container. Gases released during melting are continuously pumped off and collected. After melting, which lasts about 30 min, the gases are extracted over continuously stirred water for an additional 6.5 h. If the pH value of the melt water is >7, phosphoric acid is added under vacuum until the pH value is 4. This guarantees a complete extraction of CO₂ and carbon in carbonates, as calculated and verified by different tests. The extracted gases are collected in two fractions. The first fraction is collected during the first 20 min of the melting process until about two-thirds of the ice is melted. The remaining gases are collected in the second fraction. The gas composition of the first fraction corresponds approximately to the gas composition in the air bubbles (Berner unpublished) and allows estimation of CO₂ concentration of air in the bubbles. The second fraction includes CO₂ formed by carbonates in the melt water. To extract the CO₂ content of the air in the bubbles alone, a new fast dry-extraction method was developed, by which the ice is crushed into small pieces in an evacuated container between two plates equipped with an array of steel needles. The gases are released into a laser absorption cell where CO₂ concentration is measured with a laser spectrometer (Zumbunn and others submitted for publication*). The sample size is 1 g; the

extraction time lasts <1 min. The short extraction time and a container temperature of -20°C minimize contamination with CO₂ desorbed from the walls of the extraction container. The short time involved is one of the big advantages of this mechanical method over other mechanical methods. Pieces of the crushed ice have diameters between 0.1 and 1 mm. The gas yield is about 75% compared with results from the melt-extraction method. The small sample size enables the study, with high spatial resolution, of the distribution of CO₂ concentration in bubbles.

ANALYTICAL METHODS

The gas samples, which are extracted from 300 g of ice using the vacuum-melting technique, are measured in a Hewlett Packard 5880 A gas chromatograph. N₂, O₂, and Ar are measured with a thermal conductivity detector. CO₂ is catalytically transformed into methane and then measured with a flame ionization detector. The detection limit for CO₂ is about 2×10^{-13} m³ s.t.p., for N₂, O₂ and Ar about 10^{-11} m³ s.t.p.

The CO₂ concentration in the very small gas samples extracted using the cracker method is measured with a laser absorption spectrometer. The light source is a PbS-Diode laser emitting at 4.3 μm. For this wavelength, the CO₂ molecules have a strong vibrational transition. The laser beam passes through an absorption cell filled with the sample gas and a monochromator, which filters out the undesirable modes. The intensity of the light leaving the monochromator is measured with a PbSe-detector.

The absorption cell has an optical path length of 150 mm. An air sample extracted from 1 g ice corresponds to a pressure of 60 to 100 Pa in the cell. The pressure can be measured with an accuracy of 1 Pa. The accuracy of the CO₂ concentration measurement at this low gas pressure is about 1%. The overall accuracy, including uncertainties regarding the extraction, is estimated to be 2%. Both analytical methods are based on a calibration with three World Meteorological Organization CO₂ in air standards (320 ppm, 342 ppm, 380 ppm) prepared at the Scripps Institution of Oceanography, San Diego, California, U.S.A.

RESULTS

Before the analyses, 1 kg samples were cleaned by removing a 10 mm layer from all sides with a microtome knife. The cleaned ice was cut into one 300 g sample for vacuum melting and into several 1 g samples for the dry extraction. The measured results for samples from Camp Century and Byrd station are listed in Tables III and IV. The CO₂ concentrations for the first melt-extraction fraction (measured with the gas chromatograph) and the concentration obtained with the laser spectrometer are plotted in Figures 1 and 2. The results measured with the laser spectrometer are shown with the lowest and highest value and with the median value for each depth. Since we consider the risk of contamination with CO₂ to be higher than that of CO₂ loss, we believe that the lowest values are at least as significant as the median values. In Figures 1 and 2, the curves are drawn through the means of the three lowest values measured with the laser spectrometer. The CO₂ concentrations measured for the first extraction fraction show the same trend as earlier measurements on different ice samples of the same ice cores (Berner and others 1980). The laser spectroscopic results confirm this general trend. The laser median values, however, are about 30 ppm lower than the first extraction

values. There are two reasons for this: (1) a small part of the CO₂ in the first extraction fraction (estimated to be on the average 5 to 10 ppm) originates from carbonates and CO₂ dissolved in the ice, and (2) about 20 to 25 ppm is due to a desorption of CO₂ from the walls of the melt extraction system (Zumbrunn unpublished).

The melt water of samples from Camp Century from depths below 1 100 m was alkaline and had to be acidified during the extraction to recover the CO₂ completely. This explains the much higher values of the total CO₂ concentration for this depth interval compared to results given earlier. (Berner 1980). The strong alkalinity of these samples probably also influenced the CO₂ concentration of the first extraction fraction and possibly even the CO₂ concentrations of the gases obtained by the dry extraction method. These influences are discussed later in more detail.

The data of the Camp Century samples given in Figure 1 are based on measurements and calculations by Hammer and others (1978). For Byrd station, age calculations are more difficult (Johnsen and others 1972) and no age scale is given in Figure 2. If a synchronous global change of the atmospheric CO₂ concentration caused the CO₂ shifts in the two cores, we would conclude that the ice from Camp Century at a depth of 1 130 m has the same age as ice from Byrd station at a depth of 1 100 m.

As a test for our methods a section 110 mm long from a depth of 103 m of the North Central, Greenland ice core was cut into 22 samples of 1 g each. The

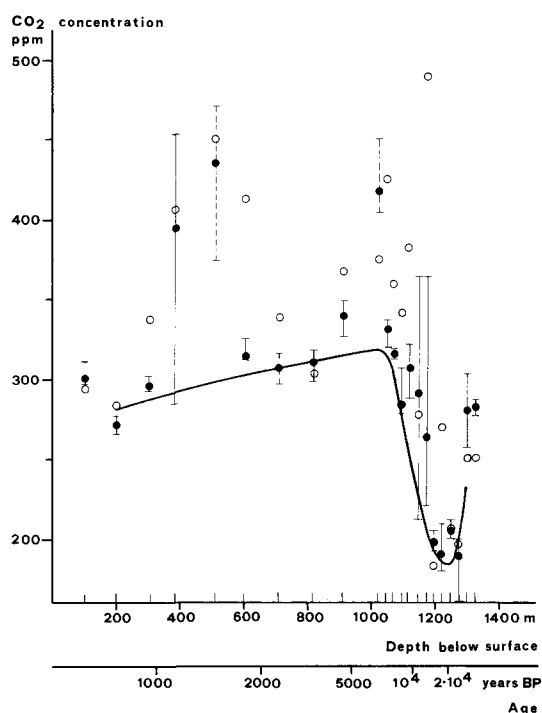


Fig.1. CO₂ concentration in ice samples from Camp Century:

- CO₂ concentration of the first extraction fraction
- CO₂ concentration of the dry extracted gases measured with laser spectroscopy. Black dot indicates median value. Highest and lowest results (T, ⊥) are also indicated. The smoothed curve connects 3-value running means of the lowest values measured with the laser for each depth.

*Submitted for publication: Zumbrunn, R., Neftel, A., Oeschger, H. CO₂ measurements on 1 cm³ ice samples with an IR-laser spectrometer combined with a new dry extraction method. *Earth and Planetary Science Letters*

TABLE III. RESULTS OF ANALYSES OF ICE FROM CAMP CENTURY, GREENLAND

Depth below surface (m)	Total gas content (mg kg ⁻¹)	O ₂ /N ₂ -ratio (%)	Ar/N ₂ -ratio (%)	CO ₂ -conc. 1st fraction (ppm)	CO ₂ -conc. total (ppm)	C-content total (μg kg ⁻¹)	C-content 2nd fraction (μg kg ⁻¹)	CO ₂ -concentration measured with laser spectroscopy									median value (ppm)
								1 (ppm)	2 (ppm)	3 (ppm)	4 (ppm)	5 (ppm)	6 (ppm)	7 (ppm)	8 (ppm)		
101.8	106.5	26.83	1.20	294	355	20.3	3.5	311	296	302							302
201.3	109.8	26.53	1.18	284	353	20.8	4.1	271	266	277							277
309.6	124.0	26.86	1.20	338	400	26.6	4.1	302	293	296							298
383.2	95.7	26.21	1.19	407	471	24.1	3.3	284	421	369	453						395
510.9	72.4	26.19	1.19	450	577	22.4	4.9	452	471	419	374						435
607.9	102.8	26.46	1.19	413	476	26.2	3.5	325	305	310	316						315
710.1	107.8	26.73	1.20	339	446	25.8	6.2	297	305	310	316						307.5
819.0	101.3	26.78	1.20	304	367	19.9	3.4	318	299	311	311						311
910.9	105.5	26.79	1.20	368	468	26.5	5.7	327	349	330	349						339.5
1021.4	91.0	26.03	1.19	375	427	20.8	2.5	426	450	405	410						418
1047.9	82.6	25.51	1.18	426	539	23.9	5.1	320	332	337	332						332
1069.2	100.9	26.93	1.20	360	414	22.4	2.9	315	316	317	317						416
1095.2	81.8	26.25	1.19	342	416	18.3	3.3	279	307	284	284						284
1115.7	94.5	26.29	1.18	383	436	22.1	2.7	288	307	322	322						307
1146.3	74.8	26.41	1.19	278	368	14.7	3.6	212	276	253	306	364					264
1173.2	102.8	26.92	1.21	490	992	15.6*	27.6	270	267	364	301	247			221	261	261
1197.5	104.3	26.77	1.20	184	2758	154.1*	143.8	193	205	199	181						199
1220.7	99.4	26.60	1.19	271	489	26.0	11.6	185	210	198	198						191.5
1248.8	97.1	26.67	1.20	207	410	21.3*	10.5	212	203	201	206						204.5
1272.3	93.5	26.46	1.19	197	476	23.8*	13.9	159	200	180	199						190
1298.6	96.3	26.84	1.20	251	385	19.9	6.9	280	258	282	303						281
1324.6	96.1	26.08	1.18	251	315	16.2	3.3	278	287	283	285						284

TABLE IV. RESULTS OF ANALYSES OF ICE FROM BYRD STATION, ANTARCTICA

Depth below surface (m)	Total gas content (mg kg ⁻¹)	O ₂ /N ₂ -ratio (%)	Ar/N ₂ -ratio (%)	CO ₂ -conc. 1st fraction (ppm)	CO ₂ -conc. total (ppm)	C-content total (μg kg ⁻¹)	C-content 2nd fraction (μg kg ⁻¹)	CO ₂ -concentration measured with laser spectroscopy									median value (ppm)
								1 (ppm)	2 (ppm)	3 (ppm)	4 (ppm)	5 (ppm)	6 (ppm)	7 (ppm)	8 (ppm)	9 (ppm)	
101.0	112.5	26.62	1.19	286	326	19.6	2.4	275	265	263	273						269
201.7	110.3	26.76	1.19	313	364	21.5	3.0	270	301	285	275						280
297.5	111.3	26.68	1.19	290	363	21.6	4.4	261	252	263	263						263
401.5	103.4	26.44	1.19	327	411	22.8	4.7	270	269	269	268						269.5
503.8	96.5	26.10	1.18	384	525	27.1	7.3	502	260	416	410						412
600.3	101.9	26.49	1.19	343	468	25.5	6.8	270	262	262	412						412
700.9	87.5	25.91	1.17	354	482	21.7	5.1	397	323	327	303						266
797.7	94.5	25.64	1.17	297	482	24.4	7.6	283	290	264	277						286.5
902.4	110.0	26.42	1.18	306	394	23.2	5.2	253	268	264	370						266
1005.5	113.7	26.57	1.19	323	461	28.1	8.4	284	283	293	268						286.5
1051.1	109.0	26.70	1.19	321	424	24.8	6.0	240	279	267	251						288.5
1150.1	110.4	26.50	1.19	282	321	18.9	2.3	229	211	219	239						257
1251.1	109.4	26.52	1.19	242	329	19.3	5.1	227	228	222	227						219
1346.6	114.9	26.60	1.19	242	305	18.8	3.9	183	208	196	257						227
1444.5	111.9	26.45	1.18	243	364	21.8	7.3	208	210	222	227						196
1544.7	108.8	26.65	1.19	253	333	19.4	4.7	221	218	222	227						219.5
1652.4	109.0	26.49	1.18	239	271	15.8	1.9	228	227	201	201						206
1751.7	108.6	26.78	1.19	240	309	18.0	4.0	218	200	220	229.5						229.5
1853.4	107.2	26.49	1.19	252	299	17.2	2.7	239	220	227	226						226
1956.1	106.0	26.18	1.18	255	330	18.7	4.3	224	228	228	268						261
2150.6	104.0	26.11	1.18	261	355	19.8	5.2	262	260	252	268						261

* These samples were acidified after the first extraction.

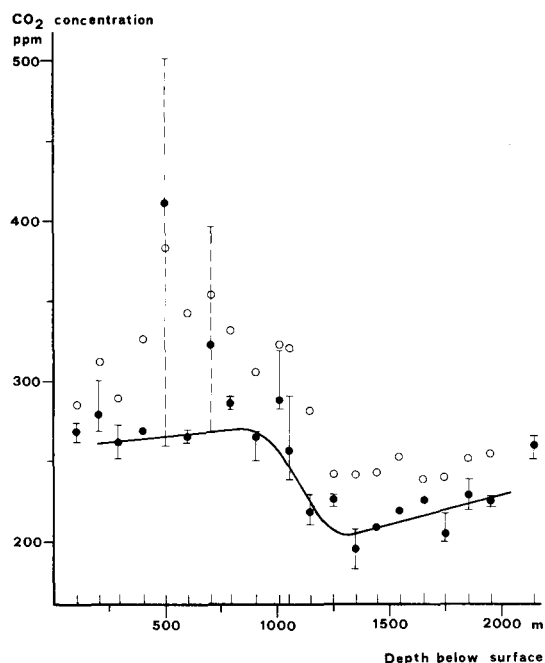


Fig.2. CO₂ concentrations in ice samples from Byrd station:

○ CO₂ concentration of the first extraction fraction

● CO₂ concentration of the dry extracted gases measured with laser spectroscopy. Black dot indicates median value. Highest and lowest results (T, ⊥) are also indicated. The smoothed curve connects 3-value running means of the lowest values measured with the laser for each depth.

gases were extracted by the dry extraction method and analysed with the laser spectrometer. The CO₂ concentration measured in this young ice is 271±9 ppm, which agrees with estimates of the pre-industrial atmospheric CO₂ concentration. Good reproducibility is shown by the small standard deviation.

DISCUSSION

Based on the agreement between the estimated value for the pre-industrial CO₂ content of the atmosphere and the laser results for the samples from North Central, Greenland, we conclude that the CO₂ concentration in air bubbles of young ice corresponds within about 10 ppm with the atmospheric CO₂ concentration. The results obtained for samples from Camp Century and Byrd station show at a certain depth a well-marked transition from lower to higher CO₂ concentrations. The transition occurs in both cores at the same depth interval as the shift from lower to higher δ¹⁸O values (Berner and others 1980).

The lowest CO₂ concentrations in bubbles from Camp Century coincide with the highest alkalinity of the ice (pH = 8.5). The alkalinity of the melt water delays the CO₂ extraction by the vacuum-melting method and causes the CO₂ concentration of the first extraction to be low. We cannot exclude that the alkalinity also causes a lowering of the CO₂ concentration in the dry-extracted gases. If the alkalinity caused the whole shift from low to high values at 1130 m, a different explanation (such as bubble disappearance) is necessary for the corresponding

shift in the Byrd core, since this shift is not connected with any change in the alkalinity.

The shift in the CO₂ concentration values in the core from Byrd station coincides approximately with the disappearance of the bubbles (Gow 1968). The bubbles do not disappear at the corresponding depth in Camp Century (Miller 1969). We feel that the coincidence of the shift from low to high CO₂ concentration values in two ice cores from different locations, both where the δ¹⁸O of the ice shifts, is more easily explained by a shift from a lower to a higher CO₂ concentration in the atmosphere at the end of the last glaciation than by independent separate explanations for each core.

It is difficult to give absolute values for the atmospheric CO₂ concentration because of alkalinity effects, the diffusion of CO₂ into the ice structure, and possible carbon contaminations. The CO₂ data for samples from depths between 400 and 900 m show the greatest scatter. The CO₂ variance at these depths is probably caused by contamination.

The cores from this depth interval show many small cracks. For three samples, traces of bore-hole fluid (diesel oil and trichloroethylene) were observed in the melt water. We assume that some of these samples were contaminated with bore-hole fluid and that additional CO₂ was produced in the ice core during storage. It is highly desirable that in future core drillings more and even better suited ice cores will become available. Then it should be possible to give estimated absolute values for the CO₂ concentration of the atmosphere in the last 50 ka.

ACKNOWLEDGEMENTS

We would like to express our most sincere thanks to Drs C C Langway and B L Hansen who envisaged the potential of ice cores for studies of this kind and made them available for the present investigations. This work has been supported by the Swiss National Science Foundation, the US National Science Foundation Division of Polar Programs, US National Science Foundation, and the US Department of Energy.

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