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Formation and behaviour of organic iodine

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Abstract

The report presents experimental studies on the formation of organic iodine in severe reactor accidents. The analyses were performed to evaluate the amount of alkaline chemical needed for effective pH control of containment water during the accidents. The formation of organic iodine in solutions used in the filtered venting system and the absorption of iodine compounds in the solutions were studied. Experiments for the formation of organic iodine on painted surfaces were also performed.

Keywords

Organic iodine, filtered venting system, pH control, methyl iodide

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1. Introduction

Iodine is one of the most important fission products released in a nuclear reactor accident. The main reason for this is that a significant fraction of iodine may exist in a volatile form. The isotope ^{131}I has an 8 days half-life, short enough to have a high specific activity and long enough to persist for significant time after the accident. The biological activity of iodine increases the health hazard as it concentrates in the human thyroid.

The iodine released from the fuel is commonly assumed to be in the form of CsI, which dissolves into water as iodide. In acidic conditions all iodine in the water is oxidised to volatile I_2 . Elemental iodine also reacts with low molecular weight hydrocarbons forming volatile organic iodides.

The volatile elemental iodine is relatively easy to trap with filters, but low-molecular weight organic iodides are more difficult to absorb in filters and may therefore be more prone to be released to the environment. This makes the prevention of the formation of organic iodine an important part of the severe accident management.

In BWR containment, cables with high amount of organic insulator material are situated below the pressure vessel. The pyrolysis of this material will release HCl into the containment water thus decreasing the pH of the water /1/. Without control, the pH of containment water can be as low as 2 a couple of hours after the accident and stay low. A high amount of volatile organic compounds is also released in the pyrolysis and will react with elemental iodine. When pH of the water is kept above 7, the formation of I_2 is decreased and also the formation of organic forms of iodine is slow, but it is nevertheless the main component of the released iodine /2/.

In the present work the thermal hydraulic conditions in the containment during severe accident were calculated with MELCOR 1.8.3 and the pH behaviour of the containment lower dry well was simulated with *ChemSheet*, which is a novel thermochemical multi-phase routine, operating in Excel spreadsheets. In *ChemSheet* the chemical equilibriums are solved with Gibbs energy minimisation and all intensive and extensive

thermodynamic properties of the given chemical processes can be calculated. Several models for non-ideal solutions, among them the Pitzer model for concentrated aqueous solutions can be used. Thus, ChemSheet is appropriate for solving aqueous pH-chemistry in multi-component solutions respective for the accident scenario. The calculations were made to evaluate the amount of NaOH needed to keep the containment $\text{pH} > 7$ as a part of severe accident management. NaOH is supplied to the containment by the containment water filling system.

The formation of organic and elemental iodine is also possible in the filter solution through which the fission products are vented in case the venting system is operated. Experiments were performed in this study to evaluate iodine volatilisation in the filter solutions used in Olkiluoto NPP.

The second source for organic iodine in a case of severe reactor accident is the reaction of elemental iodine with painted surfaces. In this study, experiments were performed to study this formation using paints and water composition typical for Loviisa NPP.

2. Behaviour of containment pH in severe accident

2.1 ChemSheet simulations

ChemSheet is a novel simulation technique, which can be used in the Microsoft Excel ® spreadsheet software /3/. Thus ChemSheet offers a non-programming environment for repetitive complex equilibrium calculations and even for simulation of time-dependent processes. The evaluation of the simulation data can straightforwardly be done with the versatile mathematical and graphical tools of the commercial spreadsheet program.

pH calculations were performed to find out the amount of 50 % solution of NaOH needed to keep the pH >7 during an accident in Olkiluoto 1 and 2. The filling of lower drywell with water as well as the temperature and pressure were calculated by MELCOR. The filling of the containment starts about one hour after the station black out, which was chosen as a design-basis scenario. In this scenario the lower drywell is flooded from wetwell starting about 30 min after the start of accident and cannot be performed by using alkaline solution. The scenario included a very early release through the filtered venting system, 1h 37 min after the total loss of power.

2.2 HCl release

When the pH control in the containment is calculated, the most important factor is the amount of chlorine released in the pyrolysis of the cable insulators. This release is thought to be in the form of HCl, which is dissolved into the containment water and decreasing the pH. In a severe accident including the reactor vessel rupture the molten core flows on the cables. The total amount of chlorine in cable insulators is 470 kg. In the hypothetical release all the chlorine of the cable insulators in the lower drywell was estimated to be released in the form of HCl in 10 minutes in the pyrolysis and in addition to that 25 % of the chlorine of the insulators in upper drywell within next 24h due to radiolysis.

Conservative release was estimated on the base of the results of previous studies /1/. In these studies the highest release 20% of Cl was reached in the case of fast pyrolysis when the insulator material was cut into small, a few mm³, pieces. This amount was released in about 10 min. In these experiments there was no noticeable difference between 300°C and 500°C. When heating was continued 24 h, a few more percents of chlorine were released. In higher temperatures 800°C and 1000°C 50-60 % of chlorine released was in the form of high molecular weight organic compounds, which do not decrease the pH of the water same way as HCl. According to these experiments the conservative release of HCl was estimated to be 25% of total chlorine in 10 min and 25% during next 24 h in pyrolysis. The radiolytic release of HCl from the upper drywell was estimated to be 25 % in 24 h.

2.3 Results of the pH simulation

In the following figures are presented the results of pH behaviour calculations in dry well by using different HCl release and various amounts of 50% NaOH-solution. The marked points in the graphs are the following:

1. Flooding of lower drywell
2. Containment water filling starts
3. Vessel failure
4. Containment filtered venting starts

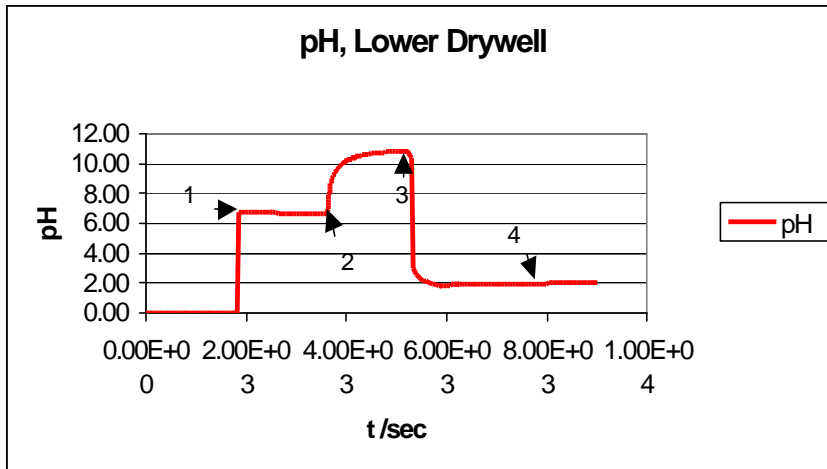


Figure 1. Lower drywell water pH, hypothetical HCL release, 1.6 m^3 50% NaOH solution.

In the first case the impact of 1.6 m^3 50 % NaOH-solution added into spray water was calculated (Figure 1). 1.6 m^3 of NaOH-solution cannot keep the pH of dry well high before the release through scrubber filter in the case of hypothetical release. In the case of conservative release of HCl the pH in lower dry well decreases below 4 after the ex vessel release, but is increased above 9 before the release through scrubber filter (Figure 2).

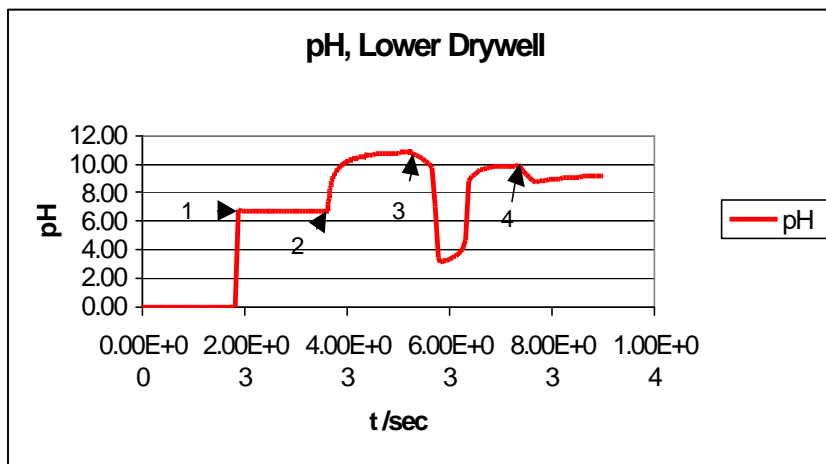


Figure 2. Lower drywell water pH, conservative HCL release, 1.6 m^3 50% NaOH solution.

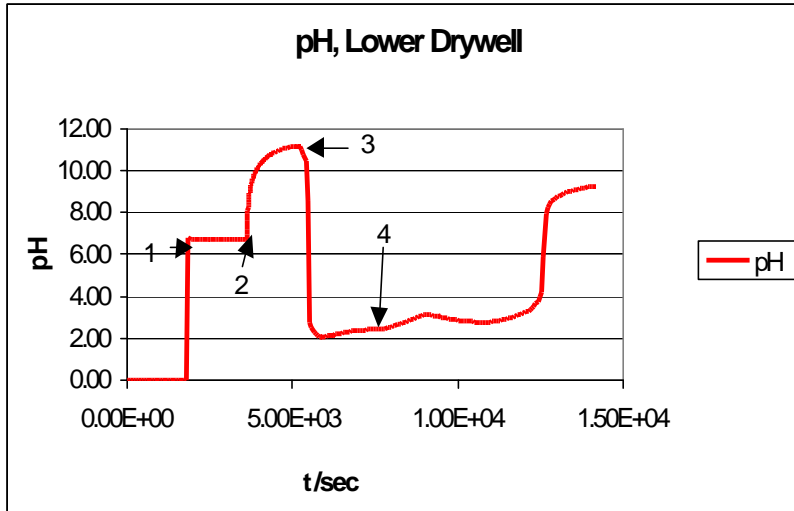


Figure 3. Lower drywell water pH, hypothetical HCL release, 4.8 m^3 50% NaOH solution.

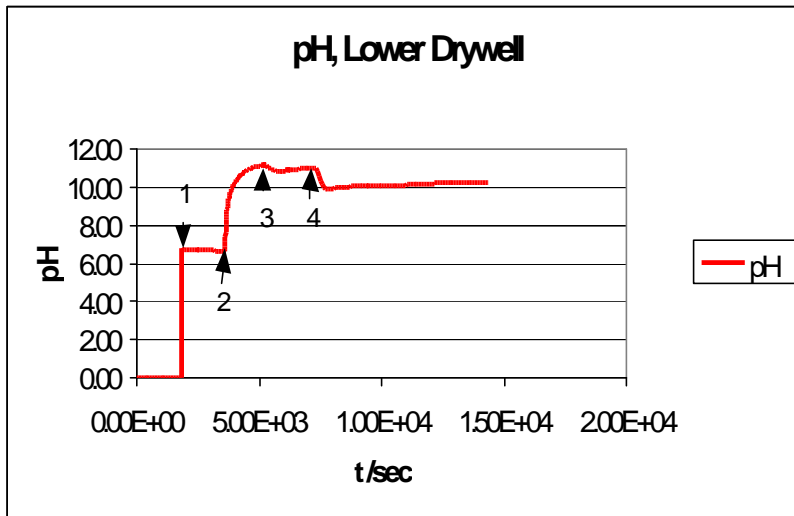


Figure 4. Lower drywell water pH, conservative HCL release, 4.8 m^3 50% NaOH solution.

When the volume of NaOH-solution is increased to 4.8 m³, in the case of hypothetical HCl release pH drops below 4 after the ex vessel failure and does not increased above 7 until 1.5 h after the release through the filter (Figure 3). In the case of conservative HCl release pH stays above 10 after the ex-vessel release and even after the release through scrubber filter (Figure 4).

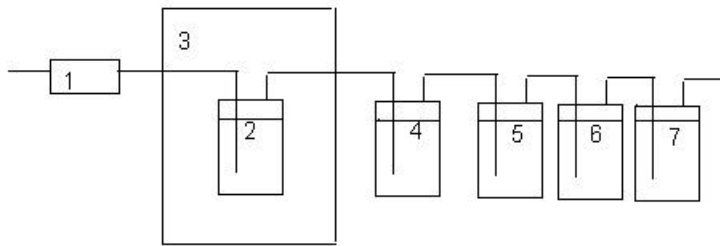
3. Formation of organic iodine in scrubber filter

The radiation field in the scrubber filter is rather high after the ventilation of the containment in the severe accident management. The iodine comes into the filter solution in aerosol form as CsI, as I₂ or as organic compound. The CsI and elemental iodine dissolve into the water phase as I and may form volatile iodine, because of the radiolysis. In this work the experiments were performed to study the volatility of inorganic iodine and formation of organic iodides in the filter solutions in the presence of methane in water solution with high pH and in the solutions used in scrubber filters.

3.1 Experimental

The apparatus used for bubbler experiments is presented in Figures 5-7. It consists of a Gammacell irradiation facility having a dose rate of 0.63 kGy h⁻¹ calibrated using Fricke dosimetry. The reactor vessel was a glass bottle and the sample gas was bubbled through the solution with a flow rate of 6l/h 0.27 mmol CH₄/h. The amount of water was 500 ml in these experiments. The iodine was added into the solution as CsI and ¹³¹I was used as a radioactive tracer.

The gas mixture used in these experiments was 1 % CH₄ in N₂. The sample solutions were bubbled at least one hour with nitrogen before the start of irradiation.



- 1 Flow meter
- 2 Reactor vessel
- 3 Irradiation facility
- 4 NaOH-solution (2g/l)
- 5, 6, 7 Ethanol

Figure 5. Schematic diagram of bubbler tests.



Figure 6. Reaction vessel.



Figure 7. Iodine traps, NaOH and ethanol in bubblers.

Figure 6 shows the reaction vessel, 500 ml of reaction solution and gas flowing through the solution. Figure 7 shows the traps used to absorb different forms of iodine into solution. The amount of solution in each trap was 200 ml.

Inorganic iodine was trapped by a 2g/l solution of NaOH. To validate the efficiency of the trap pure nitrogen gas was used in one experiment instead of methane gas mixture as a sparger gas. The results showed that all iodine released from the solution was retained in the NaOH trap. However, the previous experiments showed that one ethanol trap was not sufficient for retaining all organic iodides in long experiments. Consequently, three traps were installed in a row to guarantee that no organic iodides were lost from the system. This was confirmed by calculating the activity balance in each experiment. The activity of the first ethanol trap was followed by on-line gamma measurement. The amount of released organic iodide and elemental iodine was calculated by measuring the ^{131}I in various traps and the sample solution after the experiment. The amount of radioactive tracer used in these experiments was so high that the detection limit $<0.00002\%$ of total iodine in a trap was measured.

The experiments performed focused on the formation of organic iodine in solutions with high pH. These were made to study the organic iodine formation in scrubber filters. By bubbling a gas mixture (N_2 99% and CH_4 1%) through a CsI-solution in a gamma radiation field, the formation of organic iodides was monitored.

The concentration of iodine in the experiments in water was 4.5×10^{-5} M. The tracer of the sample solution amounted to 100 kBq/ml. The NaOH -solution was used to adjust the pH of the solution.

Experiments were also made in a solution corresponding to the scrubber filters. The previous filter solution contained 5g/l NaOH and 2g/l $\text{Na}_2\text{S}_2\text{O}_3$. In the solution now in use, the amount of $\text{Na}_2\text{S}_2\text{O}_3$ was increased to 35g/l.

The effect of chlorine to the formation of organic iodine in filter solution was studied by making the experiments in solutions containing different amounts of chlorine. Chlorine

was added to the solutions as HCl, and the concentration of the solutions was 1, 0.1 and 0.01 M. The precipitation of sulphur was noticed in 1M and 0.1M HCl.

The temperature in all experiments was 80°C.

In one experiment in water at pH 9 the gas mixture was circulated. (Experiment 3) The gas was returned to the reactor vessel after the traps. The total amount of gas was 300 ml containing 0.0134 mmol CH₄.

3.2 Results

The results of the tests performed at pH 9 are presented in Table 1. According to these results at pH 9, the formation of organic forms of iodine is slow. The fraction of methane used to form methyl iodide is only 2×10^{-4} %/h. The circulation of the gas had no effect on the fraction of CH₄ /h used to form methyl iodide. These results indicate that the fraction of methane reacting with iodine in water will increase if the delay time in the water is increased.

Table 1. Formation of organic iodine in bubbler tests at pH 9.

Experiment	1	2	3
	4.65h	6.1h	4.2h
Formation of organic iodine (% of total iodine)	0.00592	0.00787	0.00022
Formation of organic iodine (%/h)	0.0013	0.0013	0.000053
Formation of organic iodine total, 10 ⁻¹⁰ mol	26.6	35.4	0.99
Formation of organic iodine, 10 ⁻¹⁰ mol/h	5.72	5.81	0.239
Fraction of CH ₄ used to form CH ₃ I (% x 10 ⁻⁴)/h	2.13	2.17	1.8

The results of experiments made in a solution corresponding to the solutions used in scrubber filter are presented in Table 2. The previous filter solution contained 5g/l NaOH and 2 g/l Na₂S₂O₃ and formation of elemental and organic iodine was detected. Increasing the amount of Na₂S₂O₃ to 35g/l brought the organic iodine formation below

the detection limit or very near it. Also the amount of elemental iodine was decreased. The effect of chlorine concentration of the solution had no noticeable effect on the formation of organic iodine.

Table 2. Organic iodine formation, scrubber filter tests.

$\text{Na}_2\text{S}_2\text{O}_3$	[Cl^-]	% of I as I_2/h	% of I as CH_3I
2 g/l	-	0.005	0.0027
35 g/l	-	0.00007	0.00004
35 g/l	0.01 M	0.0002	<0.00002
35 g/l	0.1 M	0.0001	0.00002
35 g/l	1.0 M	<0.00002	<0.00002

4. Absorption of methyl iodine into scrubber filter

The previous studies /2/ indicated that volatile organic iodine compounds might be formed in larger quantities than previously assumed. However, the effectiveness of the new solution with high concentration of $\text{Na}_2\text{S}_2\text{O}_3$ to trap organic iodine compounds has not been experimentally tested.

In severe accidents methane is formed in reaction of boron carbide from the control rods with steam as well as in the pyrolysis of the cable insulators. In pyrolysis also ethane and small amounts of other hydrocarbons are formed. Organic compounds can react with iodine in radiation field forming volatile organic iodides. The amount of other organic iodides than methyl- or ethyl-iodide is small and long carbon chains are also split in the radiation field forming mostly methyl- and ethyl-iodides. Methyl- and ethyl-iodides are volatile and water insoluble, even at high pH. Thiosulphate-ion in basic solution is known to improve the absorption of these compounds in water solutions.

4.1 Experimental

The Gammacell ^{60}Co gamma source was used in the experiments for the production of methyl-iodide. The experimental set up was similar with the one used in chapter 3. Nitrogen gas containing 1% CH_4 was lead through a CsI solution with ^{131}I as radioactive tracer in radiation field of 0.6 kGy/h. In the radiation field in acidic solution CH_3I was formed. The gas mixture went then through a NaOH solution to trap the elemental iodine. To control that elemental iodine did not pass the trap, a second trap with NaOH- solution was added. No ^{131}I was found in this solution showing that only methyl-iodide passed the NaOH trap.

After NaOH -solution the gas mixture containing N_2 , CH_4 and CH_3I was bubbled through the filter solution (5g/l NaOH + 35g/l $\text{Na}_2\text{S}_2\text{O}_3$) and finally through two ethanol traps to absorb the methyl-iodide escaping from the filter solution. ^{131}I was used as a radioactive tracer in the CsI solution and the absorption was calculated by comparing the ^{131}I activities in different solutions.

Parallel experiments were performed in radiation field and without radiation to study the effects of radiation to the absorption of methyl-iodide. There was no difference between these two experiments. The breaking of methyl-iodide in radiation is probably so slow reaction, that the delay-time of the gas mixture in the filter solution is too short for it. Basically it could improve the methyl-iodide absorption by breaking it to methane and elemental iodine. In this form iodine is easily trapped in filter solution.

TVO delivered for the experiments MELCOR calculations of the conditions in the filter during the release. These calculations were used to plan the experimental conditions. The flow rate of the gas was not as fast as in accident, but the distance which gas mixture flows in the filter solution is shorter in the experiments. The experiments were made using three different gas flow rates and in two different temperatures. The height of the absorption vessel was normally 9 cm. One experiment was performed using 90 cm vessel for the filter solution to test the influence of the delay time of the gas mixture in the filter solution.

4.2 Results of the methyl-iodide absorption tests

Table 3. The results of the absorption tests.

Gas flow l/h	Temperature °C	CH ₃ I in filter solution % of total CH ₃
6	23	60 ± 5
1	23	68 ± 3
10	23	61 ± 5
6	80	65 ± 5
10	23	77*

* High absorption vessel (90 cm)

5. Organic iodine production on painted surfaces

5.1 Experimental

The formation of organic iodine on painted surfaces in the radiation field was studied by irradiating painted concrete blocks in glass bottles (Figure 8). The block was partly in water having the same chemical composition as the sump water in Loviisa reactors. The water contained 2.9 g boric acid, 6.7 g sodium metaborate, 0.5 mg NH₃, 2.2 mg KOH, 0.2 g HCl and 5.6 mg iodine per kg. In the experiments the gas phase/water ratio was 42, approximately same as Loviisa reactors, 25-50. Iodine inventory of Loviisa reactors is 7.3 kg giving the I concentration 3.6 mg/kg if all iodine is released and water volume is 2000m³. The dry/wet surface ratio in Loviisa is 0.2 and in our experiment 2.4.

The total volume of the reaction vessel was 215 cm³ and the volume of sump water 5 cm³. The block was 3 cm x 3 cm x 2 cm, and the dry/wet surface ratio 2.42. 10MBq of ¹³¹I was used as radioactive tracer in each experiment.

Gammacell ⁶⁰Co source was used to irradiate the samples during the experiments. The radiation dose was 0.63 kGy/h. The temperature in the experiments was 25 °C

The production of organic iodides was measured by leading a slow flow of N₂, 0.1 l/h through the bottle and trapping the inorganic iodine into a NaOH-solution and organic iodides into ethanol. The irradiation times in the experiments were between 19.5 and 166 hours corresponding to the radiation doses of 12.3 and 105 kGy. In one experiment air was used instead of nitrogen. The activities of trap solutions were measured and the production of elemental iodine and organic iodides was calculated.

5.2 Results

The results of the tests are presented in the Tables 4 and 5.

Table 4. Volatile iodine formation on painted surfaces. * Experiment with air flow.

Dose kGy	% of iodine released as organic compound	% of iodine released as organic compound/kGy	% of iodine released as I ₂	% of iodine released as I ₂ /kGy
14	0.012	0.00086	0.021	0.0015
57	0.023	0.00040	0.039	0.00068
88	0.03	0.00034	0.054	0.00061
104	0.11	0.00106	0.33	0.0032
27*	0.019	0.00070	0.17	0.0063

The formation seems to be faster at the beginning of irradiation. The painted surfaces were washed after irradiation. The surface, which had been in the water, was first washed with NaOH-solution (2 g/l) and then with ethanol. The surface in the gas face during the irradiation was treated in a similar way. The ¹³¹I activity of these solutions was counted and the amount of iodine in different fractions was calculated. The results of these tests are presented in Table 5. Most of the iodine was in the sump water after the experiment, 70-90 % of the original amount. The amount of iodine on the surface is difficult to measure precisely because of the inhomogeneous sample geometry. The values in Table 5 are maximum values. The production rate of organic iodine in these experiments is on the same level as in the experiments with similar dose rate and total radiation dose reported by Belval-Haltier and Taylor /4/

Table 5. Fraction of Iodine found on the painted surfaces after the irradiation.

* Experiment with air flow.

Dose kGy	% of iodine, wet surface washed with ethanol	% of iodine, wet surface washed with NaOH	% of iodine, dry surface washed with ethanol	% of iodine, dry surface washed with NaOH	% of iodine left on the surfaces (after washings)
14	0.53	2.3	0.3	0.4	17
57	0.12	0.8	0.3	0.2	18
88	0.83	6.2	0.5	0.2	28
104	0.73	2.7	0.7	1.2	23
27*	0.32	1.4	0.3	0.2	10

According to the results wet surface is trapping several times more inorganic iodine per cm^2 than the dry surface. Also organic iodine is enriched on the wet surface but the difference is not as clear as in the case with inorganic iodine.

Fraction of volatile iodine after 100 kGy irradiation is 0.33 % of total iodine. In case of Loviisa this would result in gaseous iodine concentrations of 0.6 mg/m^3 . The increase of temperature can somewhat increase the amount of volatile iodine [5]. However, such direct extrapolations are not possible, since the experimental conditions do not replicate the containment geometry correctly. In particular, the surface/volume ratios are in the experiment much smaller than in the containment. The dry surface/total volume ratio in the experiment was $1.4 \times 10^{-3} \text{ m}^{-1}$, while in containment it is 0.42 m^{-1} . The wet surface/total volume in the experiment was $0.57 \times 10^{-3} \text{ m}^{-1}$, while in containment it is 0.083 m^{-1} . Consequently the adsorption of iodine on the painted surfaces in the containment has larger impact on iodine volatility and therefore the experimental results may be treated as upper values.

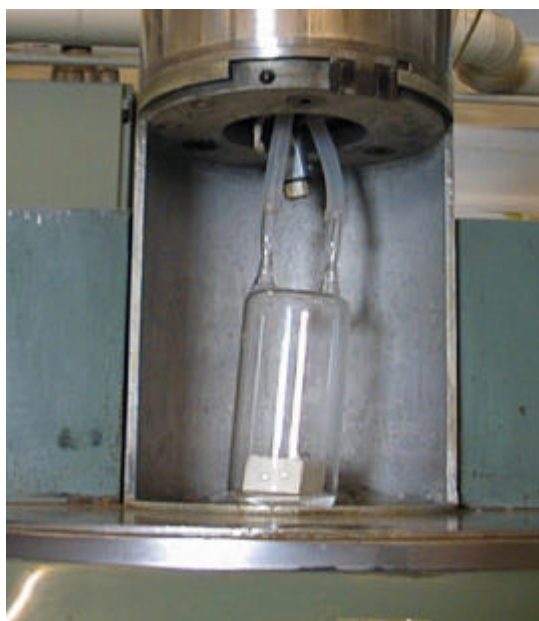


Figure 8. Formation of organic iodine in radiation field on painted surfaces. Experiment vessel.

6. Conclusions

Iodine behaviour in severe reactor accidents is strongly dependent on the containment water pH. In particular this is true for boiling water reactor plants, because in all severe accidents not involving primary system break all releases from reactor are vented into the containment through a suppression pool. Depending on water pH, the pool can act as a sink or source of volatile iodine.

The analysis performed as a part of this study showed that relatively large quantities of alkaline chemicals are needed to counter balance the effect of acids released during the accidents, such as HCl. The analysis also indicated that such quantities could be introduced into the containment in timely fashion. Consequently, such a system has been designed and installed at Olkiluoto NPP in 2001. The reduction of iodine releases has been striven for also by using a modified solution in the containment venting system filter. This solution, which was originally selected for greater tolerance against HCl releases during accidents, was shown to be very effective also in reducing iodine volatility in the filter. The new filter solution with increased sodium- thiosulphate concentration makes the formation of organic iodine in filter negligible and absorbs the inorganic iodine effectively. However, the now tested solution has only limited methyl iodine retention capability underlining the need for prevention of organic iodine formation in the containment with all tools available, including efficient pH control.

According to the experiments in this study, the absorption of organic iodine in the filter is improved by increasing the temperature and delay time of the gases in the solution. The ten times higher column containing the filter solution improved the absorption from 61 to 77 %, when the speed of the gas flow was the same. Decreased gas flow also improved the methyl-iodide absorption. After the improvements in the filter solution a notable part of organic iodine is still passing the filter and released to the environment.

The experiments with Loviisa paints indicated that the paints do not significantly differ with respect to their impact on iodine behaviour from similar type of paints used in other countries. The experimental conditions did not correspond to real accident conditions in all respects, for example temperature and surface/volume ratio were lower

than in real case. Therefore the results cannot be directly extrapolated to accident conditions, but instead can be used as a part of modelling development.

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Abstract	The report presents experimental studies on the formation of organic iodine in severe reactor accidents. The analyses were performed to evaluate the amount of alkaline chemical needed for effective pH control of containment water during the accidents. The formation of organic iodine in solutions used in the filtered venting system and the absorption of iodine compounds in the solutions were studied. Experiments for the formation of organic iodine on painted surfaces were also performed.
Key words	Organic iodine, filtered venting system, pH control, methyl iodide