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NEW DEVELOPMENTS IN THE USE OF RADIOISOTOPES FOR DATING AND GEOPHYSICAL STUDIES

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ABSTRACT

Radioisotope measurements give key information regarding problems of men's future as nuclear waste disposal and the CO_2 increase due to fossil fuel combustion. In view of this special efforts regarding low level counting and the direct detection of low abundance isotopes (concentrations of 10^{-12} - 10^{-16}) are highly desirable. Very low backgrounds obtained with high pressure filled proportional gas counters in an underground laboratory are given and compared with estimates based on a semiempirical background formula. Then recent developments regarding high energy mass-spectrometry using accelerators are discussed. Samples representing only about one milligram of carbon can be used for ^{14}C-dating in a range of up to 50 000 - 70 000 years.

Due to new developments the radioisotopes ^{39}Ar, ^{81}Kr and ^{36}Cl are already or will be applicable to studies of environmental processes. The characteristics of these isotopes are discussed and the present state of studies based on them is given.

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INTRODUCTION

In these years the field of isotope application to earth sciences is experiencing a considerable upswing. Some of the reasons are:

a) Man is interfering in natural cycles in a no more negligible manner: - artificial radioactivity is produced due to nuclear weapon testing and also nuclear industry.

- The use of fossil energy results in the production of the CO_2 and SO_2. During the last decades an increase in the atmospheric CO_2 has been observed. Model calculations indicate that already for the beginning of the next century a significant climatic change is to be expected because of the alteration of the terrestial radiation balance based on an assumed exponential increase of the atmospheric CO_2 level.
- Nuclear and other waste is produced which needs to be stored outside the biosphere. This leads to clear questions which are asked to scientists, such as
  - By how much will the atmospheric ^{14}C and CO_2 levels rise for assumed input functions?
  - Will there be either natural or anthropogenic climatic changes leading to socioeconomic problems?
  - What does an optimum solution for the deposition of industrial waste look like?

To find answers to questions of this kind, isotope studies are of unique value because they yield information which combined with model calculations lead to a quantitative understanding of environmental processes and their history.

b) There has been a considerable progress regarding the measuring technique of radioisotopes:

- By improving the conventional detection method natural ^{14}C-activities could be measured with a precision of 2‰, yielding insight into the history of the modulation of cosmic radiation by solar and terrestrial magnetism (e.g. Radiocarbon 1980).

- Considerable reductions of the background of low level counting systems enabled the measurements of isotopes with much lower specific activities than that of ^{3}H and ^{14}C. Examples are ^{37}Ar and ^{39}Ar.
- By the use of accelerators several laboratories in USA achieved mass-spectrometric measurements of isotopes with abundance ratios of 10^{-12} to 10^{-16} (e.g. Radiocarbon 1980). Measurements of ^{14}C, ^{10}Be and ^{36}Cl are now possible on much smaller samples than hitherto were used. This will not only result in an enlargement of the spectrum of isotope applications. Also a new group of scientists will be attracted by the field of natural isotope research and will contribute with new methods and skills.

In this paper some recent developments in the use of radioisotopes for studies of processes in nature are discussed.

The environmental system

The ultimate aim of natural isotope research is the quantitative understanding of the processes taking place in the environmental system which is schematically shown in Fig.1.
Isotopes give information on circulation and mixing processes in the atmosphere. Natural and artifical radioisotope production leads to a labelling of atmospheric constituent components. Some of the radioisotopes live long enough to be detectable in systems exchanging relatively rapidly with the atmosphere like the oceans, the biosphere, the water on the continents and the glaciers. Radioisotope measurements on samples from these systems enable the determination of turnover times and dilution processes,since due to radioactive decay and dilution the specific activities of all these isotopesare smaller in these systems than in the atmosphere.

Phase transitions, diffusion processes and chemical reactions lead to isotopic fractionation providing additional information e.g. on temperature(stable isotopes of the water molecule) or on origins from the different subsystems. (E.g. the carbon in the different exchanging systems such as the atmosphere, biosphere, ocean and sediments has different $^{13}C/^{12}C$ ratios. $\delta^{13}C$ measurements on atmospheric samples therefore help to disentangle contributions from the different reservoirs).

In each of the systems mentioned above records are formed such as in tree-rings, peat bogs, sediments and natural ice in which past isotopic ratios are preserved. Their measurements provide information on the history of the systems.

Low level counting

The natural concentrations of cosmic ray produced radioisotopes are very low.
Orders of magnitude of concentrations are:

10^{-2} dpm / g H_2O for ^3H in groundwater or ocean water
10^{-8} dpm / g H_2O for ^{39}Ar, ^{32}Si in groundwater or ocean water
10^{-1} dpm / g ice for ^3H, fission products in ice
10^{-7} dpm / g ice for ^{39}Ar, ^{32}Si, ^{14}C, fission products in ice
10^{-8} dpm / g ice for ^{36}Cl, fission products in ice

If samples with such low radioactivity concentrations are to be measured by counting the radioactive decay rates, attempts are necessary to reduce the background values of the detectors.
In our institute at Bern we have the possibility to work in an underground laboratory, about 70 m of water equivalent below the cellar of the building. In the following the background results for proportional gas counting systems are summarized. In Fig. 2 the backgrounds obtained in the institute are compared with those obtained in the underground laboratory. Over the range of counter volumes from 22 cm^3 to > 1000 cm^3 background reductions by a factor of 2 to 4.5 were obtained.

In Fig. 3 we give for several counters the results of our attempt to estimate the different background components:

- surface activity of the counter wall ($S_{\alpha\beta}$)
- muons not detected by the anticoincidence systems ($\alpha \cdot \phi$)
- γ-induced electrons ($S\gamma e$)

The background reduction is mainly due to the reduced number of not cancelled muons (The muon flux is by a factor of 11 lower in the underground laboratory than in the cellar) and due to a reduced γ-flux. The latter indicates that in a normal laboratory with radioactively pure shielding the γ-flux is to a great extent due to interaction of cosmic radiation with the shield material. We have the impression that in the underground laboratory at least half of the remaining background is due to radioactive contamination of the counter and shield material.

The underground laboratory backgrounds obtained and displayed in Fig. 3 are lower than those reported by most other authors. Similarly low backgrounds are obtained by Stuiver in an underground laboratory (Stuiver et al. 1976) and by Nydal (Nydal et al, 1975), making use of a special shield as well as by Tans and Mook (Tans,1978).

The backgrounds we expect in the underground laboratory for copper counters of different size and filled up to different pressures with different counting gases can be estimated with the following empiric formula:

$$\begin{aligned} BG \cong\ & 4 \cdot 10^{-4}\ (\text{cpm/cm}^2) \times O \quad (O = \text{surface } (\text{cm}^2)) \\ & + 10^{-2}\ (\text{cpm/g}) \quad \times m \quad (m = \text{mass of counting gas (g)}) \end{aligned}$$

Examples are backgrounds for ^{39}Ar counters with P - 10 (90 % argon, 10 % methan) filling gas:

a) $V = 16\ \text{cm}^3$, $O = 50\ \text{cm}^2$, filling pressure 28 at → 0,72 g Ar;
$BG = 0.03$ cpm

b) $V = 10^3 \text{cm}^3$, $O = 600\ \text{cm}^2$, filling pressure 4.5 at → 7,2 g Ar;
$BG = 0.5$ cpm

The calculated backgrounds correspond well to the measured ones. The formula given above, which includes a surface α,β-contamination of $2 \cdot 10^{-4}$ cpm/cm^2, clearly demonstrates the importance of the surface effect. The smaller the counter the larger is its relative importance. An optimum ratio of background to sample size is therefore obtained if the sample is counted using a filling pressure as high as possible (see example a).

Even with such extremely low backgrounds many possible applications of environmental radioactivity studies cannot be made use of, since too large samples would be required. For radiosotopes with a long half-life the measurement of the radioactivity makes pure use of the potentially available information:

N_{obs}	=	λ	$\cdot$	N	$\cdot$	Δt
Number of atoms observed		decay constant		Number of atoms in the sample		period of measurement

and

$$\frac{N_{obs}}{N} = \lambda \cdot \Delta t = \frac{1 \cdot 3x10^{-2}}{T_{1/2}} \qquad T_{1/2} = \text{half-life in years}$$

Table 1: ratio of observed number of atoms to number of atoms in sample. It shows which fraction of isotopes with different half-life decays during a counting time of one week.

Isotope	$T_{1/2}$ (y)	N_{obs}/N
^{39}Ar	270	$5 \cdot 10^{-5}$
^{14}C	5730	$2,3 \cdot 10^{-6}$
^{81}Kr	210000	$6,3 \cdot 10^{-8}$
^{36}Cl	310000	$4,3 \cdot 10^{-8}$
^{10}Be	1500000	$8,9 \cdot 10^{-9}$

Mass-spectrometry basedon the use of accelerators

Many scientists have been aware of the relatively bad use which is made of the available information when long lived isotopes are measured by determining their radioactive decay rate (Oeschger 1970).

In 1977 two laboratories in USA published first results on the determination of radioisotopes using accelerator based mass-spectrometry (Muller 1977 and Bennet 1977).

In conventional mass-spectrometry, ions are first accelerated to several KeV and then separated by magnetic and electric fields according to their mass to charge ratio. The highest sensitivity ever achieved for ^{14}C measurements was $1:10^{11}$ (Anbar 1974) which is still one order of magnitude above the modern $^{14}C/^{12}C$ ratio in the atmosphere. The main contributions to the background limiting the sensitivity of a conventional mass-spectrometer are:

- contaminating molecules with masses equal to those of the atoms which are actually searched for
- contaminating particles which reach the detector because of wall or gas scattering
- contaminating particles reaching the detector after some improbable charge exchanges.

The use of accelerators increases the sensitivity of mass-spectrometry by several orders of magnitude.
Due to the high energy of the ions (∿MeV/amu) all molecules are completely dissociated and this background component is eliminated. In addition nuclear detectors (ΔE, E) can be used to determine the total kinetic energy E and the rate of energy loss dE/dx.

Fig. 4 shows the experimental arrangement of an accelerator based mass-spectrometer which is under construction at the Laboratorium für Kernphysik, Eidg. Technische Hochschule, Zürich (Suter, 1979).

In the sputter ion source negative ions of the sample to be measured are formed and accelerated to 60 KeV. The magnetic mass analyser selects the mass of the ions to be counted. In the EN-Tandem accelerator (6 MV) the negative ions are accelerated towards the terminal where electrons are stripped off. Positive ions are formed according to a charge state distribution. After a second acceleration back to ground potential the ions leave the tandem and enter the electrostatic deflector which selects the particles with the proper E/q (energy/charge) ratio. The final magnetic mass analyser separates the particles according to their ME/q^2. The high intensity beam currents of stable isotopes (^{12}C, ^{13}C, ^{9}Be, ^{35}Cl, ^{37}Cl) are measured with Faraday cups. The rare isotopes (^{14}C, ^{10}Be, ^{36}Cl) are counted in a gas-ΔE-E-counter determining unambigously the atomic number Z and the mass M.
The isotopic ratios are determined by injecting the different isotopes sequentially.

To avoid variations in the isotopic ratios due to instabilities of the terminal voltage the isotopes are injected in pulses of very short duration (∿ 0.5 ms). The fast change from one isotope to another is achieved by varying the energy of the beam through the inflection magnet.

Table 2 shows some characteristics of the system shown in Fig. 4.

overall efficiency	10^{-2}
neg. ion current	50µA C^-, equivalent 0.5 µg/min
count rate of 5730 y carbon sample	∿ 1000 counts/min
measuring times	10 - 30 min
size of C-sample	< 1 mg

Assuming an efficiency of 10 % for the production of negative ions an overall efficiency of the system of 1 % should be achieved. The current of negative ions leaving the ion source is expected to be 50 µA which is equivalent to a carbon flux of 0.5 µg/min. This leads to a count rate of about 1000 ^{14}C-atoms per minute if the sample has an age of one half-life (5730 y).

The time needed to date one sample is expected to be between 10 and 30 min. depending on its age. The sample size necessary is less than 1 mg carbon.

The dating period is limited by contamination problems and the not yet known background of the system to probably 50000 - 80000 years.

Examples of radioisotopes with low activities

For the solution of some problems (like nuclear waste disposal, CO_2 increase with possible climatic consequences) it is necessary to use the information of the full spectrum of radioisotopes. Beside the traditionally used isotopes ^{3}H, ^{18}O and ^{14}C in hydrology e.g. ^{39}Ar, ^{32}Si, ^{36}Cl, ^{81}Kr, ^{85}Kr, $^{234}U/^{238}U$ and others should be considered (Davis, 1978). In the following short summaries regarding three of these isotopes with low activities are given.

^{39}Ar

^{39}Ar is produced exclusively by cosmic radiation. Its atmospheric specific activity has been measured to be 0.107 ± 0.005 dpm/lt argon (Loosli, 1981). Its half-life of 269 years allows dating in a range between about 30 and 1000 years. As noble gas isotope ^{39}Ar has a great potential for geophysical applications.

^{39}Ar has been successfully applied for ice dating on samples from Greenland and Antarctica, showing that the obtained results are in agreement with ages determined by other methods (Loosli, 1981). For the 20 ice samples which have been measured until now an amount of a few tons of ice was molten and the gases were extracted in situ. About 2 lt of argon were separated from the gases and afterwards purified. Simultaneously the ^{85}Kr activity was determined for the same samples to estimate a possible contamination by ambient air during collection and processing. Based on the ^{85}Kr results for some of the samples significant corrections of the ^{39}Ar results had to be made, whereas for the others an upper limit of a few % of modern ^{39}Ar activity contribution by contamination could be estimated.

The ^{39}Ar activity is mainly measured in the high pressure counters described in table 3.

Table 3 Proportional Counting System for ^{85}Kr and ^{39}Ar

Counter		Isotope	Filling pressure (bar)	Composition of gas	Background (B) (counts/min)	Net modern counting rate (S)	S^2/B
Volume (cm^3)	Material						
22	Cu	^{85}Kr	5	P-10	0.03	70% yield	
						(counts/min)	
100	Lucite	^{39}Ar	18	P-5	0.12	0.13	0.14
100	Cu	^{39}Ar	19	P-5	0.10	0.18	0.32
50	Cu	^{39}Ar	36	P-5	0.08	0.14	0.24

^{39}Ar was also applied to dating of groundwater samples. Contamination of the samples by ambient air was usually below a few percent as determined by ^{85}Kr results. A decrease of the specific activity with increasing water "age", measured e.g. by ^{14}C, was observed (Loosli and Oeschger, 1979). But if ^{14}C- and ^{39}Ar-model ages are compared discrepancies are observed. For some aquifers the apparent discrepancies might be explained by mixing of different water masses (Loosli and Oeschger, 1979), but for other aquifers the discrepancies cannot be explained by mixing alone.

At present two explanations have to be considered: In aquifers with relatively high neutron-fluxes and potassium-contents ^{39}Ar may be produced underground to such a degree that the in situ produced ^{39}Ar activity in the water exceeds the cosmic ray produced level. Such high activities have been measured recently for a few aquifers in granitic rocks (Loosli, 1981). On the other hand aquifers were found for which underground production of ^{39}Ar can be neglected: two samples collected in Ingolstadt, FRG, showed ^{39}Ar activities below 7 % modern (statistical counting error).

On the other hand the interpretation of ^{14}C results is not without problems too. Regarding uncertainities of the initial ^{14}C concentration and further net carbonate dissolution in the aquifers information is obtained e.g. by using ^{13}C measurements and evolution models based on chemical analyses (e.g. Reardon and Fritz, 1978). In these model calculations, however, a chromatographic delay of ^{14}C due to isotope exchange between solved and solide phase is not included. This would lead to an overestimation of the real water ages by the ^{14}C-model ages. At present we feel that both explanations - underground production for ^{39}Ar and isotope exchange for ^{14}C - have to be studied and understood in more details.

The application of ^{39}Ar for studying ocean water mixing and circulation is at the beginning. Mainly due to the half-life corresponding ideally to characteristic times of ocean processes this isotope looks very promising. Untill now only a few samples extracted from ocean surface water were measured for ^{39}Ar and ^{85}Kr. Whereas samples from Monaco showed 100 % modern ^{39}Ar activity indicating, as expected, gas equilibration between atmosphere and ocean surface, two samples collected at Mc Murdo sound, Antarctica, yielded only about 75 % modern ^{39}Ar-activity (Loosli, 1981). This probably has to be explained by assuming that the sampled upwelling water was not yet in isotopic equilibrium with the atmosphere. Until now ^{39}Ar was not included more often in oceanographic studies because large amounts of ocean water ($\sim$ 1.5 m^3) have to be collected and degassed. If in the next future the ^{39}Ar activity could be measured by resonance ionisation spectroscopy (RIS, see chapter on ^{81}Kr), the amount of water could be reduced by about 3 orders of magnitude. It can be expected that then similar to ^{14}C and ^{3}H ^{39}Ar would be included in most isotope investigations in oceanography.

^{36}Cl

^{36}Cl is a radioactive isotope with a half-life of 3.1 x 10^5 y. It originates mainly from three different sources:

- spallation reactions of cosmic ray particles with atmospheric argon
- capture of cosmic ray produced neutrons by ^{35}Cl in seawater and rocks
- capture of neutrons produced during nuclear weapon tests by ^{35}Cl in seawater

The direct influx of ^{36}Cl associated to extraterrestrial material is negligible.

The ^{36}Cl produced by spallation of ^{40}Ar in the stratosphere (2/3) and the troposphere (1/3) gets attached to aerosols and is removed from the atmosphere by rainfall and dry deposition with residence times of weeks to months. Lal and Peters (Lal, 1967) estimated the global cosmic ray induced production rate of ^{36}Cl to be $1.1 \cdot 10^{-3}$ atoms / cm^2sec. The deposition rate depends on the latidude due to cut off effects caused by the geomagnetic field and latitude dependent injection of stratospheric air into the troposphere.
The concentration of ^{36}Cl in surface ice and rainwater samples ranges from 10^6 to 10^7 atoms ^{36}Cl/kg (Finkel, 1980).
Although first measurements by β-counting of ^{36}Cl were reported in 1955 (Davis, 1955) and the potential suitability of this isotope as chronometer and tracer in geophysics was pointed out, only little further work has been done since that time because of the difficulties to measure the extremely low specific activity.
By the development of the new high energy mass-spectrometry based on accelerators the sample size necessary for a ^{36}Cl measurement has been reduced from a few tons to about 1 kg of water or ice opening a broad field of applications:

The polar ice sheets represent ideal archives of the precipitation of at least the last 10^5 years. Studies of the isotope composition of ice cores provide information about the time variations of the production of isotopes in the atmosphere, extending similar studies of ^{14}C in tree rings back to 10^5 years.
Due to its half-life ^{36}Cl is suggested to be a very promising isotope for dating purposes covering the entire Pleistocene era (10^4 - 10^6 y) possibly combined with ^{10}Be or ^{26}Al. Recently the advantages of ^{36}Cl for dating of ground water have been pointed out in connection with the search for suitable nuclear waste storages (Davis 1978). First measurements of ^{36}Cl in environmental water smples have been published recently (Elmore 1979).

^{81}Kr

^{81}Kr is a cosmic ray produced isotope with a half-life of $2{,}1 \cdot 10^5$ years. As noble gas isotope and conservative tracer it is therefore ideally suited for dating of very old groundwater and possibly polar ice. It furthermore could be applied together with ^{36}Cl, ^{10}Be, ^{26}Al and ^{14}C in ice samples to give information on the cosmic ray production rates of isotopes and their time variations.
The specific atmospheric activity was measured recently to be about 0.07 dpm/lt krypton (Kuzminov and Pomansky, 1980), corresponding to about $4 \cdot 10^{-9}$ dpm/lt H_2O. For the activity measurements on groundwater by counting therefore an amount of 10^2-10^3 m^3 of water would have to be outgassed, which is considered to be too great an effort. But if the relatively large number of ^{81}Kr atoms could be used directly about 600 atoms per lt of H_2O with a Kr : ^{81}Kr ratio of(about $2{,}5 \cdot 10^{12}$: 1), much smaller water samples would have to be processed. Since negative krypton ions cannot be produced the present Tandem accelerator based measuring technique is not applicable. However a measuring technique which is a combination of laser spectroscopy and mass-spectrometry seems feasible (Hurst et al, 1980). The laser would select the desired element, the mass-spectrometer the desired mass. Although the development and testing of such a system looks

very difficult and unexpected problems cannot be excluded, due to the ideal characteristics of ^{81}Kr the effort seems worthwile. Such a system even could possibly be applied to the measurement concentrations, although additional problems have to be solved due to the low ^{39}Ar:^{40}Ar ratio of about $8 \cdot 10^{-6}$ in a modern sample.

Conclusions:

Due to developments in measuring techniques in addition to ^{3}H and ^{14}C a series of other isotopes are becoming applicable to studies of environmental processes.

Among the new applications are

- ^{39}Ar measurements on ocean water samples to determine the age distribution of ocean water and to get information on the degree of isotope equilibration of surface water. Both informations are important for estimates of the uptake of excess CO_2 by the oceans.
- Determination of the age distribution of groundwater from potential nuclear waste disposal sites. ^{36}Cl and ^{81}Kr are isotopes with half-lives of ∿ 200 000 years comparable to the desired time period during which nuclear waste should remain separated from the biosphere.
- ^{36}Cl and ^{10}Be measurements on polar ice core samples are expected to yield information on the last 100 000 years history of solar and terrestrial magnetism.

  This information will be used to investigate a possible relationship between solar activity and climate.

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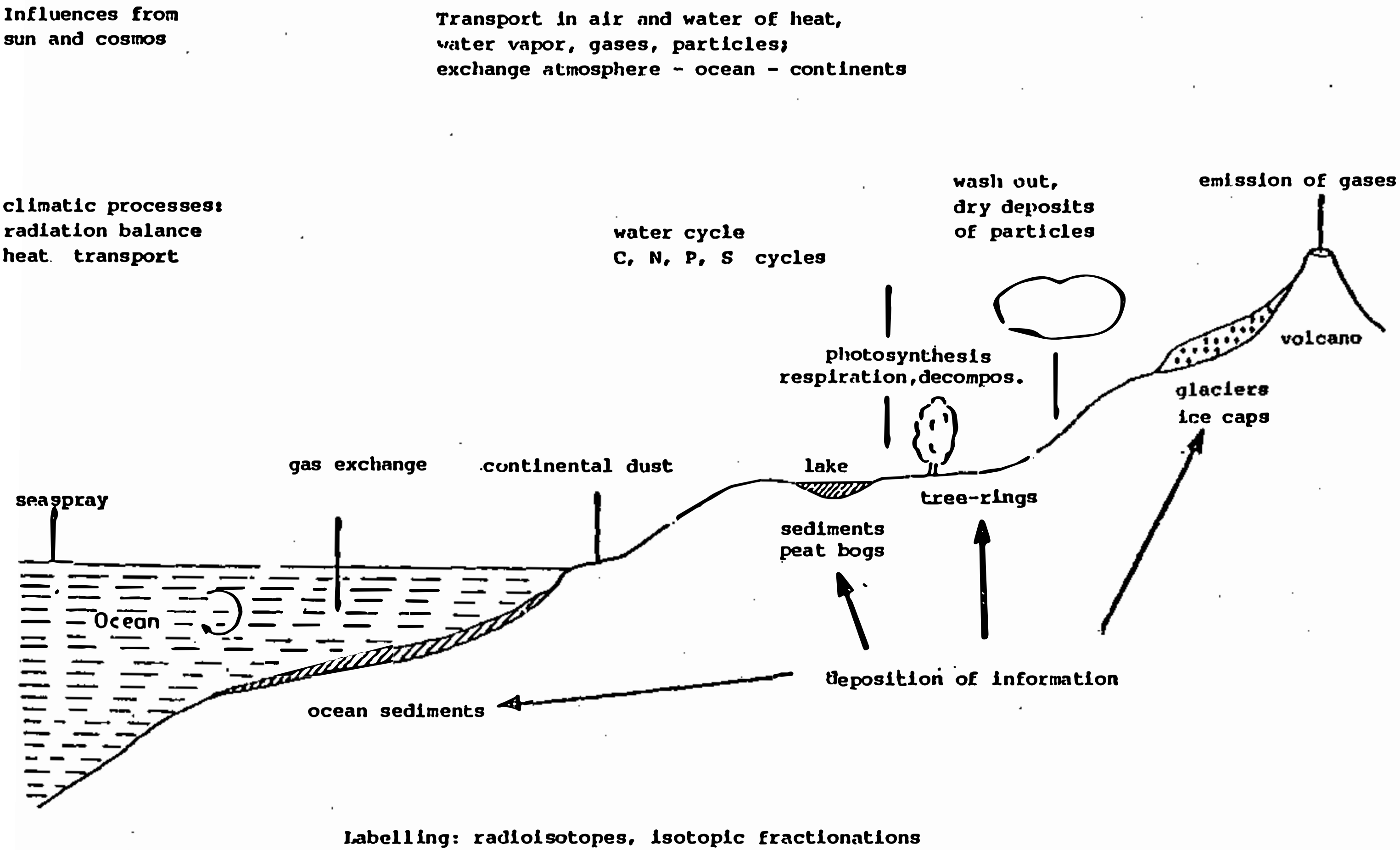
Fig. 1 Environmental System



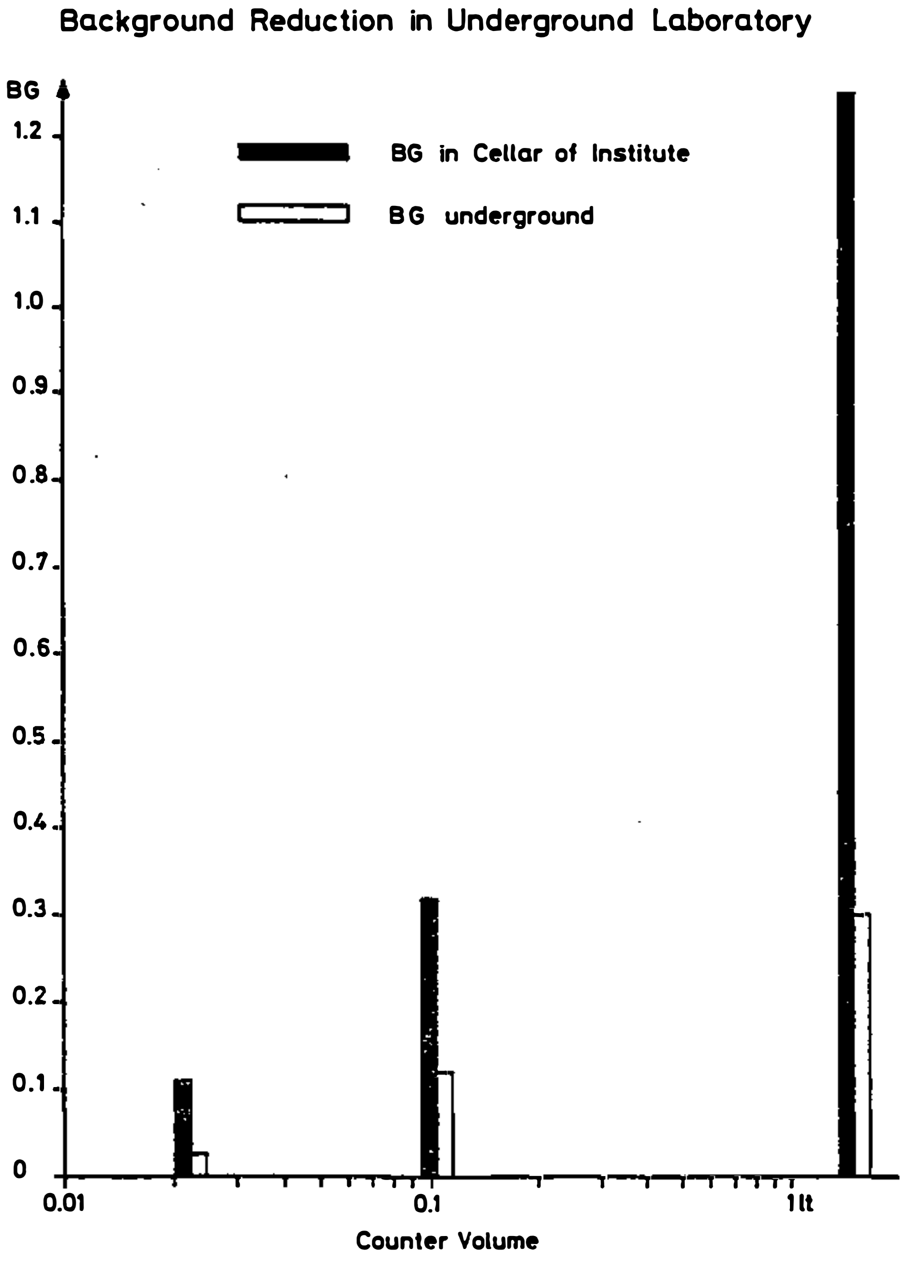
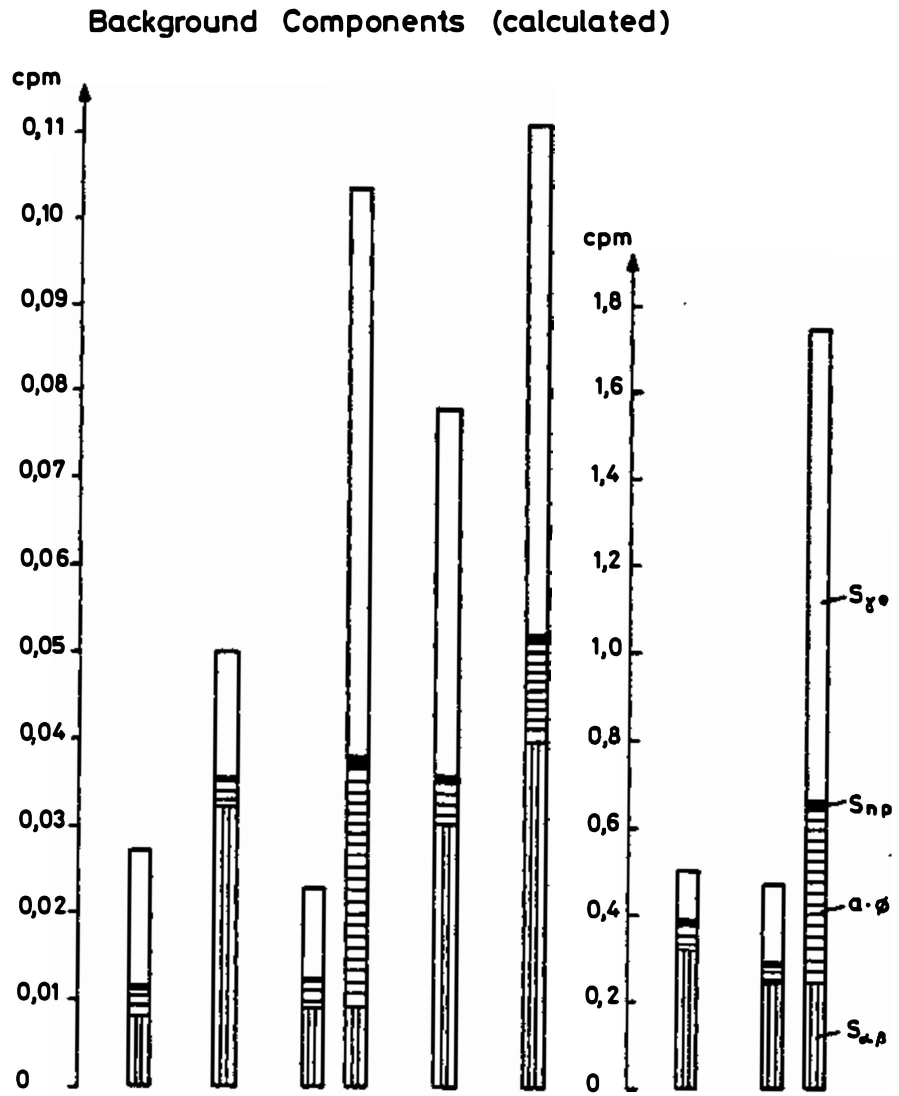


Fig. 2 Background reduction in an underground laboratory. For different counters reduction factors of 2-4.5 could be obtained by moving underground. As examples, background values are given for three counters: a 22cc Cu counter filled with 5 at P-10, a 100cc Plexi counter filled with 17 at P-5, and a foil counter with internal anticoincidence for ^{14}C filled with 1.6 at CH_4.



counter	16-06	16-01	22	1(50cc)	2(100cc)	Cu(1lt)	Plexi (1lt)
pressure [at]	28	26	5	36	19	4,5	4,5
$S_{\alpha\beta}\left[\times 10^{-4} \frac{cpm}{cm^2}\right]$	1,6	6,5	2,3	3,5	2,4	5,2	3,9
Location	ug	ug	ug I	ug	ug	ug	ug I

Fig. 3 Calculated background components. For proportional counters with different volumes (16, 22, 50, 100, 1000cc) and different filling gas pressures, the estimated background components are given for our underground laboratory (ug) and the laboratory in the Institute (I). $S_{\gamma e}$ is the component induced by γ-rays in the counter volume or in the wall; S_{np} is the component induced by neutrons, $a \cdot \phi$ is the component proportional to the muon flux, and $S_{\alpha\beta}$ is the component from self-activity in the counter wall. All counters are constructed from copper with the exception of the 1 lt plexi counter.

Fig. 4 Schematic diagram of the high energy mass-spectrometer at Zurich