NICKEL RELEASE REGULATIONS, EN 1811:2011 – WHAT’S NEW?

BACKGROUND:
EN 1811 is the internationally recognised test method which was devised almost 12 years ago to determine the rate of nickel release from jewellery, spectacle frames and other items. The test was devised to assess whether articles comply with the requirements of the EU Nickel Directive which restricts the release rate of nickel from articles intended to come into direct and prolonged contact with the skin and with post assemblies intended to be inserted into piercings in the body.

Sensitisation to nickel can result in severe allergic reactions. The objective of the Nickel Directive is to reduce the incidence of nickel dermatitis by preventing non-compliant items from reaching the marketplace.

The EU Nickel Directive was originally introduced in 1994, became enforceable in 2000 and was amended and updated in 2005. The 2005 requirements were subsequently amalgamated into the REACH regulation [Ref: REGULATION (EC) No 1907/2006, Entry 27 of Annexe 17 (REACH)]. REACH is a European Union regulation concerning the Registration, Evaluation, Authorisation and restriction of Chemicals. It came into force on 1st June 2007 and replaced a number of European Directives and Regulations with a single system. The Annex XVII of REGULATION (EC) No 1907/2006, came into force from 1 June 2009)

Under the legislation any manufacturer, importer, wholesaler or retailer in the supply chain will commit an offence if:

- They sell a post assembly intended to be inserted into a pierced part of the human body unless the rate of nickel release from the post assembly is less than 0.2 μg/cm²/week;

- They sell jewellery items, including certain metal clothing fasteners, if the rate of nickel release from those parts of the product which come into direct and prolonged contact with the skin is greater than 0.5 μg/cm²/week.
They sell any jewellery items, including certain metal clothing fasteners, where these have a non-nickel coating unless the coating is sufficient to ensure that the rate of nickel release from those parts of the product which come into direct and prolonged contact with the skin does not exceed 0.5 μg/cm²/week for a period of at least two years normal use of the product.

**CHANGES TO EN 1811:1998**

There are many factors which can affect the level and rate of nickel release from a nickel alloy. Apart from the actual nickel content and composition of the alloy these include surface finish, non-homogeneity, and plating among others. As a consequence, results from testing according to the EN 1811:1998 + A1:2008 standard test method have sometimes been shown to be subject to variability and uncertainty. This is acknowledged by the fact that the EN 1811:1998 method requires that the test result be multiplied by a correction factor of 0.1 in order to compensate for these variables and try to obtain a more consistent and comparable nickel release figure. Effectively this means that an article recording an actual release rate of as much as 5.0μg/cm²/week or 2.0μg/cm²/week, depending upon type of article, when tested by EN 1811:1998 would still be deemed to be compliant to the Directive once the correction factor of 0.1 had been applied.

In 2010, the EU granted a mandate to CEN TC 347 TG 1 (the European Committee tasked with revision of the international standard) to examine the EN 1811:1998 test procedure and the related uncertainties. In order to protect the health of the consumer it wanted to develop and introduce a more reliable / robust procedure, remove or reduce the adjustment factor of 0.1 and deliver a test method which was more accurate and reproducible.

Following the work undertaken by the CEN Committee the 1998 standard has now been superseded and replaced with EN 1811:2011.

**SUMMARY OF DIFFERENCES BETWEEN THE 1998 AND THE 2011 STANDARD:**

The main difference between the 1998 and the 2011 standard is that the existing correction factor of 0.1 has been replaced by an uncertainty of measurement ($u_{t,r} = 46 \%$, $u_{t,r}$ is related to the relative reproducibility). This has the effect of effectively lowering the compliance limit for nickel release, so products which were currently compliant with the regulations may cease to be when tested to the revised standard.

The new standard also features a ‘no decision’ category of result due to the move away from the use of a correction factor and adoption of an uncertainty of measurement approach to assessing test results. ‘No decision’ captures the range of results where, after testing to the revised standard, no clear decision is possible, due to interpretation of the measurement of uncertainty of results. A “No decision” verdict does not mean that the article is not compliant; but it means the supplier cannot claim that it is compliant. Under such circumstances the manufacturer and/or importer shall be made aware by the laboratory carrying out the test that the material composition of the tested article may be unsuitable and consideration should be given to the use of alternative materials.
Other significant changes:

- The scope was expanded to include all post assemblies which are inserted into pierced parts of the human body.
- The preparation of the test solution has changed; ammonium hydroxide is no longer used and it has been replaced by sodium hydroxide. The reason for this is that ammonium hydroxide is unstable and can change concentration with time in storage and also by evaporation, even during the test procedure and, therefore, contributes uncertainty to the activity of the test solution. Sodium hydroxide is a more relatively stable alkali and unlike ammonium hydroxide, it does not form complexes with nickel and the resulting test solution will be less active and, therefore, less nickel will be released using the revised 2011 procedure.
- The procedure for measuring the surface area has now been defined in greater detail to reduce variability, especially for complex articles.
- The standard also details that the 18 carat white gold nickel alloy ‘quality control sample’ (previously referred to as the ‘reference disc’) should only be tested once. The previous practice was to abrade the surface to remove the nickel depleted layer and re-use; this practice contributed to variability as it was very difficult to determine how much to remove and, therefore, the nickel depleted layer may not always have been entirely removed. This new practice should improve the reproducibility of quality control sample test results.
- The procedure for immersion of the test article in the synthetic sweat solution is defined in more detail with the objective of reducing variability.
- The method of calculating the nickel release (that is the calculation formula) is unchanged but NO adjustment factor (previously x0.1) is applied in the formula used in the 2011 standard.

Revised criteria:
The changes are based on the results of round robin testing by a number of laboratories. If a laboratory can show that the measure of uncertainty of its own results are different from those obtained by the round robin trial (46%) then it can set its own values for ‘non-compliance’, ‘compliance’ and values where ‘no decision’ is possible. For more information with regard to this calculation please see end of document.

In order to determine whether a tested article is compliant /non-compliant or falls into the ‘no decision category’, specific values, calculated using a 46% measurement of uncertainty have been provided in the 2011 standard.
As can be seen from the comparison table below, this means that results from some articles which were previously deemed to be compliant after the correction factor had been applied will now be “no decision” or non compliant.
COMPARISON OF ACTUAL RELEASE VALUES En 1811 1998 VS 2011

For articles that shall show compliance with the migration limit of 0.5 μg/cm²/week an article (assuming a measurement of uncertainty of 46%) will be deemed to be -

<table>
<thead>
<tr>
<th>Standard before correction factor of 0.1</th>
<th>Standard 2011</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Noncompliant</strong> when nickel release, μg/cm²/week is</td>
<td>&gt; 5.00</td>
</tr>
<tr>
<td><strong>Compliant</strong> when nickel release, μg/cm²/week is</td>
<td>≤ 5.00</td>
</tr>
<tr>
<td><strong>No decision</strong> when nickel release, μg/cm²/week is</td>
<td>Not applicable</td>
</tr>
</tbody>
</table>

For articles that shall show compliance with the migration limit of 0.2 μg/cm²/week an article (assuming a measurement of uncertainty of 46%) will be deemed to be -

<table>
<thead>
<tr>
<th>Standard before correction factor of 0.1</th>
<th>Standard 2011</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Noncompliant</strong> when nickel release, μg/cm²/week is</td>
<td>&gt; 2.00</td>
</tr>
<tr>
<td><strong>Compliant</strong> when nickel release, μg/cm²/week is</td>
<td>≤ 2.00</td>
</tr>
<tr>
<td><strong>No decision</strong> when nickel release, μg/cm²/week is</td>
<td>Not Applicable</td>
</tr>
</tbody>
</table>

UK Implementation of the 2011 standard:
BSI recognise that the supply chain will need some time to ensure goods comply with the new standard. They have therefore elected to delay the withdrawal of the British standard BS EN 1811:1998 until March 2013, or whenever the new standard, BS EN 1811:2011 becomes mandatory by its inclusion in the REACH Directive, whichever happens earlier. However, other EU member states may choose to withdraw BS EN 1811:1998 and introduce the revised standard immediately. This could cause confusion for those trading in several EU countries. For these companies and all those with average stock turn, complying with the revised 2011 standard is the safest option.

Spectacle Frames and Sunglasses:
Spectacle frames and sunglasses which were earlier included in the scope of EN 1811:1998 + A1:2008 standard are now excluded from the 2011 standard. The exemption was requested by the spectacle frame industry on the grounds that spectacle frames are medical devices that have complex shapes, different materials and coatings, all of which contribute to the uncertainty of measurement. Additionally, because coated articles have to be subjected to EN 12472 (corrosion and wear treatment phase) before the EN 1811 test, this creates further uncertainty. The spectacle frame industry has applied for a mandate to develop a more appropriate nickel release test for complex, coated spectacle frames and sunglasses. Until a more appropriate nickel release test for
such complex, coated spectacle frames and sunglasses is developed, EN 1811:1998 + A1:2008 has been republished unchanged as EN 16128:2011.

MORE INFORMATION FOR THOSE WHO ARE INTERESTED IN THE CALCULATION:

How the values detailed in the 2011 standard have been calculated:
The results of an inter-laboratory comparison were undertaken according to ISO 5725 which gave as an estimate for the combined measurement uncertainty a value of $u_{t,r} = 46\%$. The performance characteristic $u_{t,r}$ is related to the relative reproducibility’s of the surface area determination $s_{R,r}^2 (area)$ and the analytical determination of the nickel release $s_{R,r}^2 (Ni)$ by the following equation:

$$u_{t,r}^2 = s_{R,r}^2 (area) + s_{R,r}^2 (Ni)$$

In order to determine whether a tested article is non-compliant with its respective limit [i.e. 0.2 µg/cm²/week limit or 0.5 µg/cm²/week limit, respectively] a statistical test is applied. This test decides whether a determined nickel release value significantly exceeds its limit, it is one-sided and determines whether the lower boundary of the confidence interval at a significance level of 0.05, also known as guard band (*See Note*), is equal to, or larger than the limit. The following equations demonstrate how to calculate the values given in the revised standard

$$r_{\lim} \leq \bar{r}_{\text{meas}} \left(1 + k(\alpha) \cdot u_{t,r}\right)$$

$$r_{\lim} \geq \bar{r}_{\text{meas}} \cdot \left(1 + (k(\alpha) \cdot u_{t,r})\right)$$

Where

$k(\alpha)$ is the expansion factor for the chosen significance level/guard band (i.e. 0.05 [See Note*]) and the one sided t-test which gives a corresponding value of 1.65 for such significance level.

[Note: What significance level of 0.05 means*:- In statistical term it means that rate of false non compliant results is 5 % i.e. the probability of a false rejection is 5 %. Please note statistically a value of 1.65 x measurement uncertainty ($u_{t,r} = 46\%$) will give a probability of 5% false rejection (and a value of 2.33 x measurement uncertainty implies a probability of 1%).

$r_{\lim}$ correspond to the 0.2 µg/cm²/week limit (0.5 µg/cm²/week limit, respectively)

$\bar{r}_{\text{meas}}$ is the mean of the replicates of the nickel release determinations

$u_{t,r} = 46\%$.

The above equation shows the relationship between the measured value, the expanded measurement uncertainty and the limit values.

Example 1: when nickel release value is $\leq 0.2$ µg/cm²/week or $\geq 0.2$ µg/cm²/week (when correction factor of $x0.1$ is applied in the formula), then corresponding compliant / non-compliant value based on 46% uncertainty at significance level of 0.05 but in absence of correction factor of $x0.1$ is
\( r_{\text{lim}} \leq \frac{0.2}{(1 + 1.65 \times 46/100)} \) i.e. \( r_{\text{lim}} \leq 0.11 \mu g/cm^2/\text{week} \\
\( r_{\text{lim}} \geq \frac{0.2}{1 + 1.65 \times 46/100} \) i.e. \( r_{\text{lim}} \geq 0.35 \mu g/cm^2/\text{week} \)

The above example shows that when uncertainty of 46% is taken into account then for articles that shall show compliance with the migration limit of 0.2 \( \mu g/cm^2/\text{week} \) an article will be deemed to be compliant when the nickel release value is less than or equal to 0.11 \( \mu g/cm^2/\text{week} \). For nickel release values that are greater than 0.11 \( \mu g/cm^2/\text{week} \) but less than 0.35 \( \mu g/cm^2/\text{week} \) no clear decision is possible for compliance of this test article. The article will be treated non-compliant if nickel release values that are greater than or equal to 0.35 \( \mu g/cm^2/\text{week} \)

Example 2: when nickel release value is \( \leq 0.5 \mu g/cm^2/\text{week} \) or \( \geq 0.5 \mu g/cm^2/\text{week} \) (when correction factor of x0.1 is applied in the formula), then corresponding compliant / non-compliant value based on 46% uncertainty at significance level of 0.05 but in absence of correction factor of x0.1 is
\( r_{\text{lim}} \leq \frac{0.5}{1 + 1.65 \times 46/100} \) i.e. \( r_{\text{lim}} \leq 0.28 \mu g/cm^2/\text{week} \\
\( r_{\text{lim}} \geq 0.5 \times (1 + 1.65 \times 46/100) \) i.e. \( r_{\text{lim}} \geq 0.88 \mu g/cm^2/\text{week} \)