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Corrosion of metals and alloys — Procedures to determine and estimate runoff rates of metals from materials as a result of atmospheric corrosion

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National foreword

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Corrosion of metals and alloys — Procedures to determine and estimate runoff rates of metals from materials as a result of atmospheric corrosion

Corrosion des métaux et alliages — Modes opératoires pour déterminer et évaluer le taux d'entraînement par les eaux de ruissellement des métaux présents dans des matériaux soumis à la corrosion atmosphérique

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 17752 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

Introduction

Runoff testing is carried out in order to obtain data on the release rates of metals of different materials under atmospheric exposure conditions. It involves exposure of specimens at a test site and, in addition, continuous collection of runoff water samples and is therefore more demanding than standardized corrosion tests.

Standardized corrosion tests cannot be used for obtaining these data since, on a long-term perspective, the runoff rate is always less than or equal to, and frequently much less than, the corrosion rate. The difference is the metal incorporated in the corrosion products. In contrast to standardized corrosion tests, runoff testing can be performed on real product surfaces or on any kind of surface-treated material, e.g. pure metals, alloys or different coatings (i.e. metallic coatings, metal-including organic coatings), as long as the history and surface finish and characteristics of the specimen are documented.

The result of this field test is the metal release rate to the collected rain water that has impinged the material surface. Further possible transformations of the metal in its different chemical forms and interactions with the environment are beyond the scope of this International Standard.

Procedures to establish runoff rates can be carried out either by determination (normative) based on exposure of specimens or by estimation (informative), taking into account the conditions in a particular application.

Corrosion of metals and alloys — Procedures to determine and estimate runoff rates of metals from materials as a result of atmospheric corrosion

1 Scope

This International Standard specifies procedures to determine and estimate runoff rates of metals from metals, alloys and coatings under atmospheric conditions carried out in the open air.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4221, *Air quality — Determination of mass concentration of sulphur dioxide in ambient air — Thorin spectrophotometric method*

ISO 4226, *Air quality — General aspects — Units of measurement*

ISO 4543, *Metallic and other non-organic coatings — General rules for corrosion tests applicable for storage conditions*

ISO 8565, *Metals and alloys — Atmospheric corrosion testing — General requirements*

ISO 9169, *Air quality — Definition and determination of performance characteristics of an automatic measuring system*

ISO 9225, *Corrosion of metals and alloys — Corrosivity of atmospheres — Measurement of environmental parameters affecting corrosivity of atmospheres*

ISO 9226, *Corrosion of metals and alloys — Corrosivity of atmospheres — Determination of corrosion rate of standard specimens for the evaluation of corrosivity*

3 Requirements for test specimens

3.1 Types of specimen

The exposed surface area of the specimen should be of such a magnitude that it provides sufficient water runoff volumes to enable reproducible results to be determined from the test, minimizing edge effects and any loss of runoff water. Rectangular specimens in the form of flat sheets shall be sized at least 100 mm × 300 mm (300 cm^2). The surface area of the specimens can be larger, provided that they can be accurately evaluated and all runoff water continuously collected. The specimen thickness shall be adequate to ensure that the specimens will endure the intended test period. A useful thickness is between 1 mm and 3 mm. The surface of the specimen tested should represent real products as closely as possible.

3.2 Specimen preparation and handling

Because atmospheric corrosion and metal runoff tests may extend over many years, it is important to ensure that specimens are clearly identified and records of data (collected total runoff water volumes, pH, and measured total metal concentrations) are carefully kept.

Surface damage shall be avoided. All specimens, in particular coated materials, shall be free from surface damage. To minimize surface contamination and to ensure surface uniformity, the uncoated specimens should be cleaned prior to exposure, if possible, depending on the material. A convenient procedure is degreasing in acetone and isopropylic alcohol. Abrasion is not recommended since such a surface is not representative for real product conditions and can largely influence the extent of released metals. Fingerprints during specimen handling can influence the results and shall be avoided by using cotton gloves and by handling the specimens at the edges.

The reverse side of each specimen shall be covered by an adhesive tape and the cut-edges shall be sealed with lacquer (metal-free) or wax in order to avoid metal release from these parts of the specimen. Sealing of cut-edges is primarily essential for coated materials.

A specimen holder without a metal specimen made of an inert material with the same area should be exposed in parallel to continuously collect impinging rainwater for background deposition rates of the metals of interest.

3.3 Marking of specimens

Mark the specimens in such a way that no confusion during the exposure is possible. It is recommended to mark both the sample holder and the specimen.

This can be accomplished by notching (which shall be on the back side of the specimen). Other marking procedures may be used, provided that the requirements of legibility and durability are met. The area affected by marking shall be minimized. Any method of identification used on the specimens shall not influence, or otherwise interfere with, the results of the test; this is particularly important where notching is used to identify coated specimens. All notching on coated specimens shall be made prior to coating.

3.4 Number of specimens

For each type of material, one specimen is sufficient as long as the surface area is equal to or exceeds 300 cm^2 (see 3.1).

3.5 Storage

The time period between specimen preparation and start of exposure should be kept as short as possible to minimize any corrosive effects induced on the specimen during storage. Such effects can influence runoff results, in particular for specimens releasing very low concentrations of metals. The recommended time between specimen preparation and start of exposure is one week, in particular for bare metal and alloy surfaces.

Since runoff tests can be conducted on any kind of surface, it is essential to document the storage time and storage conditions prior to testing. If the specimens are to be analysed for corrosion products and surface morphology after exposure termination, the recommended maximum time between the end of exposure and surface evaluation is two weeks to ensure no changes in surface characteristics and to avoid further oxidation. Naturally this period shall be kept as short as possible, in particular for short-term exposures.

During storage of the test specimens before exposure, care shall be taken to avoid mechanical damage and contact with other specimens. A room with controlled temperature and relative humidity of 50 % or less shall be used for storage purposes. Particularly sensitive specimens shall be stored in a desiccator or sealed in plastic bags with a desiccant. (See ISO 4543.)

3.6 Specimen data records

For each series of test specimens, record the data that are needed for the assessment of the runoff effects (see Clause 8). These records shall include the following:

a) in the case of bare metals or alloys:

- chemical composition including main and minor alloying elements;
- exposure geometry and surface area;
- surface finish characteristics (e.g. surface roughness profile, etc.);

- specimen history (e.g. storage conditions, pretreatments, surface ageing).

b) in the case of coatings or other product surfaces, in addition:

- specification of the basis metal (substrate), if available;
- specification of the coating application method(s);
- specification of the coating material(s), chemical composition;
- coating thickness.

NOTE 1 The composition can vary from point to point on the specimen, as can the morphology of coating exposed to the test.

NOTE 2 Visual and photographic records of the specimen conditions prior to and during testing can be made.

4 Atmospheric-corrosion test sites

4.1 Test site requirements

The atmospheric-corrosion test site shall provide facilities for open-air exposure, i.e. direct exposure to all atmospheric conditions and atmospheric contaminants (see ISO 8565).

Test sites shall be selected so that the testing area will normally be exposed to the full effects of the weather. The presence of buildings, structures, trees and certain geographical features (rivers, lakes, hills or hollows) might cause unintended shelter of exposure to wind, sources of pollution or sunlight.

Unless the effects from man-made or natural features are to be an intended part of the exposure programme, such features in the vicinity of the test site, that could influence the results of the test, should be avoided; otherwise their presence shall be reported. Similarly, the presence of low-growing shrubs and other plants may affect the temperature and humidity distribution over a given test site. Therefore these shall be absent or controlled to a maximum height of 0,2 m or by placing test frames on well-drained ground or on gravel, concrete or paved foundations.

4.2 Test site locations and security

The exposure site shall be selected so that it represents uniform conditions of some atmospheric site with specific characteristics, for example, a rural, urban, marine or industrial environment. If possible, it shall be located at or near a site where on-going meteorological and environmental measurements of temperature, humidity, rainfall characteristics (rainfall quantity, rain intensity), gaseous and/or particle contaminants are performed, for instance by environmental or health organizations. Near-point sources of emission of corrosive substances shall be avoided. Ideally, the test site shall be fenced or in other ways protected from damage caused by the public or by animals and from growing plants reaching the specimens.

4.3 Exposure frames

The function of the exposure frames is to maintain the test specimen securely in position without undergoing significant deterioration or influencing the corrosion or runoff of the test specimens attached to them. Metal sections or wood may be used, provided that they have adequate strength and durability. If necessary, additional protection can be provided by paint coatings over suitably prepared and primed metal surfaces. Frames may also be constructed from suitably protected and maintained wood. The choice of materials and design of the supporting framework shall not influence or otherwise interfere with the results of the tests.

The test frames shall also be designed to enable specimens to be exposed at an angle of 45° from the horizontal. The design of the frame shall be such that test specimens are not affected by water that runs off the test frame or other specimens, or by splash water from the ground. The minimum height shall be chosen to prevent both splashing by rainwater and burial in snow-drifts and should be not less than 0,5 m.

5 Test site characterization

It is recommended to characterize the atmospheric conditions at the test sites. This characterization shall be carried out by direct measurements of the corrosion rate of standard specimens according to ISO 9226, together with measurement of the environmental parameters at the site or collection of the environmental data from other sources.

Recommended environmental data for the characterization of the atmosphere are as follows:

— air temperature, °C;

— relative humidity, %;

— amount of precipitation, mm/a;

— pH of precipitation based on the annual precipitation-weighted average of H^+ concentration of precipitation;

— SO_2 concentration, µg/m^3, or SO_2 deposition rate, mg/(m^{2}d);

— Cl^- deposition rate, mg/(m^{2}d), usually only for marine test sites;

Other factors can be collected or measured depending on the specific requirements of the test.

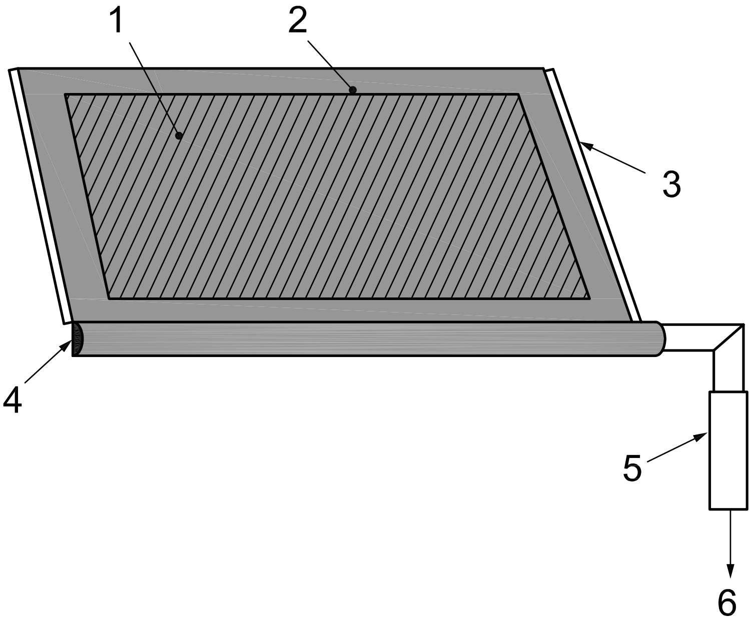
The characterization of the test site atmosphere shall be performed according to ISO 4226 and ISO 9169. Concentration measurements shall be performed, preferably according to ISO 4221, and deposition measurements according to ISO 9225.

The characterization according to ISO 9226, together with environmental data, enables the determination or estimation of the atmospheric corrosivity category.

In order to compare the results of the runoff measurements, it is recommended to perform direct measurements of corrosion attack on the front side of specimens identical to those used in the runoff exposure, with the back side of each specimen covered by an adhesive tape and cut-edges sealed with wax or lacquer as described in 3.2.

6 Operating conditions

Each specimen shall be mounted single-sided on a fixture equipped with an inclined gutter into which the runoff water, which has impinged on the specimen, is collected. From the gutter, the runoff solution is transported to the container (see Figure 1). The fixture, the gutter as well as container, shall be made of some inert material, such as polymethylmetacrylate or polyethylene. The width of the fixture extending the specimen shall be held as small as possible to avoid dilution of runoff from the specimen, usually <0,5 cm.



Key

1	panel
2	fixture of inert material
3	inert material edge
4	stop edge
5	silicon tube
6	polyethylene vessel for runoff water collection

Figure 1 — Recommended fixture of inert material for runoff rate measurements

The test specimens shall be placed in such a way that:

- contact does not occur between individual test specimens and any material that can affect their corrosion or runoff under the test conditions:
- corrosion products and runoff water containing released metals from the surface do not drip from one test specimen onto another;
- the total runoff water impinging the specimen surface is collected;
- the collection vessel for runoff water is easy to change periodically;
- there is easy access to the surfaces of the test specimens;
- the test specimens are protected from falling, accidental contamination or destruction;
- all the test specimens are exposed to the same conditions, as far as possible, with uniform access of air from all directions;
- the surface of the test specimen, in general, faces south in the northern hemisphere and north in the southern hemisphere, but the direction of other sources that can influence the corrosion attack, such as the ocean, can be taken into consideration;
- the inclination of the test specimens is preferably 45° to the horizontal.

7 Test procedure

7.1 Duration of tests

The total period and the season of the exposure depend on the type of test specimen and the purpose of the test. A total exposure time of at least one year and preferably five years is recommended for runoff rate determinations. For coated specimens, the period of exposure shall not be less than five years.

7.2 Periodic collection of runoff samples

The runoff water in the collection vessel shall be collected continuously. The vessels shall be changed before being completely filled to ensure no loss of released metals and enable determinations of actual amounts of precipitation impinging on the specimens, amounts influenced by prevailing exposure conditions and wind directions. If the collection vessel is overfilled, an estimate of the impinging rainfall volume shall be made. This can be based on meteorological measurements of rainfall units during the specific sampling period, or estimated based on similar previous sampling periods. The time period for changing collection vessels depends on the amount of precipitation, prevailing precipitation conditions and the volume of the vessels used. Depending on rain characteristics, a volume of (2 to 5) L is recommended. If evaporation of collected runoff water takes place, runoff water collection should be performed more frequently to minimize this effect (e.g. by coverage of the collection container with aluminium foil). Metal runoff measurements from specimens releasing low (µg/L) concentrations of metals, typically coated specimens, shall be conducted using acid-cleaned collection vessels to avoid any metal contamination through soaking in pure 10 % HNO_3 for at least 24 h, followed by rinsing at least four times with ultra-pure water, and then dried in ambient laboratory air before use.

Runoff water shall be collected from an inert identical specimen holder exposed in parallel at identical exposure conditions and collection-time periods to determine background metal concentrations. Measured concentrations should be subtracted from the runoff data generated from the specimens.

After collection, the collected runoff solutions shall be transported to the laboratory for determination of the total runoff water volume and pH (at ambient laboratory temperature) and analysis of the total metal content. Before analysis, the runoff solution shall be acidified to a pH less than 3 by using, for example, HNO_3 or HCl. Acidification is performed to dissolve possible metal complexes formed on the walls of the collection container and conserve the runoff water. After acidification, the runoff solutions can be stored for up to one month (or longer) before analysis (preferably in dark conditions).

7.3 Evaluation of results

Depending on the metal(s) to be analysed, determination of metal concentrations exceeding 1 mg/L is normally performed by means of flame atomic absorption spectroscopy (FAAS). Analysis of metal concentrations in the range of µg/L and upwards, can be accomplished by means of graphite furnace atomic absorption spectroscopy or inductively coupled plasma/atomic emission spectroscopy.

By adding the measured released metal content from each sampling period (metal concentration multiplied by the collected runoff water volume) after subtracting the background content measured for each sampling period, the total amount of released metal can be calculated and expressed in $g/(m^2a)$. An alternative unit for expressing runoff rate is $\mu m/a$. The former is more appropriate when presenting actual metal runoff rate data, the latter possibly when comparing data with corrosion rates.

The runoff rate is based on the total volume of precipitation impinging the surfaces, expressed either as L/m^2 (based on the exposed area of the specimen inclined 45° from the horizontal, largely influenced by the prevailing wind direction) or as mm precipitation (L of rain per m^2 of horizontal area), impinging on the surface during the total exposure period. The meteorological unit “mm precipitation” is mainly used for metal runoff predictions and comparison between runoff data for different sites and surface inclinations and orientations.

The runoff rate can be calculated using Equation (1):

$$\mathrm{MR} = \frac{m}{A \cdot t} \tag{1}$$

where

MR is the metal runoff rate, g/(m^{2}a);

m is the total mass of metal in the collected runoff, g;

A is the metal surface area, m^2;

t is the exposure time, a.

Here, the total mass is the sum of each individual mass obtained from the different sampling events after subtraction of the mass originating from background deposition. A total exposure time of at least one and preferably five years is recommended for runoff rate determinations.

Even though the runoff water in the collection vessel shall be changed regularly and before the vessel is completely full, it may happen on rare occasions due to seasonal effects that the vessel is overfilled due to an unexpected amount of rainfall. In this case, it shall be assumed that the measured metal concentration of the rainwater is representative of the entire sampling period. The total amount of precipitation for the sampling period should, in this case, be based on meteorological measurements of rainfall units, if available from nearby meteorological stations, measured during the specific sampling period, or estimated based on similar previous sampling periods.

8 Test report

The test report shall contain at least the following information:

a) data on conditions of specimen storage prior to exposure;

b) data on the test specimens, including preparation, inclination and direction of specimens exposure and any deviations from the recommended operating conditions;

c) the test site characterization (see Clause 5);

d) the dates of exposure and collection time periods of runoff samples;

e) information on specific sampling periods with overfilled collected vessels, unexpected exposure conditions, e.g. storm events, hail, etc.;

f) information on total metal concentrations, pH and volumes of collected runoff water for each sampling period and specimen, and for the inert sample holder exposed in parallel for background deposition measurements;

g) the quantitative results of assessment of runoff rate, in g/(m^{2}a);

h) annual rainfall quantities in mm/a;

i) background deposition levels of the metal of interest, based on parallel runoff water measurements on a specimen holder without any specimen in g/(m^{2}a).

Annex A
(informative)

Conditions affecting estimation of runoff rates of metals from bare materials as a result of atmospheric corrosion in a particular application

A.1 General

The procedure described in this International Standard for determination of runoff rates of metals is useful for comparative purposes, for example, between different types of materials, different types of corrosion-protective measures, or between different atmospheric environments. Such data can also be used to establish long-term trends as a result of changes in pollutants at a given outdoor environment.

The data, however, represent a normalized exposure situation and are not in every case directly applicable for estimating the runoff rate in a particular application or exposure situation. Furthermore, if there is a lack of measured runoff data, it is sometimes necessary to make an estimate of the runoff rate. Many parameters are known to influence both the corrosion rate and the runoff rate, including, for example, the precipitation volume, sheltering effects, wind conditions, specimen age and orientation, and emission source.

Some relevant information or experience for such transformations or estimations is briefly listed below. More detailed information is given in References [1] to [17].

The runoff rate is always less than or equal to the corrosion rate on a long-term perspective and is frequently significantly less than the corrosion rate for bare metal surfaces. Even so, corrosion data can, for specific situations, be used for assessing conservative upper estimates of the runoff. This can be especially useful in areas known to have very low corrosion rates (see A.9).

When calculating a conservative upper estimate of the runoff rate based on corrosion data obtained in the usual way, it is necessary to take into account the difference in corrosion attack of the reverse and front side of the specimen. If they are equal, the runoff rate is always lower than the corrosion rate, as described before. However, if the corrosion rate of the reverse side is negligible compared to that of the front side, a conservative upper estimate implies that the runoff rate is always lower than twice the corrosion rate, since the corrosion rate is always expressed taking into account the total area, both front and reverse.

A.2 Effect of exposure time

Runoff rates of metals from outdoor construction materials that develop corrosion products, exhibit relatively constant or slightly decreasing values over longer (>years) periods of time. During specific rain episodes of given rainfall characteristics, however, the runoff rate can, depending on prevailing exposure conditions prior to the rain event, be very high during the first period of the episode, the so-called first flush, after which the rate decreases and becomes relatively constant during the remaining episode. When integrating many rain episodes, the accumulated amount of metal runoff from bare surfaces with corrosion products increases approximately linearly with time for a given test site, from which follows a relatively constant runoff rate, irrespective of exposure time. As long as prevailing environmental conditions do not change dramatically between different years of exposure at a given exposure site, the released amount of metals per given rainfall unit is relatively constant for bare metal surfaces.

A.3 Effect of evolution of corrosion products

Bare construction materials often develop corrosion products with time of a relatively porous character, a process which depends on prevailing environmental conditions and time of exposure. The corrosion products are able to retain humidity and environmental pollutants for longer time periods compared to a more compact surface oxide. Measured runoff rates from such corrosion products exhibit, however, only slightly higher values.

This observation holds true although the corrosion products may possess different chemical composition and density. However, from a risk-assessment perspective, these differences are small.

A.4 Effect of precipitation volume

Given the pollution concentration and the surface inclination, the runoff rate is strongly correlated with the precipitation volume impinging the surface, which act as a medium to both dissolve and transport dissolved metal species from the surface. For certain bare metals, a preliminary very rough and conservative approximation is that the total amount of runoff is linearly proportional to the total precipitation volume. However, the actual amount of runoff depends on the specific material and is strongly related to prevailing environmental and pollutant conditions prior to a rain episode, and of rain characteristics such as intensity, composition and pH.

A.5 Effect of surface orientation

The runoff rate from a building with surfaces (roofs, facades, etc.) of different geometry, orientation and inclination is largely influenced by the prevailing wind conditions that determine the precipitation volume impinging each surface. Hence, when comparing the runoff rate from two surfaces, one facing south with 45° inclination according to the normalized procedure and the other one facing any given direction with 45° inclination, the comparison should be made by normalizing the amount of runoff for each surface with the actual collected impinging precipitation volume. It shall be added that the prevailing wind conditions also influence the deposition of corrosion-stimulating atmospheric constituents.

A.6 Effect of surface inclination and degree of sheltering

Given all other parameters and assuming the effect of prevailing wind direction to be small, the runoff rate decreases with increasing inclination to the horizontal. This is caused by differences in the projected surface area, which collects the highest precipitation volume when being inclined horizontally and which collects a lower precipitation volume the higher the degree of inclination. Hence, when comparing the runoff rate from two surfaces with different inclinations, differences in precipitation volume hitting each surface shall be compared and the proportionality between runoff rate and precipitation volume shall be assumed. Based on this general rule and for the simplified case when precipitation is vertical, the runoff rate of a surface inclined at angle θ can be estimated from the runoff rate measured at 45° inclination using the relation

$$\text{Runoff rate } (\theta) = \text{Runoff rate } (45^\circ) \times \cos(\theta)/\cos(45^\circ)$$

The total runoff rate from any building or other outdoor construction will be the result of added runoff rates from each individual surface. Based on the results from Reference [5], it is estimated that the weighted runoff rate of surfaces in general does not exceed the runoff rate from the normalized procedure, which therefore can be said to represent an upper limit. The weighted runoff rate from an average building, with both facades and roofs, is typically at least 25 % lower than that obtained from the normalized procedure. This conclusion is based on model roof exposures and is possibly applicable only to their particular exposure situations. On sound scientific grounds, however, the results may be applicable to other exposure situations as well, at least in the tempered climate zone and with no extreme emission sources present. A significantly less conservative number of approximately 50 % has been suggested elsewhere [6].

A.7 Effect of sulfur dioxide (SO_2) concentration

The importance of the corrosive gaseous pollutant SO_2 on the extent of metal runoff rates has been documented for some bare metals in the scientific literature, and limited predictive models have been established. However, it is evident that SO_2 influences both directly the dissolution process of constituents of the corrosion products during “dry” conditions, as well as indirectly via rain pH during precipitation events.

A.8 Effect of chlorides

No relations have been published so far on the effect of chlorides on metal runoff rates. However, recent findings suggest that even though chlorides are known to largely influence the corrosion rate, their effect on the runoff rate seems to be limited. Due to limited data so far, predictions of runoff rates are difficult to make. However, even highly corrosive sites show relatively constant or decreasing runoff rates with time for bare metal surfaces, and at levels significantly lower than reported corrosion rates.

A.9 Effect of temperature

There are no published relations describing the effect of temperature on runoff. However, as stated in A.1, the runoff rate is always less than or equal to the corrosion rate and is frequently significantly less than the corrosion rate. Furthermore, the information on the effect of temperature on corrosion is less scarce and this information should be taken into account. For example, when the temperature is very low, corrosion can also be very low. Therefore, when estimating the runoff rate, it is generally advisable to compare this estimate with reliable estimates of the corrosion rate for a similar situation and make sure that these two estimates comply with the rule that the runoff rate is always less than or equal to the corrosion rate.

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