

ACCELERATOR MASS SPECTROMETRY AND THE PREHISTORIC OCCUPATION OF THE COAST OF RIO DE JANEIRO - BRAZIL

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Abstract

The Accelerator Mass Spectrometry (AMS) technique is being used to determine the radiocarbon age of ceramic, bones, charcoal, nuts, shell and teeth samples from different archeological sites within the coast of Rio de Janeiro. Most of the ages were found to be within the 500 – 8000 years range. These data are being analyzed in order to map the occupation of Rio de Janeiro by the Indians, during prehistoric period. The 8000 years old sample is of particular interest.

1- The AMS Technique

The Accelerator Mass Spectrometry (AMS) technique is a very powerful method for mass spectrometry that allows the determination of concentrations with a sensitivity down to 1 atom of isotope in 10^{15} normal atoms, from samples of a few milligrams.

In the AMS technique, a Tandem accelerator is used as a magnetic and electrostatic analyzer, and it is an ideal tool for the determination of extremely low concentrations of rare isotopes, with sufficient energy to enable the counting and unambiguous identification of each ion, using techniques from Nuclear Physics. Good references about the experimental details and applications of the AMS technique are found in the proceedings of the International AMS Conferences, published by Nuclear Instruments and Methods in Physical Research B [1-4].

In Brazil the technique is being implemented at the 8 UD Tandem of the University of São Paulo, and there are plans to have a ^{14}C - AMS line at the new 1.7 MV Tandem facility in Rio de Janeiro.

The interaction of cosmic radiation with the atmosphere and surface layer of the Earth results in the production of small quantities of long lived radioactive nuclei such as ^{10}Be , ^{14}C , ^{36}Cl and ^{26}Al . These nuclides enter the geological cycle and become incorporated in organic materials, ground water and sediments. They decay sufficiently slowly to be useful in dating or natural features in the environment (their half-lives are, for example: ^{10}Be - 1.51×10^5 y, ^{14}C - 5730 y, ^{26}Al - 7.4×10^5 y and ^{36}Cl - 3.01×10^5 y). However, due to their very long half-lives, the determination of their concentration by measuring the radiation emitted during their decay is not possible, except for large samples of ^{14}C . A much better method to deal with these isotopes is the direct measurements of the concentration of the undecayed isotopes, as it is done by AMS. As no decay is detected, one does not need to wait thousands of years, in average, for the decay of one atom. In the conventional method, large amount of sample is needed to compensate the long half-life. With AMS, the measurements take few minutes, and very small amounts of sample are used. For example, 5 grams of ^{14}C can be replaced for less than 1 mg. Therefore, archaeological samples do not need to be spoiled, and extremely low radioactive material may be used for biological and medical studies. An ordinary mass spectrometer has a limited sensitivity of $1: 10^9$, in long measurement times. With AMS, within a few minute's measurements, a sensitivity better than $1: 10^{15}$ is achieved, since the typical beam currents are of the order of 10^{-6} - 10^{-9} A.

The concentrations of the rare isotopes are determined by the beam current intensity in Faraday cups and conventional nuclear detectors. From the determination of the ratio between the concentrations of the radioisotopes and the corresponding stable ones, one can derive the age of the sample that corresponds to the time interval in which the sample is no longer in equilibrium, from its assimilation to its radioactive decay. For ^{14}C , this time interval is called radiocarbon age.

2- The Experimental AMS Set-up

The experimental set up used in AMS may be divided in the following parts: sample preparation, ion source, accelerator system, control of the stable and radioactive beams and detection system. Figure 1 shows a layout of the AMS system [5]. The operation of such devices for AMS purposes is much more difficult and precise than for normal Nuclear Physics applications. In the following, a brief description of the main features and characteristics of each part will be given, in order to discuss some of the difficulties and possible achievements of the method.

a) Sample Preparation: This is a fundamental part in the AMS technique. When one wants to date a very small sample, one has to be sure about the correct collection of the material to be analyzed. It is essential that the non-physicists who collect and prepare the samples, at their initial stages, work in collaboration with the nuclear physicists who will run the AMS experiments. The sample preparation is complex and depends on very specific techniques for each different element or application.

b) Ion Source: The most suitable ion sources used for AMS purposes are of the sputtering type, and it is highly recommended to use multi-cathode sources, since it is important to be able to change, in a very fast way, many different samples within a few minutes periods. The ion source must have high and stable currents; high efficiency; very low memory (it has to be cleaned very often). As the sample dimensions are of the order of millimeters, the focusing properties of the source must be excellent.

c) Acceleration System: The extracted negative ions leaving the source must be pre-accelerated by a stabilized voltage of the order of 100 kV. Then, the beam is focused and controlled by electrostatic devices. It follows an analyzer magnet, responsible for the mass separation. Other electrostatic devices focus the beam into the accelerator tube. The magnetic field of the analyzing magnet must be extremely constant. A Tandem accelerator consists, basically, of a low energy accelerator tube, a high voltage terminal with a stripper device and a high-energy acceleration tube. The usual energy control system for the stabilization of the machine uses the current-slits feedback mode. However, this procedure can not be applied for the extremely low intensity unstable beams used for AMS purposes. Therefore, one has to rely on the stabilization via the generating voltmeter (VGM). If the terminal voltage and beam transmission rate are not very stable and constant, the system is useless for AMS purposes. It is very important that the

transmission of the acceleration system, including the stripper, is constant for periods longer than the cycle of measurements. After the acceleration region, a high-energy analyzer magnet selects the charge state of the beam and rejects the others, as well as molecules. Due to the very low intensity of the radioactive beams, the beam profile monitors and Faraday cups, placed along the beam tube, are not useful to measure these beams.

d) The handling and measurement of radioactive beams: There are two possibilities for the injection of stable and unstable beams in AMS: simultaneous and sequential injections. In the first, Faraday cups positioned out of the central beam position are used. They measure the intensity of the stable beams that are transmitted through the analyzer magnet simultaneously with the radioactive beam. In the second, one has to change periodically some parameters of the acceleration system, in order to focus different isotopes. The simultaneous injection is used, in general, in laboratories where very few different beams are accelerated.

In the sequential injection it is usual to accumulate spectra during periods smaller than one minute for the stable beams, and of the order of 5 to 20 minutes for the unstable ones. Isotopic ratios are obtained by alternatively selecting the stable isotope and measuring its beam current in a removable Faraday cup, and then by measuring the extremely low radioisotope counting rate in a detector placed at the beam direction. The time interval for the changing of parameters of the acceleration system, for the swapping of beams, and to place and/or remove the Faraday cup in front of the detector, must be less than one minute. Therefore, automatic systems, computer controlled, must be developed. Standard and blank samples are run periodically for normalization of the isotopic ratio and measurement of the background, respectively. Accuracy better than 1% can be obtained in the determination of the relative isotopic concentrations.

e) Detection System: A major problem when dealing with ultra high sensitive AMS is the presence of isobaric contamination on natural samples, like ^{10}B in ^{10}Be , ^{26}Mg in ^{26}Al and ^{36}S in ^{36}Cl . In all these examples, the chemical separation of the contaminants, in the levels needed for AMS purposes, is very difficult. Therefore, one needs a detection system that is able to separate elements that differ by one unit of charge and mass. The most widely used detector for charge identification is the gas filled ionization chamber, combined with a solid state detector. The specific energy loss of the incident particle in the gas is proportional to its Z^2 . The complete particle identification may be obtained by combining telescopes E - ΔE or Bragg detectors with time of flight devices. More complex systems, as multi-anode ionization chambers, magnetic

spectrometers, electrostatic detectors, gas-filled magnets and velocity filters are usually used in the application of the AMS technique.

3- The Pre-Historic Occupation of the Coast of Rio de Janeiro

The main objective of this research project is to improve the present knowledge on the occupation of the Brazilian coast by the Indians in the prehistoric period, studying archeological sites located in Rio de Janeiro.

Studies show [6-13] that the prehistoric occupation of the Brazilian central-south coast has started with fishermen and mollusk collector populations, who have settled down in the neighborhood of lagoons, between 6000 yrs BP and 1000 yrs AC. They built huge shell, sand and ground mounds found in several bays and lagoons from Rio Grande do Sul to Bahia States – the so called *sambaquis* (the word is of Tupi etymology, *tamba* meaning shellfish and *ki* to pile up). Although they also used to fish and hunt, the shells played a special role in their lives as it was used intentionally to build their settlement. They coined the term that describes the main characteristic of the sites – the accumulation of great quantities of mollusk shells.

These cultures searched for the sea and its seafood, leaving traces of their daily activities and funeral rituals. As the *sambaquis* could be easily seen in the landscape, these mounds were noticed by the first Europeans who arrived in Brazil during the 16th century. From then on, these cultures have been intensively investigated and many studies are concerned with their social functioning structure. Figure 2 shows a *sambaqui*, which had heights from 4 m to 25 m [9]. In these sites one finds weapons, tools, adornments, food remains, ashes and charcoal from old fires, as well as human graves and traces of old houses.

Between 6500 and 4000 years B.P. changes of climate, such as higher temperatures and humidity made possible other types of territory exploration. In this period many groups moved to the places where life conditions were better. Those groups, dedicated to horticulture and pottery, chose the Rain Forest as their preferential habitat. Pottery is the most important archaeological evidence and the pieces have extremely diverse characteristic decoration.

One of aims of the present research is to understand those societies functioning structure, spatial ordering, interaction with the environment and their transformation processes, from the

study of cultural remainder originated by those ancient population. It is intended to analyze the region occupation process dynamics and search for possible links between the pre-pottery population socio-cultural system destabilization, from the study of the relation between fishermen-collectors group (builders of shell mounds) and horticulturist, ceramics manufacturers group. Another objective is to compare information from the study area to data available from the entire national territory in order to explain questions about the origin center of these groups and their expansion routes.

The geographical identification of prehistoric routes depends exclusively on the relation between location/characterization of the archaeological sites and the radiocarbon dating.

This project joins different Brazilian research groups as IPHAN - Instituto do Patrimônio Histórico e Artístico Nacional (Institute of National Historic and Artistic Property), IAB - Instituto de Arqueologia Brasileira (Brazilian Institute of Archeology) and the National Museum of Federal University of Rio de Janeiro.

4. Sample Collection and Preparation

The complex process of sample preparation [14,15] will not be fully described in this paper. Its main steps consist of pre-treatment, chemical processing and graphitization. After these steps, the small graphitized samples are placed at the ion source, to be accelerated.

For radiocarbon dating a wide variety of materials can be used. The samples analyzed in this project are of several types, including ceramic, bones, charcoal, nuts, shells and teeth. They have been collected from different archeological sites within the coast of the State of Rio de Janeiro, Brazil.

Samples to be measured by AMS have to be reduced to graphite. Prior to the graphitization, the samples have to be pre-treated in order to remove contaminants that could interfere with the results. Depending on the material either the organic or the inorganic carbon can be the source of carbon to be dated. The fraction of interest is presumed to be made of the original carbon, left at the sample “death”, i.e., when no more carbon was exchanged with the environment. Other sample fractions are considered to be contaminants. And so is any carbon that might have added to the sample during collection, handling, storing or even the pre-treatment.

The samples were taken to the PRIME Lab. of the Purdue University, Indiana, in order to be prepared for AMS use. The cleaning of the samples begin with it examination under microscope. The gross impurities are removed with forceps or razor blade depending on the hardness of the material. For samples in extremely bad conditions, such as some bone samples that had been excavated, it may be necessary to wash it with ultra-pure water and even using an ultrasonic cleaner.

The chemical pre-treatment [15] depends on the sample material:

- a) *Inorganic samples*: For shell samples which are mostly made of calcium carbonate, the surface carbon is considered to be a contaminant, as it might have exchanged with the environment after an unknown period being submerged. The whole sample is treated with a small amount of hydrochloric acid at 95 degrees C to remove surface carbonates. Then, the samples are rinsed with ultra-pure water and dried. Vials with samples are evacuated and filled with phosphoric acid to produce carbon dioxide.
- b) *Organic samples*: For ceramic, charcoal and nut samples* the inorganic fraction is considered to be a contaminant. Also, the organic carbon from fulvic and humic acids originated in the decomposition process have to be removed for they may have added to the sample at a time different from its death. Samples are reduced to 1 to 2 mm thickness using a razor blade. The amount of sample needed depends on the material. After rinsing with ultra-pure water, the sample is treated with hydrochloric acid to remove the inorganic fraction. A base treatment, with sodium hydroxide, is done to remove the fulvic and humic fraction. Finally, another acid treatment removes the inorganic carbon that can have added to the sample during the base treatment. The whole chemical processing is done at 95° C. The sample is then dried and combusted at 900° C in a evacuated quartz tube. Copper oxide is used as an oxidizing agent and a silver foil to remove sulphur compounds released during the oxidation process.
- c) *Bone samples*: For teeth and bone samples only the collagen fraction is considered to be preserved. Bone contains 20% organic matter, from which 88% is collagen. The bone is demineralized with hydrochloric acid, then treated with sodium hydroxide to remove humic and fulvic acids. The process is performed at room temperature for the collagen to

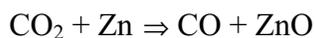
* Also for peat, wood, seed and soil samples. But those were not included in this research project.

remain intact. After the base treatment, hydrochloric acid is added and the sample is heated at 58° C overnight for the gelatin to dissolve. The liquid is filtered and then purified by ion exchange chromatography. (In case the sample is very badly preserved, single aminoacids can be separated and measured.) Collagen is dried and combusted as other organic samples. This procedure is still under development at PRIME lab and the bone and teeth samples are still to be measured.

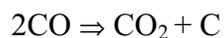
Obtained the carbon dioxide either by combustion of organic samples or by the dissolution in acid of carbonate samples, the gas is injected in a vacuum line in order to be purified and transferred to the graphitization tube. A mixture of dry ice and ethanol is used to trap water from the gas and a liquid nitrogen trap freezes the carbon dioxide while other gases are discarded. Part of the CO₂ is collected to have the isotopic ratio ¹³C/¹²C measured by conventional mass spectrometry since most accelerators don't have enough resolution.

The graphitization tube consists of a long quartz tube with a small tube, with iron, inside. A dimple in the bigger tube prevents the small one to touch its bottom, where the zinc is placed. In vacuum only the small tube is heated to 725° C to remove any contaminants in the iron. The zinc is then heated to 550° C to be distilled from the bottom to form a surface area layer in the upper part of the big tube. The bottom is sealed and removed.

The carbon dioxide is transferred to the graphitization tube with a liquid nitrogen trap. Finally, the tube is sealed and heated in the oven for 700° C for 10 hours. It is left to cool and heated again to 700° C for 10 more hours. At this temperature the CO₂ is reduced by the reaction:



The carbon monoxide decomposes forming graphite by the iron-catalyzed reaction:



And if the zinc is enough, all CO₂ produced by this reaction is converted to graphite in repeated cycles until there is no more CO₂. After the process the graphite is deposited in the inner tube with the iron and they are both pressed into the sample holder to be measured in the accelerator.

5 - Sample Transmission through the Accelerator and Measurements of ^{14}C / ^{13}C

The 8 UD Tandem accelerator of the PRIME Lab was used. Figure 1 shows a layout of the AMS system [5]. It has a 8 cathod SNICS ion source. The detector used was a conventional gas proportional counter. The terminal voltage used was 6MV, and C^{4+} (30MeV) were selected by the high-energy magnetic analyzer. The typical contaminants, the molecules $^{13}\text{CH}^-$ and $^{12}\text{CH}_2^-$ and the isobaric ^{14}N are either dissociated or separated by their final energies and/or different energy loss at the detector. The accelerator system parameters were adjusted as the following:

The $^{13}\text{C}^{4+}$ was adjusted for the terminal voltage at 7MV (35.15 MeV), corresponding to the same magnetic rigidity as the $^{14}\text{C}^{4+}$ at 6MV (30.13 MeV). The optimum values for the beam magnetic focusing devices (deflector magnets, quadrupole magnets and steering magnets) were then determined. Then, the $^{13}\text{C}^{4+}$ was adjusted with the terminal voltage at 6MV and the triplet electrostatic lens. After that, these values were kept fixed, and the high-energy analyzing magnet and quadrupoles were varied. The intensities of ^{12}C and ^{13}C beams were measured by a Faraday cup. The $^{13}\text{C}^{4+}$ beam was adjusted by varying the mass selector, analyzing magnet and the high-energy quadrupoles. Finally, these devices were adjusted to return to the initial conditions, to select the ^{14}C beam.

The cycle times, for the sequential beam injection, were two periods of ^{14}C counting, of 3 minutes each, the blank samples were measured at two periods of 5 minutes each, and the standard samples in two periods of 2 minutes each. Between each of those periods there were 10 seconds counting of the stable beams, ^{12}C and ^{13}C . In Primelab is obtained a 1% precision for $^{14}\text{C}/^{13}\text{C}$ ratios.

6- Radiocarbon Age Determination

The radiocarbon age of a sample S [16] is calculated from its specific activity A_S and the specific activity of a known standard from 1950 $A_{(1950)}$, both normalized to $\delta^{13}\text{C}=-25\%$

$$\text{Age} = -T \ln (A_{S[-25]}/A_{(1950)[-25]})$$

where $T = 8033$ years is the ^{14}C mean life, adopted by international convention, and determined by Libby[17]. This value corresponds to the half-life of 5568 years, although later the value of 5730 years was found to be more correct [18].

The specific activity is proportional to the ratio of ^{14}C to total carbon in the sample:

$$A \propto ({}^{14}\text{C}/({}^{12}\text{C} + {}^{13}\text{C}))_{[-25]}$$

Which can be approximated as the ratio of ^{14}C to ^{12}C to better than 1 part in 10^4 . At the PRIME Lab accelerator it is measured the ratio ${}^{14}\text{C}/{}^{13}\text{C}$, which can be related to ${}^{14}\text{C}/{}^{12}\text{C}$ by:

$$({}^{14}\text{C}/{}^{12}\text{C})_S = ({}^{14}\text{C}/{}^{13}\text{C})_S ({}^{13}\text{C}/{}^{12}\text{C})_S = ({}^{14}\text{C}/{}^{13}\text{C})_S (1 + \delta^{13}\text{C}_S/1000) ({}^{13}\text{C}/{}^{12}\text{C})_{\text{PDB}}$$

where PDB is a reference standard [19].

The ratio between the isotopic ratios of natural and standard samples is called “Fraction of Modern” (F) and it is determined by the expression:

$$F = \{ ({}^{14}\text{C}/{}^{13}\text{C})_{S[-25]} / ({}^{14}\text{C}/{}^{13}\text{C})_{(1950)[-25]} \}$$

The measured ${}^{14}\text{C}/{}^{13}\text{C}$ ratio is corrected by its $\delta^{13}\text{C}_S$ to give:

$$({}^{14}\text{C}/{}^{13}\text{C})_{S[-25]} = ({}^{14}\text{C}/{}^{13}\text{C})_{S[\delta]} \{ (1 + 25/1000) / (1 + \delta^{13}\text{C}_S/1000) \}$$

The oxalic acid standard used for the experiments is the NBS SRM 4990C, that relates to the 1950 standard by:

$$({}^{14}\text{C}/{}^{13}\text{C})_{(1950)[-25]} = 0.7404 ({}^{14}\text{C}/{}^{13}\text{C})_{(\text{NOX})[-17.8]}$$

The radiocarbon age is then:

$$\text{Age} = -T \ln F =$$

$$= -8033 \ln \{ ({}^{14}\text{C}/{}^{13}\text{C})_{S[\delta]} / 0.7404 ({}^{14}\text{C}/{}^{13}\text{C})_{(\text{NOX})[-17.8]} \} \{ (1 + 25/1000) / (1 + \delta^{13}\text{C}_S/1000) \}$$

7- Determination of the Ages

In this work, *sambaqui* samples from different archeological sites within the coast of the Rio de Janeiro State were analyzed. Table 1 shows the results of the AMS - radiocarbon age determination. Most of the present results are in agreement with other dating of *sambaquis* from the Brazilian coast. This fact shows that both the South and Southeast Brazilian coasts were occupied at roughly the same period. The analysis of these ages, associated with archaeological information, allows the study of how these cultures begin to diversify and spread regionally, searching for a better adaptation to the local natural resources. Also, these results give the background to the interpretation of possible reoccupation of the sites, leading to the understanding of possible contacts with other groups, their destiny and finally the destabilization of their way of life, which seems to be related with the occupation of the territory by ceramics manufacturer groups.

However, the dating from Sítio do Algodão, Angra dos Reis, corresponding to 7860 ± 80 years BP, showed a very unexpected result, as it is strongly shifted from the chronological period in which these sites are inserted, since the eldest *sambaquis* date 6000 years. In the literature [9, 10] there are just two dating for the *sambaquis* from the Central-South coast with this age: the one of 7.958 ± 224 years BP for the Sambaqui of Camboinhas, Rio de Janeiro and the one of 7.803 ± 1300 years BP for the Sambaqui of Maratuá, São Paulo. These two dating have always been seen with some discredit by the Brazilian archaeologists [9].

12.000 to 10.000 years ago there would have happened the last glaciation period. Since then, the temperature has been increasing progressively till it reached the presents levels 6.000 to 5.000 years ago. This shift in temperature would have melted the great continental ice cores increasing gradually the ocean levels. Thus, an expressive area of the coastal was submerging, modifying the coasts and changing the fauna and flora. At this time portions of the land that today are covered by the sea used to be exposed and at the disposal of the hunter/collector groups. This sea behavior allows arising the hypothesis that these groups might have reached the coast before 6.000 BP, providing that such older *sambaquis* would today be submerged and destroyed. Though this is a hypothesis of intricate corroboration as archaeologists can only deal with the evidences, considering the 6000 PB date as the initial period of occupation of the Central-South coast by *sambaqui* constructors. Therefore, the present result for the Sítio do Algodão, through the AMS technique, with small experimental uncertainty, is in agreement with

the two earlier results, and consequently re-opens the discussion on the chronology of the *sambaquis*.

8- Conclusions

The precise dating of the lower level of the Sambaqui do Algodão situated in a small island at the Ribeira Bay, Angra dos Reis, Rio de Janeiro, obtained by AMS technique, corresponding to 7860 ± 80 years BP is much older than the expected age for a *sambaqui*. This very important result re-opens the question on the human occupation of the Brazilian Central – South coast.

The present work is one example of how the methods and instrumentation usually used in Heavy Ion Nuclear Physics, such as Tandem Accelerators, detection systems and the powerful Accelerator Mass Spectrometry Method have many important applications. The implementation of this technique is quite difficult and complex, and it is essential the collaboration with scientists from other areas.

Acknowledgments

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Figure Captions

Figure 1. Layout of the AMS Primelab system [5].

Figure 2. Sambaqui: shell mounds presents on the Brazilian coast where prehistoric period groups lived accumulating great quantities of mollusk shells [9].

Tables

Table 1. Results of the AMS - radiocarbon age determination of the samples from the Coast of Rio de Janeiro.

PRIMELAB ID	ARCHEOLOGICAL SITE	LOCATION	SAMPLE MATERIAL	AGE (B.P.)
00 0670	Malhada	Cabo Frio	Ceramic	2500 ± 110
00 0671	Malhada	Cabo Frio	Shell	4400 ± 80
00 0672	Corondó	São Pedro d'Aldeia	Charcoal	3520 ± 80
00 0676	Camboinhas	Niterói	Shell	4230 ± 80
00 0677	Algodão	Angra dos Reis	Charcoal	7860 ± 80
00 0678	Peri	Angra dos Reis	Charcoal	1420 ± 80
00 0680	Caieira	Angra dos Reis	Charcoal	1520 ± 80
00 0681	Manitiba I	Saquarema	Shell	3730 ± 80
00 0682	Manitiba I	Saquarema	Shell	3880 ± 80
00 0683	Camboinhas	Niterói	Shell	4180 ± 80
00 0686	Morro Grande	Araruama	Charcoal	510 ± 160
00 0688	Morro Grande	Araruama	Charcoal	2600 ± 160
00 0689	Ilha da Boa Vista	Cabo Frio	Nut	3740 ± 160
00 0690	Ilha da Boa Vista	Cabo Frio	Nut	1620 ± 80

Figure 1

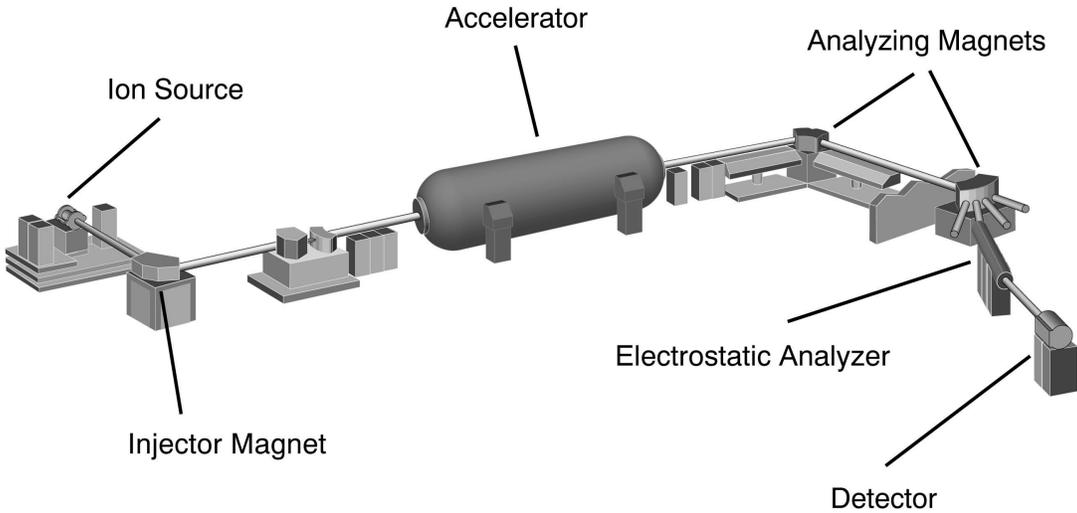


Figure 2

