ASSESSING THE QUALITY OF THERMALLY DRILLED DEEP ANTARCTIC ICE CORES FOR TRACE ELEMENTS ANALYSIS

by

Boutron C.F.1,2, Patterson C.C.2 and Barkov N.I.3

- 1 Laboratoire de Glaciologie et Géophysique de l'Environnement du C.N.R.S., Domaine Universitaire, B.P. 96, 38402 Saint-Martin d'Hères cédex, France.
- Division of Geological and Planetary Sciences 170-25, California Institute of Technology, Pasadena, California 91125, U.S.A.
 - ³ Arctic and Antarctic Research Institute, UL Beringa 38, 199226 Leningrad, U.S.S.R.

ABSTRACT

Concentrations of Pb, Zn, Na, Mg, K, Ca, Fe and Al have been measured in successive veneers of ice mechanically chiselled progressing from the outside to the very center of various sections of the Dome C and Vostok deep Antarctic ice cores. Mean elemental contamination present in the outside layer of the cores was found to range from 0.3 ng/g (Al) up to 20 ng/g (Na) for the Dome C core, and from 5 ng/g (Al) up to 290 ng/g (Zn) for the Vostok core. Contrasting outside - inside curves were observed for the various elements. Plateaus of concentrations were obtained in the inner parts of the cores sections in all cases for Na and Mg, and in most cases for K, Ca, Fe and Al. For Pb and Zn, on the other hand, plateaus were observed only for part of the sections, which confirms that for these two heavy metals it is of utmost importance to study in details the variations of the concentrations from the outside to the inside of each core section if reliable data are to be obtained.

1 - INTRODUCTION

During the past 25 years, extensive efforts have been devoted to reconstruct the past variations of the composition of the earth's atmosphere during the last 150,000 years through the analysis of various elements or compounds in deep Antarctic or Greenland ice cores. Among the elements or compounds which have been investigated are major sea derived or soil derived elements such as Na, Cl, Mg, K, Ca and Al (see for instance Cragin et al. (1977), Petit et al. (1981) and De Angelis et al. (1987), sulfate and nitrate (see for instance Herron and Langway (1985), Legrand and Delmas (1988) and Legrand et al. (1988) and heavy metals such as Pb, Cu, Zn and Cd (see for instance Boutron and Patterson (1986), Boutron et al. (1987) and Batifol et al. (1989). Unfortunately, deep ice cores are always contaminated on their outside during drilling, field processing, transportation and storage. The severity of this outside contamination varies greatly from one core to another and from one element or compound to another. It is especially high for cores drilled in fluid filled holes. This contamination will penetrate more or less deeply into the ice cores, depending on numerous factors.

If reliable data are to be obtained from the analysis of these cores, it will be first of utmost importance to study in full details for each element or compound how deep contamination has intruded into the cores in order to determine whether the inner parts of the cores are free of contamination or not and are therefore suitable for elemental analysis. It will then be necessary to develop ultraclean laboratory procedures to get these inner parts of the cores without transferring outside contamination to these inner parts.

We present here comprehensive data on the contamination characteristics of various sections of two thermally drilled deep Antarctic ice cores. These data were obtained by investigating the variations of the concentrations of Pb, Zn, Na, Mg, K, Ca, Fe and Al from the outside to the inside of these sections.

2 - EXPERIMENTAL

2.1 - Description of the core sections

We have studied 10 sections of the 905 m Dome C core (depths ranging from 172.8 to 796.9 m) and six sections of the 2,083 m Vostok core (depths ranging from 499.1 to 2,026.3 m). The Dome C core was thermally drilled in a dry hole (Lorius and Donnou, 1978; Lorius et al, 1979). The Vostok core was thermally drilled in hole filled with kerosene and freon (Kudryashov et al., 1984 a, b; Barkov et al., 1988). Each section was 15-30 cm in length. The diameter was 10.5 cm for all the Dome C sections, 10.8 cm for the Vostok sections from the surface down to 1,500 m, and 9.1 cm for the Vostok sections deeper than 1,500 m. Full sections were available for the present study, except a shallow-chord shaving which was cut in the field with a band saw for continuous measurements of oxygen and hydrogen isotopes. No special precautions were taken for the handling of these sections in the field, which means that they have been in contact with leather or woollen gloves and with wooden or plastic gutters, and that they have been exposed to contaminated air in the drilling trench before being sealed in non acid cleaned polyethylene bags.

2.2 - Mechanical chiselling of the core sections

The chiselling took place inside a cooled double walled conventional polyethylene tray flushed with cooled nitrogen (Boutron and Patterson, 1983) placed inside the ultraclean Patterson's laboratory (Patterson and Settle, 1976) at California Institute of Technology (C.I.T.). We mechanically chiselled 4 to 6 successive veneers of ice in progression from the outside to the interior of each core section, using stainless steel chisels. The ends of the ice sections were also shaved. Each veneer layer was approximately 6-10 mm thick, depending upon the number of veneer layers. The remaining inner core obtained after the chiselling was completed, was usually 3 to 4 cm in diameter and 10-20 cm in length.

Sophisticated ultraclean procedures were used to prevent entrainment of the huge contamination existing on the outside of the core sections to the successive veneer layers and to the final inner core. These procedures were similar to those previously described in full details by Boutron and Patterson (1983). Various improvements were however effected. These improvements included the use of several stainless steel chisels instead of a single chisel, which allowed better cleaning

of each chisel between its use for different veneer layers. Conventional polyethylene collection trays and 1 liter beakers were used for the collection of the ice chips and the melting of each veneer layer or inner core instead of quartz trays and beakers in the earlier study. Also, the cleaning procedures were improved: the second acid bath was 0.1 % ultrapure HNO3 from U.S. National Bureau of Standards, instead of 1% electronic grade HNO3 from John Frederic Smith.

2.3 - Analytical procedures

Each veneer layer and each inner core were analysed separately. They were allowed to thaw overnight at room temperature in 1 liter conventional polyethylene beakers. Ultrapure HNO3 from U.S. National Bureau of Standards was then added to make a 0.1 % HNO3 solution. The solution was allowed to sit for 2 hours. About half of this solution was then analyzed for Pb by Thermal Ionization Isotope Dilution Mass Spectrometry (I.D.M.S.) at C.I.T.. The other half was transported frozen to the Laboratoire de Glaciologie et Géophysique de l'Environnement (L.G.G.E.), where a small sub-aliquot (about 2 ml) was analyzed for Zn, Na, Mg, K, Ca, Fe and Al by Graphite Furnace Atomic Absorption Spectrometry (F.A.A.S.) without preconcentration (multiple (up to 10) 50 µl injections were used for Zn). The analytical precision was better than 10 % for Na, Mg, K, Ca, Fe and Al. For Pb and Zn, it was better than 10 % for the high concentrations of the Last Glacial Maximum, but it was up to 50 % for the very low Holocene concentrations.

2.4 - Chiselling blanks

Contamination introduced by the

mechanical chiselling was directly measured using an artificial ice core of frozen ultrapure water. This ice core (diameter: 10 cm; length: 20 cm) was prepared by freezing about 1.6 l of C.I.T. ultrapure quartz distilled water (Q.D.W.) of known composition (Pb: 0.16 pg/g; Zn: 2.5 pg/g; Na : 40 pg/g ; Mg : < 3 pg/g ; K : < 60pg/g; Ca : < 10 pg/g; Fe : < 15 pg/g; Al: < 20 pg/g) in an ultraclean 2 liters conventional polyethylene bottle. The bottle was then cut using an acid-cleaned stainless steel scalpel, and the artificial ice core so obtained was chiselled according to the procedure described in section 2.2. One of the veneer layers and the inner core were then analyzed separately, thus allowing to determine how much Pb, Zn, Na, Mg, K, Ca, Fe and Al were added by the chiselling procedure.

The chiselling blanks were found to vary greatly from one element to another, Table 1. For the inner core, they range from 0.05 ng for Pb up to 25 ng for Al. Surprisingly, very low values were obtained for Fe (≤ 7.5 ng), despite the fact that a stainless steel chisel was used. When compared with the typical elemental content of Antarctic ice, Table 1, the chiselling blanks are found to be extremely small for Na, Mg, K, Ca and Fe. For Pb, Zn and Al however, the chiselling blanks can be up to 75 % of the elemental content of Antarctic inside veneer layers or inner cores.

3 - C O N T A M I N A T I O N CHARACTERISTICS OF THE DOME C AND VOSTOK CORES

3.1 - Contamination on the outside of the core sections

Table 2 gives the concentrations measured in the first (outside) veneer layer and in the

| | | | ng in a typical 200g veneer layer or inner core | | | |
|---------|--|---|---|------------|--|--|
| Element | ng introduced into the veneer layer ^a | ng intoduced into the inner core ^b | Holocene ice ^C | LGM ice of | | |
| Pb | 0.05 | 0.05 | 0.13 | 3 | | |
| Zn | 1.5 | 1.0 | 1.7 | 10 | | |
| Na | 45 | 15 | 3500 | 1 7000 | | |
| Mg | 8.5 | 5 | 500 | 3300 | | |
| K | 10 | 10 | 400 | 2800 | | |
| Ca | <5 | <5 | 230 | 5700 | | |
| Fe | <7.5 | <7.5 | 180 | 5200 | | |
| AI | 190 | 25 | 290 | 8500 | | |

 $^{^{}a}$ 375 g ice veneer made with 28 strokes of the stainless steel chisel - b 515 g inner core - c Calculated from mean concentrations measured in the inner cores of sections 172.8 m, 300.6 m, 373.9 m and 451.9 m of the Dome C core - d Calculated from mean concentrations measured in the inner cores 527.2 m, 545.1 m, 602.2 m, 658.2 m., 670.5 m and 704.2 m of the Dome C core.

<u>Table 1</u> - Comparison of chiselling blanks (measured by processing an artificial ice core of known composition) with typical trace elements content of Holocene and Last Glacial Maximum (LGM) Dome C ice.

| Core section | Pb | Zn | Na | Mg | K | Ca | Fe | AI |
|--------------|--------------|-----------------|------------|-------------|-------------|-------------|--------------|--------------|
| | pg/g | pg/g | ng/g | ng/g | ng/g | ng/g | ng/g | ng/g |
| Dome C | 2320 | 8600 | 42 | 4.7 | 35 | 10 | 18 | 7.3 |
| 172.8 | (0.76) | (6.1) | (13) | (1.9) | (1.5) | (1.0) | (0.48) | (0.95) |
| 300.6 | 720 | 1 700 | 29 | 3.1 | 15 | 3.3 | 5.4 | 4.0 |
| | (0.47) | (9.3) | (16) | (2.45) | (2.6) | (1.2) | (1.1) | (1.6) |
| 373.9 | 772 | 4020 | 47 | 2.8 | 40 | 7.9 | 7.7 | 4.3 |
| | (0.94) | (4.9) | (7.5) | (1.05) | (1.5) | (0.48) | (0.48) | (1.4) |
| 451.9 | 646 | 1300 | 47 | 6.6 | 13 | 5.2 | 4.3 | 3.1 |
| | (0.43) | (6.0) | (34) | (5.1) | (2.5) | (2.0) | (1.5) | (1.9) |
| 500.5 | 668 (3.8) | 1840 (16) | 68 (53) | 11 (8.4) | 24 (5.2) | 11 (6.2) | 10 (6.8) | 7.1 (8.0) |
| 545.1 | 340 | 1830 | 83 | 18 | 30 | 37 | 21 | 25 |
| | (10.2) | (32) | (63) | (16) | (9.0) | (23) | (17) | (21) |
| 602.2 | 1030 | 4360 | 111 | 22 | 19 | 27 | 18 | 17 |
| | (11.4) | (40) | (102) | (22) | (14) | (21) | (20) | (37) |
| 670.5 | 707 | 2080 | 92 | 27 | 17 | 60 | 38 | 44 |
| | (29.3) | (63) | (75) | (22) | (12) | (26) | (31) | (38) |
| 775.7 | 45000 | 37600 | 128 | 17 | 46 | 47 | 106 | 39 |
| | (7.2) | (10.5) | (55) | (10) | (4.2) | (7.0) | (4.3) | (6.0) |
| 796.9 | 1100 | 4000 | 80 | 11 | 19 | 7.5 | 9.0 | 5.6 |
| | (1.2) | (7.8) | (58) | (10) | (4.1) | (4.7) | (2.9) | (4.4) |
| Vostok | 20600 | 99000 | 1 40 | 35 | 61 | 180 | 108 | 35 |
| 499.1 | (38) | (33) | (92) | (19) | (10) | (18) | (9.7) | (24) |
| 851.6 | (3.1) | 400000 (1 4) | 76 (65) | 16 (11) | 34 (6.3) | 39 (8.9) | 39 (6.6) | 29 (32) |
| 1 425.3 | 20000 | 300000 | 82 | 20 | 59 | 200 | 89 | 38 |
| | (2.4) | (3.8) | (34) | (6.4) | (3.4) | (5.0) | (2.8) | (7.1) |
| 1775.4 | (2.6) | 220000 (3.0) | 52 (15) | 16 (2.4) | 51 (1.7) | 56 (2.4) | 135 (1.3) | 25 (3.6) |
| 1850.4 | 1 5700 | 400000 | 56 | 11 | 49 | 71 | 114 | 10 |
| | (1 0.6) | (12.5) | (8.3) | (1.3) | (0.7) | (0.43) | (0.2) | (0.9) |
| 2026.3 | 31 400 | 300000 | 156 | 40 | 79 | 31 0 | 263 | 55 |
| | (20) | (24) | (98) | (27) | (25) | (51) | (44) | (95) |

Table 2 - Dome C and vostok deep Antarctic ice cores: comparison of concentrations measured in the first (outside) layer with concentrations measured in the inner core. For each section and each element, the upper value gives the concentration measured in the first layer, and the lower value (in brackets) gives the concentration measured in the inner core.

inner core for each of the Dome C and Vostok core sections. It must be emphasized that the first (outside) veneer layer was about 6 to 10 mm thick; the corresponding concentrations listed in Table 2 then represent mean concentrations over that thickness interval. The concentrations present on the very outside surface of the core sections were then probably much higher. Table 3 shows the mean concentration differences, C, between the first (outside) veneer layer and the inner core for the Dome C and Vostok cores. Since the inner core concentrations are in most cases close to the original concentrations in the ice. these C values represent the mean contamination which was present in the outside layer of the ice cores.

For the Dome C core, the C values range from 0.3 ng/g for Al up to 20 ng/g for Na. When compared with the mean original concentrations in the ice, the relative importance of this contamination changes considerably from one element to another: for Pb and Zn, it represents about 150 fold the original concentrations; for K, about 4 fold; for Al, about 1.05 fold. Especially for Fe, Al and Zn, part of this contamination is likely to originate from the various components of the thermal drill used at Dome C: the melting head (bare spiral resistance wire made out of a Fe - Cr - Al alloy), the Al head support, the stainless steel barred (FE, Cr, Ni, Mo), the galvanized steel cable (Fe, Zn) and the Al winch drum (Gillet et al, 1976; Lorius and Donnou, 1978). But another part of this contamination is probably added to the ice cores after they have been removed from the drill. This last contamination is thought to originate from the dust and gases present in the drilling trench, and from the dirtiness of the various

items which contact the cores in the drilling trench (gutters into which the cores are placed for logging, bandsaw used to cut the continuous slice for oxygen and hydrogen isotopes measurements, gloves and clothes of the operators, polyethylene bags used to pack the cores...). These items are likely to have been contaminated by urban air during manufacturing and in the home laboratory, by the walls of the various cases in which they were packed during transportation to the field, by contaminated marine air in the holds of the supply ships, by exhaust gases and particles from aircrafts, caterpillar vehicles, field power generators... This contamination is then probably a very complicate blend of various components, including anthropogenic dust and gases, soil dust, seasalts... Finally, some contamination might have been added to the cores during transportation back to the home laboratory and during storage, especially when the polyethylene bags used for the packing of the core sections were not tightly sealed or were damaged.

For all the investigated elements, the C values measured for the Vostok core are higher than those obtained for the Dome C core, Table 3. The effect is especially pronounced for Zn (2 orders of magnitude difference between the Vostok and Dome C values) and for Pb, fe, Ca and Al (1 order of magnitude difference). It is rather small for Na and K. This stronger contamination of the outside of the Vostok core is probably mainly due to the fact that it was drilled in a fluid filled bore hole (Kudryashov et al, 1984 a, b). This fluid was a mixture of kerosene and freon (Barkov et al, 1988). Unfortunately, we have no direct data on the elemental content of this fluid. It is moreover unlikely that a

| | Pb | Zn | Na | Mg | K | Ca | Fe | AI |
|----------|------|-----|----|-----|----|-----|-----|-----|
| Dome C a | 0.92 | 3.3 | 20 | 1.9 | 18 | 9.3 | 5.6 | 0.3 |
| Vostok | 22 | 287 | 42 | 12 | 48 | 128 | 114 | 4.9 |
| | | | | | | | | |

^a Except section 775.7 m. This section was probably badly contaminated because of severe technical problems encountered with the drill near that depth.

Table 3 - Mean concentration differences, \triangle C, between the first (outside) layer and the inner core for nine sections of the Dome C core and six sections of the Vostok core. All \triangle C values are given in ng/g.

good prediction of this elemental content can be made from the few available data on the elemental content of kerosenes and freons from other parts of the world (see for instance Smith et al, 1975) since impurities in the Vostok fluid probably did not come only from the original liquids. Part of them were probably added to these liquids both from the walls of the containers, barrels and pipes during their transportation to Vostok and from the drill itself and its cable during drilling operations.

3.2 - Outside -inside variation profiles

As first proposed by Ng and Patterson (1981), the investigation of the variations of the elemental concentrations from the outside to the inside of each core section as a function of radius is an unique way to determine how deep outside contamination has penetrated into each core section. Continuous decrease of concentrations from the outside to the inside indicates that outside contamination has penetrated to the very center of the core section: the analysis of this very center will then allow to get only upper limits of the original elemental concentrations in the ice. On the other hand, if an unambiguous plateau of concentrations is observed for at least two consecutive inner layers, this clearly indicates that outside contamination has not penetrated to the central parts of the core section: the analysis of the inner layers for which a plateau is observed will then allow to get the original elemental concentrations in the ice, providing of course that satisfying blank corrections have been made for the chiselling and analytical procedures.

For Na and Mg, excellent plateaus of concentrations were observed for all the Dome C and Vostok sections, Table 4. In

most cases, the plateau was reached from the second layer from the outside, as illustrated in Figure 1 a, b, which means that for Na and Mg, contamination did not penetrate beyond about 6 to 10 mm from the outside for most Dome C and Vostok core sections. For Na, the only two exceptions were for two Dome C sections (373.9 and 775.7 m), for which the plateau was reached only from the third layer from the outside (but the Na concentration for the second layer was only slightly higher than that in the third layer). For Mg, this last situation occured only for the 373.9 m Dome C section and for the 1,775.4 m Vostok section (but as for Na, the concentration in the second layer was only slightly higher than that in the third layer). For Mg, we even had two situations (Dome C 545.1 and 602.2 m, Figure 1 c) where the plateau started from the first layer (flat Mg concentration profile from the very outside to the center).

For K, Ca, Fe and Al, excellent plateaus of concentrations were obtained for most core sections, Table 4. However, for each of these elements, we did find a few core sections for which no plateau at all was obtained: Dome C 172.8 m for Fe and Al: Dome C 373.9 m for Fe; Dome C 775.7 m for K and Al; Vostok 1,850.4 m for Ca and Fe. When a plateau was obtained, it started at variable distances from the outside of the cores: in a very few cases, it started from the first layer, as illustrated in Figure 2 a (flat concentration profile from the very outside to the center). In many cases, the plateau started from the second layer, Figure 2 b. Finally, in some cases, the plateau was obtained only for the two most central layers of the core, Figure 2 c. It is interesting to note that for a given core section, the situation was in many cases changing from one element to another. For instance, for Dome C 172.8 m, the plateau started from the second layer for Ca and from the fourth

| - | | | | | | | | |
|-----------------|----|----|-----|----|-----|----------------|----|----|
| Core Section | Pb | Zn | Na | Mg | K | Ca | Fe | Al |
| Dome C | | | | | | | | |
| 172.8 | | | + | + | + | + | - | • |
| 300.6 | • | + | + | + | + | + | + | + |
| 373.9 | - | - | + | + | + | + | - | + |
| 451.9 | + | + | _ + | + | + | + | + | + |
| 500.5 | + | + | + | + | + | + | + | + |
| 545.1 | + | + | + | + | + | + | + | + |
| 602.2 | + | + | + | + | + | + | + | + |
| 670.5 | + | • | + | + | + | + | + | + |
| 775.7 | - | • | + | + | • | + | + | - |
| 796.9 | + | + | + | + | + | + | + | + |
| Vostok | | | | | | | | |
| 499.1 | + | - | + | + | + | + | + | + |
| 851.6 | - | • | + | + | + | + | + | + |
| 1 425.3 | - | - | + | + | + | + | + | + |
| 1775.4 | • | - | + | + | + / | + | + | + |
| 1850.4 | • | • | + | + | + | - | - | + |
| 2026.3 | + | - | + | + | + | ₂ + | + | + |
| | | | | | | | | |

<u>Table 4</u> - Investigation of the outside - inside variations of the concentrations of eight elements in various sections of the Dome C and Vostok cores. Symbol + indicates that a satisfying plateau of concentrations was obtained in the inner part of the core for at least two consecutive veneer layers. Symbol - indicates that no satisfying plateau was observed.

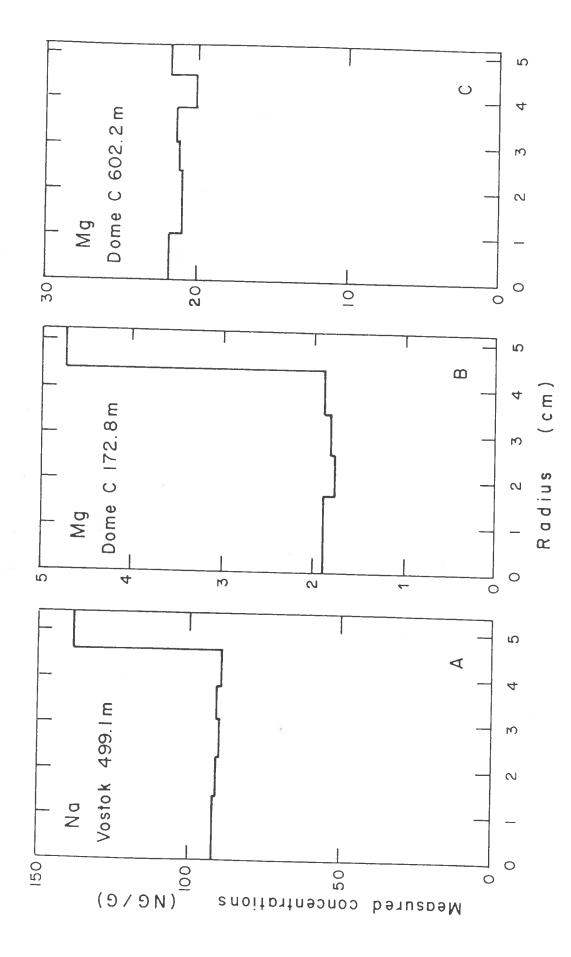


Figure 1 - Variations of elemental concentrations from the outside to the inside of three core sections as a function of radius. a) Na, Vostok 499.1 m. b) Mg, Dome C 172.8 m. c) Mg, Dome C 602.2 m.

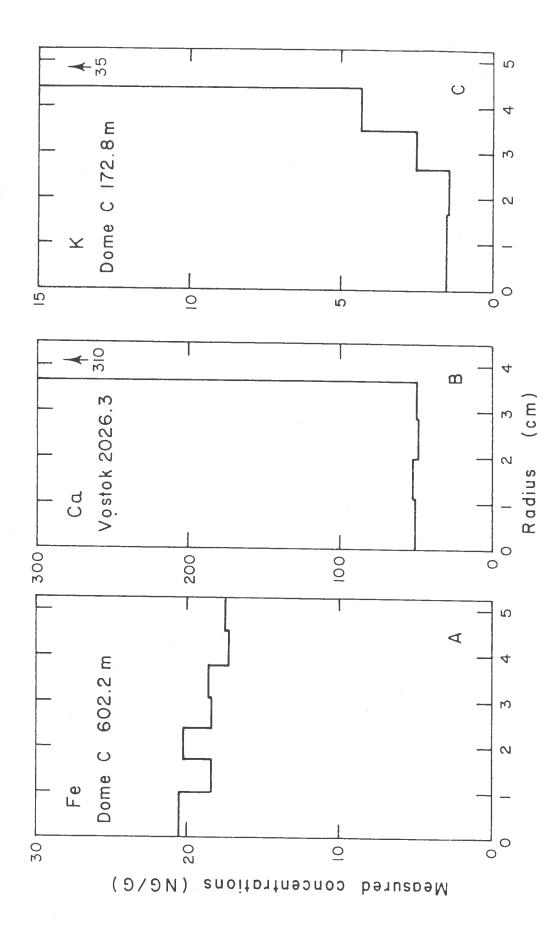


Figure 2 - Variations of elemental concentrations from the outside to the inside of three core sections as a function of radius. a) Fe, Dome C 602.2 m. b) Ca, Vostok 2,026.3 m. c) K, Dome C 172.8 m.

layer for K, and no plateau at all was obtained for Fe and Al.

Much more contrasting situations were observed for Pb and Zn, whose concentrations in Antarctic ice are several orders of magnitude lower than those of Na, Mg, K, Ca, Fe and Al (pg/g instead of ng/g level). For Pb, we found good plateaus for only six out of the ten Dome C core sections, and for only two out of the six Vostok sections, Table 4. For Zn, we also found good plateaus for six Dome C sections, but we did not get a plateau for any of the six Vostok sections, Table 4. When a plateau was obtained, it started at variable distances from the outside. In some cases, it started from the second layer, but in most cases was observed only for the most central layers of the cores. In some cases, Pb and Zn behaved similarly: for instance, a plateau was observed for both metals for Dome C 500.5 m, Figure 3 a; and no plateau was obtained for both metals for Dome C 172.8 m, Figure 3 b. But in other cases, they behaved differently: for Dome C 300.6 m, a plateau was obtained for Zn, but not for Pb, Figure 4 a; for Vostok 499.1 m, on the other hand, the opposite situation happened: no plateau was obtained for Zn, while a plateau was observed for Pb, Figure 4 b.

4 - CONCLUSIONS

Our results clearly indicate very contrasting situations for the eight elements investigated in the present study. For Na and Mg, our data suggest that outside contamination probably never penetrates beyond about 1-2 cm from the outside of the cores, both for Dome C and for Vostok: reliable Na and Mg values can then be obtained for both cores after discarding this outside part of the cores. For K, Ca, fe and

Al, the situation is not so clear: if in most cases, reliable data can be obtained from the analysis of the inner parts of the cores, however the fraction of the core to be discarded is rather variable. Moreover, in some cases even the most central parts of the cores appear to be contaminated. Finally, for Pb and Zn, the situation appears to be much more complicated: for these two metals, our data confirm that it is mandatory to draw complete outside-inside concentration profiles for each section in order to get reliable data whose quality can be clearly assessed.

Our data are for eight elements only. It will be necessary in the near future to perform such detailed studies for various other elements or compounds. Such investigations will be especially interesting for heavy metals such as Cd, Cu, Ag, Hg, Bi and Ir, whose concentrations are at or below pg/g level. Only when detailed reliable outside-inside variation profiles have been obtained for these heavy metals will it be possible to get reliable data on their concentration changes in Antarctic ice cores in order to reconstruct their past pre-man natural fluxes.

Our data are for thermally drilled cores only. It will be necessary in the near future to perform similar detailed studies for electromechanically drilled ice or firn cores in order to determine the contamination characteristics of such cores.

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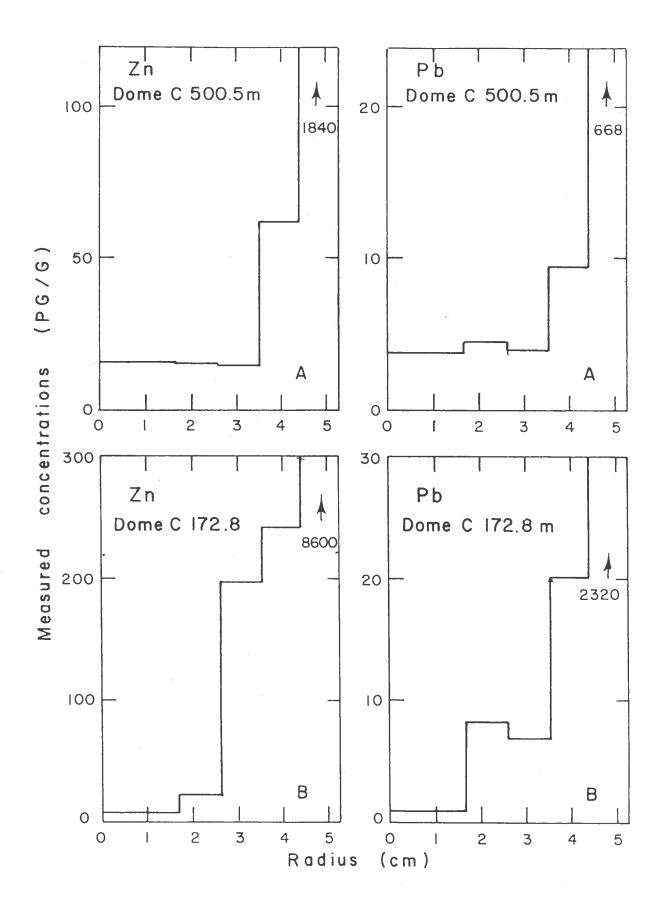


Figure 3 - Variations of Zn and Pb concentrations from the outside to the inside of two Dome C core sections as a function of radius. a) Dome C 500.5 m. b) Dome C 172.8 m.

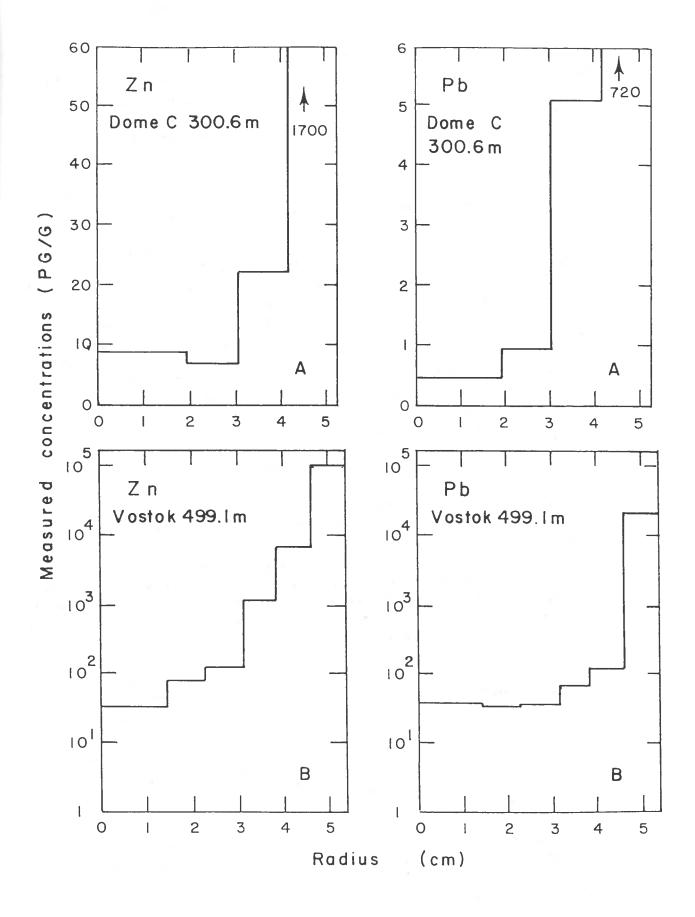


Figure 4 - Variations of Zn and Pb concentrations from the outside to the inside of one Dome C core section and one Vostok core section. a) Dome C 300.6 m. b) Vostok 499.1 m.

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