Characterization and Assessment of Dermal and Inhalable Nickel Exposures in Nickel Production and Primary User Industries

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The aim of this study was to measure the levels of nickel in the skin contaminant layer of workers involved in specific processes and tasks within the primary nickel production and primary nickel user industries. Dermal exposure samples were collected using moist wipes to recover surface contamination from defined areas of skin. These were analysed for soluble and insoluble nickel species. Personal samples of inhalable dust were also collected to determine the corresponding inhalable nickel exposures. The air samples were analysed for total inhalable dust and then for soluble, sulfidic, metallic, and oxidic nickel species. The workplace surveys were carried out in five different workplaces, including three nickel refineries, a stainless steel plant, and a powder metallurgy plant, all of which were located in Europe. Nickel refinery workers involved with electrolytic nickel recovery processes had soluble dermal nickel exposure of 0.34 μg cm⁻² [geometric mean (GM)] to the hands and forearms. The GM of soluble dermal nickel exposure for workers involved in packing nickel salts (nickel chloride hexahydrate, nickel sulphate hexahydrate, and nickel hydroxycarbonate) was 0.61 μg cm⁻². Refinery workers involved in packing nickel metal powders and end-user powder operatives in magnet production had the highest dermal exposure (GM = 2.59 μg cm⁻² soluble nickel). The hands, forearms, face, and neck of these workers all received greater dermal nickel exposure compared with the other jobs included in this study. The soluble nickel dermal exposures for stainless steel production workers were at or slightly above the limit of detection (0.02 μg cm⁻² soluble nickel). The highest inhalable nickel concentrations were observed for the workers involved in packing nickel powder (GM = 0.77 mg m⁻³), although the soluble component comprised only 2% of the total nickel content. The highest airborne soluble nickel exposures were associated with refineries using electrolytic processes for nickel recovery (GM = 0.04 mg m⁻³ total nickel, containing 82% soluble nickel) and those jobs involving contact with soluble nickel compounds (GM = 0.02 mg m⁻³ total nickel content, containing 76% soluble nickel). The stainless steel workers were exposed to low concentrations of relatively insoluble airborne nickel species (GM = 0.03 mg m⁻³ total nickel, containing 1% soluble nickel). A statistically significant correlation was observed between dermal exposures for all anatomical areas across all tasks. In addition, the dermal and inhalable (total) nickel exposures were similarly associated. Overall, dermal exposures to nickel, nickel compounds, and nickel alloys were relatively low. However, exposures were highly variable, which can be explained by the inconsistent use of personal protective equipment, varying working practices, and different standards of automation and engineering controls within each exposure category.

Keywords: dermal exposure; dermal exposure assessment; dermal exposure measurement; EASE; EU chemical regulation; exposure assessment for existing substances; inhalable dust; nickel

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INTRODUCTION

Industrial exposures to nickel and nickel compounds by inhalation, ingestion, and skin contact may occur in production plants making metallic nickel, nickel compounds, and nickel alloys as well as downstream use such as battery manufacturing and electroplating operations. Consumer exposure to nickel ions can occur through prolonged, intimate skin contact with high nickel-releasing items, e.g. some jewellery, buttons, and body piercing materials.

Nickel may cause allergic contact dermatitis in humans if there is sufficient dermal exposure (i.e. above a threshold level) to nickel ions. However, different types of nickel metal and nickel alloys release nickel ions at different rates, so it is important to consider the metallurgical composition of nickel-containing substances when evaluating exposure and risks (Flint, 1998; Robinson et al., 2000). It is widely recognized that the ability to release nickel ions is an important factor in the potential for a nickel-containing material to induce nickel sensitization or elicit an allergic response in nickel-allergic individuals (Ung and Furst, 1983; Lidén et al., 1996; Lidén and Carter, 2001). Consequently, the European Union (EU) Nickel Directive (94/27/EC, amended 2004/98/EC) (European Commission, 1994, 2004) has been enacted to decrease this source of consumer exposure in EU member states. EU regulations (CEC, 2004) now specify a maximum nickel release rate of 0.5 µg Ni cm⁻² week⁻¹ for products intended to be used in direct and prolonged contact with the skin (0.2 µg Ni cm⁻² week⁻¹ for items intended for skin piercing).

Nickel compounds have been categorized as ‘carcinogenic to humans’ (Group 1), on the basis of inhalation exposure risks, specifically for lung, nasal cavity, and paranasal sinuses, whereas metallic nickel and nickel alloys are categorized as ‘possibly carcinogenic to humans’ (Group 2B) for the same exposure route (IARC, 1990; DEPA, 2005; Straif et al., 2009).

The EU Existing Substances Regulations (CEC, 1993) required a high-level comprehensive evaluation of health risks relating to the production and use of nickel and several nickel compounds (i.e. nickel sulphate, nickel chloride, nickel nitrate, and nickel carbonate) within the European Community. This was done to identify if existing workplace controls were adequate or if further measures were necessary to control the health risks. The strategy and methods used for the occupational exposure assessment and overall risk assessment process is described in the Technical Guidance Document (ECB, 2003).

There are published data on inhalable nickel exposure from previous studies in nickel production and primary user industries (e.g. Kiilunen et al., 1997a,b; Werner et al., 1999) and a comprehensive review of independent and industry-funded airborne nickel exposure studies was prepared to aid setting occupational exposure limits in Europe (NiPERA, 1996). The presence of different airborne nickel species has also been reported in primary nickel production sites (e.g. Andersen et al., 1998; Vincent et al., 2001).

The availability of dermal exposure data has been limited to a few measurements from the electroplating industry (Bavazzano et al., 1994) and consumer studies (e.g. Bang Pedersen et al., 1974; Fournier and Govers, 2003), which are not of direct relevance to primary production industries. In addition, predicted exposures using the Estimation and Assessment of Substance Exposure (EASE) model (HSE, 1996) and so-called ‘analogous data’ obtained from the zinc industry (Hughson and Cherrie, 2005) produced very high estimates, which were considered to be unrealistic by industry stakeholders. A clear need was therefore identified for the collection of reliable dermal exposure measurements to inform the formal regulatory risk assessment.

The aim of this study was therefore to produce baseline dermal exposure levels for predefined processes/tasks for the main production processes and end-user applications where there was potential for dermal nickel exposure. This paper summarizes the development of a dermal exposure method and collection of workplace exposure samples from five different workplaces, including three nickel refineries producing a range of nickel metal products and nickel compounds; a stainless steel production plant, which used nickel briquettes and nickel cathode plates in the process; and a powder metallurgy facility using nickel powders to produce aluminium nickel cobalt (AlNiCo) magnets. Measurements of airborne nickel species were also collected to supplement the existing data set for this industry. The workplace surveys were carried out during 2004–2005 and all sites were within EU member states.

MATERIALS AND METHODS

Workplace descriptions

Exposure measurements were collected for several areas/tasks in each workplace. These are briefly described as follows:

- Nickel Refinery 1—produced nickel products by grinding nickel matte and then using chemical
leaching to produce nickel sulphate solution. Metallic nickel was recovered using an electrolytic process known as electro-winning, where nickel was deposited onto cathodes. The cathodes were cut into squares and then packed in drums. Nickel briquettes were manufactured by compressing and sintering nickel metal powder and the operators were tasked with packing these into bulk containers. Nickel sulphate hexahydrate and nickel hydroxycarbonate were produced using fully enclosed chemical processes. The nickel compounds were fed to individual storage silos and then dispensed to automatic bag filling stations for packing into 25- or 1000-kg bags.

- Nickel Refinery 2—produced nickel metal and nickel metal powder products using the Mond process, i.e. by production and decomposition of nickel carbonyl gas. Only the nickel powder-packing process was included in the survey.
- Nickel Refinery 3—produced nickel metal and nickel chloride hexahydrate crystals by recovering elemental nickel from nickel matte in a hydrometallurgical process. The leaching plant, electrolysis plant, and nickel chloride-packing areas were assessed. It should be noted that the electrolytic process at Refinery 3 liberated chlorine gas and was therefore more highly controlled than the electro-winning process at Refinery 1.
- Stainless steel plant—produced stainless steel from recycled stainless steel using nickel briquettes, nickel plate, and high nickel content alloy material to adjust the nickel content of the melt. The tasks assessed included alloy handling operators, raw material inspectors, and DC-arc technicians.
- Powder metallurgy facility—produced AlNiCo magnets. The rough pattern was formed by compression of the metal powder mixture. These were sintered in tunnel furnaces and then machined to size. The tasks assessed included nickel powder operators, hydraulic press setters, and grinding machine operators.

Most refinery tasks were highly automated and, in many instances, supervised or operated by a small number of individuals. Some manual tasks were undertaken, for example, cleaning, mechanical repairs to the production equipment, transferring products, and removing and tying bags during powder filling tasks. The electro-winning task at Refinery 1 involved significant manual involvement during removal of cathodes from the electrolysis tanks. A variety of ventilation, respiratory protective equipment (RPE) and personal protective equipment (PPE), and hygiene measures were used at the sites visited. In general, these were considered to be adequate, but there were instances where the operators did not always use the recommended RPE and PPE, including gloves.

Due to the high level of automation and the small number of workers available for sampling, it was necessary to collect repeat samples from some workers over consecutive days to obtain more measurements for each task.

Since the purpose of the study was to obtain measurements for specific process-task categories, the data were first grouped into seven specific categories, based on the type of process and the nickel-containing material being used. These were identified as follows:

**Refinery processes:**
- Front-end refinery processes (raw material handling/leaching)
- Electro-winning/electrolysis (nickel in solution)
- Packing solid nickel metal products (cutting nickel cathodes and packing nickel briquettes)
- Packing nickel compounds (soluble nickel salts)
- Packing nickel metal powders (insoluble nickel powder)
- Powder metallurgy (magnet production)
- Stainless steel production

**Sampling strategy**

The sampling method was designed to quantify the mass of nickel in the skin contaminant layer (Schneider et al., 1999). Furthermore, laboratory procedures were developed to separate soluble and insoluble nickel species from the exposure samples and thereby allow separate quantification for both dermal and inhalation exposure measurements. In the case of dermal exposures, this is because the risk of induction or elicitation of nickel sensitization is dependent on the skin absorption of the available free nickel ions. In the case of inhalation exposure, it is important to differentiate between soluble and insoluble nickel because soluble nickel compounds have been classified as respiratory sensitizers.

In most of the workplaces, there were high levels of automation, with a relatively small number of workers available for sampling. Therefore, all personnel working on the given shift on the processes of interest were invited to participate in the sampling survey. For this study, it was assumed that workers were clean as they reported for work. No attempt was made to assess the level of contamination on
the skin of workers before the beginning of the working shift or after washing up at the end of the day.

**Dermal sampling method**

Dermal exposure measurements were obtained using a removal method, i.e. samples were collected from the skin contaminant layer at predetermined anatomical locations. This followed the general strategy developed for dermal exposure assessment of zinc compounds as described in an earlier study (Hughson and Cherrie, 2005).

The dermal exposure samples were collected using commercial moist wipes using a template with an open aperture of 25 cm² pressed onto the skin. Each area of skin was wiped with three sequential wipes and a clean template was used on each occasion. For each subject, samples were collected on three separate occasions during the working day, i.e. before break times and at the end of the shift. Workers were instructed not to wash their hands until after the sampling was done. For each of these three sampling periods, the palms and backs of both hands and both forearms were sampled. At the last sampling period, additional samples were collected from the neck, face (perioral region), and chest (Table 1). It was not possible to use the template for sampling the perioral region, but this area was estimated to be equivalent to 25 cm². A total of 12 individual dermal samples and one field blank sample were collected for each worker per shift. The field blank was collected to check for adventitious contamination and comprised three wipes that were handled in the same way as the exposure samples but were not wiped over the workers’ skin.

**Analysis of dermal samples**

All wipe samples were transferred to the laboratory in 250-ml wide-neck glass jars. The wipes contained in each sample jar were analysed to determine the soluble and insoluble nickel content using a variation of a published method (Zatka et al., 1992). Firstly, the samples were treated with 0.1 M ammonium citrate to dissolve any soluble nickel species. The wipes in each sample container were covered with the ammonium citrate and left to soak for 3 h. The resultant solution was vacuum filtered through a 1-μm membrane filter and then made up to 100 ml using deionized water in a volumetric flask. The filtrate was therefore assumed to contain the soluble nickel fraction. Secondly, the filter and remainder of the wipes were covered with 10% nitric acid, heated to near boiling point for 3 h, cooled, vacuum filtered, rinsed and then made up to 100 ml volume. This portion was assumed to comprise the insoluble nickel fraction. It should be noted henceforth that the terms ‘soluble’ and ‘insoluble’ nickel refer to their solubility in 0.1 M ammonium citrate solution and solubility tests in other liquids may of course produce different results.

All dermal exposure samples were analysed for nickel by the Institute of Occupational Medicine (IOM) using inductively coupled plasma atomic emission spectroscopy in accordance with a documented in-house method, based on Occupational Safety and Health Administration (OSHA) method 121 (OSHA, 1991).

All sample masses were blank corrected by subtracting any nickel detected on laboratory blanks and field blanks. The analytical recovery was checked by analysing blank wipe samples spiked with known masses of nickel for each batch of samples analysed. A correction was therefore applied to exposures sample measurements to adjust for incomplete analytical recovery. All dermal exposure results are expressed as a skin surface loading (micrograms per square centimetre for both soluble and total nickel content). The total nickel content was derived from the sum of the soluble and insoluble nickel measurements.

The skin surface loading for each sample was calculated and an average of each set of three was calculated as an estimate of average daily exposure. A skin surface loading for the combined surfaces of the hands and forearms was also calculated for comparison with predicted exposures obtained from the

<table>
<thead>
<tr>
<th>Anatomical area</th>
<th>Sample period</th>
<th>No. of samples¹</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>First break</td>
<td>Mid-shift break</td>
</tr>
<tr>
<td>Palms of both hands</td>
<td>✔</td>
<td>✔</td>
</tr>
<tr>
<td>Backs of both hands</td>
<td>✔</td>
<td>✔</td>
</tr>
<tr>
<td>Forearms (left and right)</td>
<td>✔</td>
<td>✔</td>
</tr>
<tr>
<td>Neck</td>
<td>✔</td>
<td></td>
</tr>
<tr>
<td>Face (perioral region)</td>
<td>✔</td>
<td></td>
</tr>
<tr>
<td>Chest</td>
<td>✔</td>
<td></td>
</tr>
<tr>
<td>Total number of samples per subject</td>
<td></td>
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</tr>
</tbody>
</table>

¹Each sample comprised three wipes taken from each of the anatomical areas. Therefore, each of the first three samples contained six wipes.
EASE model (HSE, 1996), using the mean surface areas for hands (840 cm²) and the forearms (1140 cm²) (EPA, 1997). These surface areas were used in preference to the corresponding European Centre for Ecotoxicology and Toxicology of Chemicals values (Zaleski and Gephart, 2001) because they corresponded to the surface areas previously used in the EU risk assessment and Technical Guidance Document (ECB, 2003).

Dermal sampling method validation

Commercial wet wipes (Jeyes ‘Sticky Fingers’ Wet Ones) were previously evaluated as suitable for recovering samples of skin contaminant layer (Hughson and Cherrie, 2005) and were selected for consistency with the previous study. Tests were carried out to determine blank levels for varying numbers of wipes (one, three, and six wipes) and to establish a detection limit for the analytical method.

The analytical recovery was determined by preparing a number of spike samples. The spike levels were selected to correspond to surface contamination levels in the range 0.5–1000 µg cm⁻². Since the normal area of sampling was 25 cm², samples of nickel were prepared for ~12.5, 25, 250, and 2500 µg.

This was achieved in two ways. Firstly, spike samples were prepared by adding known amounts of nickel onto the surface of the wipes. This was done by weighing out quantities of nickel powder (INCO Type 255) onto plastic boats. The powder samples were removed from the boats using three consecutive wipes and then placed into a glass beaker. It proved difficult to weigh out nickel dust samples of <250 µg accurately and consistently, so the spike ranges for nickel powder were limited to 250 and 2500 µg spike levels.

Spike samples were also prepared using 10 µg ml⁻¹ stock solution of nickel sulphate hexahydrate (Sigma–Aldrich product No. 467901) in 10% nitric acid. The solutions was dispensed by pipette onto clean wipe samples and left overnight to stabilize. These were acid digested and analysed as previously described. All four spike levels (12.5, 25, 250, and 2500 µg) were tested in this way.

Additional tests were carried out to evaluate whether the amount of nickel taken up into the ammonium citrate solution from nickel powder was influenced by the time of treatment in the ammonium citrate. Spike samples were prepared using two different types of nickel powder (INCO Type 123 and 255 powder) and at two different spike levels (250 and 1000 µg). INCO Type 123 powder has a spherical particle morphology with mean particles size of ~8 µm, whereas the Type 255 powder has a filamentary structure with mean particle size of ~20 µm. The spike samples for each powder type were soaked in the ammonium citrate for 10 min, filtered, rinsed, and the filtrate and solid residues collected for analysis. This was repeated for soak times of 60, 180, and 720 min.

Tests were carried out to determine the dermal removal efficiency of the sampling method. This was done by applying pre-weighed quantities of nickel powder onto a pre-marked 100-cm² section of cured leather, which was intended to act as a surrogate for human skin. These tests were performed using sample masses at levels of 200, 500, and 1000 µg. The nickel powder sample was placed onto the section of surrogate skin and spread out across a defined surface area of 25 cm². The surface deposits were then removed using three successive wipes. Each wipe was stored and analysed separately in order to evaluate the relative efficiency of each wipe. The overall sampling efficiency was calculated as the ratio of total recovered nickel to the amount deposited. This procedure was repeated using a solution of nickel sulphate hexahydrate in solution applied to the surrogate skin surface using a pipette. The surface was allowed to dry for 3 h and the recovery tests were carried out as described above.

The background levels of nickel on the skin of 10 non-occupationally exposed volunteer subjects were assessed using the sampling methodology for the hands and forearms. This was carried out to assign baseline levels for non-occupationally exposed individuals. The volunteers were recruited from scientific and administrative staff from the IOM.

Sampling and analysis of inhalable dust and nickel compounds

Inhalable dust sampling was carried out using personal sampling apparatus in accordance with Health and Safety Executive method MDHS 14/3 (HSE, 2000). This involved using an IOM inhalable dust sampler loaded with a pre-weighed cassette containing a 25-mm quartz fibre filter. The quartz fibre filters used for the air sampling were chosen for compatibility with the analytical procedure, which is described in more detail by Zatka et al. (1992).

The sampling apparatus was fitted to the worker near the start of the working shift and left running for the majority of the working day so that the inhalation exposures could be considered to be representative of average full-shift exposures.

After sampling, the IOM cassettes were reweighed to determine the total inhalable dust concentration and the samples were then shipped to INCO Technical Services, Ltd, Mississauga, Ontario, Canada, for
analysis of soluble/insoluble nickel species using the method by Zatka et al. (1992). The speciation method is based on sequential leaching of the dust sample with reagents of increasing chemical power to release nickel ions into solution. Three selective leaches, utilizing in order of increasing power, ammonium citrate/citric acid solution, hydrogen peroxide–ammonium citrate, and bromine–methanol solutions, were followed by decomposition of the final leach residue with nitric and perchloric acids. The resulting four solutions were then analysed using atomic absorption spectrometry, providing measurements of four different nickel species: soluble nickel, sulfidic, metallic, and oxidic nickel, respectively. This enabled airborne measurements for each of the different nickel species to be obtained. Measurement of total airborne nickel exposure was calculated for each sample by taking the sum of the masses of the different nickel species.

Statistical analysis

Since the purpose of the survey was to identify exposure levels for certain job types and production processes, the exposure data were firstly grouped into specific process-task categories based on the type of nickel product being used and the industry sector being considered. The soluble and total nickel dermal exposure data for each process-task category were summarized in terms of the number of measurements (n), median, geometric mean (GM), geometric standard deviation (GSD), and the range (maximum and minimum values). The inhalation exposure data for each category were also summarized in this manner for total inhalable dust and total inhalable nickel. The data were tested for normality and log transformed where appropriate. Summary statistics were obtained using Microsoft Excel 2003 and box–whisker plots were produced using SigmaPlot version 11. Correlations between log-transformed dermal exposure and inhalable dust concentrations were investigated by calculating the Pearson correlation coefficient using SigmaPlot version 11. To aid the statistical analysis of the data, sample results that were below the limit of detection (LOD) were set to half the LOD, in accordance with the approach suggested by Rajan-Sithamparanadarajah et al. (2004).

RESULTS

Method validation

The LOD for nickel, based on analysis of the blank wipe samples, was calculated as 0.018 μg cm⁻² (assuming a sample area of 25 cm²) and was rounded to 0.02 μg cm⁻² for ease of expression.

The analytical recovery efficiency for the lowest spike level of 11.06 μg using nickel sulphate solution was in the range 41–53%, which although being relatively low was reasonably consistent. The recoveries improved at the higher spike levels, with the best recovery apparent at the 27.65 μg spike level (mean 120%). The recovery efficiency for nickel powder at spike levels 250 and 1000 μg was 98 and 93%, respectively. Overall, the analytical recovery across all spike ranges was an average of 80%. All field samples were therefore corrected for incomplete recovery by analysing spike samples prepared with each separate batch of field samples.

Treatment of nickel powders in the ammonium citrate solution produced ~5% soluble nickel from the total mass of nickel powder over all soak times (10, 60, 180, and 720 min) and at both spike levels (250 and 1000 μg). It was clear from this analysis that the length of the soak time had no effect on the recovery of soluble nickel species for the nickel powders tested. The recovery tests for the soluble and insoluble nickel fractions showed that the combined nickel recovery efficiency for the sample preparation and analysis was ~95% for the insoluble nickel sample and 87% for the soluble nickel.

The results of the sampling efficiency tests showed that the majority of the nickel powder deposits on the surrogate skin media were removed on application of the first wipe, with small remaining percentages being removed by the second and third wipes. The average recovery efficiencies for nickel powder and nickel in solution were 92 and 97%, respectively. This was considered to be acceptable and the field samples were left uncorrected for sampling efficiency.

Lastly, samples were collected from the non-occupationally exposed volunteers and the total nickel on the hands and forearms were calculated. Of the 10 subjects tested, four results were below the LOD for the method (0.02 μg cm⁻²). The results showed that the background nickel levels for the hands and forearms combined were in the range <0.02–0.07 μg cm⁻², with a GM of 0.02 μg cm⁻² and GSD of 1.8.

Workplace survey results

A total of 67 sets of dermal exposure measurements were collected from 52 different workers (15 workers were sampled twice on different days). A total of 1547 dermal exposure samples (excluding blanks) were collected for nickel analysis. When
analysed, 205 of these were less than the LOD of 0.02 μg cm\(^{-2}\). All samples were corrected for blank levels, field blanks, and for analytical recovery efficiency. The data were tested for normality and found to have a log-normal distribution. Log transformation was performed to allow summary statistics to be presented.

Although one of the study objectives was to collect a personal inhalable dust sample for each dermal exposure measurement, due to some workplace restrictions it was only possible to collect a total of 62 air samples from a possible total of 67.

The results of the Pearson correlation tests showed a high level of correlation (for all tasks) between the hands and forearms and the neck, face, and chest, respectively. All upper body exposure measurements (neck, face, and chest) were highly correlated with each other. The highest correlations were for the hands and face (\(r = 0.754\)), forearms and face (\(r = 0.732\)), and face and neck (\(r = 0.644\)). Except for the chest and forearm test, all \(P\) values were highly significant, with \(P < 0.001\) (Table 2).

When the dermal exposure data were compared with the inhalable dust concentrations, the correlations were not statistically significant. However, dermal exposure for all the anatomical areas was observed to be highly significantly associated with airborne nickel levels, with all \(P\) values < 0.001.

**Dermal exposure measurements**

The summary of dermal exposure data for the seven process-task categories are presented in Table 3, for each anatomical area sampled and for both soluble and total nickel content. Since the regulatory risk assessment for nickel and nickel compounds focuses on dermal exposure to the hands and forearms, these specific exposure data for soluble and total nickel species are illustrated for convenience in box-plot diagrams of Figs 1 and 2, respectively, for each task category. In these plots, the boundary of the box closest to zero indicates the 25th percentile, a line within the box marks the median, and the boundary of the box furthest from zero indicates the 75th percentile. Whiskers (error bars) above and below the box indicate the 90th and 10th percentiles. It should be noted that a minimum number of data points is required to compute each set of percentiles. At least three points are required to compute the 25th and 75th percentiles, five points to compute the 10th percentile, and six points to compute the 5th, 90th, and 95th percentiles. If SigmaPlot is unable to compute a percentile point, that particular point is not drawn. Solid dots beyond the error bars indicate outlying data points.

The workers involved with recovery of nickel by electro-winning/electrolysis processes had soluble nickel exposure of 0.34 μg cm\(^{-2}\) (GM) and total nickel dermal exposure of 0.56 μg cm\(^{-2}\) (GM) for the hands and forearms combined, illustrating that while the dermal exposures were fairly low, the proportion of soluble nickel to total nickel content in the skin contaminant layer was relatively high. These workers used PPE fairly consistently, both due to the risk of physical abrasion from sharp metal surfaces and to protect themselves from the corrosive properties of the process liquors.

The workers involved in packing water-soluble nickel compounds (nickel chloride hexahydrate, nickel sulphate hexahydrate, and nickel hydroxycarbonate) had marginally higher dermal exposures, having GM values of 0.61 μg cm\(^{-2}\) (GSD = 3.0) (soluble nickel) and 1.17 μg cm\(^{-2}\) (GSD = 4.0) (total nickel) for the hands/forearms combined. For this group of workers, the use of PPE was more

<table>
<thead>
<tr>
<th>Anatomical area/correlation test</th>
<th>Forearms</th>
<th>Neck</th>
<th>Face</th>
<th>Chest</th>
<th>Inhalable dust</th>
<th>Inhalable Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hands</td>
<td>0.766</td>
<td>0.567</td>
<td>0.754</td>
<td>0.548</td>
<td>−0.109</td>
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</tr>
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<td>0.401</td>
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<td>0.536</td>
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<td>0.464</td>
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<tr>
<td>Significance (two tailed) ((P))</td>
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<td>&lt;0.001</td>
<td>0.001</td>
<td>0.438</td>
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<tr>
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<td>Face</td>
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</tbody>
</table>
variable, which perhaps explains the greater variability in the measured data and the high number of outliers.

The highest overall dermal nickel exposures were recorded for workers involved with packing of nickel metal powder. The dermal nickel exposures for the hands/forearms of these workers were \( \text{GM} = 2.59 \mu g \text{ cm}^{-2} \) (soluble nickel) and \( \text{GM} = 8.73 \mu g \text{ cm}^{-2} \) (total nickel) with GSDs of 1.6 and 1.8, respectively.

This group of workers, together with those at the magnet production plant that handled nickel powders, had exposures to the face and neck regions, which were greater than those observed for most of the other process-task categories.

There was considerable variation in dermal exposure levels within the powder metallurgy workers, confirmed by high GSD (3.4–8.1). This is explained by the different type of jobs carried out by workers.

Table 3. Summary of dermal nickel exposures by exposure category and anatomical area sampled

<table>
<thead>
<tr>
<th>Task</th>
<th>Anatomical area</th>
<th>( n )</th>
<th>Soluble nickel (( \mu g \text{ cm}^{-2} ))</th>
<th>Total nickel (( \mu g \text{ cm}^{-2} ))</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Median</td>
<td>GM</td>
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<tr>
<td>Front-end refinery</td>
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<td>6</td>
<td>0.24</td>
<td>0.24</td>
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<tr>
<td></td>
<td>Neck</td>
<td>6</td>
<td>0.21</td>
<td>0.14</td>
</tr>
<tr>
<td></td>
<td>Face</td>
<td>6</td>
<td>0.62</td>
<td>0.70</td>
</tr>
<tr>
<td></td>
<td>Chest</td>
<td>6</td>
<td>0.24</td>
<td>0.15</td>
</tr>
<tr>
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<td>0.34</td>
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<tr>
<td></td>
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<tr>
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<tr>
<td></td>
<td>Chest</td>
<td>12</td>
<td>0.05</td>
<td>0.04</td>
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<tr>
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<td>7</td>
<td>0.26</td>
<td>0.27</td>
</tr>
<tr>
<td></td>
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<td>0.33</td>
<td>0.24</td>
</tr>
<tr>
<td></td>
<td>Face</td>
<td>7</td>
<td>0.70</td>
<td>0.55</td>
</tr>
<tr>
<td></td>
<td>Chest</td>
<td>7</td>
<td>0.04</td>
<td>0.05</td>
</tr>
<tr>
<td>Packing nickel compounds</td>
<td>Hands and forearms</td>
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<td>0.54</td>
<td>0.61</td>
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<td></td>
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<td></td>
<td>Face</td>
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<tr>
<td></td>
<td>Chest</td>
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<td>Powder metallurgy (magnet production)</td>
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<td>0.08</td>
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<tr>
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<td>Neck</td>
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<td>0.17</td>
<td>0.08</td>
</tr>
<tr>
<td></td>
<td>Face</td>
<td>8</td>
<td>0.17</td>
<td>0.18</td>
</tr>
<tr>
<td></td>
<td>Chest</td>
<td>8</td>
<td>0.10</td>
<td>0.08</td>
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<tr>
<td>Stainless steel production</td>
<td>Hands and forearms</td>
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<td>0.02</td>
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<tr>
<td></td>
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<td>0.04</td>
<td>0.03</td>
</tr>
<tr>
<td></td>
<td>Face</td>
<td>13</td>
<td>0.03</td>
<td>0.03</td>
</tr>
<tr>
<td></td>
<td>Chest</td>
<td>13</td>
<td>0.01</td>
<td>0.02</td>
</tr>
</tbody>
</table>

Notes: There was an incomplete set of measurements for one worker involved in nickel powder packing. This resulted in seven sets of hand/arm data and six measurements for face, neck, and chest, respectively. It is emphasized that the exposures for the hands and forearms are an average of measurements obtained from the palms and backs of both hands and both forearms. Also, the individual measurements of the hands and forearm exposures (not detailed here) are averages of the levels recorded at three periods over the working shift.
within this particular exposure category and the fact that many measurements were below the LOD. It was observed at the time of survey that workers involved in setting up the hydraulic presses had direct contact with nickel dust on machine surfaces, whereas workers involved in final processing of the magnets did not. Overall, the dermal nickel exposure for the hands/forearms for this group was \( GM = 0.08 \mu g \text{ cm}^{-2} \) (GSD = 3.4) (soluble nickel) and \( GM = 1.69 \mu g \text{ cm}^{-2} \) (GSD = 5.2) (total nickel).

The workers in stainless steel production who were potentially exposed to nickel had the lowest exposures overall \( [GM = <0.02 \mu g \text{ cm}^{-2} \) (GSD = 1.8) (soluble nickel) and \( GM = 0.11 \mu g \text{ cm}^{-2} \) (GSD = 2.7) (total nickel)]. Although these workers had potential for direct contact with nickel briquettes and nickel cathodes, as used by the refinery workers who produced them, the very low dermal exposures in comparison can be explained by the use of bulk loaders, grabs, and other mechanical handling methods in common use in the stainless steel industry.

Results of inhalable dust monitoring

These inhalation exposure measurements are detailed in Table 4 using the same exposure categories as the dermal exposure data. These are also illustrated in box-plot diagrams (Figs 3 and 4).

The results of the analysis of nickel species in the air samples are summarized in Table 5, where the quantity of soluble, sulfidic, metallic, and oxidic nickel is expressed as a percentage of the total nickel content in the airborne dust samples. The GM value is used to summarize the particular nickel species in each exposure category and corresponding GSD is included to illustrate the variability within each group.

As with the dermal exposures, the stainless steel workers had very low inhalable total nickel exposures with \( GM = 0.03 \text{ mg m}^{-3} \) (GSD = 2.3), although the total inhalable dust levels were the highest overall at \( GM = 3.9 \text{ mg m}^{-3} \) (GSD = 2.1). This confirms that while the process was generally dusty, the nickel content of the airborne dust was low.

The inhalable nickel exposures for the electrowinning/electrolysis workers gave a GM value of 0.04 mg m\(^{-3}\) with a GSD = 3.0. The relatively high variability within this group can be explained by low exposures occurring at Refinery 3 (0.01–0.02 mg m\(^{-3}\), \( n = 3 \)) compared to those at Refinery 1 (0.03–0.18 mg m\(^{-3}\), \( n = 9 \)). This was because different processes were used and primary containment at Refinery 1 was less efficient.

Inhalable nickel exposures in the powder metallurgy facility were also highly variable (\( GM = 0.05 \text{ mg m}^{-3} \), GSD = 3.9) for the same reasons of variability in the dermal exposures, i.e. some workers had direct exposure to nickel powders, whereas other did not.
The inhalable total nickel exposures for the nickel powder-packing operators at Refinery 2 were the highest with GM = 0.77 (GSD = 3.0). This is of course due to the high nickel content of the powder, the mobile nature of the powder products being used (i.e. small particle size), and its tendency to become airborne.

Considering the nickel content of the inhalable dust (Table 5), the exposures in the electro-winning, electrolysis processes and tasks involving packing of nickel compounds (nickel chloride, nickel sulphate hexahydrate, and nickel hydroxycarbonate) had the highest proportions of soluble nickel, with few other nickel species being present. The nickel powder packers and all the workers in the powder metallurgy plant were exposed mainly to metallic and other relatively insoluble nickel-containing species (e.g. oxidic nickel and nickel alloys). Workers involved with packing solid nickel metal products (cathode cutting operators and nickel briquette workers) were also exposed mainly to metallic and oxidic nickel species but also had some soluble nickel present. Workers in refinery front-end processes (leaching plant and raw material operators) had similar exposure profiles, characterized by significant levels of sulfidic nickel.

The exposure profiles for the stainless steel workers were quite different from all the other workers in this study. The most common nickel species present in this group was oxidic nickel. This is true for all the stainless steel workers regardless of task, which indicates that the main exposure determinant was the process dust within the plant itself.

**DISCUSSION**

The results of the method validation showed that the sampling procedure, sample preparation, and analytical methods produced relatively consistent results for recovery of soluble and insoluble nickel species from test samples. This is in line with a previous study using the same general method for zinc and zinc powder products (Hughson and Cherrie, 2005). When this method was used in the field, the amount of soluble nickel recovered compared to the total nickel was not always consistent, taking into account the physical properties of the nickel products in the workplace and the inhalation speciation data for these same areas. For example, considering the nickel powder workers in Refinery 2, approximately one-third of the total dermal exposure comprised soluble nickel. In contrast, the powder metallurgy workers had much lower soluble nickel exposure (~5%), even though the same powder product was used. Considering the results of the laboratory validation using the same nickel powder, the soluble nickel comprised only 5% of the total nickel recovered. Conversely, the soluble nickel levels for the workers exposed to nickel compounds were lower than expected, considering that these products are practically 100% water soluble. Also, some of the variability may be due to relatively low analytical recovery at levels approaching the LOD. These discrepancies indicate the need for further research to identify other factors that may potentially influence the sampling and analytical methods used. In particular, the efficacy of ammonium citrate solution as the desorption medium needs to be evaluated in more detail since recent reviews of the Zatka method on airborne dust samples suggests that ammonium citrate may also extract a portion of the less soluble nickel substances (Luk et al., 2000; Conard et al., 2008; Olle et al., 2009).

In examining the dermal exposure data together, there were some interesting associations between the nickel levels on different anatomical areas. As might be expected, the dermal nickel levels for the hands and forearms were strongly correlated, but there were also strong correlation between dermal exposure for all anatomical areas and airborne nickel levels. Previous studies have indicated the possibility of some association between inhalable levels of
workplace contaminants and dermal exposure levels, e.g. Vermeulen et al. (2000).

There was some variability in dermal exposure levels within-task categories, which is consistent with other dermal exposure studies, e.g. Kromhout et al. (2004). The variability may be caused by a number of workplace factors, including the intermittent and inconsistent use of protective clothing and variation in working practices and standards of engineering controls within exposure categories. Furthermore, a significant number of samples were below the LOD, and while a large number of samples were collected, these translate into relatively small amount of measurements for each exposure category. The results from the study overall make it difficult to study within-worker and within-task variability and so this has not been done.

Initial drafts of the EU risk assessments used dermal exposure estimates obtained from the EASE model (HSE, 1996) and so-called ‘analogous data’ obtained from the zinc industry (Hughson and Cherrie, 2005). This preliminary assessment strategy produced a dermal nickel exposure estimate of 1400 µg cm\(^{-2}\) for workers involved in refinery processes and it was recognized that the assumptions inherent in this approach were probably not valid. Indeed, the preliminary exposure estimate was far in excess of the highest nickel exposures recorded in this study. The only other sources of measured dermal nickel exposures were from studies of nickel release from coins (Bang Pedersen et al., 1974; Fournier and Gover, 2003). The data from these studies suggested that exposures for this low-level exposure scenario could be \(~\)200 µg over the area of both hands. By

![Fig. 3. Inhalable dust exposures by work process/task.](image)

![Fig. 4. Inhalable nickel exposures by work process/task.](image)

Table 5. Airborne nickel species by process area

<table>
<thead>
<tr>
<th>Process/task</th>
<th>n</th>
<th>Airborne nickel species (percentage of total nickel content)</th>
<th>GM (and GSD) values</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Soluble Sulfidic Metallic Oxidic</td>
<td></td>
</tr>
<tr>
<td>Front-end refinery processes</td>
<td>6</td>
<td>25 (1.6) 44 (1.5) 3 (6.8)</td>
<td>13 (3.1)</td>
</tr>
<tr>
<td>Electro-winning/electrolysis</td>
<td>12</td>
<td>82 (1.5) 1 (8.7) &lt;1 (6.3)</td>
<td>1 (9.5)</td>
</tr>
<tr>
<td>Packing nickel metal products</td>
<td>7</td>
<td>21 (1.5) 1 (7.0) 30 (1.2)</td>
<td>41 (1.4)</td>
</tr>
<tr>
<td>Packing nickel compounds</td>
<td>12</td>
<td>76 (1.3) 2 (12) &lt;1 (1.0)</td>
<td>3 (12)</td>
</tr>
<tr>
<td>Packing nickel powders</td>
<td>7</td>
<td>2 (2.3) 1 (2.1) 33 (1.5)</td>
<td>60 (1.3)</td>
</tr>
<tr>
<td>Powder metallurgy (magnet production)</td>
<td>8</td>
<td>1 (10) 3 (6.3) 42 (2.1)</td>
<td>35 (1.4)</td>
</tr>
<tr>
<td>Stainless steel production</td>
<td>10</td>
<td>1 (10) 1 (8.8) &lt;1 (7.2)</td>
<td>89 (1.1)</td>
</tr>
</tbody>
</table>
extrapolation, this could imply a surface skin loading of 0.14 μg cm\(^{-2}\). This is at the lower level of some of the industrial exposure measurements reported in this study. Clearly, the results of our study have provided useful exposure measurements to enable more robust regulatory risk assessments to be performed for nickel and nickel products.

It is possible to compare the present data set with the 0.3 μg cm\(^{-2}\) suggested empirical no observed effect level (NOEL) for elicitation of nickel dermatitis in nickel-sensitive individuals (DEPA, 2005 based on Uter et al., 1995). This value was derived based on occluded, patch testing with nickel sulphate; dermal reactions were read after 24–48 h after removal of the patches. The soluble nickel exposure for the nickel powder-packing task was well in excess of the NOEL value with over half of the workers surveyed in this process-task having average hand/forearm dermal exposures above this threshold. In view of the low number of occupation-related cases of nickel dermatitis among metal workers (Shum et al., 2003), it is perhaps likely that the overall absorbed dose is less than that suggested by the measured exposure levels. This may be because these industrial exposures are less concentrated and lack the same skin occlusion and duration of contact, compared to, say, a nickel-plated button or jewellery in direct contact with the skin. These particular factors are among those previously identified as affecting uptake and hence influencing the risks of dermatitis (Robinson et al., 2000).

In addition, the threshold NOEL used in the risk assessment was based on a level for elicitation of sensitization reactions in nickel-sensitive subjects. The threshold for induction of nickel sensitivity is expected to be higher than the threshold for elicitation since this is generally true for dermal sensitizers (United Nations, 2007) and the induction threshold may not be exceeded by the exposure levels noted in this study. The EU risk assessments of nickel, nickel sulphate, nickel carbonate, and nickel chloride used the data collected in this study to characterize the risk for dermal nickel sensitization and concluded that there is no need for further risk reduction, testing, or risk reduction beyond existing measures. This was because the threshold value is based on prolonged (48-h) contact under occlusion, which is overly conservative for workplace exposure.

Analysis of airborne dust and nickel exposures has helped with interpretation of dermal exposure measurements and also provided additional information regarding the nickel species present in the included task-process categories. Referring to Fig. 4, it is clear that the majority of airborne nickel exposures were below the UK workplace exposure limit (WEL) of 0.5 mg m\(^{-3}\) for nickel and water-insoluble nickel compounds (HSE, 2005). The task of greatest concern is that of nickel powder packing in Refinery 2 where exposures were in the range 0.13–2.81 mg m\(^{-3}\) (total nickel). These data compare quite well with some historical exposure data of nickel powder packing (NiPERA, 1996), indicating that further effort is required to reduce airborne nickel levels for this process. These results indicate that the mechanisms of exposure are related to the mobility of the powder and the difficulty of applying effective controls where the tasks require manual intervention.

In contrast, dermal and inhalation exposures were controlled to a higher standard for nickel compound packing. In this case, it is appropriate to compare the results with the WEL of 0.1 mg m\(^{-3}\) for water-soluble nickel compounds (HSE, 2005) and it is clear that the majority of inhalable nickel exposures for nickel compounds (nickel chloride, nickel sulphate hexahydrate, and nickel hydroxycarbonate) were less than the WEL. This high level of control was largely due to the use of automatic handling equipment in this process and is supported by previous airborne nickel exposure studies (NiPERA, 1996).

The measured exposures for total inhalable nickel for the electro-winning process in Refinery 1 were in the range 0.03–0.18 mg m\(^{-3}\) (comprising ~80% soluble nickel) and included several excursions above the 0.1 mg m\(^{-3}\) UK limit. These measurements are similar to previous data for this process reported by Kiilunen et al. (1997b), although the data cannot be directly compared due to the different monitoring strategies used. However, it does suggest the need for further improvements to prevent emission of nickel aerosol into the workplace.

For the remaining processes, it is necessary to consider the presence of soluble nickel and other species in the workplace air. The analysis detailed in Table 5 confirms the very low content of soluble nickel in nickel powder packing, powder metallurgy, and stainless steel production and also the high levels of soluble nickel in the electro-winning and nickel compound-packing processes. The front-end refinery processes was characterized by the presence of ~25% soluble nickel in the total airborne nickel. By applying a factor of 0.25 to the measured inhalable nickel exposures, it can be shown that all samples were ≤0.1 mg m\(^{-3}\) WEL. A similar analysis for the packing of solid nickel products (21% soluble nickel) indicated that several measurements exceeded the 0.1 mg m\(^{-3}\) limit.
The speciation of the airborne nickel exposure samples shows a high degree of variability as indicated by the GSD values in certain categories. This is largely due to many values at or below the LOD and also a relatively small sample size in some cases. Workers in refinery front-end processes (leaching plant and raw material operators) had similar exposure profiles, characterized by significant levels of sulfidic nickel. This is due to the high sulphur content of the raw materials used in the nickel production process. This finding corresponds to that of Vincent et al. (2001) and Creely and Aitken (2008), where the authors studied nickel species in the upstream side of nickel refining processes, i.e. from raw materials handling, nickel matte crushing, and grinding through to roasting in the smelters.

Also there are detectable levels of oxidic nickel, which is unexpected considering the processes involved. This may be because the Zatka method speciates by solubility, with oxidic nickel being the most insoluble nickel species. The assumption is made that all the nickel substances would fall into one of the four major categories and that these substances would be dissolved based on increasing strength of leaching solution with the following order: (i) soluble, (ii) sulfidic, and (iii) metallic. Nickel that remains undissolved after the third leach is considered to be oxidic nickel. However, the review by Luk et al. (2000) suggests that a portion of the oxidic nickel in the speciation assay may actually be nickel alloy, due to the slower rate of nickel ion dissolution when present in combination with other metals.

As noted in the sampling strategy, no attempt was made to assess the levels of ‘take-home’ nickel contamination, so it is conceivable that some of the dermal samples contained nickel that was originally deposited on previous working shifts. Since the aim of the study was to investigate the levels of nickel on the skin, this does not invalidate our results, but it would be an interesting exercise to utilize this sampling method to evaluate the efficiency of washing and other decontamination procedures on site.

CONCLUSIONS

While there may be some discrepancies in the sample results, the survey has produced measurements of dermal nickel exposures in the primary production and primary user industry where there were only very limited existing data.

Overall, this study has shown that the highest nickel exposures occurred in nickel powder packing, due in part to the highly mobile nature of the product. Some additional attention will be required to prevent emission to the workplace, such as better use of ventilation and possibly robotic or other automated handling equipment. Recovery of nickel by electrolytic processes resulted in both dermal and inhalation exposures to soluble nickel compounds. Our study has shown that further improvements are required to control emission of nickel aerosol to the workplace in one of the two refineries included in the survey, where the local exhaust ventilation was not particularly effective.

The results show that the industrial exposures were certainly numerically higher than previous measurements of consumer exposure, but were much lower than exposure predictions from the EASE model and analogous data sets from the zinc industry. This indicates the need for exposure measurements in regulatory risk assessments and demonstrates that modelling, although a cheap and convenient approach, may not generate exposure predictions of sufficient quality to satisfy all stakeholders.

The dermal nickel exposures were highly variable, and it is concluded that this was largely due to differences in observed working practices, variable use of PPE, and application of varying standards of engineering controls within the exposure categories. It is also clearly demonstrated that the most effective control measure for reducing both dermal and inhalation exposures is the use of automatic, remote materials handling processes, particularly for substances that are mobile and prone to produce airborne dust.

Generally, the results showed that it is not just the hands and forearms that become exposed. Other areas including the face, neck, and chest had measurable levels of nickel substances, which is likely to be due to deposition of nickel from the air or from touching of the neck and face areas with contaminated hands or gloves. This is supported by the statistically significant relationship between airborne nickel and dermal exposures and the relationship between exposures for all anatomical areas. The pattern of dermal nickel deposition on the hands and face combined with observed working practices indicates that there is potential for inadvertent ingestion of nickel and nickel compounds. In order to better understand the significance of this route of exposure, further research is needed.

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REFERENCES


