Report No. 42

RESULTS FROM THE MULTI-POLLUTANT PROGRAMME
CORROSION ATTACK ON CARBON STEEL AFTER 1, 2 and 4 YEARS OF EXPOSURE
(1997-2001)

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July 2003

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Summary

This report details the experimental method and gives results of evaluation of carbon steel corrosion after exposure in unsheltered and sheltered position at 30 test sites for 1 year, 2 years and 4 years in period 1997-2001 in frame of the UN ECE ICP Effects on Materials. The report summarises the results of evaluation of corrosion gains, corrosion losses and evaluation of selected corrosion active water soluble compounds in rust layers. The preliminary results of 1 year and 2 years’ exposure had been presented in Report No 35.

The results for corrosion mass loss for 1, 2 and 4 years exposure are presented in Table 12-13 and below the mean values.

Mean and limited values of corrosion loss of carbon steel in period 1997-2001 (g.m⁻²):

<table>
<thead>
<tr>
<th></th>
<th>1 year</th>
<th>2 years</th>
<th>4 years</th>
</tr>
</thead>
<tbody>
<tr>
<td>open exposure</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mean</td>
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<td>210</td>
<td>309</td>
</tr>
<tr>
<td>Min.</td>
<td>53</td>
<td>82</td>
<td>147</td>
</tr>
<tr>
<td>Max</td>
<td>324</td>
<td>462</td>
<td>677</td>
</tr>
<tr>
<td>shelter exposure</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td>50</td>
<td>81</td>
<td>138</td>
</tr>
<tr>
<td>Min.</td>
<td>13</td>
<td>27</td>
<td>53</td>
</tr>
<tr>
<td>Max</td>
<td>103</td>
<td>176</td>
<td>303</td>
</tr>
</tbody>
</table>

In the first phase of programme carbon steel samples had been exposed only for 1 year period so there are no data to compare. This situation made difficult to perform a systematic validation of previous formulated D/R function.

Analysis of corrosion active compounds of rust layer is significant information of interaction between environment and corroded material. Rust water extract pH values are not characteristic parameter for evaluation of rust layers properties from different environmental exposures. The conductivity of rust water extract is much significantly affected by environmental conditions. The most concentrated soluble ions in adherent rust layer both in open and shelter exposure are sulphates. The second significant ion in rust layer is chloride. The concentration is higher for shelter exposure in all cases. The concentration of nitrate in adherent rust layer both in shelter and open exposure was non-significant.
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Introduction

The UN ECE International Co-operative Programme was performed to a quantitative evaluation of the effect of sulphur pollutants in combination with NO\textsubscript{x} and other pollutants as well as climatic parameters on the atmospheric corrosion of important materials. The programme was based on an international field exposure on 39 test-sites in a wide geographical zone of the 13 Signatories of the Convention on Long-Range Transboundary Air Pollution. The basic field exposure was performed in 1987-1995.

Within 1985 - 1995 the environmental situation have been changed. The SO\textsubscript{2} concentrations significantly decreased in many European countries and the pollution situation changed to multipollutant. This phase of exposure programme (1997-2001) is focused on the possible synergetic effects of NO\textsubscript{x}, SO\textsubscript{2} and O\textsubscript{3} on a range of materials including carbon steel, zinc, copper, bronze, painted steel, glass and calcareous stone. The following list names the responsible sub-centres of this programme:

- Swedish Corrosion Institute, Stockholm, Sweden (main centre)
- SVUOM Ltd., Prague, Czech republic
- Swiss Federal Laboratories for Material Testing and Research, Dübendorf, Switzerland
- Bavarian State Department of Historical Monuments, Munich, Germany
- Norwegian Institute for Air Research
- Building Research Establishment, Garston Watford, United Kingdom
- Institute for Chemistry, Academy of Fine Art, Vienna, Austria

SVUOM has been subcentre responsible for exposure and evaluation of samples of carbon steel in the frame of programme. The corrosion of unalloyed carbon steel is influenced to an high extent by environmental conditions mainly by acidifying airborne pollutants. Other results of evaluation of degradation of other materials exposed in this programme are reported by responsible sub-centres [1 - 6].

1. Methods

1.1. Characterisation of samples

Plates of unalloyed carbon steel (with C < 0.2 %, P < 0.07 %, Cr < 0.07 % according to CSN 11373) with dimensions 100 x 150 x 0.5 mm were used, triplicate per exposure period.

Before the exposure the samples were degreased in alkaline degreaser, rinsed with ethanol, dried and weighed.

1.2. Exposure of samples

Methods prescribed in the Technical manual to the UN ECE International Co-operative Programme on Effects on Materials including Historic and Cultural Monuments [7] were applied for exposure of samples on test sites. Samples were exposed on racks in open atmosphere (unsheltered exposure) according to ISO 8565 and at special ventilated shelters which prevented rainfall reaching the samples. For each exposure there had been used triplicate samples.
The list of test sites for the multi pollutant programme is in Table 1 and Figure 1. Exposure period has been 1, 2 and 4 years (1997-2001).

Table 1 - The list of test sites

<table>
<thead>
<tr>
<th>Country</th>
<th>Test site</th>
<th>Type of atmosphere</th>
</tr>
</thead>
<tbody>
<tr>
<td>the Czech Republic</td>
<td>1 Prague</td>
<td>urban-industrial</td>
</tr>
<tr>
<td></td>
<td>3 Kopisty</td>
<td>industrial</td>
</tr>
<tr>
<td>Finland</td>
<td>5 Ahtari</td>
<td>rural</td>
</tr>
<tr>
<td>the Federal Republic of Germany</td>
<td>7 Waldhof-Langenbrugge</td>
<td>rural</td>
</tr>
<tr>
<td></td>
<td>9 Langenfeld</td>
<td>rural</td>
</tr>
<tr>
<td></td>
<td>10 Bottrop</td>
<td>industrial</td>
</tr>
<tr>
<td></td>
<td>41 Berlin</td>
<td>urban</td>
</tr>
<tr>
<td>Italy</td>
<td>13 Rome</td>
<td>urban</td>
</tr>
<tr>
<td></td>
<td>14 Casaccia</td>
<td>rural</td>
</tr>
<tr>
<td></td>
<td>15 Milan</td>
<td>urban-industrial</td>
</tr>
<tr>
<td></td>
<td>16 Venice</td>
<td>urban</td>
</tr>
<tr>
<td>Norway</td>
<td>21 Oslo</td>
<td>urban</td>
</tr>
<tr>
<td></td>
<td>23 Birkenes</td>
<td>rural</td>
</tr>
<tr>
<td></td>
<td>44 Svanvik</td>
<td>rural</td>
</tr>
<tr>
<td>Sweden</td>
<td>24 Stockholm South</td>
<td>urban</td>
</tr>
<tr>
<td></td>
<td>26 Aspvreten</td>
<td>rural</td>
</tr>
<tr>
<td>the United Kingdom</td>
<td>27 Lincoln Cathedral</td>
<td>urban</td>
</tr>
<tr>
<td></td>
<td>46 London</td>
<td>urban</td>
</tr>
<tr>
<td>Spain</td>
<td>31 Madrid</td>
<td>urban</td>
</tr>
<tr>
<td></td>
<td>33 Toledo</td>
<td>rural</td>
</tr>
<tr>
<td>the Russian Federation</td>
<td>34 Moskow</td>
<td>urban-industrial</td>
</tr>
<tr>
<td>Estonia</td>
<td>35 Lahemaa</td>
<td>rural</td>
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<tr>
<td>Portugal</td>
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<td>urban</td>
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<td>Canada</td>
<td>37 Dorset</td>
<td>rural</td>
</tr>
<tr>
<td>France</td>
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<tr>
<td>Israel</td>
<td>43 Tel Aviv</td>
<td>urban</td>
</tr>
<tr>
<td>Switzerland</td>
<td>45 Chaumont</td>
<td>rural</td>
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<tr>
<td>USA</td>
<td>47 Los Angeles</td>
<td>urban-marine</td>
</tr>
<tr>
<td>Belgium</td>
<td>49 Antverps</td>
<td>urban-marine</td>
</tr>
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</table>
Figure 1 – Map showing location of test sites
1.3. Measurement of environmental data

Basic climatic parameters (temperature, relative humidity and intensity of sunshine radiation), concentration of gaseous pollutants (SO\(_2\), NO\(_x\), O\(_3\)) and precipitation (amount, pH, conductivity, amount of SO\(_4^{2-}\), NO\(_3^-\), Cl\(^-\), and NH\(_4^+\), K\(^+\), Ca\(^{2+}\), Mg\(^{2+}\) if available) were measured on each test site. All data were reported to, and completed by the Norwegian Institute for Air Research - NILU, which also checked the quality of the data [8].

The environmental data collected in the course of the multi-pollutant programme confirms the decreasing concentration of sulphur dioxide in atmosphere.

1.4. Evaluation of corrosion attack

The visual evaluation of layer of corrosion products was done to indicate non uniform corrosion process.

The basic information about corrosion behaviour of carbon steel in different environmental conditions gives the value of corrosion gain and mass loss. After exposure samples were weighed and the corrosion products were removed using pickling method according to ISO 8407 (Annex A - solution C3.5. and C9.5.). In consecutive pickling cycles the corrosion layer was completely removed without dissolving the base metal. The repetition of the procedure stopped if the surface appears clean. The corrosion losses had been obtained by gravimetric method.

For selection samples of carbon steel the amount of water soluble salts in corrosion products was performed by spectrophotometric analysis. There were analysed corrosion products from samples exposed at test sites No 1, 3, 10, 27, 40, 41, 43, 46 and 49, it means test sites with high corrosion losses and test site 33 as the one with low corrosivity.

1.5. Evaluation of corrosion active characteristics of rust layer

Analysis of corrosion active components and evaluation of some other characteristics of rust layers gives an important information on interaction of environment with the steel surface and on expected function of rust in corrosion system including possible protective coatings. Analysis was performed on two rust sub-layers separate.

Non-adherent rust sub-layer had been got by mechanical removal by fine steel brush separately for upper and ground side of exposed samples. The content of active components had been analysed after extraction by deionised water for 24 hours.

The sample with adherent rust layer was immersed in deionised water for 24 hours and the water extract had been analysed. The concentration of soluble ions was determined by spectrophotometric method. The pH value and conductivity were measured too.
2. Results

2.1. Visual evaluation

An illustrative evaluation was done in combination with a digital photo of each sample. The visual evaluation of carbon steel samples after 1 and 2 years of exposure is presented in Report No 35.

2.1.1 Samples exposed at open atmosphere (unsheltered)

The layers of corrosion products of carbon steel had formed a typical structure, colour and thickness according exposure conditions. The surface layer of samples of carbon steel exposed on test site No. 43 had been affected by erosion (sand storm) - Figure 2.

The layer of corrosion products had very firm structure from test site with relatively "dry" climate (test site No 10, No 15, No 21, No 43) and on the other hand very rough structure from test sites with relatively "wet" climate (test site No 16, No 27).

2.1.2 Samples exposed at shelter

After 4 years of exposure there are samples with corrosion layer formed on 80 - 95 % of surface - there are samples from test sites: No 5, No 7, No 21, No 24, No 26, No 31, No 33, No 35 and No 45 - Figure 3.

The samples at test site No 47 had been exposed at open atmosphere only.

2.2. Mass gains of corroded carbon steel

Mass gains of exposed samples represent differences of mass of corrosion products originated at impact of environment and part of them removed by run-off erosion and fall out of non adherent particles and sub-layers. Mass increase of exposed steel samples gives a complex and not well-defined information. Mass changes of exposed samples of carbon steel exposed at open atmosphere and under shelter are summarised in Tables 2 - 5 and Figures 4 and 5.

For exposure at open atmosphere the negative values of corrosion mass gain were found for test sites:

<table>
<thead>
<tr>
<th>exposure</th>
<th>test site No</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 year exposure</td>
<td>43</td>
</tr>
<tr>
<td>2 years exposure</td>
<td>10, 16, 27, 36, 43, 44, 46</td>
</tr>
<tr>
<td>4 years exposure</td>
<td>1, 3, 7, 9, 10, 16 23, 24, 27, 34, 36, 41, 43, 44, 46, 49</td>
</tr>
</tbody>
</table>

For exposure under shelter the negative values of corrosion mass gain were found for test sites:

<table>
<thead>
<tr>
<th>exposure</th>
<th>test site No</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 year exposure</td>
<td>3, 10, 36, 49</td>
</tr>
<tr>
<td>2 years exposure</td>
<td>1, 3, 10, 36, 41, 44, 49</td>
</tr>
<tr>
<td>4 years exposure</td>
<td>1, 3, 10, 15, 27, 34, 36, 40, 41, 43, 44, 46, 49</td>
</tr>
</tbody>
</table>
Figure 2 - Samples from No 43 exposed at open atmosphere

Figure 3 - Samples exposed under shelter - the surface partly covered by corrosion products - examples

No 5  No 21  No 26
No 31  No 35  No 45
Table 2 - Mass gain of carbon steel after 4 years of exposure at open atmosphere

<table>
<thead>
<tr>
<th>Test site</th>
<th>MC$_{57}$ (g)</th>
<th>MC$_{58}$ (g)</th>
<th>MC$_{59}$ (g)</th>
<th>average (g)</th>
<th>MC (g.m$^{-2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Prague</td>
<td>-0.1482</td>
<td>-0.0377</td>
<td>-0.0701</td>
<td>-0.09</td>
<td>-2.84</td>
</tr>
<tr>
<td>3 Kopisty</td>
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<td>-0.6685</td>
<td>-0.7861</td>
<td>-0.86</td>
<td>-28.57</td>
</tr>
<tr>
<td>5 Ahtari</td>
<td>1.0254</td>
<td>0.9996</td>
<td>0.9996</td>
<td>1.01</td>
<td>33.61</td>
</tr>
<tr>
<td>7 Waldhof-Langenbrugge</td>
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<td>-0.3178</td>
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<td>-7.69</td>
</tr>
<tr>
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</tr>
<tr>
<td>13 Rome</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>14 Casaccia</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
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</tr>
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</tr>
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<td>1.3304</td>
<td>1.27</td>
<td>42.33</td>
</tr>
<tr>
<td>33 Toledo</td>
<td>0.9262</td>
<td>0.9796</td>
<td>1.0838</td>
<td>1.00</td>
<td>33.22</td>
</tr>
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<td>-0.094</td>
<td>-0.07</td>
<td>-2.39</td>
</tr>
<tr>
<td>35 Lahemaa</td>
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<td>0.2136</td>
<td>0.34</td>
<td>11.41</td>
</tr>
<tr>
<td>36 Lisbon</td>
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</tr>
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<td>37 Dorset</td>
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<td>2.50</td>
</tr>
<tr>
<td>40 Paris</td>
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</tr>
<tr>
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<td>-10.0860</td>
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<td>-344.40</td>
</tr>
<tr>
<td>44 Svanvik</td>
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</tr>
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<td>-0.8109</td>
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<td>-49.39</td>
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<td>2.6514</td>
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</tr>
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<td>-0.2969</td>
<td>-0.0965</td>
<td>-0.59</td>
<td>-19.70</td>
</tr>
</tbody>
</table>
Table 3 - Comparison of mass gain after 1, 2 and 4 years of exposure at open atmosphere

<table>
<thead>
<tr>
<th>Test site</th>
<th>MC$_1$ (g.m$^{-2}$)</th>
<th>MC$_2$ (g.m$^{-2}$)</th>
<th>MC$_4$ (g.m$^{-2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Prague</td>
<td>55,7</td>
<td>36,3</td>
<td>-2,8</td>
</tr>
<tr>
<td>3 Kopisty</td>
<td>79,7</td>
<td>36,7</td>
<td>-28,6</td>
</tr>
<tr>
<td>5 Ahtari</td>
<td>23,3</td>
<td>31,0</td>
<td>33,6</td>
</tr>
<tr>
<td>7 Waldhof-Langenbrugge</td>
<td>40,0</td>
<td>25,3</td>
<td>-7,7</td>
</tr>
<tr>
<td>9 Langenfeld</td>
<td>23,0</td>
<td>0,7</td>
<td>-41,7</td>
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<td>18,7</td>
<td>-79,0</td>
<td>-185,3</td>
</tr>
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<td>13 Rome</td>
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<td>63,3</td>
<td>-</td>
</tr>
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<td>15 Milan</td>
<td>36,7</td>
<td>36,0</td>
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</tr>
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<td>-42,4</td>
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</tr>
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<td>-7,3</td>
<td>-28,3</td>
</tr>
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Table 4 - Mass gain of carbon steel after 4 years of exposure at shelter

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<th>MC&lt;sub&gt;58&lt;/sub&gt; (g)</th>
<th>MC&lt;sub&gt;59&lt;/sub&gt; (g)</th>
<th>average (g)</th>
<th>MC (g.m&lt;sup&gt;-2&lt;/sup&gt;)</th>
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Table 5 - Comparison of mass gain after 1, 2 and 4 years of exposure at shelter

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<th>MC₂ (g.m⁻²)</th>
<th>MC₄ (g.m⁻²)</th>
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<td>16,3</td>
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<td>-25,3</td>
<td>-71,5</td>
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</table>
Figure 4 - Mass gain of carbon steel after 4 years of exposure

open atmosphere

shelter
Figure 5 - Comparison of mass gain after 1, 2 and 4 years exposure

open atmosphere

shelter
The rust layer is not an homogeneous material. The detailed systematic evaluation of the properties of rust layers is being performed in SVUOM on steel with a range of composition from various atmospheric exposures [9 - 12]. Quantity of rust in the individual underlayers (fall - 1, upper non adherent layer - 2, middle semiadherent layer - 3, under adherent layer - 4) was determined. The part of fall in the complex rust layer after outdoor exposure is very high. An example is given on Figure 6. The quote of fall in the total mass of created rusts is growing with time of exposure and corrosivity of exposure conditions. The part of fall of rust on carbon steel after 5 years of outdoor exposure for upper exposed surface was 50% of all formed rust, for under surface it was even higher.

Figure 6 – Quantity of rust in the individual underlayers
A systematic study of rust layers is laborious and it was not possible to perform it within the ICP programme. Certain information on mass changes in rusts layers gives Figure 5 making possible the relative comparison for rusts from outdoor and shelter exposures. The part of fall is very high on the most aggressive test sites (No 1, 3, 10, 27, 36, 44, 46 and 49). The fall of loosen rust during outdoor exposure is realized periodically in layers and fluent in scales, what is more typical for fall of rust during sheltered exposure. Relative part of non-adherent and adherent rust sub-layers on steel samples after 4 years exposure under shelter is presented in Table 6. The adherent rust layer forms as significant part of total rust layer from ca 75% to ca 95%.

Table 6 – Comparison of non-adherent and adherent parts of rust layer in shelter exposure

<table>
<thead>
<tr>
<th>test site</th>
<th>non-adherent part* (g.m⁻²)</th>
<th>adherent part (g.m⁻²)</th>
<th>non-adherent part (%)</th>
<th>adherent part (%)</th>
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<td>7,2</td>
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Note: part removed from samples after exposure by fine steel brush. This part of rust layer does not include fall of rust during exposure.

2.3. Corrosion losses of carbon steel

Corrosion losses for individual samples of carbon steel exposed in open atmosphere and under shelter evaluated for 4 years’ exposure (1996/2001) are summarised in Table 7 and Table 8 and presented in Figure 7. After 4 years of exposure in open atmosphere in this period test sites with the greatest corrosion losses of samples were No. 43 (Tel Aviv) and No. 10 (Bottrop).
Table 7 - Corrosion losses (g/m$^2$) of carbon steel after 4 years exposure in open atmosphere (period 1997/2001)

<table>
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<tr>
<th>Test site</th>
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Table 8- Corrosion losses (g/m²) of carbon steel after 4 years exposure under shelter (period 1997/2001)

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<th>ML&lt;sub&gt;69&lt;/sub&gt;</th>
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Figure 7 - Corrosion loss of carbon steel exposed 4 years

open atmosphere (unsheltered)

shelter exposure
2.4. Analysis of corrosion products

Analysis of corrosion active compounds of rust layer is significant information of interaction between environment and corroded material. In rust layer the corrosive agents are cumulated in adherent layer.

The two analysis had been performed – for non-adherent and adherent rust sub-layers. The results are in Table 9 - 11 and in Figure 9. The pH value, conductivity and concentration of soluble ions (sulphates, chlorides and nitrates) were evaluated in water extract of corrosion products of selected samples of carbon steel exposed in period 1997/01 (test sites with relatively highest pollution of environment only). The concentration of soluble salts in extracts was determinated by spectrophotometric method.

Rust water extract pH values are not characteristic parameter for evaluation of rust layers properties from different environmental exposures. During extraction, strong changes in pH value occur, pH values obtained by measurement after 24 hours of extraction. Non-adherent rust layer pH values are less acid (5,0 – 6,7 for open exposure and 4,8 – 6,6 for shelter exposure with exception of No. 1) than for adherent rust layer (3,1 – 4,0 for open exposure and 3,8 – 4,7 for shelter exposure).

The conductivity of rust water extract is much significantly affected by environmental conditions. Conductivity of non-adherent rust layer for open exposure ranges from 8 to 33 \( \mu \)S/cm and for shelter exposure from 25 to 60 \( \mu \)S/cm. Conductivity of adherent rust layer for open exposure ranges from 115 to 613 \( \mu \)S/cm and for shelter exposure from 145 to 545 \( \mu \)S/cm. There is one exception – test site No. 1.

The non-soluble part of non-adherent rust layer is about 95 – 96% for open exposure and about 98,5% for shelter exposure. Test sites with the highest soluble fraction in rust layer had been No. 40 and No. 41.

In Tables 10 and 11 the comparison of amount of soluble salts are summarized.

The amount of soluble salts contained in non-adherent rust layer is expressed as percentage in 1 g of rust product. The most concentrated ions in adherent rust layer both in open and shelter exposure is sulphate about 0,40%. The second significant ion in rust layer is chloride about 0,05%. The concentration of nitrate was non-significant about 0,01%. Higher concentration of sulphates and nitrates was found in upper non-adherent rust layer on the contrary to higher concentration of chlorides in adherent rust layer.

The most concentrated soluble ions in adherent rust layer both in open and shelter exposure are sulphates. The amount of sulphate is higher in shelter exposure in majority cases. The highest value was found for test site No 46 – ca 630 mg/m\(^2\). The second significant ion in rust layer is chloride. The concentration is higher for shelter exposure in all cases. The highest value was found for test site No 27 and No 46 – ca 75 mg/m\(^2\). The concentration of nitrate in adherent rust layer both in shelter and open exposure was non-significant – below 20 mg/m\(^2\). Also the concentration of ammonium ion is very low from 0,1 to 3,3 mg/m\(^2\).
Table 9 - Comparison of pH and conductivity of rust layers

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<th>conductivity (µS/cm)</th>
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Table 9 - Continue

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Table 10 - Comparison of soluble ion amount in non-adherent rust layer

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Table 11 - Comparison of soluble ion amount in adherent rust layer

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</table>

Analysis of corrosion active components of rust represents an important information not only on interaction of environment with the metal but can serve for looking on alternatives of surface preparation and choice of effective anticorrosion measures. Summary (complex) characterization of stimulate activity of electrolytes on corroded steel surfaces give evaluation of conductivity and pH of water extracts.

Content of sulphates represents still the dominating component of soluble ions in rust. Content of soluble ions is not in real relation to atmospheric corrosivity. Temperature – humidity complex and/or not measured parameters are very important too.

The real stimulative action of rust layers depends on activity of sulphate or chloride nests. Ions in nests are very stable and may be not satisfactory extracted.
Figure 8 - Content of water soluble ions in corrosion products in non-adherent rust layer

open exposure - upper non-adherent layer

open exposure - ground non-adherent layer

shelter exposure – non-adherent layer
3. Treatment of results

3.1. Comparison of corrosion losses after 1, 2 and 4 years

In Tables 12 and 13 there is a comparison of corrosion losses after 1, 2 and 4 years of exposure in open atmosphere and under shelter. In Tables 14 and 15 and Figure 10 corrosion rate on individual test sites is listed from the highest to the lowest one. There is seen from this that corrosion rate of carbon steel is decreasing during exposure time on all test sites.

Table 12 - Comparison of corrosion losses \((\text{g/m}^2)\) of carbon steel after 1, 2 and 4 years’ exposure in open atmosphere (period 1997/2001)

<table>
<thead>
<tr>
<th>Test site</th>
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Table 13 - Comparison of corrosion losses (g/m²) of carbon steel after 1, 2 and 4 years’ exposure under shelter (period 1997/2001)

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Figure 9 - Comparison of average values of corrosion loss with limit values after 1, 2 and 4 years of exposure

open atmosphere (unsheltered)

shelter exposure
Table 14 - Comparison of corrosion rates (g/m² a) of carbon steel after 1, 2 and 4 years’ exposure in open atmosphere (period 1997/2001)

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</table>

C 2, C 3 .....corrosivity category according to ISO 9223
Table 15 - Comparison of corrosion rates (g/m² a) of carbon steel after 1, 2 and 4 years’ exposure under shelter (period 1997/2001)

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C 2, C 3 …..corrosivity category according to ISO 9223
Figure 10 - Comparison of corrosion losses (g.m$^{-2}$)

**open exposure (unsheltered)**

![Graph showing corrosion losses for open exposure over 4 years, 2 years, and 1 year.](image)

**shelter exposure**

![Graph showing corrosion losses for shelter exposure over 4 years, 2 years, and 1 year.](image)
Figure 11 – Differences between corrosion losses of carbon steel in shelter and open exposure

Average values for 1, 2 and 4 years of exposure

Results after 4 years of exposure for individual test site
3.2. Environmental data

In Tables 16 - 18 there are summarized environmental data for 1, 2 and 4 years of exposure [8]. The pollution of $\text{SO}_2$ had been significantly lower in the second phase of exposure programme than in the first one, but the reduction was smaller than during the first phase of programme. $\text{NO}_x$ had been much more significant gaseous pollutant that others. The amount of $\text{O}_3$ was rather remaining.

Table 16 - Average environmental parameters for exposure period 1997/98

<table>
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<th>Test site</th>
<th>CLIMATE</th>
<th>GASES</th>
<th>PRECIPITATION</th>
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<tbody>
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Table 17 - Average environmental parameters for exposure period 1997/99

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</table>
Conclusion

Evaluation of corrosion attack of carbon steel after 1, 2 and 4 years represents an important step in the ICP on materials activities as such exposure of this material was not included in the first phase of exposure programme. Repeated one-year exposures of carbon steel were realized only in that period.

The evaluation presented in this report implicated:
- visual evaluation of exposed samples,
- evaluation of mass gain of corroded samples,
- evaluation of corrosion losses after 4 years of exposure,
- analysis of corrosion layers,
- comparison of 1, 2 and 4 years corrosion losses.

Data base for later statistical treatment was summarized. Preliminary statistical treatment of corrosion losses of carbon steel after 1 and 2 years exposure had been presented in Report No. 35. Systematic statistical treatment of data including one-year data from the first phase of the ICP Effect on Materials is relatively simple including as parameters $\text{SO}_2$, RH and T only (chlorides and acidity of precipitation have been not found as decisive).

The equation for weathering steel was presented:

$$ML = 34 \ [\text{SO}_2]^{0.33} \ \exp\{0.020 \ \text{RH} + f(T)\}t^{0.33}$$

where $f(T) = 0.059(T-10)$ when $T \leq 10^0\text{C}$, otherwise $f(t) = -0.036(T-10)$.

Treatment of the ISOCORRAG data gives D/R function for carbon steel based on one-year exposure data but the environmental base was not complex enough in this case – TOW, T, $\text{SO}_2$, salinity had been only measured. There were presented two equations for carbon steel:

$$\text{CR} = 0.091[\text{SO}_2]^{0.56} \ \text{TOW}^{0.52}\exp(f_{Fe}(T)) + 0.158[\text{Cl}^{-}]^{0.58} \ \text{TOW}^{0.25}\exp(0.050T),\quad N = 125, \ R^2 = 0.85$$

where $f_{Fe}(T) = 0.103(T-10)$ when $T \leq 10^0\text{C}$, otherwise $f_{Fe}(T) = -0.059(T-10)$.

$$\text{CR} = 1.77[\text{SO}_2]^{0.52} \ \exp(0.020\text{RH})\exp(f_{Fe}(T)) + 0.102[\text{Cl}^{-}]^{0.62} \ \exp(0.033\text{RH} + 0.040T),\quad N = 128, \ R^2 = 0.85$$

where $f_{Fe}(T) = 0.150(T-10)$ when $T \leq 10^0\text{C}$, otherwise $f_{Fe}(T) = -0.054(T-10)$.

Parameters used in ISOCORRAG dose/response functions are listed in Table 19. All parameters are expressed as annual average.
Table 19 – ISOCORRAG environmental parameters

<table>
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<th>Symbol</th>
<th>Description</th>
<th>Interval</th>
<th>Unit</th>
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<td>T</td>
<td>Temperature</td>
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<td>°C</td>
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<tr>
<td>RH</td>
<td>Relative humidity</td>
<td>34 – 93</td>
<td>%</td>
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<tr>
<td>SO$_2$</td>
<td>Sulphur dioxide deposition</td>
<td>0.7 – 150.4</td>
<td>mg.m$^{-2}$.d$^{-1}$</td>
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<tr>
<td>Cl$^-$</td>
<td>Chloride deposition</td>
<td>0.4 – 760.5</td>
<td>mg.m$^{-2}$.d$^{-1}$</td>
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</tbody>
</table>

The statistical treatment performed by SVUOM Ltd. as sub-centre will be concentrated on deeper evaluation of the single and combined environmental effects in relation to analytical data published in this report.

**Acknowledgement**

This exposure programme is the result of co-operation between the organisations listed below. Each was responsible for gathering meteorological and pollution data, and for providing sites for the exposure of materials:

- VTT Manufacturing Technology, Espoo, Finland
- Bayrishes Landesamt fur Denkmalpflege, Munchen, Germany
- ENEA - Environmental Department, Rome, Italy
- NILU - Norwegian Institute for Air Research, Kjeller, Norway
- SCI - Swedish Corrosion Institute, Stockholm, Sweden
- BRE - Building Research Establishment Ltd., Watford, United Kingdom
- Ministerio de Fomento, Direcccion General de la Vivienda, Madrid, Spain
- Institute of Physical Chemistry, Academy of Sciences, Moscow, Russian Federation
- Technical University of Lisbon, Laboratory of Mineralogy, Lisbon, Portugal
- Ministry of the Environment, Canada
- LISA - Université Paris XII, Creteil, France
- Israel Antiquities Authority, Conservation Department, Jerusalem, Israel
- EMPA - Swiss Federal Laboratories for Materials Testing and Research, Laboratory for Metallic Materials, Dubendorf, Switzerland
- Getty Conservation Institute, Museum Service, Los Angeles, USA
- UIA - University of Antwerpen, Department Chemistry, Wilrijk, Belgium

The activity of SVUOM as a sub-centre for carbon steel was granted by Ministry of the Environment of Czech Republic, Department of Air Protection.
Reference

1. Report No 43 Results from the multipollutant programme: Corrosion attack on zinc after 1, 2 and 4 years of exposure (1997-2001), 2003

2. Report No 44 Results from the multipollutant programme: Corrosion attack on copper and bronze after 1, 2 and 4 years of exposure (1997-2001), 2003

3. Report No 45 Results from the multipollutant programme: Corrosion attack on limestone after 1, 2 and 4 years of exposure (1997-2001), 2003

4. Report No 46 Results from the multipollutant programme: Corrosion attack on painted steel after 1, 2 and 4 years of exposure (1997-2001), 2003


6. Report No 48 Results from the multipollutant programme: Evaluation of the decay to glass samples after 1, 2 and 4 years of exposure (1997-2001), 2003


