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Section A

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Identification of  $^{63}\text{Ni}$  and  $^{60}\text{Co}$  produced in a steel sample by  
thermal neutrons from the Hiroshima atomic bomb

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# Identification of $^{63}\text{Ni}$ and $^{60}\text{Co}$ produced in a steel sample by thermal neutrons from the Hiroshima atomic bomb

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## Abstract

Long-lived residual radioactivity  $^{63}\text{Ni}$  produced by the  $(n, \gamma)$  reaction was detected for the first time from a steel plate sampled at near the hypocenter of the Hiroshima atomic bomb. Nickel and cobalt were chemically separated and enriched from the steel sample. Low energy beta rays of  $^{63}\text{Ni}$  were measured with a low-background liquid scintillation counter and gamma-rays of  $^{60}\text{Co}$  were measured with a low background Ge detector. Specific activities were determined as  $0.0063 \pm 0.0004 \text{ Bq mg}^{-1}$  for  $^{63}\text{Ni}/\text{Ni}$  and  $8.70 \pm 0.46 \text{ Bq mg}^{-1}$  for  $^{60}\text{Co}/\text{Co}$  at the time of the bomb explosion. Comparisons with the calculated yield based on the current dosimetry system DS86 neutrons were also given.

## 1. Introduction

Recent low-level radioactivity measurements of the residual radioactivities  $^{152}\text{Eu}$  (half-life  $T_{1/2} = 13.5 \text{ yr}$ ) and  $^{60}\text{Co}$  ( $T_{1/2} = 5.27 \text{ yr}$ ) and atomic mass spectrometry (AMS) of  $^{36}\text{Cl}$  ( $T_{1/2} = 3 \times 10^6 \text{ yr}$ ) induced by the Hiroshima atomic bomb (A-bomb) neutrons have revealed a systematic discrepancy from the calculation based on the neutron fluence of the new dosimetry system DS86 [1]. This discrepancy must be resolved not only for the validation test of the current neutron transport calculation, but for fundamental risk estimates of radiation effects and safety standards.

Shibata et al. [2] have recently proposed a new possibility to estimate the fast neutron fluence through the  $^{63}\text{Ni}$  ( $T_{1/2} = 100 \text{ yr}$ ) measurement. Up to now, the only data concerning the fast neutron fluence were those of  $^{32}\text{P}$  ( $T_{1/2} = 14.2 \text{ d}$ ) produced by the  $^{32}\text{S}(n, p)^{32}\text{P}$  reaction (threshold energy  $\sim 3 \text{ MeV}$ ) measured by Yamazaki et al. [3] immediately after the Hiroshima bomb. Shibata et al. have pointed out that  $^{63}\text{Ni}$  produced in copper samples by the  $^{63}\text{Cu}(n, p)^{63}\text{Ni}$  reaction (threshold energy  $\sim 1 \text{ MeV}$ ) should be measurable by using a low-background liquid

scintillation counter. Since the thermal neutron capture reaction  $^{62}\text{Ni}(n, \gamma)^{63}\text{Ni}$  is an interference in the evaluation of fast neutrons, high purity copper samples such as electric wire are needed. On the other hand, Beasley and Head [4] have measured  $^{63}\text{Ni}$  in environmental samples taken from areas of the Pacific Ocean. They concluded that the nuclide was produced through the  $(n, \gamma)$  activation processes in the testing of nuclear devices.

We have previously reported [5] the specific activity  $^{60}\text{Co}/\text{Co}$  in steel samples near the hypocenter of the Hiroshima A-bomb. Up to now, many authors [5–8] have reported on the residual radioactivity  $^{60}\text{Co}$  in steel samples, but no one has yet measured  $^{63}\text{Ni}$ . The present work reports a first observation of  $^{63}\text{Ni}$  radioactivity produced in a steel sample exposed to the A-bomb. In steel samples, the fast neutron reaction  $^{63}\text{Cu}(n, p)^{63}\text{Ni}$  is negligibly small, but the thermal neutron capture  $^{62}\text{Ni}(n, \gamma)^{63}\text{Ni}$  reaction is dominant. Since  $^{63}\text{Ni}$  emits only low-energy beta-rays of maximum energy 67 keV, chemical separations of Ni and Co from the steel were carefully performed and a liquid scintillation counter was utilized for the beta ray measurement. The gamma rays of  $^{60}\text{Co}$  simultaneously separated from the same sample were measured with a low-background Ge detector. Specific activities of  $^{63}\text{Ni}/\text{Ni}$  and  $^{60}\text{Co}/\text{Co}$  were compared with the calculations for the evaluation of DS86 neutrons.

\* Corresponding author.

## 2. Experimental details and results

### 2.1. Sample preparation

Steel plates (S4) were obtained from the Hiroshima A-bomb Dome located at a ground range of  $163 \pm 15$  m northwest of the hypocenter as shown in Fig. 1. The sample was taken from the roof top at 21 m above the ground level; the slant range from the explosion point ( $580 \pm 15$  m above the ground level) was  $602 \pm 21$  m.

The steel plate of 600 g was divided into small chips by using a milling machine. The contamination due to wear out of the edged tool was checked measuring the weight of the tool. No significant wearing was observed during the operation. The chips were etched with HCl to remove the surface contamination and were dissolved in hot concentrated HCl. After cooling the solution with a total volume of 4 L, the residue was removed by filtration. Small amounts ( $\sim 50$  ml) of  $\text{HNO}_3$  and  $\text{H}_2\text{O}_2$  were added in the solution to oxidize  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$ . Solvent extraction with the isopropyl ether was applied to extract Fe ions leaving Ni and Co ions in the solution. Then the solution was heated to reduce the volume. This process was repeated over and over again, and finally 200 ml of solution was obtained. After the solvent extraction, the solution was passed through an anion exchange column containing 300 g of Dowex 1-X8 (100–200 mesh, type C1) resin. The resin was then washed with 8M HCl to collect Ni ions, 4M HCl to collect Co ions and 0.5M HCl to wash out Fe ions.

For further purification of the Ni fraction, 30% ammonium hydroxide was added to the Ni fraction to adjust the pH to 8. Then dimethylglyoxim (dissolved 1 g per 100 ml ethanol) was added to the solution to form the Ni complex. The red Ni complex can be selectively extracted to chloroform. The Ni ions were reversely extracted in

diluted HCl and were evaporated to dryness. The content of Ni in the enriched sample was determined by means of the atomic absorption method as described below. The amount of Ni in the enriched sample was 12 mg, and the recovery of Ni from the steel sample was 8%.

The Co fraction was precipitated by adding NaOH. The precipitate was collected by filtration, washed well with water and dried. Finally, 1.39 g of Co enriched sample was obtained. The content of Co in the enriched sample was determined by atomic absorption. The recovery of Co from the steel sample was 19%. Results of stable Ni and Co content for each samples are given in Table 1.

### 2.2. $^{63}\text{Ni}$ measurement

A low-background liquid scintillation counter (LSC-LB III; ALOKA Co.) was used for the  $^{63}\text{Ni}$  measurement. The Ni sample for the liquid scintillation counter was prepared as follows. Distilled water was added to the dried  $^{63}\text{Ni}$  residue, and 6 ml of the solution was taken to a 100 ml Teflon vial and mixed with 51 ml emulsive liquid scintillator (clear-sol; Nakalai tesque Co.). The stable Ni content was analyzed using 1 ml of the same solution. The Ni sample was measured repeatedly for a total of 3500 min. Examples of a  $^{63}\text{Ni}$   $\beta$ -ray spectrum and the background are shown in Fig. 2.

The efficiency calibration for the  $^{63}\text{Ni}$  measurement was performed as follows. Six standard samples containing different amount of radioactivities ( $^{63}\text{Ni}$ : 230, 460 and 921 Bq and diluted samples: 4.6, 9.1 and 22.8 Bq) were dispensed to 100 ml Teflon vials from the standard solution (AmershamNBZ44, 46.8 kBq/(g sol) with total error  $\pm 4.0\%$ ) and were prepared similarly to the sample solution. A background sample containing distilled water of 6 ml and same 51 ml of liquid scintillator was also prepared. Each calibration sample and the background sample were measured for a total of 870 min. The gross counts were calculated by setting the discrimination levels

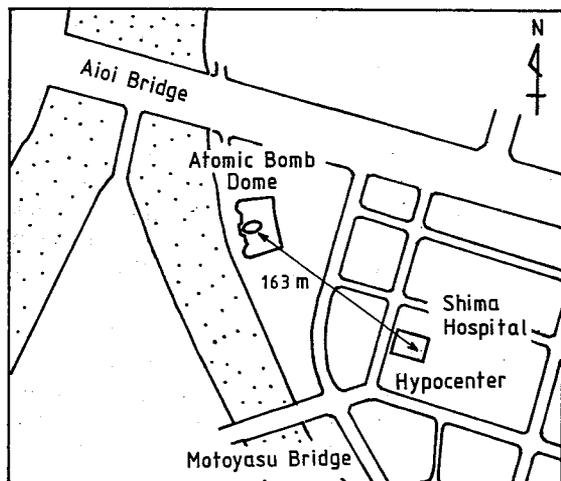


Fig. 1. Locations of the Atomic-bomb Dome and the hypocenter in Hiroshima.

Table 1  
Deduction of specific activities  $^{63}\text{Ni}/\text{Ni}$  and  $^{60}\text{Co}/\text{Co}$

Original steel weight	600 g
Elemental concentration (enriched sample)	
Ni	$1.72 \pm 0.03 \text{ mg (g sol)}^{-1}$
Co	$21.4 \pm 1.1 \text{ mg g}^{-1}$
Net counting rate	
$^{63}\text{Ni}$ (0.5–30.0 keV)	$1.76 \pm 0.11 \text{ cpm}$
$^{60}\text{Co}$ (1173 + 1332 keV)	$1.780 \pm 0.026 \text{ cpm}$
Efficiency	
$^{63}\text{Ni}$	$0.641 \pm 0.017 \text{ cps Bq}^{-1}$
$^{60}\text{Co}$	$0.079 \pm 0.0010 \text{ cps Bq}^{-1}$
Specific activity	
(February 1995) $^{63}\text{Ni}/\text{Ni}$	$0.0044 \pm 0.0003 \text{ Bq mg}^{-1}$
$^{60}\text{Co}/\text{Co}$	$0.0127 \pm 0.0007 \text{ Bq mg}^{-1}$
(August 1945) $^{63}\text{Ni}/\text{Ni}$	$0.0063 \pm 0.0004 \text{ Bq mg}^{-1}$
$^{60}\text{Co}/\text{Co}$	$8.70 \pm 0.47 \text{ Bq mg}^{-1}$

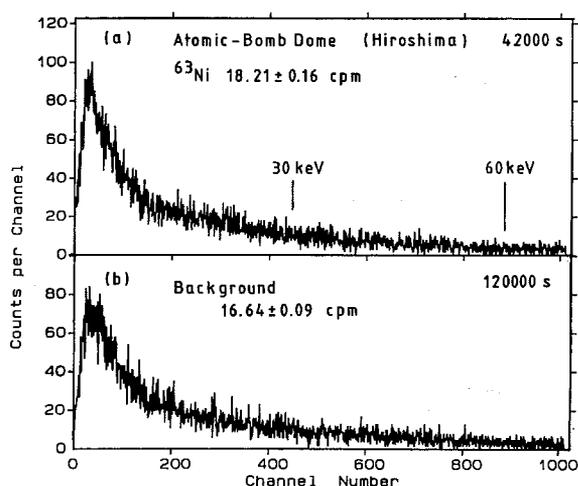


Fig. 2. Typical  $\beta$ -ray spectra measured with a low-background liquid scintillation counter. (a)  $^{63}\text{Ni}$  sample extracted from the steel plate exposed to the atomic bomb. (b) Background spectrum. The counting rate of the Ni sample in the energy region 0.5–30 keV is significantly higher than the background.

corresponding to 0.5 keV (lower level) and 30 keV (upper level), which were determined from the  $\beta$ -ray spectrum of calibration samples. Thus, the efficiency, gross counting rates of the Ni sample and the background were obtained as given in Table 1. The counting rate of Ni sample is significantly higher than that of background considering the detection limit  $3\sigma_0 = 0.23$  cpm. The Ni sample was measured with a Ge detector to check the existence of other radioactivities, but no radioactive contamination was observed. Thus, a net counting rate  $1.76 \pm 0.11$  cpm was attributed to the  $^{63}\text{Ni}$  activity. The specific activity  $^{63}\text{Ni}/\text{Ni}$  at the time of the bomb explosion as well as at the measurement (February 1995) are given in Table 1.

### 2.3. $^{60}\text{Co}$ measurement

The  $\gamma$ -ray measurement was performed with a low-background well-type Ge detector. This detector was shielded with more than 20 cm of lead and an anticoincidence system was equipped to reduce the cosmic-ray background. Details of the detector system have been described separately [9]. Contamination of  $^{137}\text{Cs}$  and  $^{60}\text{Co}$  which might be included in the detector material and shielding did not appear in the background spectrum. The Co-enriched sample was pressed into a polypropylene test tube of 13 mm diameter and 7.5 cm height, and the measurement was continued for about two days. An example of the  $\gamma$ -ray spectrum is shown in Fig. 3. Two  $\gamma$  rays of  $^{60}\text{Co}$  are clearly seen.

The calibration of the Ge detector was performed by using a standard source (Amersham CKZ24, 3.587 kBq/(g sol) with total error  $\pm 0.5\%$ ). A known amount of  $^{60}\text{Co}$

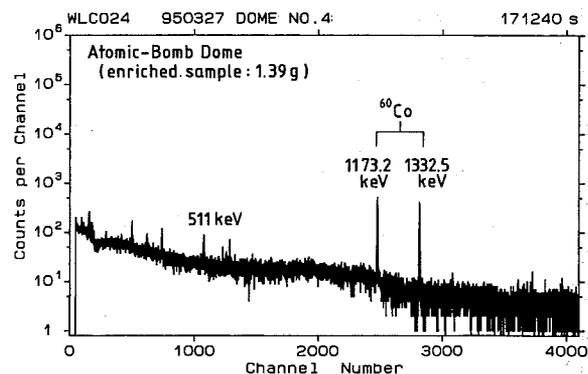


Fig. 3. Typical gamma-ray spectrum of a cobalt-enriched sample measured with a well-type Ge detector. The neutron induced radioactivity of  $^{60}\text{Co}$  is clearly seen.

102 Bq was deposited to every 0.5, 1.0, 2.0 and 3.0 g control samples (non-neutron-exposed steels) prepared in the same way. The sum of peak counts corresponding to the 1173 and 1332 keV was used for the efficiency calibration. It was determined to be  $0.0790 \pm 0.0010$  (cps/Bq) for the 1.39 g sample. The stable Co content in the enriched sample was determined by an atomic absorption method. Finally, the specific activity  $^{60}\text{Co}/\text{Co}$  at the time of the explosion was determined to be  $8.70 \pm 0.47$  Bq  $\text{mg}^{-1}$  as given in Table 1. This result is consistent with the previous value of  $10.0 \pm 1.0$  Bq  $\text{mg}^{-1}$  obtained from the same plate [5].

## 3. Discussion

### 3.1. Calculation of $^{63}\text{Ni}$ and $^{60}\text{Co}$ activation

The number of  $^{63}\text{Ni}$  atoms  $N_0$  produced in 1 mg Ni by neutrons emitted from the A-bomb can be expressed as

$$N_0 = \int N \frac{d\phi}{dE} \sigma(E) dE, \quad (1)$$

where  $N$  is the number of atoms per 1 mg Ni,  $d\phi/dE$  is the neutron fluence at a certain distance from the hypocenter and  $\sigma(E)$  is the cross section for the  $^{62}\text{N}(n, \gamma)^{63}\text{Ni}$  reaction at neutron energy  $E$ . Since the  $^{63}\text{Ni}$  production is dominant for thermal neutrons, Eq. (1) can be approximated as follows,

$$N_0 = N\phi^*\sigma^*, \quad (2)$$

where  $\phi^*$  is the thermal neutron fluence and  $\sigma^*$  is the cross section for thermal neutrons. Taking the thermal neutron fluence as  $\phi^* = 8 \times 10^{12}$  n  $\text{cm}^{-2}$  based on the calculation by Pace [10] and  $\sigma^* = 14.2$  b, one obtains  $N_0 = 4.2 \times 10^5$  atoms  $\text{mg}^{-1}$ , which corresponds to a radioactivity of  $A_0 = \lambda N_0 = 0.0093$  Bq  $\text{mg}^{-1}$  Ni ( $\lambda = \ln 2/t_{1/2}$ ).

Table 2  
Calculated activities produced by fast neutrons and thermal neutrons

Nuclide	Reaction	Activities <sup>a</sup> produced in 600 g steel sample [Bq]	Ratio to (n, $\gamma$ ) reaction
<sup>63</sup> Ni	<sup>62</sup> Ni(n, $\gamma$ ) <sup>63</sup> Ni	1.06	1.0
	<sup>63</sup> Cu(n, p) <sup>63</sup> Ni	$9.3 \times 10^{-3}$	$8.8 \times 10^{-3}$
<sup>60</sup> Co	<sup>59</sup> Co(n, $\gamma$ ) <sup>60</sup> Co	2016	1.0
	<sup>60</sup> Ni(n, p) <sup>60</sup> Co	$2.4 \times 10^{-4}$	$1.2 \times 10^{-7}$
	<sup>63</sup> Cu(n, $\alpha$ ) <sup>60</sup> Co	$2.7 \times 10^{-3}$	$1.3 \times 10^{-6}$

<sup>a</sup> At the time of bomb explosion.

Table 3  
Comparison of calculated specific activities produced by cosmic-ray induced neutrons with the measured residual activity

Nuclide	Saturation activity produced by cosmic-ray induced neutrons A [Bq mg <sup>-1</sup> ]	Measured residual activity <sup>a</sup> B [Bq mg <sup>-1</sup> ]	Ratio A/B
<sup>63</sup> Ni/Ni	$9.3 \times 10^{-27}$	0.0063	$1.5 \times 10^{-24}$
<sup>60</sup> Co/Co	$1.2 \times 10^{-14}$	8.7	$1.3 \times 10^{-14}$

<sup>a</sup> At the time of bomb explosion.

$T_{1/2}$ ). In the case of <sup>60</sup>Co, the contribution of epithermal neutrons as well as thermal ones are not negligible. An estimation from only thermal neutrons gives  $12.5 \text{ Bq mg}^{-1}$  taking the (n,  $\gamma$ ) cross section  $\sigma = 37 \text{ b}$ , however, a detailed calculation of  $14 \text{ Bq mg}^{-1}$  by Mendelsohn [11] is more reliable.

Thus, the ratio of the calculated to measured values are estimated to be 1.5 and 1.6 for <sup>63</sup>Ni/Ni and <sup>60</sup>Co/Co, respectively. These results are consistent with previous ones of <sup>152</sup>Eu, <sup>60</sup>Co and <sup>36</sup>Cl [12,13] implying that the calculated neutron fluence is overestimated near the hypocenter.

### 3.2. Contribution of the fast neutron reaction and cosmic-ray induced neutrons

The radioactivities <sup>60</sup>Co and <sup>63</sup>Ni in the steel sample are mainly produced by the thermal neutron capture process, however, they could be also produced by fast neutron reactions. According to Kimura and Hamada [14] who have chemically analysed the same steel sample, the impurities included in the steel are Cu:  $4.4 \text{ mg g}^{-1}$  sample, Ni:  $0.28 \text{ mg g}^{-1}$  sample and Co:  $0.24 \text{ mg g}^{-1}$  sample. Based on the neutron spectrum by Pace [10], neutron cross sections by Shibata et al. [15] and by Csepak et al. [16], the yield of <sup>63</sup>Ni and <sup>60</sup>Co activities in 600 g of steel sample were estimated as given in Table 2. According to these results, the fast neutron contribution is negligibly small both for <sup>63</sup>Ni and <sup>60</sup>Co.

There is another possibility that cosmic-ray induced neutrons produce <sup>63</sup>Ni and <sup>60</sup>Co activities in the steel matrix as background. Such a neutron spectrum and flux are estimated in the UNSCEAR report [17]. Taking the neutron flux as  $0.008 \text{ n cm}^{-2} \text{ s}^{-1}$ , saturation activities are

calculated as given in Table 3. Since the calculated activities are  $10^{-15}$ – $10^{-27}$  times lower than the measured values, the contribution of cosmic-ray induced neutrons is also negligible.

## 4. Conclusion

Specific activities of <sup>63</sup>Ni/Ni and <sup>60</sup>Co/Co were determined simultaneously for a steel sample exposed to the A-bomb at near the hypocenter. These activities were produced by thermal neutrons, because the estimated yield of fast neutrons and/or cosmic-ray induced neutrons are negligibly small. Comparison with calculation based on the DS86 neutrons indicates that the neutron fluence is overestimated at a point near the hypocenter. The detection of <sup>63</sup>Ni implies that the residual radioactivity measurement would be possible for a few tens of years in the future. Moreover, the evaluation of fast neutrons through the <sup>63</sup>Ni measurement in copper samples would be also promising.

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