Oil Mist and Vapour Concentrations from Drilling Fluids: Inter- and Intra-laboratory Comparison of Chemical Analyses

KAREN S. GALEA1*, ALISON SEARL1, ARACELI SÁNCHEZ-JIMÉNEZ1, TORILL WOLDBÆK2, KRISTIN HALGARD2, SYVERT THORUD2, KJERSTI STEINSVÅG3, KIRSTI KRÜGER3, LAURA MACCALMAN1, JOHN W. CHERRIE1 and MARTIE VAN TONGEREN1

1Institute of Occupational Medicine (IOM), Research Avenue North, Riccarton, Edinburgh, UK; 2National Institute of Occupational Health (STAMI), Oslo, Norway; 3Statoil ASA, NO-0243 Oslo, Norway

Received 25 August 2010; in final form 14 July 2011; published online 15 September 2011

Objectives: There are no recognized analytical methods for measuring oil mist and vapours arising from drilling fluids used in offshore petroleum drilling industry. To inform the future development of improved methods of analysis for oil mist and vapours this study assessed the inter- and intra-laboratory variability in oil mist and vapour analysis. In addition, sample losses during transportation and storage were assessed.

Methods: Replicate samples for oil mist and vapour were collected using the 37-mm Millipore closed cassette and charcoal tube assembly. Sampling was conducted in a simulated shale shaker room, similar to that found offshore for processing drilling fluids. Samples were analysed at two different laboratories, one in Norway and one in the UK. Oil mist samples were analysed using Fourier transform infrared spectroscopy (FTIR), while oil vapour samples were analysed by gas chromatography (GC).

Results: The comparison of replicate samples showed substantial within- and between-laboratory variability in reported oil mist concentrations. The variability in oil vapour results was considerably reduced compared to oil mist, provided that a common method of calibration and quantification was adopted. The study also showed that losses can occur during transportation and storage of samples.

Conclusions: There is a need to develop a harmonized method for the quantification of oil mist on filter and oil vapour on charcoal supported by a suitable proficiency testing scheme for laboratories involved in the analysis of occupational hygiene samples for the petroleum industry. The uncertainties in oil mist and vapour measurement have substantial implications in relation to compliance with occupational exposure limits and also in the reliability of any exposure–response information reported in epidemiological studies.

Keywords: analytical variability; drilling fluids; oil mist; oil vapour

INTRODUCTION

Drilling fluids are used during offshore drilling processes, among other functions, to lubricate and cool the drill stem and bit, to provide pressure support in the well, and to transport cuttings to the surface. These fluids are usually complex mixtures comprising solids and liquids, based on either oil or water. The oil-based drilling fluids generally contain a base oil with a relatively low viscosity. To recycle the drilling fluid, any cuttings are removed using a shale shaker, which is essentially a vibrating screen. The resultant cleaned and upgraded drilling
fluid is then re-injected into the well (Steinsvåg et al., 2006).

During shale shaker operations, exposure to oil mist and vapours generally occurs simultaneously. There is no internationally recognized standard method for the analysis of oil mist on filter or oil vapour on charcoal. Different sampling and analytical methods have been discussed, especially for metal working fluids (for example Volckens et al., 2000; Simpson (2003); Verma et al. (2006)). The analysis of oil mist on filter is typically performed using Fourier transform infrared spectroscopy (FTIR) using methods based on National Institute of Occupational Health (NIOSH) 5026 (NIOSH, 1996). The analysis of hydrocarbon vapour on charcoal is typically performed using gas chromatography (GC) using methods based on NIOSH 1500 (NIOSH, 2003) or MDHS 96 (HSE, 2000). Individual laboratories have adapted the methods to suit their own particular samples and circumstances. In particular, different approaches are taken to calibration, both in the choice of calibration standard and in the way that data are processed.

As part of a larger study that aimed to characterize aerosols generated from a shale shaker during drilling operations (Galea et al., 2010), we investigated the intra- and inter-laboratory variability in analytical results for oil mist and oil vapour samples. The aim of this study was to investigate inter- and intra-laboratory difference in results of oil mist on filter and oil vapour on charcoal measurements. In addition, we also undertook a limited study to investigate the sample losses of oil mist from glass fibre filters during transportation and storage.

Samples were analysed by two laboratories, one in Norway (National Institute of Occupational Health, STAMI) and one in the UK (Institute of Occupational Medicine, IOM).

MATERIALS AND METHODS

Study environment

This study was carried out in a simulated offshore shale shaker room in Sandnes, Norway (Galea et al., 2010). Two drilling fluid systems (System 2.0 and System 3.5) were used during two separate sampling campaigns. Table 1 provides further details of the two fluid systems.

Sampling method

Replicate samples of oil-based drilling mud aerosols were collected. Oil mist was sampled using the 37-mm Millipore cassette loaded with two filters in series (a glass fibre and a cellulose acetate filter) and the oil vapour using a backup charcoal tube containing 150 mg sorbent (100 mg front section; 50 mg back). The flow rate for the sampler was 1.4 l min⁻¹.

The sampling equipment was co-located and suspended at breathing zone height (~1.8 m) at a distance of ~0.65 m from the shale shaker. The sample test run was 2 h (the sample period normally used when sampling oil mist and vapours in Norway).

In the first survey, two co-located samples using the Millipore cassette and backup charcoal tube assembly were collected during each of the eight test runs, with each laboratory receiving one set of filters and tubes for each test period. In the second survey, four co-located samples using the Millipore cassette and backup charcoal tube assembly were collected during each of the eight test runs with the normal viscosity drilling mud system (System 3.5). Each laboratory analysed two sets of samples from each of these test periods (total of 16 filters and charcoal tube samples per laboratory) (Table 2).

Quality control and sample storage

Samples were rejected if the flow rate deviated by ≥5%. Field blanks were collected and handled in the same way as the other field samples except that they were not exposed to workplace air.

Immediately after sample collection, all samples were capped and placed in an on-site refrigerator where the mean temperature was 6°C [standard deviation (SD) = 1.1; range 4.8–11.6°C]. On completion of the monitoring campaigns, the capped air samples were placed in zip locked bags and transferred to the two laboratories either in a cool bag by the researcher or using next day postal delivery. All aerosol samples were stored in a refrigerator (8°C) prior to analysis.

<table>
<thead>
<tr>
<th>Drilling fluid system</th>
<th>Base oil</th>
<th>Viscosity (mm² s⁻¹ at 40°C)</th>
<th>Boiling point (°C)</th>
<th>Vapour pressure (mm Hg at 40°C)</th>
<th>Survey used</th>
</tr>
</thead>
<tbody>
<tr>
<td>System 2.0</td>
<td>A</td>
<td>2.2</td>
<td>210–260</td>
<td>&lt;1</td>
<td>Survey 1</td>
</tr>
<tr>
<td>System 3.5</td>
<td>B</td>
<td>3.3–3.7</td>
<td>250–325</td>
<td>&lt;1</td>
<td>Surveys 1 and 2</td>
</tr>
</tbody>
</table>
to analysis. The time between sample collection and analysis and the methods of delivery are shown in Table 3. In each survey, bulk samples of base oil (one for each mud system used) were distributed to each laboratory for sample calibration.

Laboratory analysis

Samples were analysed by two laboratories, one in Norway (STAMI) and one in the UK (IOM).

Oil mist samples were analysed by IOM using FTIR spectroscopy (a Perkin Elmer 1720X Series) following desorption with Freon 113 (1,1,2-trichlorotrifluoroethane). FTIR was set to scan the spectral region 3200–2700 cm\(^{-1}\) with resolution 4 cm\(^{-1}\) and two scans. Results were based on absorbance at a single wave number (2929 cm\(^{-1}\)). Calibration standards in Freon 113 were prepared using samples of the base oils provided. The limit of detection (LOD) (as determined from blank sampling media in all instances) was 0.02 mg of oil on filter.

Following desorption with carbon disulphide (CS\(_2\)), the oil vapour samples were analysed by IOM using GC with a flame ionization detector (FID). The GC consisted of a ZB-1 capillary column (30 m) and was programmed to increase the temperature from 40 to 250\(^\circ\)C at 4\(^\circ\)C min\(^{-1}\). Calibration was based on \(n\)-dodecane (C\(_{12}\)) and the area under the chromatogram over the retention times of the base oil was used to quantify the results. The LOD was 0.001 mg on each sample.

For the oil mist analyses, the STAMI laboratory used a Perkin Elmer 1600 series FTIR and scanned from 3200 to 2600 cm\(^{-1}\) with resolution 4 cm\(^{-1}\) and 16 scans. Results were based on absorbance at a single wave number (2929 cm\(^{-1}\)). Calibration standards in Freon 113 were prepared using samples of the base oils provided. For the oil vapour analyses, following desorption with CS\(_2\), the STAMI laboratory used a GC with an FID (a Perkin Elmer AutoSystem XL, detector 300\(^\circ\)C) and a Chrompack CP-Sil 8 CB capillary column (25 m × 0.25 mm inside diameter) programmed to increase the temperature from 40 to 250\(^\circ\)C at 6\(^\circ\)C min\(^{-1}\). At the STAMI laboratory, calibration standards of C\(_{12}\) in CS\(_2\) were prepared with charcoal added to the standard mixture solution, while no charcoal was added at the IOM laboratory. The LOD was 0.01 and 0.001 mg for the oil mist and vapour samples, respectively.

All results were blank corrected using the field blanks.

Sample losses during transportation and storage

Glass fibre filters were spiked using an automatic pipette with known quantities of the bulk base oils (A and B; base oil A being used in fluid System 2.0 and base oil B in System 3.5). Half of the filters were transported to Norway 12 days after spiking and returned to the IOM laboratory on Day 27. During transportation, the samples were placed in zip

### Table 3. Comparison of transport methods and times of analysis of each batch of samples in the two laboratories.

<table>
<thead>
<tr>
<th>Survey</th>
<th>Drilling mud/base oil</th>
<th>Laboratory</th>
<th>Medium</th>
<th>Delivery(^a)</th>
<th>Maximum days between sampling and analysis (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>System 2.0/A</td>
<td>STAMI</td>
<td>Filters</td>
<td>Train</td>
<td>27</td>
</tr>
<tr>
<td></td>
<td></td>
<td>IOM</td>
<td>Filters</td>
<td>Air</td>
<td>38</td>
</tr>
<tr>
<td></td>
<td></td>
<td>STAMI</td>
<td>Charcoal tubes</td>
<td>Train</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td></td>
<td>IOM</td>
<td>Charcoal tubes</td>
<td>Air</td>
<td>38</td>
</tr>
<tr>
<td></td>
<td>System 3.5/B</td>
<td>STAMI</td>
<td>Filters</td>
<td>Post</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>IOM</td>
<td>Filters</td>
<td>Air</td>
<td>17</td>
</tr>
<tr>
<td></td>
<td></td>
<td>STAMI</td>
<td>Charcoal tubes</td>
<td>Post</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td></td>
<td>IOM</td>
<td>Charcoal tubes</td>
<td>Air</td>
<td>17</td>
</tr>
<tr>
<td>2</td>
<td>System 3.5/B</td>
<td>STAMI</td>
<td>Filters</td>
<td>Post</td>
<td>16</td>
</tr>
<tr>
<td></td>
<td></td>
<td>IOM</td>
<td>Filters</td>
<td>Air</td>
<td>13</td>
</tr>
<tr>
<td></td>
<td></td>
<td>STAMI</td>
<td>Charcoal tubes</td>
<td>Post</td>
<td>28</td>
</tr>
<tr>
<td></td>
<td></td>
<td>IOM</td>
<td>Charcoal tubes</td>
<td>Air</td>
<td>10</td>
</tr>
</tbody>
</table>

\(^a\)Method of delivery of samples from site to laboratory: post—via Norwegian postal service; train—accompanied researcher via train from Sandnes to Oslo, Norway; air—accompanied researcher via car and air from Sandnes, Norway to Edinburgh, UK.
locked bags and transferred in a cool bag by the researcher, with the samples being refrigerated during storage. All filters were reweighed following standard gravimetric analysis procedures (HSE, 2000) on two occasions after return of the samples. All filters (both those transported to Norway and those remaining in the laboratory) were stored in tightly sealed metal filter storage tins and were unopened during this time. The rate of oil evaporation from filters was assessed by weighing the filters before and immediately after spiking and at set times following spiking, again following the standard gravimetric analysis procedures (HSE, 2000).

**Data analyses**

As is typical for this kind of data, the concentration distribution was skewed. Taking logs of the data resulted in an approximate normal distribution so the statistical analyses were carried out on the log-scale.

Descriptive statistics are presented in the form of geometric mean (GM) and geometric standard deviation (GSD). The ratios of the STAMI and IOM results and associated confidence interval (CI) are also given. If the CI excludes the value ‘one’, this implies that there was statistically significant difference between the results given by the two measures. This method is used to investigate differences between the two laboratories as well as within laboratories (based on data collected in Survey 2). For Survey 2, where there were two side-by-side samples analysed by each lab, the average of each pair of results was taken for the inter-laboratory analysis. Intra-laboratory precision examines how closely each lab replicates the analysis results, based on a pair of side-by-side samples, while inter-laboratory precision looks at how close the results are between the labs for the pairs of side-by-side samples (i.e. is the result of STAMI close to the result of IOM). The coefficient of variance (CV) is a measure of the dispersion of the data set, adjusted by the mean. This is expressed as a percentage and gives information about the average variation in the results obtained for the side-by-side samples.

Pearson correlation coefficients were calculated for data from the two laboratories. While these give some measure of the linear agreement between the results from the two laboratories, they cannot be used alone as high correlation can result from data that follows a straight line but not necessarily that of equality. In addition to these statistics, Bland–Altman plots were used to examine the relationship between the average of the two measurements and their difference. If there was good agreement between the measures, this plot should display a random scatter of data points.

**RESULTS**

**Oil mist concentrations**

Table 4 describes the GM and GSD of the oil mist results obtained from each of the two laboratories separately for the two surveys. There are some differences in the results provided by the IOM and STAMI laboratories, but this difference is not consistent; STAMI results for oil mist were significantly higher than IOM results in survey 1 (ratio = 1.37; 95% CI = 1.10–1.72) but not in Survey 2 (see also Fig. 1). The variation in the results between laboratories at Survey 2 (22%) was slightly better than at Survey 1 (27%), however, we would consider both to be poor.

The correlation between the STAMI results and IOM results was high. The Bland–Altman plots, ignoring any effect of survey, suggest that there are no systematic differences between the two laboratories. However, there is some evidence of a pattern when looking at each survey separately confirming earlier findings that there is a difference. In the first survey, the period between sampling and analysis was considerably less for STAMI than for IOM (by $\frac{1}{2}$ days), while in the second survey, the converse was true (3 days).

Intra-laboratory precision was only investigated in the second survey where pairs of samples that had been collected were analysed by each laboratory (Table 5). Both laboratories reported significant differences in the oil mist results for paired samples. The average ratio of results for paired samples reported by IOM was 0.70 (95% CI = 0.53–0.92) and by STAMI was 0.90 (95% CI = 0.84–0.95). The width of the confidence interval gives some initial indication

<table>
<thead>
<tr>
<th>Survey 1</th>
<th>N</th>
<th>GM mg m$^{-3}$</th>
<th>GSD mg m$^{-3}$</th>
<th>Survey 2</th>
<th>N</th>
<th>GM mg m$^{-3}$</th>
<th>GSD mg m$^{-3}$</th>
<th>Ratio</th>
<th>Mean</th>
<th>95% CI</th>
<th>CV (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>IOM</td>
<td>7</td>
<td>0.66</td>
<td>2.84</td>
<td>STAMI</td>
<td>8</td>
<td>0.94</td>
<td>2.37</td>
<td>1.37</td>
<td>1.10–1.72</td>
<td>27</td>
<td></td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>0.35</td>
<td>1.81</td>
<td></td>
<td>8</td>
<td>0.29</td>
<td>1.91</td>
<td>0.85</td>
<td>0.66–1.08</td>
<td>22</td>
<td></td>
</tr>
<tr>
<td>All</td>
<td>15</td>
<td>0.47</td>
<td>2.38</td>
<td></td>
<td>16</td>
<td>0.52</td>
<td>2.59</td>
<td>1.06</td>
<td>0.87–1.30</td>
<td>25</td>
<td></td>
</tr>
</tbody>
</table>
of the precision of each lab, with the precision of IOM being much poorer than STAMI. This is confirmed via the coefficient of variation with the variation in the results from IOM being ~30%, while STAMI were much more accurate and less variable (9%).

Vapour concentrations

It should be noted that when drilling fluid ‘System 2.0’ was tested, all the closed cassette backup charcoal tubes exhibited some breakthrough into the back section. However, the extent of the breakthrough was <25% and therefore samples were considered to be valid. To determine if this may have affected results, the data were analysed with and without these samples, where the removal of the breakthrough samples resulted in the removal of all samples taken when using drilling fluid ‘System 2.0’.

All the results for vapours, with the exception of two, are above the line of equality in the scatter plot (Fig. 2) which indicates that, although very similar, the results obtained from the IOM laboratory were typically higher than those from STAMI. There is a significant difference between the results obtained from the two laboratories (Table 6), with those from STAMI being lower, on average, than IOM (ratio = 0.81; 95% CI = 0.73–0.90). This is still the case when the samples for which there was breakthrough are removed (ratio = 0.78; 95% CI = 0.69–0.88). Again there is a suggestion that survey has an effect, with the levels being higher at Survey 1 than Survey 2, for both labs. This is confirmed through examination of the Bland–Altman plots. There also appears to be an effect of survey on the inter-laboratory precision; 14 and 24% for Survey 1 and 2, respectively.

Intra-laboratory precision in the second survey was relatively good (Table 5), with an average difference of 9% for both IOM (95% CI = 0.85–0.98) and STAMI (95% CI = 0.80–1.05). Here, the 95% CI for IOM is narrower than for STAMI indicating that IOM were more precise in analysing for oil vapour. The CV for both labs is relatively low, with IOM

Table 5. Intra-laboratory precision: the average ratio of side-by-side samples analysed at each laboratory, along with their 95% CI and CV, shown separately for IOM and STAMI.

<table>
<thead>
<tr>
<th></th>
<th>Mist</th>
<th>Vapour</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean</td>
<td>95% CI</td>
</tr>
<tr>
<td>IOM</td>
<td>0.70</td>
<td>0.53-0.92</td>
</tr>
<tr>
<td>STAMI</td>
<td>0.90</td>
<td>0.84-0.95</td>
</tr>
<tr>
<td>All</td>
<td>0.80</td>
<td>0.70-0.91</td>
</tr>
</tbody>
</table>

![Fig. 1. Left, scatter plot of the STAMI versus IOM lab results for oil mists. The line of equality is shown. Right, Bland–Altman plot of the average versus the difference in oil mist concentrations. The dotted lines are ±2 SD in the average differences between laboratories.](https://academic.oup.com/annweh/article-abstract/56/1/61/166457)
being more precise in this case (CV = 8% for IOM; 13% for STAMI).

Sample losses of oil mist from filters

Oil mist can be expected to evaporate from filters when stored after sampling (Simpson, 2003; Verma et al., 2006). Figure 3 illustrates the percentage change in the spiked filters weight on each of the weighing days following spiking, based on the average weight of five filters for each of the oils and four control filters (no base oil added). The full results are tabulated in Galea et al. (2010). The filters spiked with base oil A showed a substantial initial weight loss and then stabilized, whereas those spiked with base oil B showed a more gradual change in weight. There was no change in the weight of the control filters. The average percentage spiked filter mass prior to half of the filters being transported to Norway was 59% for filters spiked with base oil A (41% loss prior to transportation) and 83% for those spiked with base oil B (a 17% loss prior to transportation). There was no difference in the average percentage filter mass between those filters spiked with base oil A transported to Norway (59% for filters returning to the laboratory) and those which remained in the laboratory (58%). For the less volatile base oil B, the loss of volatiles appears to have continued during transport, with the average percentage of the spiked filter mass being 82% for the filters returning from Norway and 75% for those that remained in the laboratory. There was also a suggestion from the data that weight losses would have been likely to continue after the last weighing.

DISCUSSION

Replicate static samples for oil mist and vapour were collected over a 2-h sampling period from a shale shaker at concentrations ranging from 0.1 to 3.89 mg m\(^{-3}\) for oil mist and 6.16–334 mg m\(^{-3}\) for oil vapour, respectively, to assess inter- and intra-laboratory variability in analysis. Such concentrations may be encountered during offshore shale shaker operations. For example, James et al. (2000) reports airborne oil mist concentrations ranging from 0.03 to 5.52 mg m\(^{-3}\) and hydrocarbon vapour

Table 6. GM and GSD of the oil vapour concentrations, obtained from IOM and STAMI laboratories as well as the ratio of STAMI to IOM and 95% CI, separately for each survey.

<table>
<thead>
<tr>
<th></th>
<th>STAMI</th>
<th>Ratio</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>N</td>
<td>GM mg m(^{-3})</td>
<td>GSD mg m(^{-3})</td>
</tr>
<tr>
<td>Survey 1</td>
<td>7</td>
<td>160.7</td>
<td>1.88</td>
</tr>
<tr>
<td>Survey 2</td>
<td>8</td>
<td>37.4</td>
<td>1.79</td>
</tr>
<tr>
<td>All</td>
<td>15</td>
<td>73.9</td>
<td>2.59</td>
</tr>
</tbody>
</table>

Fig. 2. Left, scatter plot of the STAMI versus IOM lab results for oil vapours. The line of equality is shown. Right, Bland–Altman plot of the average versus the difference in oil vapour concentrations. The dotted lines are ±2 SD in the average differences between laboratories.
concentrations ranging from 3.2 to 96.4 mg·m⁻³ in a shaker house where the shakers were enclosed by a ventilation canopy and the other parts of the system including the fluid flow lines, cuttings ditch and shaker trough were covered or part-covered. However, given that the Occupational Exposure Limit (OEL) for oil mist is 0.6 mg·m⁻³ (12-h work shift) (PSA, 2004) and 30 mg·m⁻³ OEL (12-h work shift) for oil vapour (PSA, 2004) and that personal exposures will typically be lower than the static concentrations assessed in our variability analysis, we would recommend that an additional inter- and intra-laboratory study also be undertaken to compare the oil mist and vapour analysis at lower concentrations.

Sample losses

Slight permutations in storage conditions, transportation, and handling may contribute to the inter-laboratory differences in reported results. Sample losses also commonly occur during the collection of the sample. For example, Simpson et al. (2000) reports on a study investigating the scale and extent of sample losses from a mineral oil metal working fluid (MWF) on a filter during sampling. Losses of 5% of the filters weight were reported for oils with viscosities >18 cSt at 40°C, with losses up to 71% being observed for those with viscosities <18 cSt. Park et al. (2003) reported that conditions typical of shipment and storage (i.e. desiccation and airflow through the filter) contributed significantly to losses of MWF collected on Polyvinyl chloride filters, with higher losses reported for high viscosity oils. Menichini (1986) reported losses from aspirated filters of 1–6% mass depending on the viscosity of the oil. As such, it should be considered that oil mist concentrations being reported may be under-estimated, with the extent of the under-estimation being driven by a number of different variables. The nature of the filter may also affect the amount of mineral oil lost (Raynor et al., 2000). Progress has been done in this field with some models being developed to predict the amount of oil lost. Raynor et al. (2000) developed a model that successfully predicted significant differences in collection efficiencies for oil mist among several filter media.

Oil mist analysis

The NIOSH method 5026 (NIOSH, 1996) provides a good starting point for the analysis of oil mist. STAMI adhered more closely to the method for quantifying results from the FTIR response than IOM. The IOM would normally quantify over a broader section of the spectrum because the peak ratios may vary between the individual samples in a batch. The differences in the method of processing FTIR spectra may have a significant impact on reported results for oil mist for some oil formulations. The size and direction of difference will depend on the individual oil. The differences in normal practice between the two laboratories does not, however, account for the differences in results reported by the laboratories when following the same quantification method. Some of the differences in the results reported in each survey may be consistent with differences in the storage temperature (for example in the second survey samples issued to STAMI were delayed in the postal system and held at room temperature during 10 of the 16 days between sampling and analysis) and the time gap between sampling and analysis and the associated loss of more volatile
components from filters as higher results were reported in each case by the laboratory that undertook the analysis first.

The poorer within-laboratory performance for IOM as well as between laboratory precision suggests that there is some fundamental difficulty in the analytical method that has remained unidentified. Problems with continuing evaporation of oil from filters (particularly for Oil B) during handling prior to analysis may be a contributing factor. It is possible that the efficiency of desorption from filter has not been uniform although the base oils appear to dissolve readily in the desorption medium. It is also possible that the paired samples were not as well matched as would have been anticipated from the sample collection regime. Menichini (1986) identified sample contamination as a potential source of error associated with infrared analysis but it is difficult to identify when and how samples in this study could have been contaminated. There are no formal proficiency testing schemes for oil mist on filter and therefore little information about the wider extent of inter-laboratory differences in measurement results.

Oil vapour analysis

There is no consensus method for the quantification of oil vapour on charcoal. The methods normally used by the two laboratories involved in this study were different. STAMI would normally calibrate using the alkane that best approximates the oil composition but would quantify over the same interval of the chromatogram and this was the approach adopted by both laboratories for the purposes of this study. The IOM laboratory would usually base their calibration on measured aliquots of an oil sample provided by the client and quantify samples on the basis of the area under the chromatogram over the same retention times as for the oil. For the samples analysed in this study, the method normally used by IOM would have given results that were ~25% higher than those actually reported.

There is a significant difference between the results obtained from the two laboratories, with those from the STAMI laboratory being lower, on average, than the IOM laboratory. It is not clear why this has arisen but a difference in calibration factor seems a possible explanation as the results for the paired samples analysed by the two laboratories are well correlated. There was a slight difference in the method used to prepare calibration standards by the two laboratories as STAMI added charcoal to the standards. This would, however, be expected to lead to higher rather than lower estimates of the mass of collected oil vapour. Huynh et al. (2009) note the problems of interference from other volatile species present in workplace air when using simple GC analysis to quantify oil vapour on charcoal and suggest the use of GC–mass spectrometry to allow subtraction of the interfering phases from the oil vapour determination. However, given that both laboratories used similar column conditions, it seems unlikely that interferences account for the observed differences in reported results.

Intra-laboratory precision was relatively good for both laboratories suggesting that the handling and analysis of oil vapour samples collected on charcoal is associated with less uncertainty than for oil mist samples.

CONCLUSIONS AND RECOMMENDATIONS

This study has demonstrated that the use of different approaches for the chemical analysis of oil mist and vapour samples, including the choice of calibration method, can give rise to potentially important differences in reported results.

The precision of oil mist analysis is relatively poor with much better precision being demonstrated for oil vapour on charcoal. Sample handling may have contributed to the poor precision in the oil mist results. There is a requirement to undertake further measurements of oil mist on filter over a range of concentrations in order to better characterize intra- and inter-laboratory variation before finally developing a clear specification as to how the measurements should be made. It would be desirable for the members of the occupational hygiene community to work in partnership to develop and publish a method for the analysis of oil mist and oil vapour which also includes specifications for the storage, transport, and handling of filters prior to analysis for oil mist in order to reduce losses.

In the absence of existing laboratory analysis proficiency testing schemes for oil mist on filter and oil vapour on charcoal, the petroleum industry should consider supporting the development of a suitable proficiency testing scheme for laboratories involved in the analysis of occupational hygiene samples for the industry.

The uncertainties in oil mist and vapour measurements have clear implications in relation to compliance with OELs and may also affect the reliability of any exposure-response information reported in epidemiological studies.

FUNDING

The research project was funded by Statoil ASA, Norway and partly funded by the Confederation of
Acknowledgements—We would like to thank all the Cubility staff at the Sandnes test centre, and in particular Ken Pettersen, for their help and assistance throughout the on-shore centre testing periods. Thanks are also due to those individuals responsible for the laboratory analysis of the collected samples at the IOM: Carolyn McGonagle, Andrew Cameron and David Third. Thanks to Vegard Peikli and Esther Saetvedt (Statoil ASA) for their helpful and constructive advice and comments throughout the project.

REFERENCES


