

## CORROSION BEHAVIOUR OF METALLIC CONTAINERS DURING LONG TERM INTERIM STORAGES

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## ABSTRACT

Two main corrosion phenomena are encountered in long term interim storage conditions: dry oxidation by the air when the temperature of high level nuclear wastes containers is high enough (roughly higher than 100°C) and corrosion phenomena as those encountered in outdoor atmospheric corrosion when the temperature of the container wall is low enough and so condensation is possible on the container walls. Results obtained with dry oxidation in air lead to predict small damages (less than 1µm on steels over 100 years at 100°C) and no drastic changes with pollutants. For atmospheric corrosion, first developments deal with a pragmatic method that gives assessments of the indoor atmospheric corrosivities.

*Keywords: Nuclear waste, storage, atmospheric corrosion, dry oxidation.*

## 1- INTRODUCTION

The corrosion behaviour of metallic materials is generally well predicted for periods of a few tens years, thirty to fifty years. For longer periods of time, one to a few centuries, predictions are less usual and probably more uncertain: laboratory and model studies are needed to validate corrosion rates and mechanisms over these large periods of time, and so to have more confidence in the corrosion behaviour. As long term interim storage of nuclear wastes could last one to several centuries, a specific research and development program has been defined and is conducted on the corrosion behaviour of metallic containers during long term interim storage. This program is called COCON (Corrosion of CONTainers).

The durable functions attributed to containers during long term interim storage are mainly related to the radionuclide tightness and to the handling or the retaking of the containers. To secure these two functions over hundred years standing leads:

- to predict the corrosion behaviour of metallic materials of container walls over that period, i.e. to describe and to determine the corrosion mechanisms and phenomena, to give corrosion rates and to secure the extrapolation methods used for prediction.
- and to define a strategy of corrosion monitoring, as the environment of the interim storage (air) may be open to the outside fluctuations, in order to manage the differences from the predictions.

The COCON research and development program includes also the two main conditions expected today in interim storages:

- For high radioactive elements, like nuclear glasses or spent fuel elements, the temperature of the container walls will be high (100°C to 300°C) at the beginning of the interim storage, high enough to avoid any condensation on these walls. During that period, the corrosion phenomena will be due only to dry oxidation by the cooling air.
- After the decrease of the radioactivity of these high radioactive elements, or during the initial period of the storage of the radioactive wastes with low thermal power, the temperature of the container walls will be nearly the same as the temperature of the cooling air. The thermal inertia of the containers, the fluctuations of air temperature and humidity will probably lead to the condensation of water on the containers. Wet and dry cycles will be possible and the corrosion phenomena will be as those encountered in atmospheric corrosion.

These two main corrosion phenomena (dry oxidation by air and indoor atmospheric corrosion) are investigated in the R&D COCON program. First investigations were performed on steel materials which are widely used as container materials. Results of the bibliographic reviews and of the first experimental and model works are presented in this paper.

## 2- DRY OXIDATION

Dry oxidation is one possible mode of degradation of the container candidate materials at the moderately elevated temperatures (50°C – 300°C) expected for the external container walls. Models are required to predict the corrosion attack over long periods but to have a high reliability, they must be based not only on experimental short term data but also on a mechanistic approach.

Few data on dry oxidation of unalloyed or low carbon steels are available in the literature. Moreover, they mainly concern the first steps of the oxide film build up and have been obtained for very short oxidation periods or low oxygen partial pressures. They must therefore be completed by more relevant data for our study. Two main theories of dry oxidation have been developed with the common approximation that only one of the partial processes involved controls the rate of the whole oxidation process. The Wagner theory [1] for the high temperature oxidation in which the transport of the reactants in the oxide layer is mainly accomplished by thermally activated diffusion via point defects leads to a parabolic law of the oxide layer growth. The Cabrera-Mott theory [2] and the derived ones for low temperatures or thin oxide films considers that the reactant transport is due to the electric field across the oxide film. Various oxide layer growth rates (parabolic, logarithmic or cubic) are deduced from these theories. Even some attempts have been made to develop models taking into account all the partial processes of the oxide growth, lot of developments have to be performed to have such models.

The first objective of the experimental works is to obtain data over a longer period of time than those given in the literature at different temperatures between 100°C and 500°C in order to determine if an extrapolation of these data is possible by using an apparent activation energy. These data are then interpreted using allowable oxidation models and used for long term oxidation prediction. The influence of some pollutants in the atmosphere on the oxidation will also be presented.

### 2-1 Experimental procedure

The experimental procedures are detailed in [3, 4]. The investigated materials were pure iron and a low alloyed steel. The material chemical compositions are given in the Table 1.

Tableau 1: *chemical composition of the investigated materials (weight %)*

	C	Mn	Si	S	P	Ni	Cr	Mo	Cu	Al	Fe	Sn	N
Iron	0,001	0,05	0,01	0,003	0,004	0,019	0,013	0,001	0,005	0,001	bal.	0,002	0,003
Steel	0,403	0,692	0,214	0,0007	0,09	0,014	0,330	0,030	0,005	0,013	bal.		

Oxidation tests have been performed in quartz reactors heated by electrical furnaces for 260 to 305 hour periods at temperatures between 373 K and 673 K. The atmospheres were flowing air with 2% vol. H<sub>2</sub>O (named reference atmosphere), wet air (12% vol. H<sub>2</sub>O) or reference atmosphere with a pollutant 10 vppm HCl or 2 vppm SO<sub>2</sub>. The specimen oxidation has been evaluated by performing weight measurements, observations with optical and electron microscopes and analyses by means of electron microprobe, XRD (X-ray diffraction), RBS (Rutherford backscattering spectroscopy) or XPS (emission of photo electrons), depending on the scale thickness. The oxidation kinetics under air with 2% H<sub>2</sub>O have been also determined by continuously recording the weight gain of a specimen by means of a symmetrical thermobalance

### 2-2 Results in the reference atmosphere

The main results obtained in the reference atmosphere (air with 2%vol. H<sub>2</sub>O) are gathered in the Table 2. The oxide thickness have been determined by XPS or RBS for oxidation temperatures lower than 300°C and deduced from weight gain measurements at 300°C and higher temperatures. The oxides which have been identified by X rays analyses are Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub>, at 300°C or higher, in agreement with the data reported in the literature. As the expected oxidation kinetics at temperatures lower than 300°C are very low and the oxides are roughly the same whatever is the temperature, so the first modelling approach was to work at higher temperatures and to extrapolate the data at lower temperatures. As for most of the literature data about iron oxidation in air or oxygen at temperatures higher than 300°C report that the oxide growth kinetics follows a parabolic law as predicted by the Wagner theory, we have assumed the same type of law for oxide growth kinetics. Therefore, from the following equation:

$$W^2 = K_p t$$

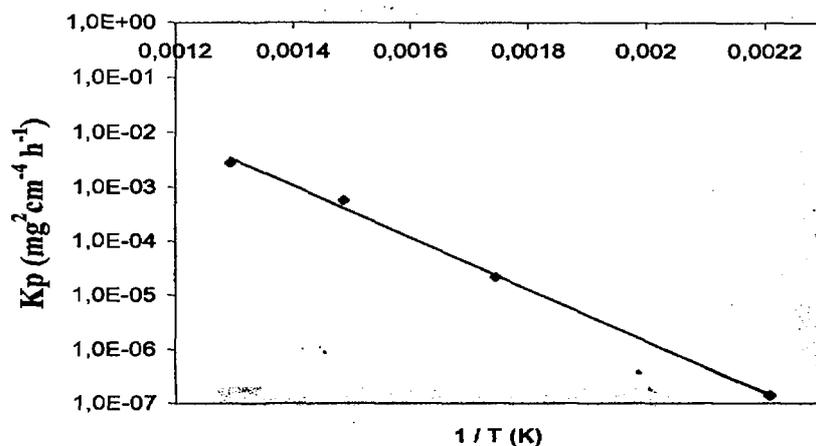
W being the weight gain,  $K_p$  the parabolic constant and t the time, with  $K_p = K_0 \exp(-E_a / RT)$ , where  $K_0$  is a constant and  $E_a$  an apparent activation energy. we have calculated a  $K_p$  value at 300, 400 and 500°C and from an Arrhenius plot we have deduced the  $K_0$  and  $E_a$  values. The Figure 1 shows such a plot. The apparent energy deduced from these data is between 90 and 120 kJ mol<sup>-1</sup> which is in agreement with the 100-150 kJ mol<sup>-1</sup> values from the literature for iron oxidation in dry air or oxygen.

Table 2 : Iron and steel oxide thickness measured after oxidation tests

Temperature	Duration	Oxide thickness	
		Iron	Steel
20°C		55 Å	50 Å
100°C	260 h	135 Å	130 Å
180°C	260 h	385 Å	550 Å
300°C	260 h	0.15 µm	4500 - 10000 Å
400°C	260 h	1,25 µm	3.3 µm

Using the  $K_p$  values extrapolated to lower temperatures, oxide layer thickness of 60 Å and 4 Å for 180°C and 100°C oxidation temperatures respectively are obtained. It is in good agreement with the experimental value obtained at 180°C (80 Å) but lower than the 100°C experimental value (35 Å). From these  $K_p$  data and assuming that the oxidation mechanism and kinetics remain the same during all the considered period, we can expect a 1 µm thick oxide layer on iron after 100 years at 150°C under the reference atmosphere. At 300°C, which is the maximum expected temperature, the calculated oxide thickness is 40 µm.

Figure 1: Parabolic constant deduced from oxidation of iron for 260 h in the reference atmosphere



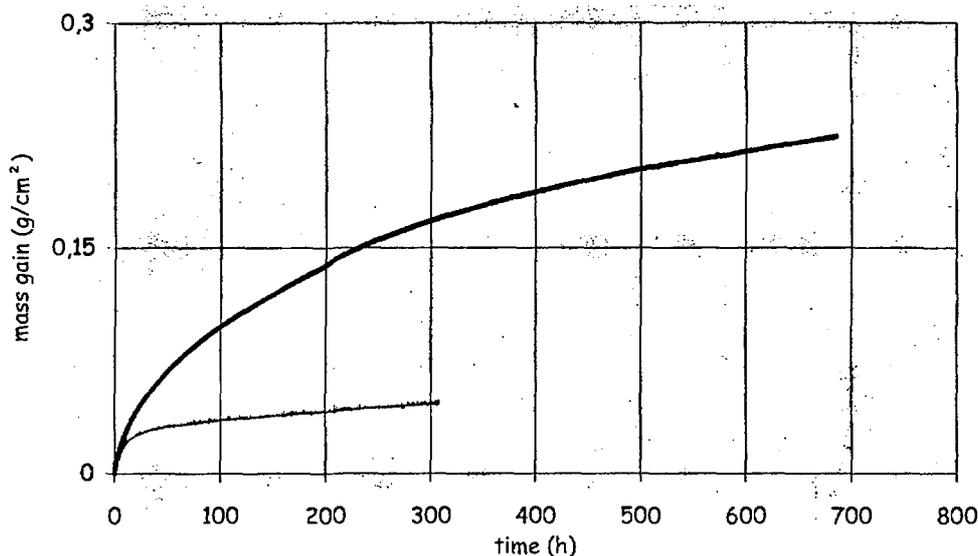
The limitation of the confidence in this extrapolation comes from the absence of experimental kinetics law. We have therefore determined the oxide thickness variations with the time at 300°C for iron and steel. They are shown on the Figure 2.

The oxide growth kinetics at 300°C on iron is better fitted by a parabolic law if only the first 300 h oxidation are considered. This is the most reported type of law reported in the literature. However, if we take into account all the data, the results are better fitted by a logarithmic law. This result has never been observed at this temperature for such long oxidation time and oxide thickness and requires to be confirmed.

As far as the steel is concerned, the oxide thickness variations with time at 300°C and 250°C are well fitted by a logarithmic law. An extrapolation over a 100-year period leads to predict the formation of a 1 µm thick oxide layer at 300°C on steel by using logarithmic laws. It is less than what is predicted by Larose and Rapp [5] but they use a parabolic law.

All these calculations converge to predict that the damage due to dry oxidation should be limited and acceptable for the external container walls. However, all these extrapolations are based on the same assumption that no desquamation of the oxide layer arises and oxide growth mechanism which is not yet well defined in the considered temperature and the environmental conditions remain unchanged during all the interim oxidation storage period. A model, taking into account several elementary processes involved in the oxide growth process must be developed in order to increase the reliability of the extrapolations of the short term data over long periods.

Figure 2: Oxidation kinetics in the reference atmosphere at 300°C of iron (upper curve) and of unalloyed steel (lower curve)



### 2-3 Results with pollutants

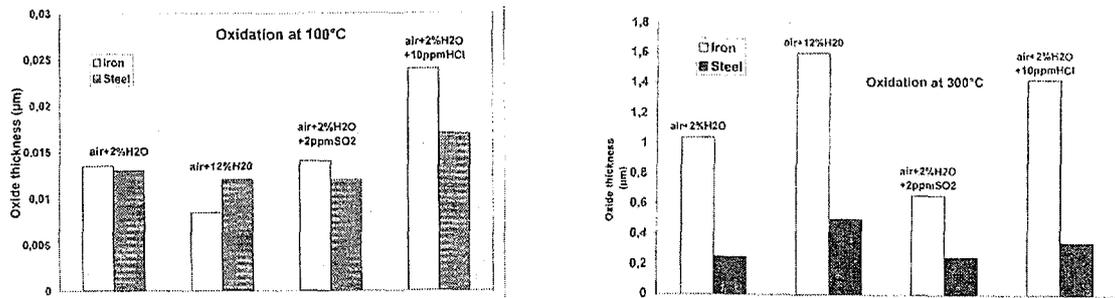
The effect of pollutants has been investigated at 100°C and 300°C. Similar results have been obtained for the two materials. The X rays analyses of the specimens oxidised at 300°C in the polluted atmospheres do not show other constituent than  $\text{Fe}_2\text{O}_3$  and  $\text{Fe}_3\text{O}_4$  in the oxide layers. XPS analyses show that the specimens oxidised in the wet atmosphere have higher hydroxyl content near the gas/oxide interface than those oxidised in the reference atmosphere.  $\text{SO}_x$  ( $x=3$  or  $4$ ) has been detected in the first superficial 15 Å of the oxide layer of the specimens oxidised in the presence of  $\text{SO}_2$  containing atmosphere. Specimens oxidised in the HCl containing atmosphere exhibit an oxide layer inside which chlorides have been detected. The Figure 4 shows the measured oxide thickness on the iron and steel specimens oxidised in the different investigated atmospheres. The Figure 4 reports also the weight gain variations with time of iron during oxidation at 300°C in the wet atmosphere and, for comparison, in the reference atmosphere. In these conditions, the measured oxide layer increase in wet atmosphere with time is best fitted by a parabolic law.

These results show a small thickness increase of the iron and steel oxides formed during oxidation at 300°C and 400°C for 300 h as the water content in the atmosphere increases from 2 to 12 %vol, but at 100°C. In the frame of the interim storage, we can expect that the impact of an increase of vapour content in the air is limited.

As far as the HCl pollutant is concerned, at all the oxidation temperatures, its presence in the atmosphere induces an increase of the oxide thickness and the formation of chloride in the oxide layer. The corrosion mechanism in this intermediate temperature range is probably different from that at high temperature ( $> 500^\circ\text{C}$ ) based on the high volatility of the chlorides. Longer duration tests and determination of the oxidation kinetics are planned in a further work.

The results show that, in the tested conditions, the presence of  $\text{SO}_2$  in the atmosphere does not increase the growth oxide kinetics at 100°C and 300°C and modifies only superficially at 100°C and more deeply at 300°C the iron and steel oxide layers. This pollutant seems less detrimental than HCl.

Figure 4: weight gains of iron and steels specimens oxidised at 100°C and 300°C for 260 h in various atmospheres



These first results on dry oxidation of iron and a unalloyed steel allow to propose some models for predicting the damage by dry oxidation for long periods in a reference atmosphere. Their reliability must be increased by developing more sophisticated models taking into account more numerous partial processes contributing to the whole oxidation process. Nevertheless, all our results, in agreement with those from the literature but obtained for short oxidation periods, agree to predict small damage if no change in the oxidation mechanism arises. The first data obtained with the pollutants do not predict a drastic change in the oxidation rates but they must be confirmed.

### 3- INDOOR ATMOSPHERIC CORROSION

Numerous data are available on outdoor atmospheric corrosion of iron or low alloy steels, including quite long-term testing period (i.e. over 10 years). Results from a number of atmospheric corrosion testing show that the behaviour of low alloy steels or iron can be characterized by two parameters  $K$  and  $n$  as far as a linear bilogarithmic law between penetration  $P$  and time  $t$  (i.e.  $P = K \cdot t^n$ ) is valid for outdoor conditions [6]. These laws are helpful in extrapolating results of corrosion test to long term, i.e. 20-30 years, but not for our need, the very long term behaviour on a few hundred years.

On the other hand there is a lack of data concerning indoor atmospheric corrosion. The stored containers can experience this particular kind of atmospheric corrosion being exposed to cyclic wet and dry conditions, depending on meteorological conditions among numerous parameters. Nevertheless, these interim storage indoor conditions are not so far from those of the classical atmospheric exposure, except that, of course, no rain is expected inside the disposal. So, first actions were to define how to apply outdoor data to the indoor conditions of an interim storage.

#### 3-1 Atmospheric corrosivity according to Standards

The corrosivity of the indoor atmospheres has been assessed using the classification tabulated for outdoor conditions in ISO/DIS 9223 and 9224 standards [7, 8], i.e. using one of the main corrosion parameters, the so called time of wetness  $\tau_i$ . The time of wetness, which is the time  $\tau_i$  during which  $T_i > 0^\circ\text{C}$  (Temperature) and  $\text{RH} \geq 80\%$  (Relative Humidity) at the metal surface, is a complex and composite variable. It determines the duration of the electrochemical process which take place during atmospheric corrosion. According to the ISO standards, outdoor atmospheric corrosivity  $C_i$  depends on three atmospheric parameters:

- $\tau_i$  : the time of wetness,
- $S_i$  : the airborne salinity, represented by the deposition rate ( $\text{mg}/\text{m}^2/\text{d}$ ) of chloride ion,
- $P_i$  : the sulphur-containing compounds content, represented by the sulphur dioxide  $\text{SO}_2$  level ( $\text{mg}/\text{m}^2/\text{d}$ ).

The categories of corrosivity  $C_i$  are connected with corrosion rates of well-defined metal panels after several years of exposure. The correspondence between the atmospheric corrosion rates of carbon steel and the corrosivity  $C_i$  is reported in the table 3.

Table 3: Corrosion rate ( $\mu\text{m}/\text{year}$ ) of carbon steel vs the classification of corrosivity [7]

Corrosivity		Corrosion rate ( $\mu\text{m}/\text{year}$ )		
		First year	t < 10 years	t > 10 years
C <sub>1</sub>	Very weak	0,1 - 1,3	< 0,5	< 0,1
C <sub>2</sub>	weak	1,3 - 25	0,5 - 5	0,1 - 1,5
C <sub>3</sub>	mid	25 - 51	5 - 12	1,5 - 6
C <sub>4</sub>	high	51 - 83	12 - 30	6 - 20
C <sub>5</sub>	Very high	> 83	30 - 100	20 - 90

### 3-2 Time of wetness on container wall

According to our intention to work on a textbook case, we defined a waste container, whose size and shape are quite realistic i.e. 1,5 m diameter and 2 m height. The given container is supposed to be a thin metallic canister filled with thermal powerless concrete which is assumed to control the thermal behaviour of the container which is disposed on the storage floor. The floor temperature  $T_{\text{floor}}$  of the storage is fixed arbitrarily to 13°C.

As soon as the temperature at the interface  $T_i$  is less than the dew-point, i.e. the saturation temperature of the ambient air  $T_{\text{dew}}$ , condensation begins on the container walls.

Principles of the thermal modelling and methodology used for the calculations of the time of wetness on container walls, can be found elsewhere [9]. As input data for the modelling, we used one-year climatic parameters to determine the thermal behaviour of the container. These data have been given by the Meteorological French Office for a quite humid place with low annual temperature range in the north of France (« oceanic climate »), and, by contrast, for a warm Mediterranean country with wide annual temperature range in the south of France (« Mediterranean climate »).

As the interim storage of the nuclear waste canister takes place inside an airy disposal, global heat transfer coefficients at container walls results from natural convection (no ventilation) or from forced convection if any ventilation occurs : these assumptions on the ventilation regimes leads to consider different global heat transfer coefficients  $h$  ( $h = 0,1 ; 1$  and  $10 \text{ W}\cdot\text{m}^{-2}\cdot\text{K}^{-1}$ ). The calculated time of wetness for different heat transfer coefficients are reported in table 4.

Table 4: Time of wetness (day/year) for the two site locations, on both exposed walls and for different values of the heat transfer coefficient in the air ( $h$ ).

h ( $\text{W}\cdot\text{m}^{-2}\cdot\text{K}^{-1}$ )	Oceanic climate		Mediterranean climate	
	Lateral wall	Superior wall	Lateral wall	Superior wall
0.1	214	223	125	113
1	231	241	89	80
10	255	260	95	96

The total time of wetness of the well-defined container stored in the northern place (oceanic climate) is far more important than it is in the southern location (Mediterranean climate): about twice as much (table 4). For the northern and humid location, the time of wetness increases with the heat transfer coefficient value i.e. the ventilation efficiency whereas it gets down in the southern site location.

### 3-3 Corrosivity assessment

Two opposite cases, i.e. two couples of pollutants contents  $S_i/P_i$ , have been considered for the assessment of  $C_i$ : a free pollutant site ( $S_0, P_0$ ), i.e. a rural location, and a heavy polluted site ( $S_3, P_3$ ), i.e. a marine-industrial place. The results of the numerical simulations are reported in the following table 5.

Table 5: Corrosivity classes for extreme values of  $Cl$  and  $SO_2$  contents for the two site locations

h ( $W.m^{-2}.K^{-1}$ )	Extreme pollutant parameters couples Si/Pi			
	Oceanic climate		Mediterranean climate	
	$S_0/S_1 - P_0/P_1$	$S_3 - P_3$	$S_0/S_1 - P_0/P_1$	$S_3 - P_3$
0.1	C <sub>3</sub>	C <sub>5</sub>	C <sub>3</sub>	C <sub>5</sub>
1	C <sub>4</sub>	C <sub>5</sub>	C <sub>2</sub>	C <sub>5</sub>
10	C <sub>4</sub>	C <sub>5</sub>	C <sub>2</sub>	C <sub>5</sub>

Even if the values of the time of wetness associated to the lateral and the upper face are different, equivalent corrosivity categories have been obtained for both faces (table 5). Comparing Oceanic and Mediterranean climates for weak atmospheric pollutant level and similar convection, the atmosphere seems to always be less corrosive in the south. Increasing pollutants levels increase corrosivity of the two site locations in the same way. Considering only the result of this pragmatic approach, the effect of pollutants content seems to gradually overshadow the influence of the time of wetness. Results show the huge influence of ventilation on the calculated time of wetness (table 4) and then on corrosivity (table 5).

As a general rule, results show the importance of simulations for prediction, as the long-term container behaviour seems closely linked, among other things, to the repository design, and can not thus be derived from direct extrapolations of classical atmospheric corrosion data.

### 3-4 Wet-dry cycles

The wet-dry cycles are another important corrosion parameter for these corrosion allowance materials as their number and their frequency are correlated to the corrosion rate and the rust layer transformation. Earlier investigations have shown that during a wet-dry cycle, the atmospheric corrosion of iron or low alloy steel can be divided into three stages. As initially proposed by Evans [10] and then performed by Stratmann et al. [11, 12], during the first stage (wetting), the anodic dissolution of iron is balanced by the reduction of the ferric species (namely the oxihydroxide  $\gamma$ -FeOOH) within the rust layer. In our description, we assume that the metal damage is proportional to the rust layer specific area as far as the reduction occurs at the nanopore surfaces. As the rust layer become thicker, the reducible rust layer internal surface is supposed to decrease and , in the same way, the metal damage.

After the reducible FeOOH is used up, the second stage (wet surface) begins : the oxygen reduction starts and takes place at the pore surfaces. It is assumed, in our work, that this step is a diffusion limited process. During the end of the drying, the third stage of the cycle, the reduced layer of  $\gamma$ -FeOOH and the other ferrous species are reoxidized by the oxygen and the electrolyte film is used up, stopping the corrosion process completely.

This description, which is not discussed further here, agree with the fact that the average corrosion rate decrease with time as it is obviously shown by the well-known bilogarithmic laws. The next step of this work is to model the evolution of the rust layer porosity whereas future calculations considering the previously mentioned well-defined container have to concentrate on the assessment of the main wet-dry cycle features regarding the physico-chemical processes described before. Then, in the perspective of this mechanistic approach the number of wet-dry cycles has been calculated as well as their average duration (table 6).

Table 6: Number and average duration (h) of wet-dry cycle for the two locations on both exposed walls

h ( $W.m^{-2}.K^{-1}$ )	Oceanic climate		Mediterranean climate	
	lateral wall	superior wall	lateral wall	superior wall
0.1	28 / 125	41 / 60	49 / 37,4	55 / 18,8
1	66 / 31,5	76 / 16	35 / 18,5	35 / 17,6
10	64 / 7	64 / 6	35 / 2,9	41 / 3,3

Regarding the storage location, simulations illustrate the influence of the environmental parameters : except for an hypothetical thermally isolated waste container (i.e.  $h = 0.1 \text{ W.m}^{-2}\text{.K}^{-1}$ ), the number of wet-dry cycles in the north of France are nearly twice what they are in the south. We can thus make the assumption that the number of actual interruption of electrochemical atmospheric processes (i.e. a electrolyte-free container wall) is more important in the south. The average duration of the wet phase of wet-dry cycles is nearly the same for both locations i.e. about 20 hours.

This pragmatic method gives assessments of the indoor atmospheric corrosivities according to the ISO/DIS 9223 standards. The calculation of indoor atmospheric corrosivities is of practical interest for the selection of materials and for the prediction of the corrosion behaviour of classical engineering structures. Nevertheless, this approach has a low discriminating power and do not permit reliable long-term predictions of the corrosion behaviour of waste canister. However, as far as atmospheric corrosion of corrosion allowance material like carbon or weathering steels is concerned, simulations give all the fundamental characteristics for deriving corrosion rates for future developments that should involve the other parameters among which the wet-dry cycles.

#### 4- CONCLUSION

The two main corrosion phenomena encountered in long term interim storages are investigated: dry oxidation by the air and atmospheric corrosion due to condensation on the container walls.

For dry oxidation, few data are available in the literature for the expected range of temperatures (below 300°C). The first experiments confirm the few available data of the literature, but over a longer period of exposure time, and validate an extrapolation method based on the apparent activation energy of oxidation. The results obtained with non polluted air lead to expect an uniform corrosion and low corrosion rate on iron and steel materials (less than 1  $\mu\text{m}$  is expected on these alloys exposed 100 years at 100°C in air). The effects of pollutants (chloride and sulphur compounds) do not induce large increases of these corrosion rates. These first results have to be confirmed while modelling of the oxidation mechanisms will secure the extrapolation, mainly in front of the breakaway phenomena.

Number of data are available concerning atmospheric corrosion, even during long periods of exposure (over 10 years), but nearly exclusively for outdoors exposure conditions. The use of these data with the ISO standards is possible by a thermohydraulic modelling of the indoor exposure conditions which leads to classify indoor atmosphere as function not only of the atmospheric conditions (Oceanic and Mediterranean climates have been considered), but also as function of the thermal inertia of the containers. To secure the corrosion rates deduced from this practical and pragmatic method, next developments of the indoor atmospheric corrosion modelling will include the use of semi-empiric well-known bilogarithmic laws and further the physical description of the corrosion phenomena with the effects of wet-dry cycles.

The last objective of the COCON program will be the strategy of corrosion monitoring: based on the results obtained during dry oxidations and atmospheric corrosion tests, the analyses of the corrosion oxide layers seem to be a promising strategy (thickness, structural and chemical composition). The purpose is to link the relevant oxide layer parameters to the evolution of the corrosion types and to the corrosion rates.

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