Assessment of Surface Cleanliness
By Use of the Bresle Method of Sampling

By Bo Carlsson, Swedish National Testing and Research Institute, Boras, Sweden

Excessive surface contamination by salts, oil, or grease residues on steel prior to painting can significantly reduce the service life of corrosion protective coating systems.

However, the lack of methods for sampling and chemical analysis or the shortcomings in existing methods have long constituted an obstacle to determining whether surface cleanliness is satisfactory.

Now, a remarkably simple sampling method developed by Swedish scientist Ake Bresle has changed the way a surface treatment contractor can check the chemical cleanliness of a steel surface before applying anti-corrosion paint.

**How is Sampling Performed?**

The Bresle sampling method uses a device consisting of a millimetre-thick plastic frame, known as a patch. It is illustrated in Fig. 1. One side is coated with adhesive that can adhere to the test surface. The other side consists of a latex film through which leaching or extraction liquid can be supplied to the surface using a syringe. The patches come in various sizes. One, for example, has a compartment size of 1,250 ±13 mm².

The liquid is pumped back and forth repeatedly between the syringe and the test compartment (Fig. 2) until it is transferred to a container for chemical analysis of the concentration of contamination leached from the surface. Sampling time may vary depending on the application. For sampling of soluble salts on blast-cleaned steel surfaces to assess total salt surface density, a sampling period of about five minutes with two to four pumping cycles per minute would be appropriate.

This sampling procedure enables the surface contamination within a well defined area of the test surface to be quantitatively transferred to a known volume of the leaching liquid. Different methods of chemical analysis can then be used to determine the quantity of surface contamination per unit area—either simple field analysis if a quick answer is needed, or advanced laboratory analysis if there is sufficient time to wait for the results.

The Bresle method has the advantage of minimising the risk of contamination from such sources as the operator’s fingers during sampling. In addition, the sampling procedure induces a powerful turbulence in the leaching liquid when pumped back and forth between the syringe and test compartment, increasing the dissolution rate of the surface contaminants.

**Hydrochloric Acid on Steel Surfaces Exposed to Fire**

Bresle’s method was applied in connection with the development of field analysis to trace and quantify the surface density of hydrochloric acid on steel that had been exposed to gases from a fire, such as
a PVC fire. The results of the analysis could indicate the appropriate cleaning methods to be applied.

The leaching liquid, in this case, was distilled water, which can be titrated with mercury nitrate after sampling to enable the surface density of chloride on a test surface to be easily determined. If the surface density of chloride was found to be over 100 mg/m², a thorough washing of the surface would be needed.

Today, this method of field analysis is routinely used by the fire protection service in Sweden, and a special bag containing all the equipment and consumables needed for analysis has been commercially available for a long time now.

**Salts on Steel Surfaces Prior to Painting**

When applying anti-corrosion paint to steel surfaces, the total surface density of salt is of prime interest. In order to measure the total surface density of salt in the leaching liquid of distilled water under field conditions, Bresle found the simplest method was to measure the conductivity using a field conductivity meter. The result is converted to an equivalent surface density of water-soluble salts on the surface of the steel.

How high can the surface density of salt on a steel surface be before it seriously affects the service life of a rust-protective coating? This, of course, depends on the type of paint system used.

Work is being done by an ISO working party (ISO TC 35/SC 12/WG 5, Guidance on Levels of Contamination for Steel Surfaces Before Application of Paint and Related Products) to develop general recommendations that can be incorporated in an international standard. Some paint manufacturers already specify the maximum surface density of salts that can be accepted when using their products.

Examples of typical residual surface densities of salt measured by the Bresle method and applicable to various treated steel surfaces include
- 50-100 mg/m² for a thin steel sheet with rust covering 25 to 100 percent of the total surface but from which loose rust has been brushed off; and
- 20-30 mg/m² for a thin steel sheet that has been acid-pickled or washed with distilled water.

**Determination of Surface Moisture**

Determining surface density of moisture on steel prior to painting is another application for Bresle’s sampling method. Sampling is done using a hygroscopic leaching liquid comprising a mixture of glycol and water. The refractive index of the mixture is measured before and after sampling using a mini refractometer. The difference in the measured values enables the moisture content (i.e., the quantity of water which has been removed from the surface) to be calculated.

Using this method, it is possible to measure a surface density of water on steel as low as the equivalent to a moisture film of 3 µm. Although a solvent-based paint normally can be used in the presence of water quantities up to 10 µm, not enough is known about the levels of moisture at which the performance of various coating types begins to suffer. However, it should be possible to use this method to determine maximum permissible moisture levels for application of different paint systems.

**Determination of Oil and Grease Contamination**

The Swedish National Testing and Research Institute, together with some Swedish industries, recently started a development project to make Bresle’s sampling method available for field measurement of oil and grease contamination on metal surfaces.

A simple field method of measuring oil and grease contamination on metal has interest far beyond corrosion protection alone. Inorganic surface treatment companies, steel users, and metal workers have been looking for a suitable method for a long time. This need has increased in recent years in Sweden as a result of the ban on the use of chlorinated degreasers such as trichloroethylene (TRI) and perchloroethylene (PER). In most cases, alkaline degreasing by aqueous solutions as an alternative to TRI or PER degreasing imposes more stringent requirements on the reliability of the control method for surface cleanliness of metals.

The first stage of the project is to develop a standardised test procedure that uses the Bresle method and simple chemical analysis to determine the oil and grease contamination. A method in which oil and grease residues are removed from the metal by use of cyclohexane and then selectively oxidised by chromate in an aqueous sulphuric acid solution has been found to have sufficient sensitivity and, therefore, looks promising.

The second stage is to find manufacturers of sampling equipment and suppliers of the chemicals to
make it possible to offer a commercially available kit for field use.

Finally, the third stage aims to offer users of the method help when establishing surface cleanliness criteria for quality control in connection with degreasing.

**Current Standardisation Work**

The Bresle method has attracted considerable international interest and become the object of standardisation work within ISO TC 35 (Paint and Varnishes)/SC 12 (Preparation of Steel Substrates Before Application of Paints and Related Products)/WG 2 (Surface Cleanliness) under the convenership of Prof. Einar Mattsson, the former director of the Swedish Corrosion Institute.

The Bresle method already has been standardised in ISO 8502-6 (Preparation of Steel Substrates Before Application of Paints and Related Products—Tests for the Assessment of Surface Cleanliness—Part 6: Extraction of Soluble Contaminants for Analysis—The Bresle Method). In addition, work is in progress on standardisation of no less than six methods of surface analysis for use in the field, all of which are based on the Bresle sampling method and some of which have been described in this article.

These six methods are

- field method for determination of oil and grease, ISO 8502-7;
- field method for refractometric determination of moisture, ISO 8502-8;
- field method for the conductometric determination of water-soluble salts, ISO 8502-9;
- field method for titrimetric determination of chloride, ISO 8502-10;
- field method for turbidimetric determination of sulphate, ISO 8502-11; and
- field method for titrimetric determination of ferrous ions, ISO 8502-12.

**Conclusion**

Within the Swedish National Testing and Research Institute, it is believed to be important to support this development of quality tools for surface treatment contractors. We recently have linked Bresle to our work, and have arranged the production and sale of equipment for measuring salt concentrations and surface moisture according to the Bresle method.

**References**