Development of Accelerator Mass Spectrometry at the Lund Pelletron

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Abstract

Accelerator mass spectrometry (AMS) is a highly sensitive method for counting atoms. It is used for detecting very low concentrations of both radionuclides and stable nuclides. The main advantages of AMS compared to conventional radiometric methods are the use of smaller samples (mg size) and shorter measuring times (less than one hour). In AMS, rare isotopes from a sample material placed in the ion source of an electrostatic tandem accelerator are measured by counting individual atoms with nuclear detection techniques after acceleration to energies in the MeV range. A dramatic improvement in background rejection for AMS systems has, in the best cases, led to a 10^8 increase in sensitivity for isotope ratio measurements compared to the older technique of mass spectrometry. In this report some current applications of the AMS technique at the Lund Pelletron accelerator, as well as the recent improvements of the Lund system, are presented.

1. Introduction

One of the applications of nuclear physics techniques which has been greatly beneficial and vital to other fields of scientific endeavour is accelerator mass spectrometry (AMS) [1]. The power of AMS for extremely sensitive radioisotope measurements has been demonstrated extensively over the last twenty years. AMS has not only allowed refinements in the technique of carbon dating in the fields of archaeology and quaternary geology, but has also been demonstrated to be useful e.g. for: tracing ^{26}Al in living systems; tackling hydrogeological problems by the use of ^{10}Be and ^{36}Cl; estimating ^{41}Ca production from nuclear weapons testing and following transport of ^{129}I and transuranes from nuclear facilities.

In AMS, rare isotopes from a sample material placed in the ion source of an electrostatic tandem accelerator are measured by counting individual atoms with nuclear detection techniques after acceleration to energies in the MeV range. A dramatic improvement in background rejection for AMS systems has, in the best cases, led to a 10^8 increase in sensitivity for isotope ratio measurements compared to the older technique of mass spectrometry. AMS evolved from nuclear physics laboratories and several tandem accelerators originally installed during the 60's and 70's (and therefore of insufficient energy for today's nuclear physics experiments) have been rebuilt to be used for AMS. However, the best achievements have been obtained with relatively small tandem accelerators that are designed for and dedicated to AMS measurements.

AMS is a development of traditional mass spectrometry by including an MV tandem accelerator, see Fig. 1. AMS can in principle be described by the following steps: negative ion production – selection of mass by a magnet – acceleration – charge exchange from negative to positive – acceleration – selection of [mass × velocity/charge] and/or [velocity] and/or [energy/charge] in one or several analysing units – and finally particle detection. The many steps from ion source to detector cause a strong reduction of the background. It is three especially important characteristics of AMS with tandem accelerators that provide the possibility to measure low isotope ratios, for example for $^{14}C/^{12}C$ down to 10^{-15} (see Fig. 2):

1) Interference from some isobars can be excluded by using a negative ion source, for example $^{14}N^-$, $^{26}Mg^-$, $^{55}Mn^-$ and $^{129}Xe^-$ (all with negative electron affinity) when detecting ^{14}C, ^{26}Al, ^{55}Fe and ^{129}I.

2) An important unit is the stripper system in the high voltage terminal of the accelerator where the negative ions become positive. In this process nearly all molecules will be broken up. A few molecules with maximum charge state 2+ will survive. By selecting charge states higher than 2+ by the analysing unit after the accelerator all interference with molecules will be avoided (in the case of ^{14}C, for example $^{12}CH_2$ and ^{13}CH).

3) The high final energy of the ions, tens of MeV, means that there is easy detection of the particle energy and that individual ions can be counted.

The most important advantage of AMS is that the amount of material needed is very small. For ^{14}C it requires at least a factor of a thousand less material as compared to conventional radiometric counting.

In this report some current applications of the AMS technique at the Lund Pelletron accelerator, as well as the recent improvements of the Lund system, are presented. Besides a
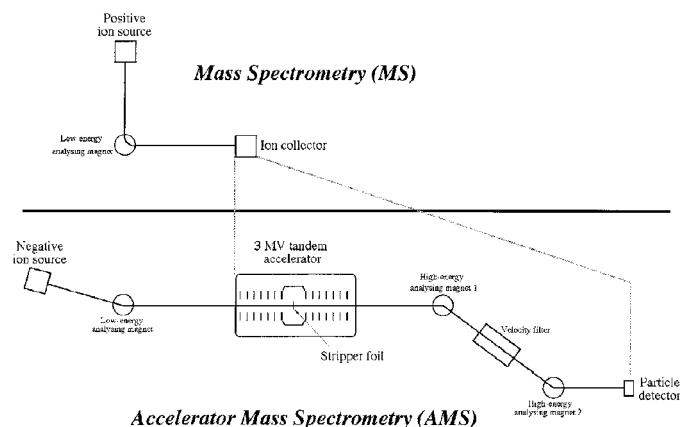

Fig. 1. Comparison between traditional mass spectrometry (MS) and accelerator mass spectrometry (AMS). The figure shows the AMS-system in Lund.
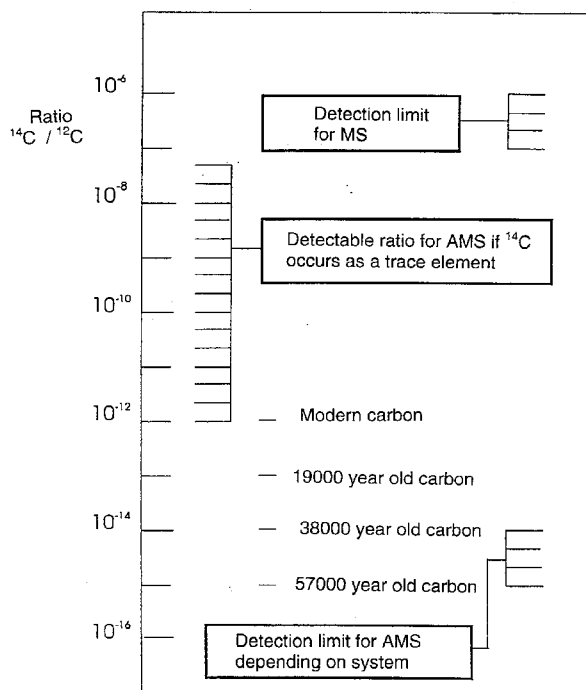
Fig. 2. Coarse comparison between the smallest detectable $^{14}C/^{12}C$-ratios determined by traditional mass spectrometry (MS) and accelerator mass spectrometry (AMS) as well as with actual concentration levels.

considerable program for radiocarbon dating, the Lund system is used for: studies of environmental releases of reactor-produced ^{14}C by analysis of air and plant material in the vicinity of nuclear installations; studies of ^{14}C-labelled compounds used in clinical nuclear medicine; application of ^{14}C in food chemistry; and the development of a new detection technique using characteristic projectile X-ray counting of ^{59}Ni to be used for nuclear waste classification.

2. The Lund AMS-apparatus

The main part of the Lund AMS-system is a Pelletron tandem accelerator (model 3UDH, produced by NEC, Wisconsin USA) with a maximum terminal voltage of 3 MV. This machine was installed in the beginning of the 1970's. Some of the modifications made so far for changing this originally nuclear structure facility to a reliable and high quality AMS facility are described below.

A caesium-sputtering, negative ion source has been designed and installed [2,3]. One of the new features of the source is a mechanism for automatically cracking the caesium glass ampoule inside the oven when the source is evacuated. In order to maximise the negative ion beam current and to allow sputtering to occur over the whole sample area, the sample holder has a mechanism for moving the sample relative the caesium beam. The moving pattern consists of two superimposed motions – one circular and one vertical – and the motion speeds are chosen such that a complete scan takes about 4 min. The source is also equipped with a multiple sample holder. Switching between different samples takes up to 3 min depending on the position on the wheel. If a running initialization of all the samples has been done, the source output is stabilised within seconds. The sample wheel may be replaced with a new wheel through a gate valve. The changing procedure takes 10 minutes. The

stability of the beam current is relatively good. The long-term stability (several days) is better than 5% and the short-term stability (less than an hour) is better than 1%. An ionization efficiency of 3% is obtained for carbon. For a carbon sample with a ^{14}C-level of 10^{-8} or less (which is relevant for AMS measurements), it is not possible to detect a memory effect. This low memory effect is obtained by protecting the samples not in use by a metal plate.

The source is followed by a remotely operated, four-blade aperture for defining the emittance of the beam emerging from the source. On the low-energy side of the accelerator all magnetic devices (except the inflection magnet) have been replaced by electrostatic devices giving the same focusing conditions for all isotopes. With the aid of beam optics calculations new lenses have been placed at optimum positions [4], which, together with a higher pump capacity just outside the tank, have increased the beam transmission through the accelerator, especially for heavy ions. Measurements of the beam transmission through the accelerator [5] and of the stripper process on gases and foils have been performed [6]. These studies have led us to a redesign of the stripper arrangement and we have just recently reported about the installation of a new gas stripper system, including terminal pumping [7]. The terminal pumps, one on each side of the stripper arrangement, are recirculating the stripper gas. The advantages of this new system compared to the old gas stripper system is that a higher beam current can be taken through the accelerator, and a better beam quality and more stable conditions are obtained during the experiments.

A magnetic quadrupole triplet, a velocity selector and a second dipole have been installed on the experimental beam line after the analysing dipole magnet. With the velocity selector mounted for vertical deflection, a good velocity resolution is obtained with insensitivity to small fluctuations in beam angle out of the analysing magnet. The image slits of the analysing magnet are kept wide open and the transmission from these to the target is close to 100%. The particle identification and measuring system consists of a surface barrier detector. A computer control system has been developed with the purpose of enhancing reliability and speeding up data taking. The control system sets and measures the fields of the inflection magnet, the analysing magnet and the velocity selector. It also operates a few beam stops and controls the change of samples and the sample motion mechanism. In this way unattended runs can be performed.

The sample preparation technique used in Lund for ^{14}C experiments is based on the production of elemental carbon by the catalytic reduction of carbon dioxide, mixed with hydrogen gas, over an iron catalyst [8]. Three slightly different systems are used, two for samples with ^{14}C-activities above the contemporary atmospheric level and one for dating purposes with samples of low ^{14}C activity. The three apparatuses are located in separate laboratories to avoid contamination of the low-activity samples. Carbon dioxide is released from carbonate samples by adding phosphoric-acid and organic matter is combusted in the presence of CuO. Samples of high activity can be diluted with ^{14}C-free CO_2 before being reduced to solid carbon. The total reduction time for each sample containing about 5 mg of carbon is about 3–4 h. By using a number of reduction units,

which can be separated from the rest of the sample prep-aration system, several samples can be processed con-currently.

3. Performance

The isotopic ratios are determined relative to a standard. The background is evaluated based on measurements of a ^{14}C free sample. The unknown sample and the standard are prepared in exactly the same way, ensuring that most systematic errors cancel.

The $^{14}C^{3+}$ ions are counted by the particle detector and the count rate is normalised to the $^{13}C^{3+}$ current, measured just in front of the last dipole. Thus the settings of the inflection magnet, the analysing magnet and the velocity filter all have to be changed when cycling between ^{13}C and ^{14}C. The samples for ^{14}C-dating are normally measured independently at least 7 times for 300 s, with each measurement divided into 5 intervals. The samples with ^{14}C-activity above the contemporary atmospheric level are measured in a similar way, although in shorter total time. In this way the short and long term stability can be monitored. One batch of 20 samples usually includes 2 oxalic acid standards and 1 background sample of processed anthracite. Each sample is normalised to the oxalic acid standard, which is measured every fifth time in the measurement sequence. A sample of known activity is put into every measurement series to check the quality of the analysis. The precision in the measurements is 1% (corresponding to ±80 y). With longer measuring times the statistical uncertainty can be further reduced, but then systematic errors must also be considered. These can depend on drift of power supplies, change of the stripper foils, beam current dependent effects from sample to sample etc. The detection limit will be set by interference in the detector. The introduction of a dipole magnet placed between the velocity selector and the particle detector has reduced the machine background from ca. 2% to <0.3% of modern carbon. The ^{14}C yield from a CuO combusted anthracite sample which has been processed as described above corresponds to 40 000 ^{14}C y. AMS has a very large dynamic range for measuring the isotope ratio. The upper limit is set by the count rate in the particle detector. However, this limit can be increased a few decades by diluting the original material. The lower limit is set by other samples that have passed the sample preparation system, by memory effect in the source, or by ions that are trans-ferred through the accelerator system giving false ^{14}C signals in the detector.

4. Applications at the Lund AMS facility

4.1 Radiocarbon dating

Many radionuclides, such as ^3H, ^{10}Be, ^{14}C, ^{26}Al, ^{32}Si, ^{36}Cl, ^{39}Ar and ^{81}Kr, are produced naturally in the terrestrial atmosphere as a result of nuclear reactions involving cosmic rays. ^{14}C, produced by reactions between slow sec-ondary cosmic neutrons and stable ^{14}N nuclei in the upper atmosphere, is quickly incorporated mainly into molecules of carbon dioxide, which are mixed throughout the atmos-phere and the hydrosphere, the latter being the main reservoir. A significant reservoir is provided by the bio-sphere because of the assimilation of carbon dioxide into plant tissue, and the consumption of these plants by animals. A secular equilibrium, constant to a first appro-ximation, is maintained in the atmosphere, hydrosphere and thus also the biosphere by the continuous production of ^{14}C in the atmosphere and the subsequent beta-decay of the radionuclide (maximum beta-particle energy 156 keV, $T_{1/2} = 5730$ y). ^{14}C constitutes about 10^{-10}% of the total amount of natural carbon, while the proportion of the stable isotopes ^{12}C and ^{13}C is 98.9% and 1.1%, respectively.

Natural ^{14}C can be used in numerous applications, of which radiocarbon dating, with the possibility for dating back to about 50 000 y [1], is among the best-known and most practised applications. Radiocarbon dating is based on the property that the activity concentration of ^{14}C in dead tissues can be used to calculate the time that has elapsed since death occurred. This is possible since all living organisms contain nearly the same proportion of radioactive carbon in their carbon stores and since, upon the death of the organism, the carbon which survives decomposition con-tinuously loses ^{14}C by its radioactive decay.

The ^{14}C dating program at the Lund AMS facility is a collaboration between the Department of Nuclear Physics and the Department of Quaternary Geology. About 200 ^{14}C datings are performed per year at the AMS laboratory. The most common types of material used for AMS dating in Lund are charcoal, wood, bone, peat, marine shells and terrestrial macrofossils.

4.2. Studies of ^{14}C releases from nuclear power plants

^{14}C is one of the radionuclides which are produced to dif-ferent degrees by neutron-induced reactions in all types of nuclear reactors. In a nuclear power facility the pro-duction of ^{14}C can occur in the fuel, the moderator, the cool-ant and the core construction materials mainly by the reactions $^{17}O(n,\alpha)^{14}C$ and $^{14}N(n,p)^{14}C$. Part of the ^{14}C created in reactors is continuously released as airborne effluents in various chemical forms (such as CO_2, CO and hydrocarbons) through the ventilation system of the power plant to the surrounding environment during normal reactor operation. The particular chemical form of the effluents depends on the type of facility. Another part of the ^{14}C pro-duced is released into the atmosphere from fuel reprocessing plants.

The environmental release of this reactor-derived ^{14}C leads to an increase in atmospheric specific activity and, hence, to an increased radiation dose to man. Because of the biological importance of carbon and the long half-life of ^{14}C, it is of interest to measure the ^{14}C releases, to study their pathways and to determine the chemical form of the effluents, as well as the incorporation of ^{14}C into living material in the environment of the power plants. It is believed that, of all nuclides released in routine operation by the nuclear power industry, ^{14}C is likely to produce one of the largest collective dose commitments [9].

An extensive investigation of the ^{14}C releases from some Swedish nuclear power plants has been performed at the Lund AMS facility. A one-year study has been performed [10] of the total air-borne ^{14}C effluents from two Swedish light-water reactors, one boiling water reactor (BWR) and one pressurised water reactor (PWR). Air emitted from
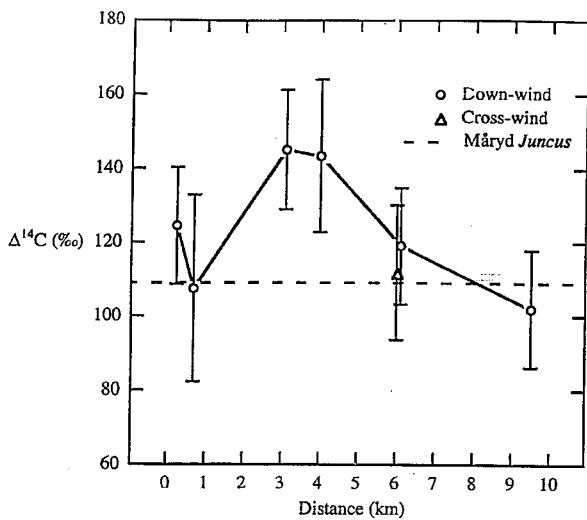
Fig. 3. The ^{14}C content of air samples collected at various down-wind distances and at one cross-wind site from the Barsebäck nuclear power plant. The 1996 year value of rush (*Juncus*) from Måryd (a "clean air" site 30 km east of Barsebäck) is also indicated. The ^{14}C content is given as the excess of ^{14}C (in ‰) compared to the 1950 year standard.

Fig. 4. The exhaled ^{14}C concentration from one volunteer at various times after oral administration of 32 nanomole ^{14}C-triolein. The dip in the curve after 6 days (A) resulted from excessive intake of food prior to sampling. This observation initiated 32 hours long and controlled fasting periods (B, C, D and E). During the fasting periods stored body fat is used and the catabolic end-product ^{14}CO$_2$ is exhaled to a higher degree than during normal intake of food. The results show that 30% of the activity is catabolised rapidly (biological half-life about 2 days), while remaining 70% of the activity has a very slow turnover (half-life several hundred days).

the stacks was collected continuously over two-week periods and the ^{14}C contents in the samples were analysed using AMS. The chemical form of the effluents has also been studied [11] and the ^{14}C concentration in willow leaves in the vicinity of a power plant has been measured [12]. Measurements of the ^{14}C activity concentration in annual tree rings and air around some Swedish nuclear power plants have been presented [13]. Further investigations are conducted to establish the ^{14}C levels in air and vegetation around nuclear facilities in Sweden as well as in some foreign sites [14].

As an example the ^{14}C content of air samples collected in the down-wind direction at various distances from the nuclear power plant of Barsebäck (south west Scania, 2 BWRs each 600 MW$_e$) is shown in Fig. 3. For comparison the measured ^{14}C content of an air sample from a cross-wind site is shown as well as the contemporary prevailing ^{14}C content of rush (*Juncus*) from a "clean air" site Måryd (30 km east of Barsebäck).

4.3 *Biomedical applications of ^{14}C*

^{14}C is used as a radioactive tracer in clinical nuclear medicine and it is used in various contexts in medical research and when testing new pharmaceuticals on volunteers. In clinical medicine, organic compounds labelled with ^{14}C are used to demonstrate metabolic abnormalities. One way of carrying out these studies is to use "breath tests" [15]. The ^{14}C-labelled compound is ingested and metabolised, resulting in the end-product carbon dioxide, which is exhaled and easily collected for measurement. The decay of the radionuclide is usually measured by gas flow counters or liquid scintillators and the activity of the sample reveals the degree of, for example, fat malabsorption. Clinically useful information is obtained from samples taken a few hours after the administration of the test compound, even if the total turnover time is much longer. A complete biokinetic study, needed for such purposes as the calculation
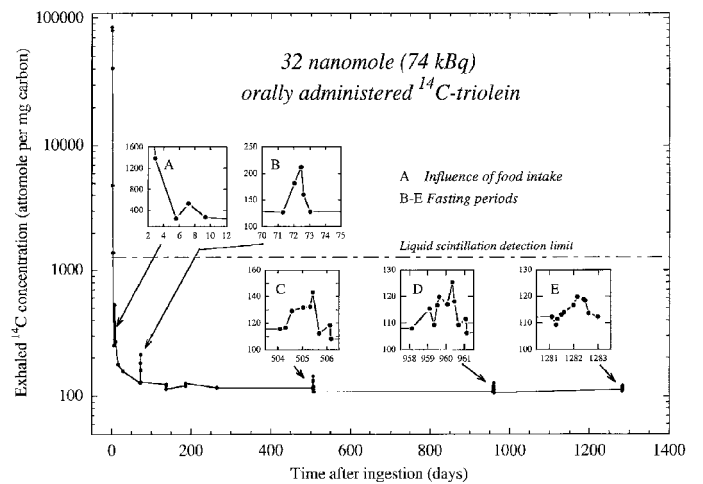
of the radiation dose, requires sampling for a much longer time, up to several months or even longer. There are considerable uncertainties in the current estimates of the absorbed doses to the body from ^{14}C-labelled pharmaceuticals, mainly due to the long half-life of ^{14}C and the difficulties involved to successfully measure with high sensitivity the long-term retention of ^{14}C in the body. Standard measuring methods, used in medical applications, are capable of detecting increased levels of ^{14}C in expired air for only a few days after ingestion. There is thus a need for a much more sensitive technique, such as AMS, for a complete study.

The AMS technique has in Lund been used to study the long-term retention of ^{14}C after a fat-malabsorption test (using ^{14}C-labelled triolein) by analysis of expired air [16] and samples of e.g. biopsies of fat, muscle and bone tissue [17]. Studies are also being performed on the long-term retention of ^{14}C after a ^{14}C-urea test [18], which is used to demonstrate abnormal activity of gastrointestinal bacteria. The use of the AMS technique makes it possible to follow the turnover of the radionuclide administered for extensive periods and also to decrease the activity administered without compromising the investigations [19]. A urea study with considerably reduced administered activity on children down to the age of four years has recently been presented [20] and a detailed biokinetic and dosimetric model study with tests of children down to the age of seven years has been reported [21].

As an example of our biomedical investigations, the ^{14}C concentration (in attomoles per milligram carbon) in expired air from one volunteer, at various times after administration of 32 nanomoles (74 kBq) ^{14}C-triolein, is shown in Fig. 4.

4.4 *Applications of ^{14}C in food chemistry*

An important topic in food chemistry technology is the interaction between foods and packaging materials, which can be

defined as chemical and/or physical reactions between a food, its package, and the environment [22]. In this way the composition, quality or physical properties of the food and/or package can be altered. Knowledge of these interactions is of importance for the development of new packaging systems [23].

Several methods, each one having advantages and disadvantages, have been and are being used in studies of interactions between foods and packaging materials [22]. One effect of the interactions is the absorption of aroma compounds into plastic packaging materials, which can damage the package and impair its protective properties. If key aroma compounds are absorbed, this can lead to a loss of the aromatic intensity of the food, a change in flavour and hence a deterioration in quality of the food. In Lund the AMS technique has been introduced into food chemistry with the aim of demonstrating that AMS can be a complementary and suitable tool for investigations of the absorption of flavours in plastic packaging materials [24] and [25].

Another possible AMS-investigation involves the reverse process, the monitoring of trace concentrations of compounds from the packaging materials that might be transferred to the food. This would be particularly valuable for cases where new synthetic packaging materials, which were not previously present in nature, are being widely introduced and used by consumers on a daily basis not only as packaging, but also for microwave cooking and serving purposes.

4.5 *Detection of ^{59}Ni in stainless steel*

In nuclear waste management ^{59}Ni is a most important radioisotope since it is produced by neutron activation in the stainless steel shielding surrounding the fuel. The total activity concentration of ^{59}Ni, as well as of other radionuclides, has to be established in preparation for final disposal. Furthermore, if the ^{59}Ni content in the steel can be measured, the integrated neutron flux at different positions in the reactor can be calculated. Because ^{59}Ni decays only via electron capture and has a very long half-life (7.6×10^4 y), it is quite difficult to measure its radiation emitted in the radioactive decay. The atom counting approach of AMS would in this case be advantageous. However, for small tandem accelerators, such as the Pelletron in Lund, the common energy or energy loss detection techniques are not able to distinguish atomic isobars for heavy elements such as Ni. One way to eliminate this problem is to combine AMS with the detection of characteristic projectile X-rays. After analysis in the AMS system the ions are stopped in a suitable target and it is possible to identify the ions by atomic number and thereby separate the isobars. In order to lower the detection limit in the case of ^{59}Ni, it is necessary to chemically reduce the content of the interfering isobar ^{59}Co, in the sample. In Lund some promising measurements using the projectile X-ray detection technique were performed some years ago [26]. The method has been further refined and a method to chemically extract nickel from stainless steel (and in this way reduce the cobalt content by several orders of magnitude) as well as a determination of the detection limit of ^{59}Ni at the Lund AMS-system have been presented [27]. An extensive investigation of the cross sections for production of characteristic projectile X-rays has recently been presented [28]. A few

active samples collected from different positions close to the core in some Swedish nuclear reactors have recently been analysed [29].

4.6 *Tests of the Exponential Decay Law*

Much experimental evidence indicates that ensembles of unstable atomic and nuclear particles decay in accordance with an exponential population decrease. However, the exponential law is not a rigorous consequence of quantum mechanics, but rather a result obtained by delicate assumptions and approximations. The estimation of uncertainties in the determination of atomic and nuclear meanlives is based on the supposition that the fundamental process is exactly governed by the exponential decay law and that the distribution of random measurement uncertainties about this exponential behaviour is exactly Gaussian. These assumptions have been tested at the level of a few percent, but as measurement accuracies increase to 0.1% and better, parameter evaluation by statistical inference must be accompanied by additional robust hypothesis testing. As discussed in a recent publication [30], possible limitations on these ultimate accuracies must be examined in the light of recent measurements and calculations, and quantitatively studied through the simulation, fitting, and correlational hypothesis testing of various models.

Toward this end, we have undertaken two types of experimental tests of the exponential hypothesis: one a study of possible deviations from a random distribution of gaussian deviations of highly precise activity data fitted to an exact exponential form, and an approach that utilizes AMS. Through the use of the AMS method, an alternative approach to the study of the exponential decay law has been made possible. Instead of the standard method in which the decay products emitted in the disintegration process are observed, one can use AMS to continuously measure the concentration of undecayed atoms still present in the sample. Preliminary tests with Na-24 (halflife 15 hours) produced at the Risø Nuclear Reactor have elucidated the technical problems that must be solved to obtain the needed accuracy.

5. Conclusions and future plans

The power of AMS for extremely sensitive radioisotope measurements has been demonstrated over the last ten to fifteen years. Only a few of all possible long-lived isotopes have at the moment been used for AMS. In this article only a few examples of all possible AMS experiments have been mentioned. An area which will grow in the future is biomedical studies. In the near future small dedicated accelerators for ^{14}C analysis with a high sample throughput will probably be available e.g. for the pharmaceutical industry. Small quantities of, for example, isotope enriched or radioactive marked chemical substances may be used. In this way the radiation dose to the test subjects will be more or less eliminated.

AMS was originally developed by nuclear physicists at existing tandem accelerator laboratories. About half of the about 50 AMS laboratories around the world originate from this category. However, during the last 5 y new 3–5 MV tandems dedicated for AMS have been installed.

In Lund further development – which has just recently been started – is in the installation of a new injector, including a spherical electrostatic analyzer (for optimal energy separation) and a 90° dipole magnet (for optimal mass separation) and provided with a high current ion-source. The main impetus for the new system is provided by our biomedical program with ^{26}Al-ions. However, the new injector will also be advantageous for our program for detecting ^{59}Ni in nuclear waste material.

Acknowledgements

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