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Studsvik Report

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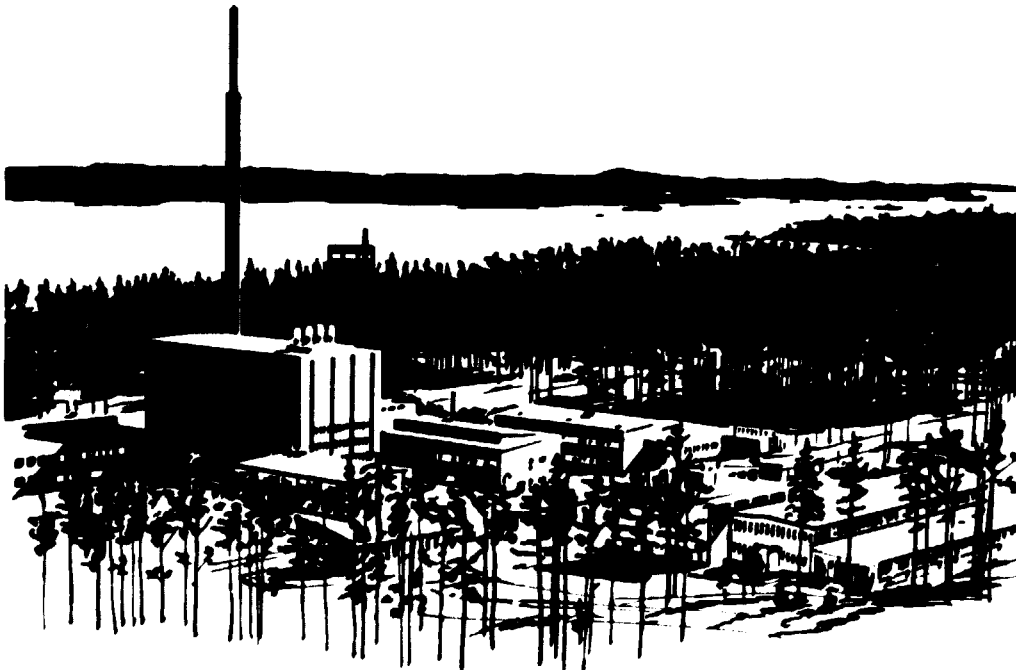
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RELEASE OF CORROSION PRODUCTS FROM CONSTRUCTION MATERIALS CONTAINING COBALT

Part 2: Inconel X750

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ABSTRACT

This report describes experimental work aimed at determining the release rate for corrosion products from 18Cr8Ni steel and Inconel X750 in BWR environments. For test purposes these environments were simulated in a high pressure loop, where irradiated samples of the materials were exposed for 720 hours. The amounts of released products were determined using gamma spectrometric analysis.

The results show that the release from Inconel X750 is higher than that from 18Cr8Ni steel. The release calculated from Co58 measurements is 7 times higher and from Co60 measurements it is 1.5 times higher. Both the filtered and the deposited fractions of the released corrosion products exhibit the same relative concentrations of Co58 and Co60.

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1. INTRODUCTION

A knowledge of release rates for metals from construction materials is very important when calculating crud and activity transport in reactor systems. The aim of the present investigation is to improve this knowledge by means of experimental measurements of release rates in carefully controlled environments. The experiments were planned in collaboration with ASEA-ATOM (1, 2, 3, 4); they were primarily intended to yield determinations of the release rates for cobalt and other metals from 18Cr8Ni steel. The first part of the investigation was concerned with materials having distinctly different cobalt contents, and the results demonstrated that the release of Co60 was proportional to the cobalt content of the material. In the second part of the investigation, measurements have been made in order to compare the release rates from Inconel X750 and 18Cr8Ni steel, which have approximately the same cobalt contents.

The investigations were carried out in the Chemistry section at AB Atomenergi, Studsvik, in collaboration with ASEA-ATOM.

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2. DESCRIPTION OF THE EXPERIMENTS

The experiments were performed in a high pressure loop where a BWR environment was simulated. The main components of the loop were a pump, a pre-heater, a vessel for maintaining pressure, samples of irradiated materials and a filter system. A diagram of the loop is shown in Figure B.1.

The water entering the loop was dosed with oxygen to a concentration of 100 - 300 $\mu\text{g l}^{-1}$. The water was heated to 280°C in the pre-heater and pressure maintenance vessel, and was then led through four parallel lines containing the active samples. The flow rate was low (Re 5 000) in two of the lines, and high (Re 20 000) in the other two. The active samples were made of 18Cr8Ni steel and Inconel X750, and they were exposed in pairs to low and high flow rates. After passing over the active samples, the water was cooled in vertical coolers with spirally wound cooling coils.

The corrosion products released in the form of particles and ions were trapped in a filter packet after the water had been cooled to room temperature. The active products in the filter packets were subsequently analyzed using gamma spectroscopy. The loop was operated as a once-through system, i.e. all the water from the loop was led to the active waste system. The loop is described in greater detail in ref (5).

2.1 Filter system

The water was filtered through \emptyset 47 mm filter packets housed in filter containers manufactured by Millipore. Beyond the filter, the pressure was reduced by means of valves to 300 kPa. The flow through each pipe and filter was set by means of needle valves, and monitored with flowmeters.

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Between the active samples and the filters there were separate pipes and coolers which were only used during the filtering periods. The products released from the active samples which deposited in these pipes and coolers were recovered by decontamination and analyzed while the loop was in operation.

2.2 Instrumentation

The specific conductivity and oxygen content of the water were monitored continuously in the main supply line for deionized water. The lower limits for the alarm signals were set at $0.5 \mu\text{S cm}^{-1}$ and 10 ppb O_2 . Oxygenated water was injected into the inlet pipe of the loop by an adjustable pump (2). A sampling line (1.1 ml s^{-1}) was taken off just before the samples (3), and the dosage was adjusted so that the oxygen content in this line was 100 - 300 ppb. The oxygen content was monitored continuously with a thallium cell, and recorded on a pen recorder. Spot checks were made of the pH value and the specific conductivity of the water that had passed through the loop. The results are summarized in Table A.1.

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3. TEST MATERIAL

In part 1 of the investigation, the release was measured from two 18Cr8Ni steels with cobalt contents of 0.027 and 0.18 %. For the present tests, new samples were made from the steel containing 0.18 % cobalt and from Inconel X750, containing 0.15 % cobalt. In both cases the initial material was in the form of steel bars. The complete compositions of the materials are given in Table A.2. The samples were sawn out in the axial direction of the steel and turned to give thin-walled tubes. The dimensions and appearance of the samples are shown in Figure B.2.

After degreasing, the Inconel X750 samples were heat-treated as follows.

1. $1\ 050^{\circ}\text{C} + 30^{\circ}\text{C}$ 15 ± 5 minutes
 $- 0^{\circ}\text{C}$
2. 705°C 20 hours
 Vacuum $5 \cdot 10^{-6}$ torr

The 18Cr8Ni samples were not heat-treated.

3.1 Irradiation and assembly

The samples were placed together in one container and were irradiated in the Studsvik R2-reactor for 16 days. The neutron flux in the irradiation position was calculated by measuring the activity in small pieces of cobalt, titanium and nickel that were placed in the container together with the samples. The values of neutron flux are given in Table A.3.

The irradiated samples were mounted in 6 x 1 mm tubes as shown in Figure B.3. Sleeves, with the same internal diameter as the samples, were placed

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just in front of the samples in the direction of flow. The function of the sleeves was to prevent the wrong flow profile arising in the inlets of the samples. The tubes were then transported inside the shielding and connected to the appropriate branch lines in the loop by means of Swagelok couplings.

In two of the branch lines, inactive samples of 18Cr8Ni steel were installed c 1 m in front of the active samples.

During irradiations, a crack formed in one of the 18Cr8Ni samples. This sample was therefore replaced by a sample of the same material that had been used in the previous experiment, and that had been irradiated 10 months earlier.

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4. EXPERIMENTAL CONDITIONS

The following values, which are typical for a BWR environment, were employed:

t, °C	280
pH	7
K, $\mu\text{S/cm } 25^\circ\text{C}$	< 1
O ₂ $\mu\text{g l}^{-1}$	100 - 300

Flow rates:	Low flow rate	High flow rate
Linear flow rate m s^{-1}	0.26	1.05
Reynolds number, Re	5 000	20 000

Samples of both 18Cr8Ni steel and Inconel X750 were exposed to each flow rate.

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5. EXPERIMENTAL PROCEDURE

The loop was operated continuously, with the exception of one 17-hour stop following 526 hours' operation. This stop was caused by valve leakage.

The filter holders were loaded with packets consisting of one Millipore filter plus ion exchange membranes. The composition of each packet was: MP, SA, SB, SA, SB, SA, where MP denotes the 0.45 μm Millipore filter, SA a cation exchanger and SB an anion exchanger. The four branch lines were filtered simultaneously and with equal flows through each filter. When the flow rate was high, one fourth was filtered, while when it was low the entire flow was filtered. Consequently the measurements of release obtained with the high flow rate have been multiplied by four. After 6 - 8 hours' filtering, the packets were removed from the filter holders and the amounts of corrosion products filtered off were determined by gamma spectrometric analysis. The measurements were made using a planar Ge(Li) detector and multichannel equipment (Nuclear Data). The background value for Co60 was 12 pCi, and measuring times of 1 - 2 hours were used. Decontamination of the pipes and filters was carried out after 1, 2 and 3 filtrations according to the following schedule:

Exposure time, h

350	Filtration
420	"
520	"
	Decontamination
600	Filtration
	Decontamination
690	Filtration
720	"
	Decontamination

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A list of the pipes that were decontaminated between the active samples and the filters is given in Table A.4. The surfaces were pretreated with an alkaline permanganate solution for about 1 hour, and an acid mixture (6 % citric acid and 3 % oxalic acid) was then circulated through the pipes for 2 hours at a temperature of 90°C. The amounts of radioactivity in the solutions were determined by measuring about 1 liter of the solution in special plastic vessels with central holes for the detector. The efficiency of this cleaning method was checked by measuring the activity remaining in the cooling tubes, using an NaI(Tl) detector.

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6. RESULTS

(All measurements of radioactivity have been corrected for decay. Reference date: 1977-03-06.)

6.1 Release rates

The release rates, expressed in nCi h^{-1} , have been summarized in Tables A.5 and A.6 for low and high flow rates respectively. In the case of the $^{18}\text{Cr}^{80}\text{Ni}$ sample exposed to a low flow rate, no measurements were obtained for Cr^{51} , Fe^{59} and Co^{58} because the isotopes had decayed for 10 months longer than in the other samples.

6.2 Deposition rates

The amounts of released products which were deposited in the pipes and coolers between the samples and filters were determined by means of decontamination and activity measurements. The deposition is given as nCi h^{-1} in Tables A.7 and A.8 and as percentages of the total release in Table A.9.

6.3 Total release

The sum of the released material, in the form of deposits and filtrates from the water, is given in Tables A.10 and A.11.

6.4 Specific activity

The specific activity of the samples was calculated with the help of the measured values for neutron flux given in Table A.3 plus empirical values for the absorption cross section. The values thus obtained are shown in Table A.12. The following reactions have been taken into consideration for Co^{58} and Co^{60} :

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	<u>mCi g⁻¹</u>	
	<u>18Cr8Ni</u>	<u>X750</u>
Co59 (n,2n) Co58	0.2	0.2
Ni58 (n,p) Co58	26	193
Co59 (n,γ) Co60	31	25.5
Ni60 (n,p) Co60	1	8
Ni61 (n,np) Co60	0	0
Cu63 (n,α) Co60	2	1
Cu63 (n,He3) Fe60 → Co60	0	0

The values for Co58 have been corrected for burn up of Co58 by reaction with thermal neutrons.

6.5 Release rates in mg dm⁻² month⁻¹

The release rates in nCi h⁻¹ were converted to mg per dm² and month, using the specific activities of Co58 and Co60 in the samples. The values obtained are given in Table A.13.

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7. DISCUSSION AND CONCLUSIONS

7.1 Corrosion rate

The inactive 18Cr8Ni samples placed about a meter in front of the active samples were weighed before and after the experiment. The increase in weight of the samples was converted to mg per dm² and month, and amounted to 9.8 mdm. It should be noted that these samples were also exposed to the water during the run-up period, when the corrosion rate is higher than for steady-state operations; consequently the above value should represent the maximum mean value for the corrosion rate in the loop.

Photomicrographs were taken of cross sections of the samples, in both a metallographic microscope and an SEM. The oxide thickness on the samples varied considerably. In some areas it was about 0.2 μm , but single crystals also occurred where the thickness amounted to 1 μm . If the thickness is converted to corrosion rate then 0.2 μm corresponds to 10 mdm.

7.2 Effect of the flow rate

There was no significant difference between the release at low and high flow rates, as can be seen from Table A.13.

7.3 Deposition

The fractions of released corrosion products deposited during sampling periods of 7, 14 and 21 hours are compared in Table A.9. With the exception of the 18Cr8Ni sample in a high flow rate, which again showed anomalous values, there is no difference between Co58 and Co60 as regards deposition behaviour. The values demonstrate that both

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sampling time and flow rate affect the deposition. With a low flow rate the deposition is greatest after 7 hours' sampling, and with the higher flow rate it is greatest after 14 hours' sampling.

If the total amounts of released corrosion products are calculated, it is found that the relative concentrations of Co60 and Co58 are the same in both the filtered and the deposited fractions.

7.4 Release from 18Cr8Ni and Inconel X750

The values in Table A.13 are not entirely comparable, since the new 18Cr8Ni sample was obviously damaged during irradiation, while the old 18Cr8Ni sample had been exposed 900 hours more than the Inconel sample in a BWR environment. Consequently, in Table A.14 the values for Inconel are compared with the values for 18Cr8Ni from part 1 of the study (5). This comparison shows that the release from Inconel is higher than that from 18Cr8Ni. Calculated from Co58 measurements it is 7 times higher, and calculated from Co60 measurements it is 1.5 times higher.

During the activation and formation of Co58 according to Ni58 (n,p) Co58, some of the Co58 nuclei near the steel surface are lost because some of the nuclei recoil out of the material. Thus, since the calculated specific activity for Co58 applies for the material as a whole, the calculated release values for Co58 can be too low. It is possible to obtain a reasonable estimate of the drop in Co58 concentration at the surface with the help of Figure B.4. The concentration at point P should be proportional to the ratio between the fraction of the spherical surface which lies within the material and the total spherical surface, if the point lies at the ori-

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gin of the sphere and R is the range of the recoiling nuclei. Thus at the surface, the concentration will be half as great as it is further down in the metal.

The magnitude of R in Figure B.4 depends on the quantity of energy transferred to the recoiling nuclei. In the irradiation position in the reactor used for this experiment, the energy of the fast neutrons amounts to max 10 MeV, and the maximum transferred energy is given by

$$\frac{E_1}{E_{\text{neutron}}} = \frac{4 A}{(A + 1)^2} ; E_1 \approx 1 \text{ MeV}$$

With the help of data from the tables given in ref (6), the range for Ni56 (1 MeV) in 15Cr15Ni steel can be calculated, giving a figure of 0.25 μm . Thus since the corrosion of the metal during the experiment amounted to c 0.1 μm (0.2 μm oxide), it is possible that the concentration gradient for Co58 near the surface of the samples had some effect on the measured values. Consequently the mdm values calculated from Co58 in Table A.14 can be multiplied by a factor greater than 1 but less than 2. This would then give an mdm value for Inconel X750 that is at most twice as large as the value calculated from Co60.

For 18Cr8Ni, on the other hand, the values calculated from Co58 are considerably lower, and even if this surface effect is taken into account the values are c 3 times lower than if they are based on Co60.

Thermodynamical calculations regarding the solubility of nickel and cobalt ferrite have been carried out by von Massow et al. (7). According to Rummery and Macdonald, cobalt is thermodyna-

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mically equivalent to nickel and probably occurs in mixed ferrites with the formula $\text{Co}_x\text{Ni}_{1-x}\text{Fe}_2\text{O}_4$, where x is much less than 1 when the cobalt content of the material is very low. Thus it seems very doubtful whether the different releases of Co58 and Co60 can be explained in terms of different solubilities for nickel and cobalt ferrite.

The results from part 1 (5) show that the release is higher at 280°C than at 160°C . In experiments with 18Cr8Ni steel up to 200°C , Brush and Pearl (8) have also found that the release increases with temperature. For magnetite, on the other hand, the solubility decreases with increasing temperature between 160°C and 280°C , as has been shown for example by Sweeton and Baes (9) and Styrikovich (10). Cobalt oxide, Co_3O_4 , also exhibits a decreasing solubility with increasing temperature (11). It may therefore be concluded that the solubility of the oxides has only a slight effect on the release, at the flow rates (0.3 and 1 m s^{-1}) adopted for this experiment.

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Table A.1

Summary of test conditions. The temperature and O₂ concentration are measured continually, pH and the specific conductivity κ are determined by spot checks

	BWR environment
Water temperature, °C	280 ± 3
pH value	6.1 - 6.6
κ , $\mu\text{S}/\text{cm}$ 25°C	0.3 - 0.6
κ , $\mu\text{S}/\text{cm}$ 25°C	0.8 - 1.1
O ₂ μl^{-1}	100 - 300

Table A.2

Composition of test material (wt %)

	18Cr3Ni Rod ϕ 30 mm	Inconel Rod ϕ 111 mm
Co	0.182	0.15
C	0.039	0.04
Si	0.48	0.06
Mn	1.86	0.08
P	0.023	-
S	0.018	0.004
Cr	18.2	14.78
Ni	8.4	73.99
Mo	0.31	-
Cu	0.087	0.02
V	0.06	-
Ti	< 0.02	2.58
Nb	< 0.01	
W	< 0.02	
Al	-	0.67
Ti	-	2.58
Fe	71	6.8

Table A.3

Irradiation data

Irradiation time		1.364 · 10 ⁶ s	
Reactor energy		16 769 MWh	
Monitor elements		Ni, Ti, Co	
Neutron flux			
$\phi_f T$ (n · cm ⁻²)		$\phi_{th} T$ (n · cm ⁻²)	
Ni	Ti	Co60	
$\sigma = 110$ mb	$\sigma = 11.5$ mb	$\sigma_{th} = 37.4$ b	
(1.98 ± 0.01)10 ²⁰	(2.12 ± 0.01)10 ²⁰	(4.04 ± 0.02)10 ²⁰	

Table A.4

Connections between the radioactive specimens and the filters

	Re 5 000		Re 20 000	
	X750	18Cr8Ni	X750	18Cr8Ni
Tube 6 x 1 mm horizontal, m	2.2	2.5	2.6	2.1
Tube 6 x 1 mm vertical, m	2.8	2.8	2.8	2.8
Tube connections, no	6	6	6	6
Coolers, tube 6 x 1 mm, m	4.0	3.8	4.0	3.8
Valves, no	2	2	2	2
Tubes total, 6 x 1 mm, m	9.0	9.1	9.4	8.7
Area in contact with water, dm ²	11.3	11.4	11.8	10.9

Table A.5

Release at low flow rate (Re 5 000)

Exposure time, h	Release rate, nCi h ⁻¹									
	18Cr8Ni					Inconel				
	Cr51	Mn54	Fe59	Co58	Co60	Cr51	Mn54	Fe59	Co58	Co60
350	-	0.7	-	-	0.7	157	0.4	1.2	28	4.6
420	-	0.6	-	-	1.2	27	0.4	-	22	3.7
520	-	0.5	-	-	1.4	211	0.5	0.9	44	7.3
600	-	0.6	-	-	1.2	12	0.6	-	21	3.6
690	-	0.5	-	-	1.3	8	0.7	-	38	6.3
720	-	0.5	-	-	1.6	17	0.6	-	49	8.3

Table A.6

Release at high flow rate (Re 20 000)

Exposure time, h	Release rate, nCi h ⁻¹ 18Cr8Ni					Inconel				
	Cr51	Mn54	Fe59	Co58	Co60	Cr51	Mn54	Fe59	Co58	Co60
350	9	29	1.2	1.0	2.8	86	4.2	1.0	34	5.1
420	41	3.3	3.9	0.8	1.4	151	0.7	0.7	23	3.8
520	82	2.0	4.1	1.2	1.4	1 300	0.7	3.0	92	15.0
600	25	1.2	1.4	7.1	0.8	476	0.6	1.2	51	8.4
690	2	1.0	-	0.1	0.2	31	0.8	-	19	3.1
720	8	1.0	0.5	0.1	0.2	30	0.8	-	20	3.5

Table A.7

Deposition rates determined by decontamination (Re 5 000)

18Cr8Ni		Deposition rate, nCi h ⁻¹				
Exposure time, h	Filtering time, h	Cr51	Mn54	Fe59	Co58	Co60
350 - 520	21	-	0.1	-	-	2.5
600	7	-	-	-	-	3.0
690 - 720	14	-	-	-	-	3.0
Inconel						
350 - 520	21	236	3.3	0.6	31	6.2
600	7	262	0.2	-	137	24.3
690 - 720	14	346	0.1	-	98	17.8

Table A.8

Deposition rates determined by decontamination (Re 20 000)

18Cr8Ni		Deposition rate, nCi h ⁻¹				
Exposure time, h	Filtering time, h	Cr51	Mn54	Fe59	Co58	Co60
350 - 520	21	1 690	5.4	2.8	445	79.7
600	7	117	-	0.8	13	2.6
690 - 720	14	31	0.2	1.1	2	1.1
Inconel						
350 - 520	21	547	1.3	4.0	157	27.0
600	7	260	0.5	-	64	11.2
690 - 720	14	431	-	-	120	20.6

Table A.9

Deposition expressed as percentage of total release

<u>18Cr8Ni</u>				
Filtering time, h	Re 5 000		Re 20 000	
	Co58	Co60	Co58	Co60
21	-	69	100	98
7	-	72	65	76
14	-	67	100	85
<u>Inconel</u>				
21	50	43	76	77
7	87	87	56	57
14	69	71	86	86

Table A.10

Total release rate (Re 5 000)

<u>18Cr8Ni</u>					
Exposure time, h	Release rate, nCi h ⁻¹				
	Cr51	Mn54	Fe59	Co58	Co60
350 - 520	-	0.7	3.4	-	3.6
600	-	0.6	-	-	4.2
690 - 720	-	0.5	-	-	4.4
<u>Inconel</u>					
350 - 520	360	3.7	1.3	62.7	11.4
600	273	0.8	-	158	27.8
690 - 720	358	0.7	-	141	25.1

Table A.11

Total release rate (Re 20 000)

18Cr8Ni Exposure time, h	Release rate, nCi h ⁻¹				
	Cr51	Mn54	Fe59	Co58	Co60
350 - 520	1 733	16.9	5.8	446	81.5
600	142	1.2	2.3	20	3.4
690 - 720	36	1.3	1.4	2	1.3
Inconel					
350 - 520	1 059	3.2	5.6	207	35.0
600	735	1.1	1.2	115	19.6
690 - 720	461	0.8	-	139	23.9

Table A.12

Specific activity of the samples, calculated using the neutron flux values in Table A.3

Sample	Spec activity, mCi g ⁻¹ steel ⁻¹				
	Cr51	Mn54	Fe59	Co58	Co60
Inconel	3 200	0.4	4.7	193	34.5
18Cr8Ni	3 950	4.3	49	22	34
18Cr8Ni*	-	4.3	-	26	14.5

* Older sample irradiated in May 1976 and exposed to
Re 5 000

Table A.13

Total release expressed as mg per dm² and month. The calculations for columns 1 and 2 refer to Co58 and Co60 respectively

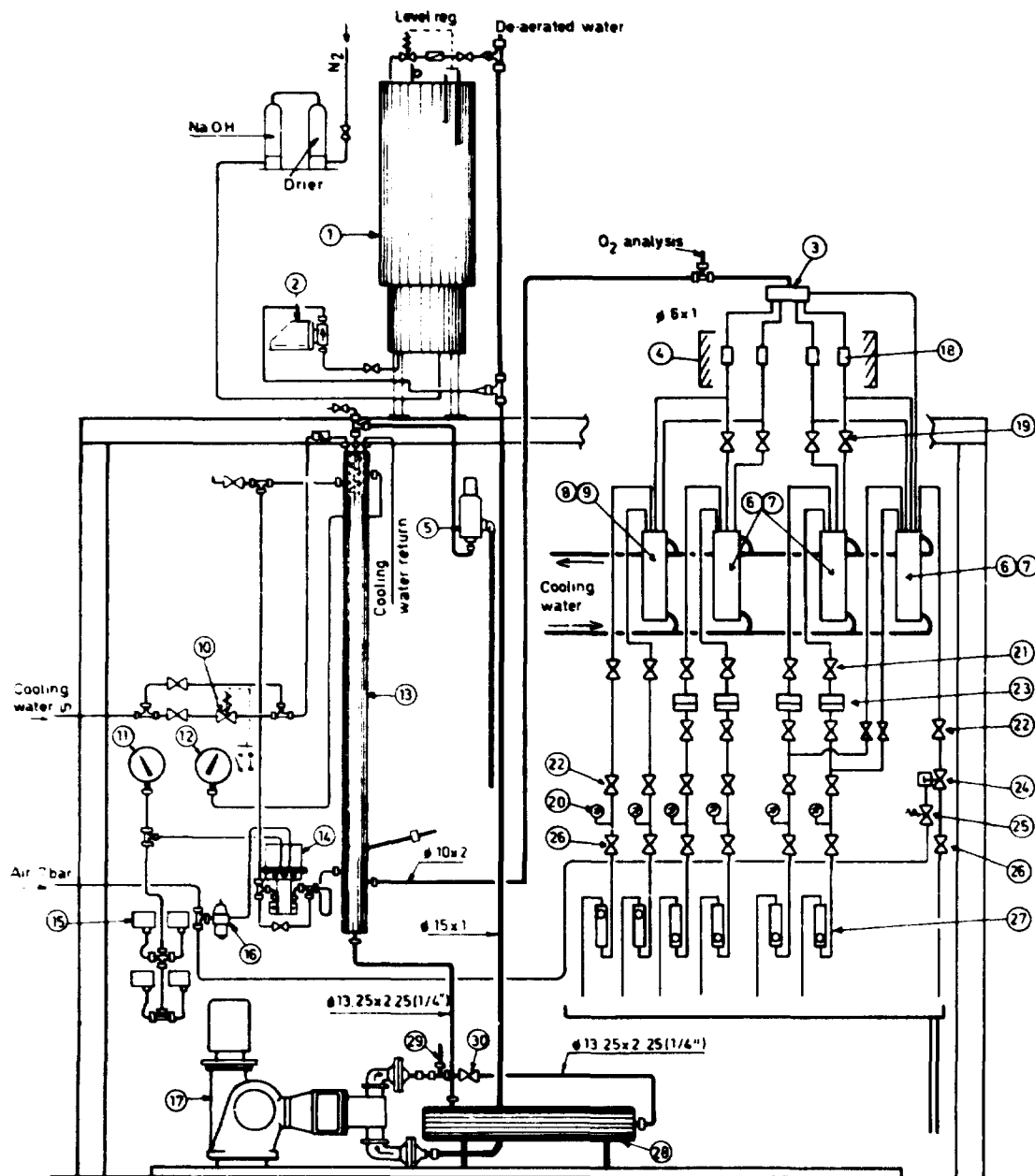
Exposure time, h	Re	18Cr8Ni		Inconel	
		1	2	1	2
600	5 000	-	3.5	9.8	9.7
700	5 000	-	3.6	8.8	3.7
600	20 000	11	1.2	7.2	6.8
700	20 000	1.2	0.5	8.6	8.3

Table A.14

Release expressed as mg per dm² and month for Inconel X750, compared with the values for 18Cr8Ni from ref (5). Columns 1 and 2 give the values calculated for Co58 and Co60 respectively

Re	mg dm ⁻² month ⁻¹			
	18Cr8Ni		Inconel	
	1*	2	1*	2
5 000	1.1	5.6	8.8	8.7
20 000	1.2	5.9	8.6	8.3

* Corrected for burn up of Co58 by reaction with thermal neutrons.



Design pressure 170 bar
 Design temp 350°C
 Max flow rate \approx 150 l/h at 170 bar/350°C
 Max pump capacity at 170 bar = 200 l/h

30	1	Ball valve	WHITEY
29	1	Overflow valve	AKA
28	1	Pre-heater	0176332
27	6	Rotameter	Fischer & Porter
26	7	fine adjustment valve	WHITEY
25	1	Magnetic valve	ASCO 8314 B35
24	1	Regulated ball valve	WHITEY
23	4	MP filter	xx450709
22	7	Pressure reduction valve	Teschm 44-10626
21	6	Valve	Gachot
20	6	Manometer	WIKKA 0-b bar
19	4	Valve	Gachot
18	4	Irradiated specimens	
17	1	Piston pump	Normadas D11 Bran & Lubbe
16	1	Pressure reduction valve	NAF 380071
15	4	Relay	NAF 350050
14	1	Level regulator	NAF 370630
13	1	Pressure vessel	0176246
12	1	Pressure regulator	WIKKA 232.160/83.21
11	1	Pressure gauge	NAF 374250
10	1	Magnetic valve	352 ABA 2150 Skinner
9	2	Cooling circuit, approx 9 m	176994
8	1	Cooler	Maskinverken
7	6	Cooling circuit, approx 3 m	276022
6	3	Cooler	Maskinverken
5	1	Safety valve	CROSBY JMB-S-A
4	4	Lead shielding	176992
3	1	Water distribution	481714
2	1	Dosage pump	Prominent Elektronik
1	1	O ₂ -water container	A0503 3.24 l/h 145556

Figure B.1

Overall drawing of the circuit.

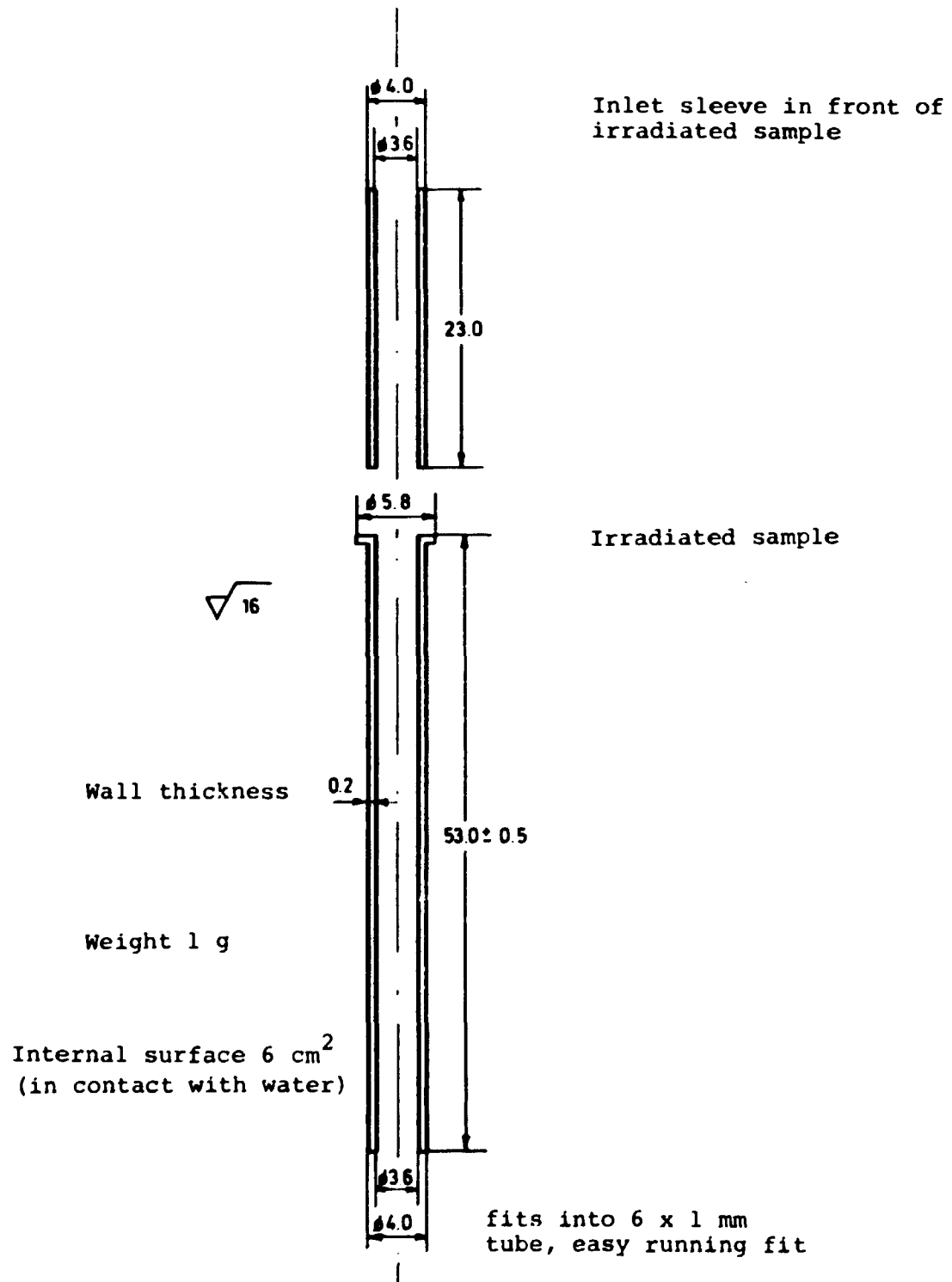
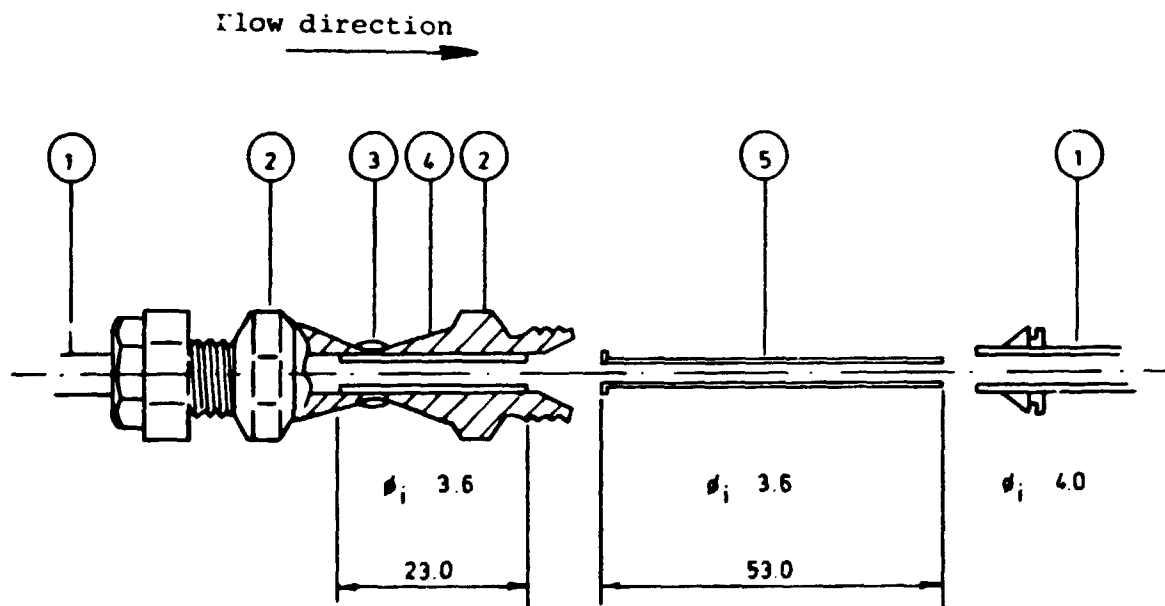


Figure B.2

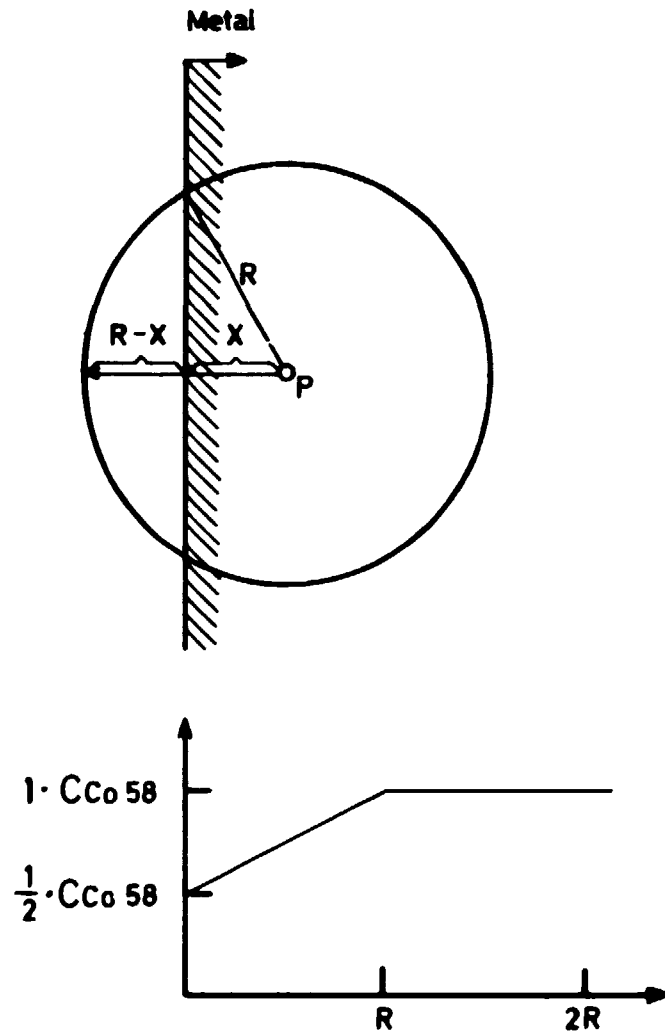
Sample plus adjacent inlet sleeve.



1. Tube 6 x 1 SIS 2343
2. Coupling Swagelok SS-6MO-1-4
3. Weld
4. Sleeve ϕ_i 3.6 mm
5. Irradiated specimen

Figure B.3

Sample assembly.



$$C_{Co^{58}} \text{ in } p = \frac{4\pi R^2 - 2\pi R(R-x)}{4\pi R^2} = \frac{R+x}{2R} \quad (0 < x \leq R)$$

Figure B.4

Diagram to illustrate Co^{58} concentration at surface of samples.



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