R-02-46

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ISSN 1402-3091
SKB Rapport R-02-46

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Abstract

^{59}Ni is produced by neutron activation in the stainless steel close to the core of a nuclear reactor. To be able to classify the different parts of the reactor with respect to their content of long-lived radionuclides before final storage it is important to measure the ^{59}Ni level. Accelerator mass spectrometry is an ultra-sensitive method for counting atoms, suitable for ^{59}Ni measurements. Improvements in the reduction of the background and in the chemical reduction of cobalt, the interfering isobar, have been made. This chemical purification is essential when using small tandem accelerators, <3 MV, combined with the detection of characteristic projectile X-rays. These improvements have lowered the detection limit for ^{59}Ni by a factor of twenty compared with the first value reported for the Lund AMS facility. Material from the Swedish nuclear industry has been analysed and examples of the results are presented.

Contents

1 Introduction

^{59}Ni is one of the nuclides that are produced by neutron activation in the stainless steel construction elements in close proximity to the fuel in a nuclear reactor. Because ^{59}Ni decays only via electron capture and has a very long half-life, traditional radiometric measurements are difficult to perform. An efficient way to measure radionuclides such as ^{59}Ni is to use accelerator mass spectrometry (AMS), which is a highly sensitive method for measuring rare isotopes /1/. The many steps from the ion source to the detector via an electrostatic accelerator and several analysing units have been found to lead to a strong reduction of the background.

In Lund an AMS system has been developed over the past years /2/, and efforts are continuously being made to lower the detection limit for various ions of interest. To measure heavy isotopes such as ^{59}Ni, one has to overcome the severe problem posed by interfering isobars, in this case ^{59}Co. To reach the desired detection level for ^{59}Ni in stainless steel, it is necessary to introduce a chemical purification step to remove the major part of the ^{59}Co, which is always present in stainless steel. In this report an improved chemical method to reduce the ^{59}Co and to prepare AMS samples from radioactive components obtained from the nuclear industry is described, and a lowered detection limit for ^{59}Ni is presented. Furthermore some results of measurements of samples from nuclear reactors are reported.

2 Development of the Lund AMS system

The Lund AMS system is built up around a 3 MV Pelletron tandem accelerator. Apart from the ^{59}Ni programme, the AMS system is used for radiocarbon dating, for studies of releases into the environment of reactor-produced ^{14}C by the analysis of air and plant material in the vicinity of nuclear installations, for studies of ^{14}C-labelled pharmaceuticals used in clinical nuclear medicine, and – in the near future – for the detection of ^{10}Be and ^{26}Al for environmental, plant ecological and biomedical studies. Since the end of the 1980's the accelerator system has been extensively remodelled to suit our various AMS programmes and to become a high-quality AMS facility /3/. One of the more recent modifications – also of importance for the ^{59}Ni programme – is the installation of a new gas stripper including terminal pumping /4/.The very limited space and the absence of electrical power in the high-voltage terminal in our accelerator, required an unusual design for the installation and powering of the two turbomolecular pumps which are parts of the new stripper /4/. The new gas stripper provides excellent beam current stability and good optical quality. A 2–3 times higher ion transmission compared to the former gas stripper system and a constant stripper-medium thickness compared to foil stripping is attained with the new system. The advantages for our AMS measurements using the new system are that a higher beam current can be taken through the accelerator, a higher transmission and more stable conditions during the experiment.

3 Detection of ^{59}Ni in waste from the nuclear industry

In nuclear waste management ^{59}Ni is an important radionuclide. It is produced by neutron activation in the stainless steel near the core. The total activity concentration of ^{59}Ni, as well as of other long-lived radionuclides, has to be established in the planning of the final disposal. Because ^{59}Ni decays only via electron capture and has a very long half-life (7.6×10^4 years), it is difficult to measure the radiation emitted in its decay. The AMS technique is in this case advantageous. However, for small tandem accelerators, such as the Pelletron in Lund, the common techniques for energy or energy loss measurements are not able to distinguish atomic isobars of heavy elements such as Ni. One way to eliminate this problem is to combine AMS with the detection of characteristic projectile X-rays emitted when the ions pass through a suitable target. In Lund this technique was introduced some years ago /5/.

As mentioned above, the most severe problem when measuring ^{59}Ni with the AMS technique, using small accelerators, <3 MV, is the interfering isobar ^{59}Co, which is always present in stainless steel. Therefore a method to extract nickel chemically from stainless steel has been developed. The chemical purification process is performed in two steps. In the first – developed during the initial stage of the nickel project in Lund /6, 7/ – the nickel is extracted from the stainless steel by dissolution in hydrochloric acid and precipitation with dimethylglyoxime. The detection limit obtained in this step is found to be set by contamination within the process. Tools and beakers of plastic and glass have to be used and the water purity is of great importance. Recently the distilled water used in this step has been replaced by distilled water with a maximum impurity concentration of the order of 10^{-9}. This latter improvement reported in ref. 7 gives – with three standard deviations – a detection limit of 12 $\mathrm{Bq/g_{Ni}}$ (Bq per gram nickel). This corresponds to a ^{59}Ni/Ni ratio of 4×10^{-9}. In the second chemical step – presented below – a further reduction of the detection limit is achieved.

Samples from the nuclear industry have activities up to about 1×10^8 Bq per gram steel, where the main radioisotope in the material is ^{60}Co. The residual products of the first chemical step contain most of this activity and are returned to the nuclear industry.

4 Recent improvements of the ^{59}Ni detection limit

The detection limit is determined by a combination of three components: the chemical purification, the mass resolution in the accelerator system and the background in the X-ray energy range, 4-10 keV, of nickel in the detector independent of the beam. The chemical purification is, at the moment, the dominating factor that sets the detection limit.

4.1 A more sophisticated chemical purification

A second and more sophisticated chemical purification process has recently been introduced for samples with a ^{59}Ni content lower than a ^{59}Ni/Ni ratio of 4×10^{-9}. This process uses the reaction between nickel and carbon monoxide to form gaseous nickel tetracarbonyl ($Ni(CO)_4$). The process is largely an adaptation of the method described in ref. 8. For details about the process developed, see the Appendices. In order to provide a high and stable beam current from the ion source the first chemical purification step, described in detail in ref. 6, as well as the second, described below, must result in samples of pure Ni.

A photo of the second system is shown in Fig. 4-1. It consists of a pear-shaped reactor flask (25 ml) with three necks, followed by a U-shaped cooling trap filled with glass beads treated with dichlorodimethylsilane solution to prevent adsorption of $Ni(CO)_4$. The different parts are connected by PVC tubes. The system ends in a thin glass tube (inner diameter about 0.5 mm) leading to the sample holder. The latter is mounted in a heater. Two gas bottles are used, one with He and the other with CO/He (1:1), both connected to the reactor. Also connected to the reactor is a peristaltic pump. As nickel tetracarbonyl is extremely toxic, the whole system is placed in a fume-cupboard.

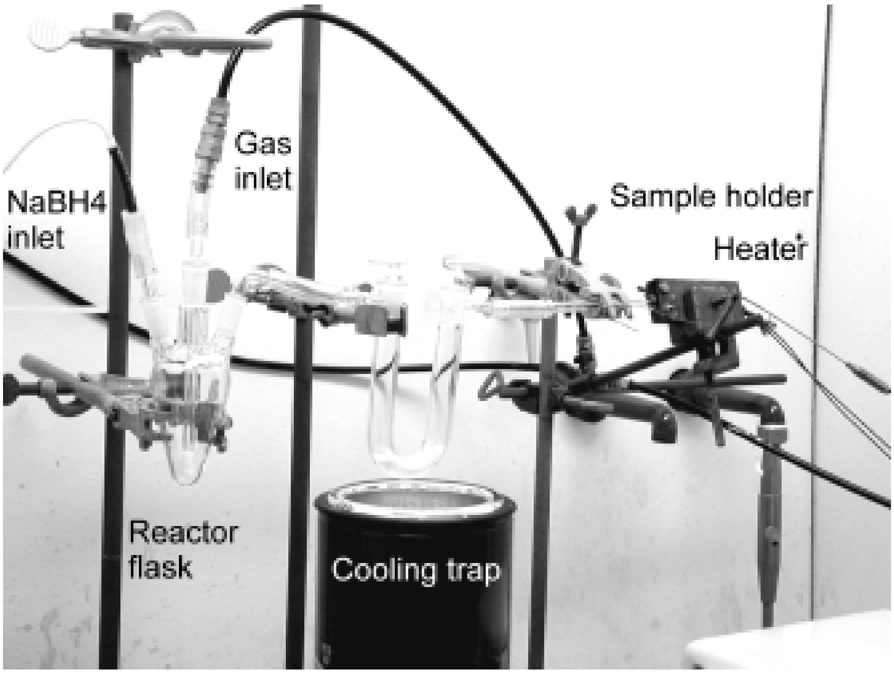

Figure 4-1. *A photo of the second and more sophisticated chemical system.*

In short, the second step is as follows. 3 mg of pure nickel from the first chemical purification step is dissolved in a beaker containing a mixture of 2 ml concentrated hydrochloric acid and 2 ml concentrated nitric acid. The solution is made basic, pH 10, with concentrated ammonia, diluted with distilled water to 13 ml in total and placed in the reactor flask. The cooling trap is rinsed with He gas, 70 ml/min for 5 minutes, ensuring that no air remains. The trap is then placed in liquid nitrogen, LN_2. Gaseous nickel tetracarbonyl is formed by leading CO/He through the solution at a flow rate of 80–100 ml/min and at the same time adding, with the peristaltic pump, a solution of $NaBH_4$, at a rate of 1 ml/min for 10 minutes. The nickel tetracarbonyl is trapped in the cooling trap with LN_2. When the cooling trap is thawed to –5 ºC, the nickel tetracarbonyl becomes liquid and starts to evaporate, while absorbed water vapour remains frozen. A He-flow of 70 ml/min transports the nickel tetracarbonyl gas to the sample holder in 20 minutes. The nickel tetracarbonyl is thermally decomposed to pure nickel, nickel oxide and gaseous CO when it hits the sample holder, which is kept at a temperature of 200 ºC. Finally, in order to reduce the nickel oxide to pure nickel, the sample holder is placed in a system which is evacuated, filled with H_2 to atmospheric pressure and heated to about 500 ºC and finally cooled down. In this procedure, as in all laboratory work, it is extremely important to use only clean equipment and high purity chemicals and to keep the environment free from any source of contamination. In this procedure the source of contamination would be any kind of metal from which Co could be extracted.

4.1.1 Difficulties during the development of the chemical process

Three main difficulties have been encountered during the development of the method: Co-contamination, a low nickel current from the sample and a low yield of nickel in the chemical process. Since it is important to have a sample holder of low Co content, different materials – aluminium, graphite and different kinds of copper – have been tested. Electrolytically produced copper turned out to best meet our requirements and has been chosen for our sample holders, even though this is still not pure enough. In order to avoid getting Co from this impure copper into the beam, copper powder of 99.8% purity is pressed into the bottom of the sample holder. At an early stage we suspected the Co to come from the atmosphere in the fume-cupboard or from the gases used in the process. This was, however, not the case, as a heated, pure nickel sample, left in the fume-cupboard for 4 hours with the gas flows on, showed no sign of elevated Co levels when measured with AMS. Another source of Co contamination turned out to be Co carried by water vapour from the solution in the reactor. This was avoided by thawing the cooling trap to –5 ºC instead of to room temperature, and thus preventing the water and the Co from escaping.

The last step of the process, the reduction in a H_2 atmosphere, was originally not included. The samples produced in this process gave an unexpectedly low beam current, a factor of 5 lower than samples of solid nickel. Further, the material in the sample holder had a black colour instead of the colour of nickel metal. It was therefore suspected that the process gave NiO instead of pure Ni, since it is known that NiO gives a current a factor of 5 lower than Ni /9/. The suspicions were confirmed when the sample was heated in an atmosphere of H_2, resulting in pure nickel metal and water vapour. A possibility was that the NiO formed as the sample was heated. Therefore a pure nickel sample was heated to 200 ºC in the heater, which, however, did not result in NiO. In order to reduce the access to O_2, we let the process take place in an atmosphere of Ar instead of air. Since this made no difference we suspect that the oxygen must come from the nickel tetracarbonyl itself.

A high yield is always desirable when carrying out chemical reactions. The process described here has a yield of about 50%. If there is any nickel left in the reactor flask when the process is finished, the solution turns slightly grey. For the most part, the extraction of Ni is complete, indicating that the loss of Ni must occur elsewhere. Some of the nickel tetracarbonyl gas is most probably carried away with the gas stream through the fume-cupboard. To reduce this loss we have placed a metal plate around the thin glass pipe and the sample holder. This modification increased the yield somewhat. Furthermore, it is important that the thin glass pipe is centred in the middle of the sample holder from which position the beam in the ion source is produced.

Initially, there was a problem with condensed air in the cooling trap. When the dewar flask with LN_2 was removed and the trap became warmer the air started to boil, which gave an undesirable overpressure in the system. This was easily avoided by allowing He (70 ml/min) to flow through the system for five minutes before the cooling trap was put in the dewar flask with LN_2.

It is of great importance that the $NaBH_4$ is stored under vacuum. Avoiding contact with moisture in the air is crucial, as otherwise the $NaBH_4$ will be destroyed.

4.2 Background

Improvements by remodelling the target detector set up in order to lower the detection limit have been made. Combining the target and exit windows into one foil has increased the detector solid angle. The geometry of the lead shield around the detector has been changed and the X-ray yield for nickel has been optimised using optimal beam energy and target material /10/. The excellent resolution of the Ge detector (145 eV FWHM at

5.9 keV) completely separates the K_α peaks of Co and Ni. The Co K_β peak does, however, influence the Ni K_α peak somewhat, but since the ratio of the K_α and K_β yields for Co is known, this influence can easily be subtracted, thus making it possible to tolerate a certain level of Co in the samples. To normalise the measurements, X-rays detected from the stable isotope ^{61}Ni (with a natural abundance of 1.14%) are used. This simplifies the normalisation compared to using the beam current of the stable isotope since neither the X-ray production rate nor the detection efficiency for the two different Ni isotopes need to be considered. The natural background obtained in the detector with the accelerator switched off corresponds to a ^{59}Ni/Ni ratio of 1×10^{-9}. It is important to optimise the beam path, otherwise the ions will hit the walls and, close to the target foil, produce X-rays that can give a higher background in the detector.

4.3 Resulting detection limit

The chemical process described in this report has decreased the Co background to a level of 2×10^{-9} for the ^{59}Ni/Ni ratio, which corresponds to 5 Bq/g_{Ni}. This is a factor of two lower than what is achieved with only the first chemical purification step. The beam current of Ni^- ions from processed samples is about 1 μA. An important feature of the new procedure is that very small amounts of Ni (down to 2 mg) can be processed. This is because the nickel is evaporated directly onto the sample holder. The first chemical process gave pure nickel powder that had to be pressed into the sample holder. This required sample sizes of at least 10 mg Ni.

5 Results from the measurements of active samples

A number of steel samples obtained from the Swedish nuclear industry have been analysed by the Lund AMS system. In Table 5-1 some of the recent results of rather active steel samples from the boiling water reactor (BWR) no.1 at the Oskarshamn nuclear power plant are presented. Samples of recirculating water from the pressurised water reactors (PWR) at the Ringhals nuclear power plant on the Swedish west coast have also been analysed. The activities found in the water were 15, 20 and 26 kBq/l (kBq per litre recirculating water) for the reactors no. 2, 3 and 4 at Ringhals, respectively. The measured values agree with the expected values. This is, to our knowledge, the first report of a measurement of ^{59}Ni in recirculating water.

Table 5-1. ^{59}Ni activities in samples taken from different positions in the BWR no. 1 at the Oskarshamn nuclear power plant on the east coast; m.t.=moderator tank; s.s.=steam separator.

Sample no.	Sample detail	Activity MBq/(g_{Ni})
1	m. t.	3.2 ± 0.8
2	m. t.	2.5 ± 0.7
3	m. t.	4.5 ± 1.1
4	m. t.	1.8 ± 0.5
5	m. t.	2.2 ± 0.6
6	Guiding rod for the m. t. head	0.30 ± 0.08
7	m. t.	2.3 ± 0.6
8	m. t.	3.1 ± 0.8
9	Rods between s. s.	0.0038 ± 0.0010
10	Guiding rod for the m. t. head	0.0063 ± 0.0016
11	m. t.	4.2 ± 1.0
12	Flange joint between s. s. and m. t. head	0.064 ± 0.016
13	Rods between s. s.	0.012 ± 0.003

6 Discussion

The detection limit obtained by AMS has been compared to radiometric measurements using the same Ge detector. As ^{59}Ni decays via electron capture a characteristic X-ray from the daughter isotope ^{59}Co will be emitted and can be detected. For a sample containing few other radioisotopes, *e.g*. steel samples, a detection limit corresponding to 20 Bq/g_{Ni} is obtained for the radiometric method.

This is somewhat higher than the AMS limit obtained for samples that have passed only the first chemical purification step and a factor of 4 higher than the detection limit for samples that have passed both chemical steps. However, for a sample containing other radioisotopes, such as a nickel sample extracted from recirculating water, the detection limit for the radiometric method is several orders of magnitude higher. By the modifications reported in this paper the detection limit has been lowered to a level of 2×10^{-9} when the highest energy available at our accelerator is used. This is comparable to the lowest limits reported in the literature for ions within the same energy range, using the detection of characteristic X-rays. Wagner *et al.* /11/, using only the first of the two chemical steps, has calculated and measured the detection limit of ^{59}Ni/Ni to be of the order of 10^{-8} for 18 MeV ions. McAninch *et al.* /12/, however, have reached detection limits of 10^{-11} for ^{59}Ni/Ni but for ion energies of 99 MeV.

The results presented in this report show that it is possible to use a small (<3 MV) tandem accelerator in combination with the detection of characteristic X-rays of the projectile and at a very low detection limit. Therefore such small accelerators are useful and economic alternatives to large accelerators and more sophisticated AMS techniques.

So far, the main goal of this work has been to reduce the cobalt levels in the samples produced. We have now almost reached levels at which the natural background and mass resolution of the AMS system set the detection limit for the ^{59}Ni/Ni ratio. Currently we have a Ni recovery of only 50% in the second chemical process, *i.e.* 3 mg Ni in the reactor yields only 1.5 mg of pure Ni metal in the sample holder. The extraction of nickel functions without flaws and cannot really be improved. However, the geometry of the last part of the experimental set-up, where the Ni is thermally decomposed directly onto the sample holder, can be improved. Furthermore, for the cooling trap, we want to reach the optimal point for the temperature. A good start would be to try using two cooling traps, one for the gaseous nickel tetracarbonyl and one for the water. The cooling trap for the water should have a fixed temperature of –5 °C when the cooling trap for the nickel tetracarbonyl is heated to room temperature.

A new injector, including a high beam current ion source, a spherical electrostatic analyser and a 90° magnet is currently being tested /13,14/. The new injector will give a better mass and energy resolution, especially for heavy ions such as ^{59}Ni.

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Appendix A1

$Ni(CO)_4$-chemistry

Preliminaries

1. Fill the burette with conc. ammonia (about 50 ml).
2. Dissolve the Ni in a mixture of 2 ml conc. hydrochloric acid and 2 ml conc. nitric acid.
3. Make the Ni solution basic to pH 10 with conc. ammonia (the acidic solution is yellow-green, basic is blue).
4. Dilute the Ni solution so that 13 ml corresponds to 3 mg Ni.
5. Pour 13 ml of the Ni solution into a measuring cylinder.
6. Mix 0.25 g $NaBH_4$ + 50 ml distilled water, make basic to pH 10.
7. Pump the $NaBH_4$ solution through the peristaltic pump.
8. Fill the dewar flask with liquid nitrogen.
9. Weigh the sample holder.
10. Place the sample holder in the heater and mount the brass plate around it.

Sample preparation

1. Pour the Ni solution into the reactor.
2. Let He through the system to get rid of the air, 70 ml/min for 5 min.
3. Raise the laboratory jack with the dewar flask containing liquid nitrogen so that the U-shaped cooling trap is properly cooled down.
4. Close the He flow and open the CO/He flow.
5. Saturate the solution with CO, 10 min at a flow of 80-100 ml/min of CO/He.
6. Add the $NaBH_4$ solution for 10 min (1 ml/min) with the peristaltic pump.
7. Close the CO flow, use only He at 70 ml/min from now on.
8. Heat the sample holder to 200 °C.
9. Lower the laboratory jack slowly.
10. Replace the LN_2 dewar flask with one containing ethanol at a temperature about –5 °C, and raise the laboratory jack.
11. Wait for 20 min.
12. Turn off the heating and the He flow, allow the sample holder to cool.
13. Weigh the sample holder.
14. Heat the sample holder in an atmosphere of H_2 to about 500 °C
15. Weigh the sample holder.

After completed sample preparation

1. Remove the cooling trap and heat it in an oven in the fume-cupboard to >200 °C in order to allow any remaining $Ni(CO)_4$ to dissociate.
2. Pump distilled water through the peristaltic pump.
3. Pour water into the $NaBH_4$ solution and dispose of it.
4. Pour the solution into the reactor in a solution of 1 M nitric acid.
5. Add water and dispose of the solution. This solution may be radioactive!
6. Rinse the reactor with conc. nitric acid.
7. Put the remaining glass parts and the PVC tubes in a washing-up bowl and pour conc. nitric acid over them and allow them to soak for some minutes.
8. Dilute this washing acid with water and dispose of the resulting liquid.
9. Rinse the parts with water and distilled water.
10. Let the parts dry.

Appendix A2
(in Swedish)

$Ni(CO)_4$-kemi

Förberedelser

1. Fyll byretten med konc. ammoniak (ca 50 ml).
2. Lös upp Ni i en blandning av 2 ml saltsyra och 2 ml salpetersyra.
3. Gör Ni-lösningen basisk till pH 10 med konc. ammoniak (sur lösning är gulgrön, basisk är blå).
4. Späd Ni-lösningen så att 13 ml motsvarar 3 mg Ni.
5. Mät upp 13 ml av Ni-lösningen.
6. Blanda 0,25 g $NaBH_4$ + 50 ml destvatten, gör basisk till pH 10.
7. Kör igenom $NaBH_4$-lösningen genom slangpumpen.
8. Fyll flytande kväve i termosen.
9. Väg provhållaren.
10. Sätt fast provhållaren i dess fäste och montera mässingsplåten runt provhållaren.

Provframställning

1. Häll Ni-lösningen i reaktorn.
2. Kör He genom systemet för att få ut all luft, 70 ml/min i ca 5 min.
3. Höj saxbordet med termosen innehållande flytande kväve så att U-röret kyls ner.
4. Stryp He-flödet och öppna till CO/He istället.
5. Mätta lösningen med CO, 10 min med flödet 80–100 ml/min.
6. Tillsätt $NaBH_4$-lösning i 10 min (1 ml/min) med slangpumpen.
7. Stryp CO, bara He med 70 ml/min fortsättningsvis.
8. Värm provhållaren till 200 °C.
9. Sänk saxbordet långsamt.
10. Byt termos till en med etanol av temperaturen ca –5 °C och höj saxbordet igen
11. Låt stå i 20 min.
12. Stäng av uppvärmningen och He-flödet, låt provhållaren svalna.
13. Väg provhållaren.
14. Hetta upp provhållaren i H_2-atmosfär så att provröret glöder för att reducera bildad NiO till Ni.
15. Väg provhållaren.

Efter avslutad provframställning

1. Ta loss kylfällan och lägg in den i ugnen och värm till mer än 200 °C för att bryta ner eventuellt återstående $Ni(CO)_4$.
2. Kör destvatten genom slangpumpen.
3. Häll vatten i $NaBH_4$-lösningen och häll ut den.
4. Töm reaktorns innehåll i 1 M salpetersyra.
5. Blanda ut med vatten och häll ut i vasken. OBS! Detta kan innehålla små mängder av aktiva restprodukter.
6. Skölj ur reaktorn med konc. salpetersyra.
7. Lägg övriga delar i diskkärlet och häll över konc. salpetersyra. Låt stå ett tag.
8. Häll vatten i diskkärlet och häll ut.
9. Skölj delarna med vatten och destvatten.
10. Låt gasinledningsröret torka i ugn, övriga delar kan lufttorka.