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Radioactivity in commercially available metals

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Abstract

Naturally occurring radioactivity in the raw materials for metal production may give rise to small amounts of radioactivity in the final products. Other possible radioactive sources in metals are recycled scrap from the nuclear industry and contamination from radiation sources erroneously ending up in recyclable scrap. A study on radioactivity levels in commercially available metals was performed. Samples from different steel, aluminium and magnesium producers in Nordic Countries were analysed. The results from the measurements are presented.

Keywords

Natural occurring radioactivity, gamma spectrometric measurements, beta measurements, neutron activation analyses, clearance, clearance levels, exemption levels

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1 Introduction

Clearance of radioactive material, in particular scrap metal, is a quite important issue, nationally as well internationally. For example, the IAEA, OECD/NEA and EU are active in this field. The volume of scrap metal cleared for recycling is expected to increase, as the nuclear installations grow older expanding refurbishment and modernisation activities. Also decommissioning of obsolete nuclear installations worldwide will add up cleared material for recycling. However, controlled clearance is not the only source of radioactivity in materials and products. Other sources are naturally occurring radionuclides, accidental smelting of radiation sources, fall-out from nuclear tests etc.

Table 1 gives international recommendations regarding exemption levels and clearance levels for some radioactive nuclides. Exemption levels are defined as levels of specific activity or dose rate below which no specific exposure assessment or exposure control needs to be undertaken for a specified natural radiation source [5]. Clearance levels are defined as values, established by national competent authorities, and expressed in terms of activity concentrations and/or total activity, at or below which sources of radiation may be released from regulatory control [2]. Concentrations of naturally occurring radionuclides in soil/rocks are given for comparison in Table 2.

Table 1. International recommendations regarding exemption levels and clearance levels.

Nuclide	IAEA exemption levels [1] Bq/kg		IAEA exemption levels [2] Bq/kg	EU exemption levels [3] Bq/kg	EU clearance levels [4] Bq/kg
	steel	aluminium	generally	generally	generally
^{60}Co	100-600	300-2000	10000	10000	100
^{137}Cs	100-600	100-600	10000	10000	1000
^{238}U	300-1000	1000-5000	10000	10000	1000
^{106}Ru			100000	100000	1000
^{226}Ra			10000	10000	10
^{210}Pb			10000	10000	10
^{232}Th			1000	1000	10
^{212}Pb			10000	10000	

Table 2. Typical levels of naturally occurring radionuclides in Nordic soil and rocks [5].

Material	^{226}Ra Bq/kg	^{232}Th Bq/kg	^{40}K Bq/kg
Normal granite	20-130	20-80	620-2400
Alun shale	100-4300	10-40	1100-1900
Sand	<4-6	2-80	150-1100
Clay	15-130	10-100	600-1200
Till	10-170	15-100	500-1299

The authorities need to know the levels of radioactivity in the environment and in materials used by man. This applies to both naturally occurring as well as man-made radionuclides. The knowledge is needed for assessing the radiological consequences of the present situation and changes expected to occur in the future. The NKS project SOS-3.3 deals with this subject. The project is called "Contamination levels in metals" and includes both an overview on clearance in the Nordic Countries and a study on radioactivity in commercially available metals. The overview on clearance has been presented in a separate report [6] and the results from the study on radioactivity in commercially available metals are presented in the present report.

2 Samples

The study was performed in co-operation with metal producers and metal producers associations within the Nordic Countries. Several samples of steel, aluminium and magnesium products from the metal producers were sent to national laboratories for analyses. The degree of reused scrap in the metal samples varied from 0 % (100 % ore based) to 100 %.

The sample weights and the dimension of the samples varied and were in some cases adjusted to and in other cases not adjusted to standard dimensions normally used at the different laboratories.

Altogether almost 200 steel samples, about 70 aluminium samples and 8 magnesium samples were analysed. For comparison also some other samples were analysed:

- slag samples from steel production
- slag samples from aluminium production
- petroleum coke samples (anode material from aluminium and magnesium production)
- steel samples from a foundry using cleared scrap from the nuclear industry as raw material
- a ferrosilicon sample

3 Measuring conditions and equipment

3.1 Gamma spectrometric measurements

Gamma spectrometric measurements were performed at Risø in Denmark, VTT in Finland, Geislavarnir in Iceland, IFE and NRPA in Norway and Studsvik in Sweden. Figure 1 shows an example of a low-level high-purity germanium (HPGe) spectrometer system used for the measurements. The HPGe detector is cooled in liquid nitrogen and placed in a lead shield in order to reduce the ambient background at the detector. The samples are filled in containers, which are placed in the lead shield close to the detector. The detector signal is recorded and analysed by a multichannel analyser, which produces the gamma spectrum. The spectral information on gamma emitting radionuclides appears in terms of peaks for which the corresponding gamma-ray energies identify the radionuclides and the peak count rates quantify the amount of radionuclide activity in the samples.

Figure 1. Low-level HPGe spectrometer system at Risø National Laboratory. The samples are placed in the lead shield on the table.

Detection limit

The lower limit of detection of a radionuclide depends on several parameters attributed to the detector, the environment, the available measurement time and the sample itself. Currie [7] has derived a formula for this limit, and it can be presented in the form of minimum detectable specific activity, D, as follows:

$$D = 4.66 * \frac{\sqrt{B/T}}{\eta \cdot \varepsilon \cdot m}, \qquad (1)$$

where

- B denotes the background count at the energy of the photopeak of the nuclide,
- η denotes the measurement efficiency taking into account the detector efficiency, the measurement geometry and the self-absorption in the sample,
- ε stands for the emissivity of the photopeak in question of the radionuclide (gamma quanta/disintegration),
- T stands for the live time of the measurement and
- m denotes the mass of the sample.

The numerical factor, 4.66, stems from the confidence level of 95 % chosen throughout this study.

As can be seen in the reported results in Section 4, all laboratories reported detection limits well below the exemption and clearance levels given in Table 1.

Measurements at Risø, Denmark

The gamma analyses of steel and aluminium samples at Risø National Laboratory were carried out using HPGe spectrometry systems, which are used regularly for analysis of low-level amounts of radioactivity in environmental samples. The efficiencies of the Ge detectors are in the range 30-40 % and the detectors are of special low-background construction. The lead shields have thicknesses of 10 cm.

The gamma spectra were analysed with software developed at Risø. The detectors were calibrated for a range of geometries with liquid solutions containing known amounts of specific radionuclides. The software analysis included corrections for coincidence summing effects and self-absorption.

The geometries of the steel and aluminium samples did not correspond exactly to the reference geometries, for which calibrations of the HPGe detectors were made. In these cases expert judgement was applied to increase the uncertainties of the results to take this into account.

Counting times of three to nine days were used.

Measurements at VTT, Finland
The steel samples analysed at VTT were measured automatically with a sample changer connected to a coaxial HPGe-detector. The relative efficiency of the detector is 25 %. The measurement time was six hours. The acquired gamma-spectra were analysed with software called SAMPO [8].

The aluminium samples were inserted manually and measured with a Ge(Li) detector for twelve hours. The relative efficiency of the detector was 11 %. The lead shield around the detector was 10 cm. The acquired gamma spectra were analysed with GammaVision 32 [9].

Measurements at Geislavarnir, Iceland
At Geislavarnir ríkisins the measurements of gamma emitting radionuclides were performed with a high purity germanium (HPGe) detector enclosed in 10 cm thick lead shielding. The relative efficiency of the detector was 45 % and all samples were measured for 3- 4 days. The program GammaVison (Version 5.10) was used for collecting the spectra, finding peaks and initial estimation of peak areas. The geometries used and composition of samples (e.g. attenuation) was not suited for routine analysis using the institute's analysis software. A special computer program was thus developed for analysing the spectra, using a mathematical programming tool, taking into account as far as possible effects of differences in geometry and sample attenuation.

The geometries of the samples were not well represented by the standard geometries that had been used to calibrate the detector. The normal calibration was thus supplemented with a new calibration better suited for the geometry used.

Measurements at IFE, Norway
Two low energy, high-purity germanium detectors were used for the gamma spectrometric measurements at IFE. All samples were counted for one day or more. The relative efficiencies for the two low energy HPGe detectors were 20 % and 40 %, respectively. The detectors were calibrated with a certified solution of gamma emitters, and Gamma Vision 32 was used for the spectrum analysis [9].

Measurements at NRPA, Norway
The measurement of gamma emitting nuclides at NRPA was performed with low level, high-purity, germanium (HPGe) detectors. The samples were counted for a minimum of 2 days. The HPGe detectors have relative efficiencies in the range of 23 % to 40 %. The detectors were calibrated with certified solutions of single gamma line nuclides. The analysis program used is developed at NRPA. It corrects for self-absorption, coincidence summing and background counting in the reported results. However, the program does not calculate detection limits. This must be done manually in accordance with formula (1), when there is need for it.

Measurements at Studsvik, Sweden
For most of the gamma spectrometric analyses performed at Studsvik software from Canberra, Genie 2000, was used. The samples were measured for between 16 and 60 hours with coaxial germanium detectors with the relative efficiency of 30 %. Some samples (foundry samples) were analysed (measuring times 11-60 hours) with a new kind of

germanium detector, a Broad Energy Germanium, connected to a software called LABSOCS. This software calculates the measurement efficiency with correction for self-absorption.

All detectors were shielded with 10-15 cm lead, which gave a very low background with no background peaks in the areas of ^{137}Cs and ^{60}Co.

The detectors were calibrated with certified water solutions. The absorption corrections were calculated theoretically. The corrections were compared with the results from the LABSOCS-calculations and the agreement was good.

The detectors were checked once a week for stability with a sealed ^{226}Ra sample.

3.2 Beta measurements

Figure 2 shows the laboratory equipment at Risø National Laboratory used for total beta measurements. Low-level gas-flow Geiger-Müller multi counters developed at Risø with five counter elements each are placed in 10-cm lead shields in order to reduce the ambient background at the detector. Furthermore, the five counters are operated in anti-coincidence with a common guard counter, which efficiently reduces the background count rate to 0.2 cpm or less. The GM counters are used regularly for measurements of ^{90}Sr and ^{99}Tc in environmental samples.

Figure 2. Low-level Geiger-Müller multi-counter system at Risø National Laboratory. The picture shows two multi counters, one on top of the other, in a 10-cm lead shield.

Steel and aluminium samples were prepared in a cylindrical geometry of 20 mm diameter and 5 mm thickness. These samples were measured in the GM counters and evaluated based on the calibration for ^{99}Tc and background correction using special steel samples. Hence these total beta measurements would give qualitative results only since reference was made to other steel samples for background and calibration for ^{99}Tc.

3.3 Neutron activation analyses

The activity concentration of ^{238}U and ^{232}Th were measured at Kjeller for a few selected samples using neutron activation analysis (see Figure 3). Neutron activation analysis has the advantage of a rather low detection limit for these isotopes, typically 1 ppm corresponding to 1.2 Bq/kg for ^{238}U and 0.5 Bq/kg for ^{232}Th.

In neutron activation of ^{238}U, the γ-emitting nuclide ^{239}Np is produced by decay of ^{239}U. The specific activity of ^{238}U is then determined using γ-spectrometry of this nuclide. Correspondingly, ^{233}Pa is used to measure the specific activity of ^{232}Th in the sample.

A small portion, about 10 mg, of each sample was irradiated with neutrons for 15 minutes in the JEEP II reactor at Kjeller. The samples were left for one week to allow for decay of short-lived radionuclides. It was necessary to let ^{239}Np decay for about one week extra before measuring the ^{233}Pa activity.

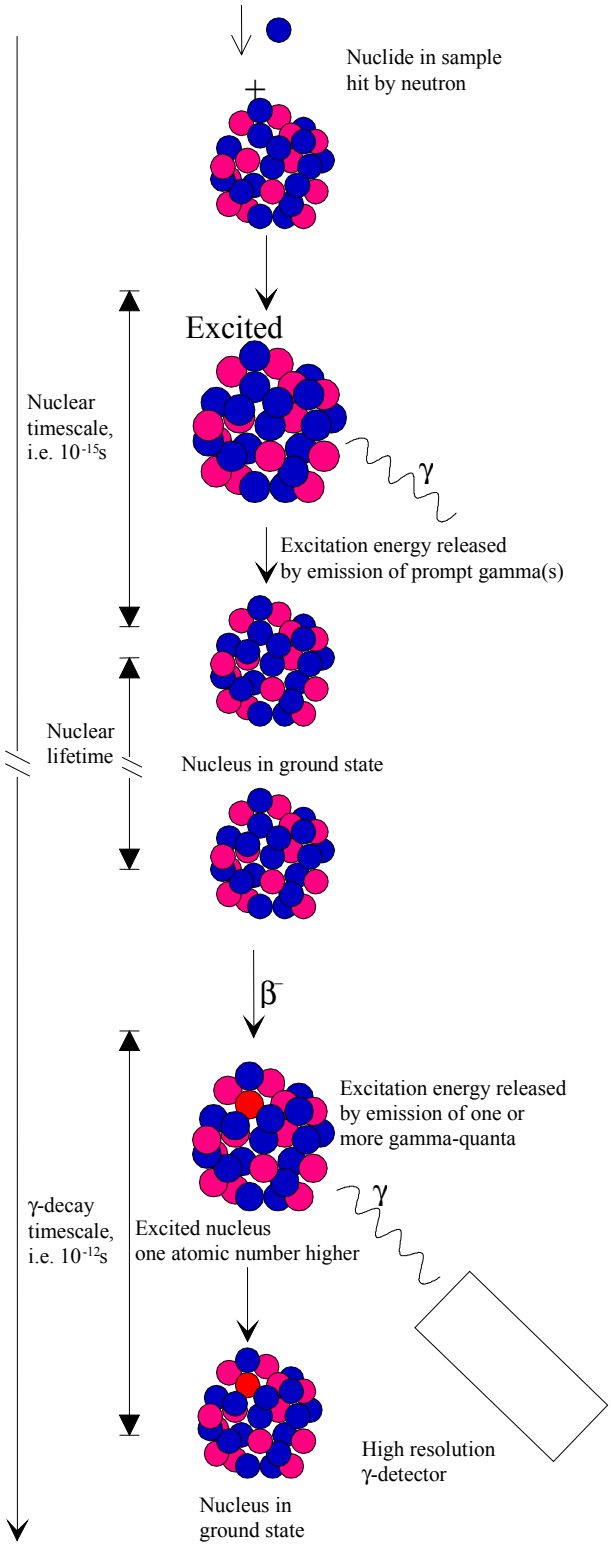

Figure 3. Principle of neutron activation analysis (NAA).

4 Results

4.1 Steel samples

Results of gamma-spectrum analyses of the steel samples are shown in Table 3. In most of the samples no activity was found, the specific activities of the samples were below the detection limits. However, five samples showed ^{60}Co concentrations above the detection limits in the range 0.03 – 0.07 Bq/kg. The detection limits were in these cases very low due to long counting times, low background and a high sample weight of 1.6 kg.

In addition beta analyses were performed of some samples prepared for the GM counter geometry (27 g samples). However, the results showed no activity in excess of the background steel samples. The detection limits from these measurements were 0.1 Bq/kg, i.e. higher than the levels of ^{60}Co found from the corresponding gamma-spectrometric analyses at Risø.

Table 3. Results of gamma spectrometric analyses of steel samples.

Laboratory	Number of samples	Sample weight (kg)	Counting time (hours)	^{60}Co concentration (Bq/kg)	Uncertainty (%)	Detection limit (Bq/kg)
Risø	2	1.6	170-190	Below detection limit		0.03
Risø	12	1.6	74-210	0.03-0.07	21-70	0.03-0.04
VTT	100	0.07-0.10	6	Below detection limit		4
IFE	4	0.1	≥ 24	Below detection limit		0.2-1.0
NRPA	2	0.1	≥ 24	Below detection limit		2.1
Studsvik	67	0.1-0.3	11-60	Below detection limit		0.1-0.9

4.2 Aluminium samples

The results of gamma-spectrum analyses of the aluminium samples are shown in Table 4. Naturally occurring uranium, thorium and radium isotopes were found in the samples.

Some samples were also measured in the GM counters (see Table 5). The total beta activities analysed were in the range 1.1 – 1.5 Bq/kg. A comparison between the results of the beta counting and those of the corresponding gamma-spectrometric analyses indicate that the beta activity is due to the presence of ^{238}U, ^{232}Th and their daughters in the aluminium samples.

Table 6 shows results of neutron activation analysis of two aluminium samples. 0.8-3.5 ppm uranium and 0.7 ppm thorium were found in the samples. The results are in accordance with naturally occurring uranium and thorium levels in aluminium [10]. The results from the neutron activation analyses can be compared with results from gamma spectrometric analyses of the same samples. The ^{232}Th activity concentrations from the neutron activation analyses are almost the same as the ^{212}Pb activity concentrations from the gamma spectrometric analyses (around 3 Bq/kg in both cases). This is logical since ^{212}Pb is part of the ^{232}Th decay chain and the nuclides between ^{232}Th and ^{212}Pb in the decay chain are rather short-lived (half-lives between 0.15 s and 5.7 y). However, no activity from ^{238}U with daughter products was found at the gamma spectrometric analyses. The ^{214}Pb activity was below the detection limit (below 0.4-0.9 Bq/kg). This can be explained by that some of the nuclides between ^{238}U and ^{214}Pb in the decay chain are long-lived (half-lives up to $2.5 \cdot 10^5$ years). Therefore, it takes a very long time to reach equilibrium between ^{238}U and ^{214}Pb if nuclides from the decay chain are removed from the material for example during a melting process. It is also very hard to detect low uranium concentrations by analysis of gamma rays from other nuclides in the decay chain. Neutron activation analysis can thus be regarded as a more sensitive method for analyses of uranium in metals than gamma spectrometry.

Table 4. Results of gamma spectrometric analyses of aluminium samples.

Laboratory	Number of samples	Sample weight (kg)	Counting time (hours)	Nuclide	Nuclide concentration (Bq/kg)	Uncertainty (%)
Risø	5	0.5	72	^{238}U ^{228}Ra	4.3-6.5 0.8-1.6	20 30
VTT	25	0.06	12		Below detection limit (^{60}Co: <3)	
Geislavarnir	5		80-90	^{228}Th/^{232}Th ^{235}U ^{238}U	0.9 0.13 2.7	30 40 30
IFE	20	0.03	≥ 24	^{212}Pb	1.4-5.8	10-20
IFE	1	0.03	≥ 24	^{212}Pb	32.1	10
NRPA	5	0.03	≥ 24	^{226}Ra ^{232}Th	1.8-8.3 1.5-5.3	10-30 10
NRPA	21	0.03	≥ 24		Below detection limit (^{232}Th: <2.7)	

Table 5. Results of total beta analyses of aluminium samples.

Laboratory	Number of samples	Sample weight (kg)	Beta activity Bq/kg)	Uncertainty (%)	Detection limit (Bq/kg)
Risø	5	0.009	1.1-1.5	2-6	0.2-0.3

Table 6. Results of neutron activation analyses of aluminium samples.

Laboratory	Number of samples	^{238}U		^{232}Th	
		ppm	Bq/kg	ppm	Bq/kg
IFE	2	0.8-3.5	10.0-43.5	0.7	2.8

Table 7. Results of gamma spectrometric analyses of magnesium samples.

Laboratory	Number of samples	Nuclide	Nuclide concentration (Bq/kg)	Uncertainty (%)
IFE	2	^{212}Pb	1.4-1.9	20-40
IFE	2		Below detection limit (^{212}Pb: <1.4)	
NRPA	3		Below detection limit (^{232}Th: <2.7)	

Table 8. Results of neutron activation analyses a magnesium sample.

Laboratory	Number of samples	^{238}U		^{232}Th	
		ppm	Bq/kg	ppm	Bq/kg
IFE	1	0.5	6.2	0.7	2.8

4.3 Magnesium samples

Naturally occurring radioactivity was also fond in the magnesium samples analysed. Results of gamma spectrometric measurements of the samples are shown in Table 7. One of the samples was also analysed by neutron activation (see Table 8). 1.4 ± 0.5 Bq/kg ^{112}Pb was found at the gamma spectrometric measurement of the sample. This result is in the same order of magnitude as the ^{232}Th concentration from the neutron activation measurement. However, ^{238}U was only detected by neutron activation analysis).

4.4 Other samples

Table 9 shows results from gamma spectrometric analyses of different samples taken from the smelting process for comparison. In addition a ferrosilicon sample was analysed. The samples contained small amounts of radionuclides from the uranium and thorium series.

Table 9. Results of gamma spectrometric analyses of samples from the process.

Type of sample	Lab	Number of samples	Nuclide	Nuclide concentration (Bq/kg)	Uncertainty (%)
Fe slag	IFE	12	^{212}Pb ^{214}Pb	3.2-9.6 15-22	10-20 10
Al slag	IFE	11	^{212}Pb	4.1-11	10-20
Al slag	IFE	1		Below detection limit (^{212}Pb: <4.6)	
Al slag	NRPA	5	^{226}Ra ^{232}Th	3.1-8.8 4.5-8.8	10 10
Al slag	NRPA	7		Below detection limit (^{232}Th: <2.7)	
Al anode	NRPA	1	^{210}Pb	47	10
Al anode	NRPA	1		Below detection limit (^{210}Pb: <19)	
Mg anode	IFE	1	^{60}Co ^{137}Cs ^{212}Pb	2.7 1.1 2	40 40 30
Foundry	Studsvik	11		Below detection limit (^{60}Co: <1.7-12)	4-30

5 Intercomparison

Three different samples were transported between the laboratories for intercomparison of the calibrations of the laboratories:

- a sample with aluminium chips from Iceland
- a slightly contaminated 573 g steel sample from Studsvik (the results were decay corrected to the date 2000-06-01)
- a slightly contaminated 242 g aluminium sample from Studsvik (the results were decay corrected to the date 2000-06-01)

The level of radioactivity in the aluminium sample from Iceland was too low to be of use for the intercomparison. The results of the measurements of the other samples are shown in Figures 4-5 and in Table 10. The agreement between the results was within 20 %, which is quite satisfactory for this kind of study.

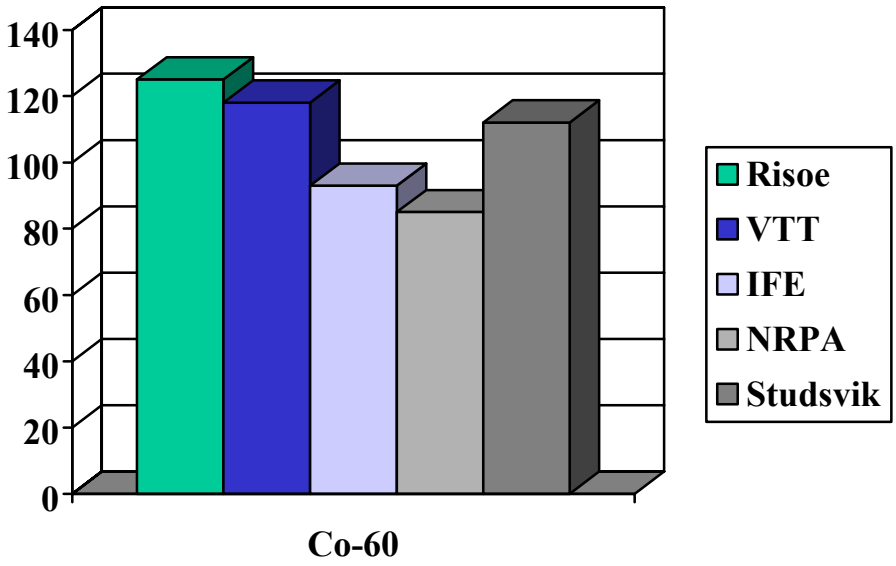

Figure 4. Results (Bq/kg) of gamma spectrometric analyses of an intercomparison steel sample from Studsvik.

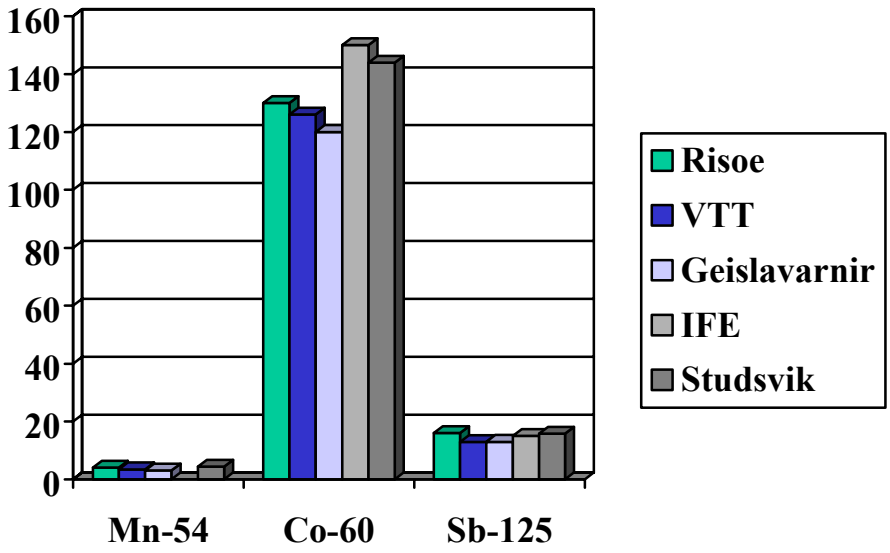

Figure 5. Results (Bq/kg) of gamma spectrometric analyses of an intercomparison aluminium sample from Studsvik.

Table 10. Results of gamma spectrometric analyses of intercomparison samples .

Lab	Steel sample	Aluminium sample		
	Co-60 (Bq/kg)	Mn-54 (Bq/kg)	Co-60 (Bq/kg)	Sb-125 (Bq/kg)
Risø	125 ± 20	4 ± 1	130 ± 30	16 ± 4
VTT	118 ± 5	3.4 ± 0.4	126 ± 5	12.9 ± 0.9
Geislavarnir		3 ± 1	120 ± 30	13 ± 4
IFE	93 ± 6	Below detection limit	150 ± 30	15 ± 3
NRPA	85 ± 5			
Studsvik	112 ± 5	4.38 ± 0.42	144 ± 5	15.8 ± 0.9

6 Discussion, conclusions and recommendations

Measurements of the radioactivity in metal samples were performed at laboratories in the Nordic countries. The samples were received from steel, aluminium and magnesium producers. No radioactivity or radioactivity levels close to the detection limits were found in the steel samples. Very low activities from the naturally occurring uranium and thorium series radionuclides were found in some of the aluminium and magnesium samples.

Most samples were analysed with gamma spectrometric equipment. However, it is not a simple task to perform low-level gamma spectrometric measurements, especially with metal samples due to heavy self-absorption of the gamma rays in the sample. This self-absorption sets a limit to the useful sample size. As the level of radioactivity in commercial metal products is generally low and close to the limit of detection, the background characteristics of the gamma spectrometer systems are very important. The background levels of the spectrometer systems at different laboratories differ.

It was only possible to analyse a limited number of samples in this study since the measurements are very time-consuming. Therefore, it could be of interest to perform measurements of more samples both from the same and also from other metal producers, possibly also in other countries.

It can be stated that, this study has found no indication of elevated radioactive contamination due to recycling of steel, aluminium or magnesium metals. It could be of interest to repeat a similar study later on in the future to see how the contamination status evolves.

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