Respiratory Exposure to Components of Water-Miscible Metalworking Fluids

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Water-miscible metalworking fluids (MWFs) are capable of causing respiratory symptoms and diseases. Recently, much emphasis has been put on developing new methods for assessing respiratory exposure to MWF emulsions. The air concentrations of ingredients and contaminants of MWF and inhalable dust were measured in 10 metal workshops in southern Finland. Oil mist was determined by infra red spectroscopy analysis after tetrachloroethylene extraction from the filter. Aldehydes were collected on Sep-Pak chemosorbents and analysed by liquid chromatography. Volatile organic compounds (VOCs) were collected on Tenax adsorbents and analysed by gas chromatography with mass spectrometric detection after thermal desorption. Endotoxins were collected on glass fibre filter and analysed by enzyme-based spectrophotometry, and viable microbes were collected on polycarbonate filter and cultured. Inhalable dust was collected on cellulose acetate filter and quantified gravimetrically. Associations between the different exposures were calculated with Spearman’s correlations. The mean concentration of oil mist was 0.14 (range <0.010–0.60) mg m⁻³. The mean total concentration of aldehydes was 0.095 (0.026–0.38) mg m⁻³, with formaldehyde as the main aldehyde. The average total concentration of VOC was 1.9 (0.34–4.5) mg m⁻³ consisting mainly of high-boiling aliphatic hydrocarbons. Several potential sensitizing chemicals such as terpenes were found in small quantities. The concentration of microbial contaminants was low. All the measured air concentrations were below the Finnish occupational exposure limits. The exposure in machine shops was quantitatively dominated by volatile compounds. Additional measurements of MWF components such as aldehydes, alkanolamines and VOCs are needed to get more information on the chemical composition of workshops’ air. New air cleaning methods should be introduced, as oil mist separators are insufficient to clean the air of small molecular impurities.

Keywords: asthma; exposure; formaldehyde; metalworking fluid; volatile organic compounds

INTRODUCTION

Water-miscible metalworking fluids (MWFs) are complex chemical mixtures consisting of petroleum oil, vegetable oil or a synthetic lubricating component and various auxiliary substances such as emulsifiers, corrosion inhibitors, extreme pressure agents, antioxidants and preservatives (NIOSH, 1998). Fluids are mixed with water to form 2–10% emulsions, which are used for cooling and lubricating the metalworking process as well as for removing metal chippings formed in machining. The chemical and physical nature of the MWF emulsions as well as their environmental contaminants, such as leaking machine oils and bacteria, make the chemistry of the fluids even more complex, as these factors enable the fluid composition to change over time.

Ingredients of MWF are well-known causes of dermatitis in machinists (Geier et al., 2004; Suuronen et al., 2007a). Epidemiological and clinical studies suggest that both chemical ingredients and microbiological contaminants in MWF may also cause various respiratory symptoms and diseases (Gordon, 2004). The reported respiratory health effects have been e.g. irritation of upper respiratory tract and eyes (Rosenman...
et al., 1997), allergic alveolitis (Gupta and Rosenman, 2006), chronic bronchitis, changes in pulmonary function and asthma (Greaves et al., 1997; Rosenman et al., 1997; Zacharisen et al., 1998) and the symptoms have been abundant even in an environment rated as fairly clean according to occupational exposure limits (OELs) (Robertson et al., 2007). In a recent Finnish study, various upper and lower respiratory symptoms were very frequent (M. Jaakkola, K. Suuronen et al., submitted for publication), although according to the Finnish Register of Occupational Diseases, incidence of occupational asthma (OA) in machinists has been about the same as in total working population (Suuronen et al., 2007a). The specific causative factor of OA has been identified in only a few cases, the reported causatives including pine oil odorant and colophony (Henry et al., 1985) and alkanolamines (Savonius et al., 1994; Pipari et al., 1998).

Respiratory exposure to both MWF emulsions and straight oils has been assessed with a variety of methods, a common one being mineral oil mist collection on a filter followed by solvent extraction and infra red spectroscopy (IR) analysis (NIOSH, 1996). It has been discussed widely that the traditional methods for oil mist collection are not sufficient when assessing exposure to water-miscible MWF. In the US, thoracic particulate, quantified with a gravimetric analysis, has been recommended as the measure of MWF exposure (NIOSH, 1998; Ross et al., 2004). However, gravimetric analysis alone is not able to differentiate soluble MWF from solid particulate originating in other sources than MWF, and thus new methods for determining all classes of MWF have emerged. The American Society for Testing and Materials (ASTM) method P-42-97 includes collection of air samples on polytetrafluoroethylene filter followed by gravimetric analysis and extraction with a ternary solvent blend (Glaser et al., 2003). In National Institute for Occupational Safety and Health (NIOSH) method 5524, an additional binary blend of methanol and water is used to enhance the removal of water-soluble components (NIOSH, 2003). Other methods include, e.g. measurement of boron or potassium (HSE, 2003) as markers of MWF. We have recently also shown that exposure to MWFs can be assessed by measuring alkanolamines (Henriks-Eckerman et al., 2007). Collecting the volatile component of the oil mist with, e.g., sorbent traps subsequent to filters has been reported in the laboratory (Simpson, 2003), but field investigations remain scarce (Woskie et al., 2003).

Most of the current methods used to assess exposure to MWF measure either the total oil component of MWF or the total MWF aerosol. Recently, we have also investigated respiratory symptoms in relation to total aerosols in metal workshops using a real-time aerosol photometer (M. Jaakkola, K. Suuronen et al., submitted for publication). Little is known about respiratory exposure for instance to the volatile constituents of MWF, and the need for new methods and strategies for assessing exposure to the airborne components of water-miscible MWF is evident. The purpose of this study was to measure small molecular weight ingredients as well as microbial impurities of MWFs capable of causing skin and respiratory sensitization and irritation. The section of this study presenting respiratory and skin exposure assessment to alkanolamines has recently been reported in detail elsewhere (Henriks-Eckerman et al., 2007).

**MATERIALS AND METHODS**

**Workplaces**

Exposure measurements were carried out in 10 metal workshops in southern Finland during the year 2004. The companies were selected from a pool of 60 metal workshops where machining was one of the main activities. In all 60 companies, an assessment of total aerosol exposure and a questionnaire concerning respiratory and skin symptoms was carried out (Suuronen et al., 2007b; M. Jaakkola, K. Suuronen et al., submitted for publication). Subsequently, the 10 workshops included in this analysis were chosen to represent different types of companies based on the MWFs, products and raw materials as well as the number of machinists in the workshop.

The 10 companies chosen for the present study were involved in different kinds of machining including the manufacturing of tools and bodies and parts for machines and vehicles. The most common processes were turning, grinding and milling. Both manual and computer numerical control machines were used, and in one machine shop, a fully automated machining centre was also used. The number of machinists in the workshops varied from 10 to 100, whereas the number of the machines ranged from 5 to 70. All the 17 MWFs observed were water miscible. Safety data sheets (SDSs) were available for 16 MWFs, and according to them, 10 (59%) were mineral oil-based soluble oils or semi-synthetic MWFs; 2 (12%) were vegetable oil-based semi-synthetic and 4 (24%) were synthetic MWFs. In seven of the 10 workshops, more than one MWF was used; the number of MWFs per workshop was one to three. One of the semi-synthetic MWFs was designed to be used without any preservatives, and another one did not contain any alkanolamines. Typically, MWFs were changed every 6–12 months, but the observed fluids’ age still varied from 1 week to almost 3 years. Ventilation measures and use of enclosures varied in the workshops, and some of the local exhaust equipment were found ineffective. Overall standard of exposure control was nevertheless found to be reasonably good throughout the companies.

**Sampling strategy**

Airborne exposure to the components of MWF was determined by personal sampling in the
breathing zone of the machinists and with stationary sampling. The workers were chosen so that different parts and processes in the workshops were evenly represented. Also, the machines producing the highest concentration of total aerosol, as measured with a real-time aerosol photometer (Data-Ram, MIE Inc., Bedford, MA, USA), were included. The number of workers with personal air sampling at each workshop was three to six, depending on the total number of machinists and machines. A maximum of three samplers were attached to the workers simultaneously. Personal air samples of volatile organic compounds (VOCs), aldehydes, endotoxins, alkanolamines (Henriks-Eckerman et al., 2007) and inhalable dust were collected from the breathing zone of the workers. A total of 42 breathing zone samples were taken for each substance. The VOCs were collected on Tenax adsorbents. Thermal desorption was used to increase the sensitivity of the analytical method compared to conventional methods with liquid desorption. Oil mist and microbes were measured from the breathing zone of the workers in the first three workshops and in the remaining seven workshops from one stationary site. The total number of samples was 21 for oil mist and 21 for microbes.

**Sampling and analytical methods**

The methods for air sampling and analysis (SFS, 1976; Palmgren et al., 1986; NIOSH, 1996; US Environmental Protection Agency, 1999; CEN, 2002; ISO, 2004) are presented in Table 1. The sampling pumps were calibrated with the relevant samplers for accurate flow rates prior to the measurements. The flow rates were checked for accuracy after sampling back in the laboratory.

**Statistical analysis**

Spearman’s correlations were used when studying associations between the exposure variables, namely oil mist, dust, endotoxins, total aldehydes, formaldehyde, total VOCs and alkanolamines (Henriks-Eckerman et al., 2007). A P-value of <0.05 was considered to indicate statistical significance.

**RESULTS**

The mean concentration of mineral oil mist in 10 metal workshops was 0.14 (range <0.010–0.60) mg m⁻³. All the oil mist concentrations were well below the Finnish OEL (5.0 mg m⁻³), and in all machine shops except for one, the oil mist concentrations were <0.2 mg m⁻³, as recommended by the American Conference of Governmental Industrial Hygienists (ACGIH, 2005). The average concentration of inhalable dust was 0.78 (<0.14–2.0) mg m⁻³, which is small as compared to the Finnish OELs for both inorganic and organic dust (10 and 5.0 mg m⁻³, respectively). In two of the 10 workshops, inhalable dust was the main impurity. The average air concentrations of the measured impurities in each workshop are presented in Fig. 1. The air concentrations of alkanolamines reported earlier

Table 1. Sampling and analysis methods of air samples in 10 metal workshops

<table>
<thead>
<tr>
<th>The measured substance</th>
<th>Number of samples</th>
<th>Sampling time</th>
<th>Approximate duration of sampling (h)</th>
<th>Approximate air flow (l min⁻¹)</th>
<th>Sampling method/collection medium</th>
<th>Analysis method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Breathing zone samples</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>VOCs</td>
<td>42</td>
<td>A</td>
<td>1</td>
<td>0.1</td>
<td>Tenax sorbent tube</td>
<td>Thermodesorption, gas chromatography–mass spectrometry (ISO, 2004)</td>
</tr>
<tr>
<td>Aldehydes</td>
<td>42</td>
<td>M</td>
<td>2</td>
<td>1</td>
<td>Sep-Pak collector</td>
<td>Liquid chromatography–mass spectrometry (US Environmental Protection Agency, 1999)</td>
</tr>
<tr>
<td>Endotoxins</td>
<td>42</td>
<td>A</td>
<td>2</td>
<td>2</td>
<td>IOM cartridge/glass fibre filter</td>
<td>Enzyme-based spectrophotometry, BIOWhittager-QCL (CEN, 2002)</td>
</tr>
<tr>
<td>Stationary samplesᵃ</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Oil mist</td>
<td>21</td>
<td>A</td>
<td>4–6</td>
<td>2</td>
<td>IOM cartridge/tetlon or glass fibre filter</td>
<td>Extraction to tetrachloromethane, IR (NIOSH, 1996)</td>
</tr>
<tr>
<td>Microbes</td>
<td>21</td>
<td>A</td>
<td>2</td>
<td>2</td>
<td>Camnea cartridge/polycarbonate filter</td>
<td>Cell culture, microscopy (Palmgren et al., 1986)</td>
</tr>
</tbody>
</table>

ᵃPersonal breathing zone samples from three workplaces and stationary samples from seven workplaces.
Henriks-Eckerman et al., 2007) are also included in Fig. 1 for comparison. The mean concentrations of viable bacteria and endotoxins were 120 (50–220) colony forming unit (CFU) m\(^{-3}\) and 18 (<1.3–290) endotoxin unit (EU) m\(^{-3}\), respectively. Fungi were found in all workshops in small amounts [average concentration 550 (<100–1600) CFU m\(^{-3}\)], and in four samples, the Finnish reference value for homes and offices of 500 CFU m\(^{-3}\) was exceeded (Reponen et al., 1992). Endotoxin concentrations were generally below the Dutch health-based exposure limit of 50 EU m\(^{-3}\), but in one endotoxin sample, the concentration was clearly higher, exceeding the Dutch legal limit of 200 EU m\(^{-3}\) (Douwes et al., 2003).

The air concentrations of aldehydes (42 samples) other than those in the VOCs are presented in Table 2. The mean concentration of total aldehydes was 0.095 (0.026–0.38) mg m\(^{-3}\). The main aldehyde, formaldehyde, constituted about half of the total aldehyde concentration, and its mean concentration was \(\sim11\%\) of the Finnish OEL of 0.370 mg m\(^{-3}\).

The concentrations of VOCs (42 samples) are presented in Table 3. Total VOCs formed the main fraction of all airborne impurities in eight of the 10 metal workshops (Fig. 1). The mean concentration of total VOCs was 1.9 (0.34–4.5) mg m\(^{-3}\). The main components were different high-boiling (150–330°C) hydrocarbons. Aromatic hydrocarbons consisted of, e.g., xylene and toluene. In organic nitrogen compounds, 5-methyl-oxazolidine, 3-methyl-oxazolidine, morpholine and morpholineborane were identified. Although the collection and analysis method used in the present study is not designed for collecting

<table>
<thead>
<tr>
<th>Company ID</th>
<th>Alkanol amines</th>
<th>VOC</th>
<th>Aldehydes total</th>
<th>Oil mist</th>
<th>Dust</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td></td>
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**Fig. 1.** Average air concentrations of dust, oil mist, aldehydes, VOCs and alkanolamines in 10 metal workshops.

**Table 2.** Average air concentrations of the identified aldehydes in 10 metal workshops collected in Sep-Pak chomsorbent (42 samples from the breathing zone)

| Aldehydes                      | Mean concentration (range), mg m\(^{-3}\) | Finnish OEL for 15 min/8 h, mg m\(^{-3}\) 
<table>
<thead>
<tr>
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<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Formaldehyde</td>
<td>0.040 (0.011–0.150)</td>
<td>1.2/0.370</td>
</tr>
<tr>
<td>Acetaldehyde</td>
<td>0.026 (0.0031–0.21)</td>
<td>46/None</td>
</tr>
<tr>
<td>Valeraldehyde (pentanal)</td>
<td>0.0096 (0.00040–0.14)</td>
<td>110/None</td>
</tr>
<tr>
<td>Methylbenzaldehyde</td>
<td>0.0081 (0.00040–0.074)</td>
<td>None</td>
</tr>
<tr>
<td>Hexanal</td>
<td>0.0044 (0.0011–0.0079)</td>
<td>None</td>
</tr>
<tr>
<td>Propionaldehyde</td>
<td>0.0022 (0.00050–0.0075)</td>
<td>None</td>
</tr>
<tr>
<td>Glyoxal (etandial) (n = 38)</td>
<td>0.0015 (0.00050–0.0054)</td>
<td>None</td>
</tr>
<tr>
<td>Benzaldehyde</td>
<td>0.0013 (0.00040–0.0041)</td>
<td>None</td>
</tr>
<tr>
<td>Butanal/isobutanal</td>
<td>0.0011 (0.00040–0.0031)</td>
<td>None</td>
</tr>
<tr>
<td>Total aldehydes</td>
<td>0.095 (0.026–0.38)</td>
<td>None</td>
</tr>
</tbody>
</table>

\(n = \) number of positive samples, if not identified in all samples.
In Spearman’s correlation test, statistically significant associations were found between (i) oil mist and alkanolamines, (ii) formaldehyde and alkanolamines, (iii) total aldehydes and alkanolamines and (iv) total aldehydes and the VOCs. The correlation coefficients ($r$) were $0.463$ (P-value $0.002$), $0.776$ ($<0.001$), $0.432$ ($0.005$) and $0.365$ ($0.017$), respectively.

**DISCUSSION**

In the present study, several small molecular weight chemicals were measured in the air of 10 metal workshops. The companies were of different sizes, and various machining techniques and MWFs were used. The overall standard of exposure varied in the companies as there were different degrees of local ventilation and MWF maintenance. However, in most of the workshops adequate care was taken to control exposure, and serious defaults were not observed. The concentrations of dusts, oil mist and bacteria were generally small when compared to the present Finnish OELs.

Mineral oil mist has traditionally been measured in machine shops in Finland. In the present study, the concentration of extractable oil mist was well below the Finnish OELs in all the samples. The highest concentration, $0.60 \text{ mg m}^{-3}$, was measured from a process with several open-face grinders connected to a central MWF system without local ventilation. In most of the samples, the concentration was also below the recommendation of the ACGIH (2005). A guidance value for mineral oil mist of $3 \text{ mg m}^{-3}$ has been used in the UK as a target value for good industrial practice. The guidance value for water-miscible MWF has been $1 \text{ mg m}^{-3}$, but it was recently withdrawn due to a multitude of asthma and allergic alveolitis cases observed in concentrations below it and considered to be caused by microbial impurities (Robertson et al., 2007). The average concentration of inhalable dust exceeded the NIOSH recommended exposure limit for total particulate mass ($0.50 \text{ mg m}^{-3}$). The value is compatible with recently reported concentrations in machining industries in North America (Abrams et al., 2000; Piacitelli et al., 2001).

Inhalable dust measurement is likely to represent total inhalable aerosol (oil mist and dust) rather than just dust, as also oil droplets may retain on the filter.

In our study, the levels of viable bacteria and endotoxins were generally lower than in some North American studies, where viable bacteria were reported in concentrations up to $8300 \text{ CFU m}^{-3}$ (Virji et al., 2000) and $148\ 500 \text{ CFU m}^{-3}$ (Sprince et al., 1997) and total bacteria up to $2.66 \times 10^9 \text{ cells m}^{-3}$ (Abrams et al., 2000); the average concentrations of endotoxin in the studies ranged from $0.27$ to $98.4 \text{ ng m}^{-3}$ ($\sim 2.7$ to $980 \text{ EU m}^{-3}$). There are no OELs for bacteria and fungi in Finland. Concentrations above the
reference values for homes and offices are often considered normal in industrial workplaces, while atypical species may be suggestive of a harmful microbial source (Reponen et al., 1992). In the present study, the only machine shop where the Dutch legal endotoxin level was exceeded was the one using MWF designed to be used without preservatives. Due to the resulting massive growth of gram-negative bacteria and because of multiple reports of endotoxin-induced respiratory symptoms (Douwes and Heederik, 1997), use of such MWFs does not seem advisable. Overall, careful maintenance of the fluid to maintain its microbiological quality as well as control measures to avoid exposure to aerosols is essential when controlling health risks due to MWF (Stear, 2005).

Finland has established OELs only for individual aldehydes, such as formaldehyde and acetaldehyde. Formaldehyde was found in all workshops. The mean concentration, 0.04 mg m\(^{-3}\), was well below the Finnish 8-h OEL and in line with another study from machine shops in Finland (Linnainmaa et al., 2003). Only few other studies of aldehydes in machine shops could be identified (Cohen, 1996; Thorne and DeKoster, 1996; Godderis et al., 2007). In a recent study from Belgium (Godderis et al., 2007), formaldehyde was found in a concentration of 0.03 mg m\(^{-3}\), whereas in another study from the US (Thorne and DeKoster, 1996), the average concentration of formaldehyde was higher, 0.22 mg m\(^{-3}\).

Aldehydes in general may originate from the oil component and fatty acid derivatives in MWF as a result of oxidative decomposition. However, the formaldehyde-releasing biocides in MWF are the most important source of formaldehyde; this was also supported by the strong correlation between formaldehyde and alkanolamines, as many of the common biocides in MWFs are composed of formaldehyde and alkanolamine derivatives. Even though individual aldehydes were found only in small quantities, the total average concentration of aldehydes may be enough to cause respiratory irritation. Formaldehyde is a skin sensitizer (Herbert and Rietschel, 2004), respiratory symptoms (Norback et al., 1995) and asthma (Piipari and Keskinen, 2005). The phenolic antioxidant 2,6-di-tert-butyl-4-methylphenol may cause allergic contact dermatitis (Flyvholm and Menne, 1990). The only ethanalamine found in the VOC analysis was triethanolamine borate, which, like other ethanalamines, is a well-known skin sensitizer (Geier et al., 2004) and may also cause asthma (Savonius et al., 1994).

In nine of the 10 metal workshops studied, the alkanolamines were also measured separately with a new filter sampling method with liquid chromatography–mass spectrometry analysis, the median concentrations for mono-, di- and triethanolamine being 0.057 (range 0.004–0.345), 0.064 (<0.004–0.180) and 0.006 (0.001–0.166) mg m\(^{-3}\), respectively. The study has been reported in detail elsewhere (Henriks-Eckerman et al., 2007). A statistically significant association was found between oil mist and alkanolamines. Alkanolamines are common in modern MWFs, their collection is easy with the newly developed method and their detection limit is low, ~0.001 mg m\(^{-3}\) for 2-h sampling. Thus, it seems that alkanolamines could be useful indicators of exposure to all classes of water-miscible MWFs.
There are some investigations of the vapour losses from aerosol samplers (Simpson, 2003; Woskie et al., 2003), but we were able to identify only one report of active sampling of VOCs in machining facilities; it had only a few samples and an apparently less sensitive method (Godderis et al., 2007). The need for evaluating the extent and composition of the volatile compounds in machine shops has been emphasized (Woskie et al., 2003), and indeed, based on the present results, VOCs provide plenty of information not only on the total volatile hydrocarbons but also on the reactive and possibly sensitizing small molecular weight compounds in MWF. In addition, the VOC method used in the present study is well established and gives reliable results with a mean analytical precision of 20% for individual volatiles at a 95% confidence level. Although the amount of VOCs was below the recommended industrial level, the total airborne impurities were dominated by volatile compounds. VOCs and small molecules in general may also increase their concentration in workplace air because they are not well retained by oil mist separators. According to a recent review of indoor investigations, the low level of total VOCs cannot be regarded as a potent reason for asthmatic symptoms (Nielsen et al., 2007). However, MWF-originated VOCs may play a role in a machining environment.

CONCLUSIONS

On the whole, there is great need for assessing and regulating exposures to water-miscible MWFs. Specific ingredients are not merely markers of total MWF exposure but important in themselves as some of them may cause respiratory sensitization or irritation even in small concentrations. We have shown that the total exposure in machine shops was quantitatively dominated by volatile compounds. In order to get more information on the chemical composition of the air in machine shops, additional measurements of VOCs, aldehydes and alkanolamines are needed. Present Finnish OELs for alkanolamines should be revised to account for multiple exposures, and the OEL for oil mist should be adjusted based on the recent development in workplaces towards a concentration clearly <5 mg m⁻³. Overall, careful maintenance of the fluid to maintain its microbiological quality is essential when controlling health hazards due to MWF. Another important step in occupational hygiene would be to develop new air cleaning methods, as oil mist separators are insufficient for cleaning the air of small molecules.

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