Determination of Triglycidyl Isocyanurate from Powder Coatings in Occupational Hygiene Samples by Gas Chromatography with Mass Spectrometric Detection

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A method for the determination of triglycidyl isocyanurate (TGIC) from powder coatings using gas chromatography with mass spectrometric (GC/MS) detection is presented. The new method is considerably easier to use than the existing methods and has superior performance. It has been novelly applied to the determination of TGIC in a variety of occupational hygiene samples (cotton swabs, gloves and whole suits). Using the GC/MS method, the following percentage recoveries were found: gloves 114 ± 1.9; swabs 73 ± 6.5; whole suit 125 ± 16.3 and 108 ± 6.8 (two powder coatings); filter 79 ± 8. The estimated limit of detection was 0.002 μg/ml.

Keywords: occupational hygiene; powder coatings; triglycidyl isocyanurate

INTRODUCTION

Triglycidyl isocyanurate [TGIC or tris(2,3-epoxypropyl)isocyanurate, CAS no. 2451-62-9] is used in polyester powder coatings as a cross-linking (curing) agent and in solder ‘mask’ inks in the printed circuit board industry. The structure of TGIC is given in Fig. 1. Occupational exposure may occur during manufacture and use of TGIC. It is toxic by inhalation and ingestion and is a severe eye irritant and a mild skin and nasal irritant. Pure TGIC and TGIC-containing powder coatings have the potential to cause skin sensitization leading to severe rashes. TGIC has been reported to induce asthma and other respiratory symptoms and can cause genetic damage.

MDHS 85 (HSE, 1997) was developed to measure TGIC in air and is based upon three similar methods (Nissan Chemicals, 1988; Work Cover Authority, 1991; Ciba-Geigy, 1991). These three methods were developed for the analysis of pure TGIC in air. MDHS 85 extended these methods to include the determination of airborne TGIC in powder coatings. All four of these methods collect air samples onto glassfibre filters and use liquid chromatography with ultraviolet/visible detection (LC/UV).

Using MDHS 85 for the determination of TGIC in powder coatings requires a more complicated sample preparation than that required for pure TGIC (see Preparation of calibration curves below). This procedure adds two precipitation and evaporation steps to remove the polyester matrix from the powder coating. For the small volumes of solvent used in MDHS 85 (<3 ml in any step) this extended method is not too time consuming. However, much larger volumes of solvent are used to desorb occupational hygiene (OH) samplers. Typical amounts used are: swabs, 25 ml; gloves, 100 ml; whole suits, 1 l. In particular, the two steps where the solvent is evaporated are extremely time consuming if a large amount of water has to be removed. For example, to keep the ratio of THF to water the same as in MDHS 85, two additions of 25 ml of water would be added to a glove extract (100 ml of THF) and then evaporated. Elevated temperature cannot be used in the drying steps as this will cause the TGIC to cross-link.

A method for the analysis of TGIC in air using gas chromatography with electron capture detection...
(GC/ECD) (OSHA, 1992) using derivatization with heptafluorobutyric anhydride has been published. It was decided to investigate the use of gas chromatography with mass spectrometric detection (GC/MS). Mass spectrometry is a more selective technique than UV detection and it is likely to be more sensitive because TGIC does not contain a strong UV chromophore.

In the work presented here a GC/MS method is developed and compared with MDHS 85. In addition, work was carried out to simplify and speed up the extraction procedure for TGIC from powder coatings and this simplified procedure was applied to OH sampling devices.

MATERIALS AND METHODS

Sampling methods

Dermal sampling. Recommended sampling methods for assessing dermal exposure to pesticides have been published by the Health & Safety Executive (HSE, 1999a,b). These methods were applied to sampling for TGIC in workplaces using powder coatings. Sampling was carried out by Health and Safety Laboratory staff during routine enforcement and survey activity by HSE Inspectors.

Square cotton gauze pads (swabs), 10 × 10 cm, are attached to operators clothing with safety pins. The positioning of these swabs is based on the standard protocol for assessing dermal exposure to pesticides developed by the World Health Organisation (WHO, 1982). This sampling method has been successfully applied in previous HSE surveys (Llewellyn et al., 1996; Garrod et al., 1998, 1999, 2000; Coldwell et al., 2003; Johnson et al., 2004).

The swab positions are shown in Fig. 2: position 1, on the hat, as close as practicable to the top of the head; position 2, over the sternum, on the outside of normal clothing; position 3, on the sternum, on the inside of normal clothing; position 4, upper surface of right forearm held with elbow bent at right angles across the body, midway between elbow and wrist, on the outside of normal clothing; position 5, front of left leg, mid thigh, on the outside of normal clothing; position 6, front of left leg, above ankle, on the outside of normal clothing; position 7, on the back between shoulder blades, on the outside of normal clothing. The swab at position 3 was placed to give information on the quantity of in-use product penetrating the top layer of clothing. To determine hand contamination, thin cotton gloves were worn under protective gloves (if worn).

As an alternative to the above approach, Tyvek® semi-absorbent disposable suits worn by operators in two factories using TGIC-based powder coatings were analysed.

Blank samples. Blank samples of all dermal samplers (swabs, gloves and suits) were bagged whilst on site and treated in the same manner as personal samples.

Bulk samples. Bulk samples (~10 g) of all TGIC powder coating formulations used were collected, so that the TGIC content of each powder used could be determined and any potential for analytical interference identified.

Package and storage. All samples taken were packed in separate plastic bags and stored at −20°C until analysed.

Experimental

Chemicals and reagents. TGIC, phosphoric acid and sodium hydroxide were purchased from Aldrich Chemical Co. (Gillingham, UK). TGIC (solid and acetonitrile solutions) has been found to decompose over time (e.g. 82% of original UV response obtained after 3 months storage at −20°C), so fresh TGIC was purchased before each set of experiments. The purity of this material was then determined using infrared spectrophotometry and LC/UV chromatography. Tetrahydrofuran and acetonitrile were purchased from Rathburns (Walkerburn, UK). All solvents...
were LC grade or better. Water was prepared using a Waters Milli-Q system (Watford, UK).

**Instrumentation.** The Waters LC system consisted of a 610 solvent delivery system, 717+ autosampler and 996 diode array detector controlled by a computer using the Millennium 32 software. The Agilent GC/MS system consisted of an HP5890 gas chromatograph, HP6890 series autosampler and HP5972 mass selective detector controlled by a computer using Chemstation chromatography software.

**Sampling media.** Cotton undergloves were purchased from RS Components (Corby, UK). Tyvek® suits were purchased from Wenaas Scotland Ltd (Aberdeen, UK). Swabs (Regal, filmated) were purchased from ARCO Ltd (Sheffield, UK). Filters (glass-fibre, GF/A 25 mm) were purchased from Whatman (Maidstone, UK).

**Preparation of calibration curves.** Calibration curves of TGIC in LC mobile phase (for LC/UV work) or TGIC in THF (GC/MS work) were prepared using standard solutions made from TGIC whose purity had been determined as described in Chemicals and reagents above. No internal standard is used in MDHS 85.

**Preparation and analysis of LC samples.** For LC analysis, samples were extracted as described in MDHS 85. A portion of the THF extract was used (filters 2 ml, swabs 10 ml, gloves 20 ml, suits 1 l) and the procedure described in MDHS 85 paragraph 63 was carried out. The treatment of a powder coating sample collected on a silanized GF/A filter as described in MDHS 85, paragraph 63 reads:

The sample filter is placed in a vial containing 2 ml of THF and mixed on a vortex mixer to dissolve the polyester coating and TGIC. Water (0.5 ml) is added to the vial to precipitate the polyester coating and the mixture thoroughly mixed on the vortex mixer. This is repeated for a second portion of water. The sample is sonicated in acetonitrile for 30 min and a portion (1 ml) transferred to a volumetric flask. The solution is dried under nitrogen and HPLC mobile phase (1 ml) is added to the sample before sonication for 30 minutes. The solution is filtered and analysed by HPLC.

It was noted that, following this procedure for the OH samplers, it took >80 h to evaporate the water from the glove extracts.

The LC method is described in MDHS 85. This uses a 100 x 4.6 mm C18 column (PhaseSep S3 ODS2 or similar) with a flow rate of 1 ml/min. The mobile phase was 90% of 1.15 g of phosphoric acid in 1 l of water, pH adjusted to 6 with sodium hydroxide, and 10% acetonitrile, run isocratically. The wavelength monitored was 205 nm.

**Preparation and analysis of GC/MS samples.** For the GC analysis all samples (filters, swabs, gloves and suits) were extracted with THF as described in MDHS 85, paragraph 63, first sentence (see above). Extraction volumes used were as stated above (Preparation and analysis of LC samples). A few millilitres of this THF extract was filtered through a 0.45 μm syringe filter into GC vials and 1 μl injected into the GC. No further sample preparation was carried out. For the workplace samples the rest of the THF extract was then processed using the LC method (MDHS 85, rest of paragraph 63 as quoted above) to enable a comparison of the two methods.

The GC/MS method used a 30 m x 0.25 mm x 0.25 μm HP5-MS column (Agilent, UK). The temperature programme was 60–300°C at 20°C/min, held at 300°C for 8 min. The injector temperature was 250°C. The GC was operated in constant flow mode (1 ml/min) with helium (99.996% purity) as carrier gas. The MS was operated in scan mode (for identification), monitoring between 50 and 500 a.m.u. and the quantitative work was carried out in selected ion monitoring mode (SIM). Ions monitored were m/z 297 [M]+ and 255 [M -C3H2O2]−, with a dwell time of 250 ms for each. A GC chromatogram of a spiked swab extract (0.1 μg, SIM mode) is shown in Fig. 3. TGIC was identified by retention time and using an ‘allowed ratio’ between the target ion (m/z 255) and first qualifier ion (m/z 297). Although target and qualifier ion ratios can vary, especially at very low concentrations, samples that differ widely from this ratio are suspect and should be reinvestigated. For forensic work, a second qualifier ion should be included, e.g. m/z 267 [M -CH2O]+ (see Fig. 4).

An internal standard (IS) was not used for the majority of this work; however, the use of an IS can often improve the precision of a method. Experiments were done to evaluate hexachlorobenzene (HCB) as an IS. An ideal IS would have the same chemical properties as the analyte, however, for TGIC this would probably mean it had the same detrimental health effects. HCB has been used as an IS previously for GC/MS analysis (Unwin and Groves, 1996) and is stable and gives a good MS response. The m/z monitored was the molecular ion 284. Table 1 shows the results of 10 replicate injections of TGIC solutions, at low and high levels, with and without the IS.

**Recovery experiments on airborne and OH samplers.** Recovery experiments were carried out on filters, swabs, gloves and suits. Filters, gloves and swabs were spiked with TGIC solutions in THF at ‘high’ and ‘low’ levels.

The sampling devices were left for ~5 h before desorption to allow any adsorption by TGIC to occur. Six sampling devices were spiked per level
and a set of each was stored in a freezer at −20°C for ∼3 weeks to determine the effect of storage. Typical desorption volumes used were: swabs, 25 ml; gloves, 100 ml. Suits (20 × 20 cm square) were spiked with known weights of two different powder coating formulations and allowed to stand for ∼16 h. They were then desorbed in 0.5 l of THF, left overnight, sonicated for 30 min and filtered into GC vials. Six replicates were performed per powder coating.

RESULTS AND DISCUSSION

Recovery experiments

The results of the recovery experiments using GC/MS and MDHS 85 are given in Tables 2 and 3. An acceptable range of recovery for routine analysis usually applied to this type of work is 60–140% (‘optimal’ range 70–110%) (Hill, 1997).
For the GC/MS method, the ‘high spike’ samples (>1 μg) which were desorbed immediately (0 days) are all within the acceptable range for routine analysis. The recoveries of the ‘low spike, immediate desorption’ samples (<1 μg) are acceptable, but the precision of analysis was much poorer at these low spiking levels. This is probably because of absorption of the TGIC by the sampling device. A similar problem, noted in MDHS 85, resulted in the recommendation to silanize the filters used for airborne monitoring of TGIC. However, this is not practicable for the OH samplers. The ‘high spike’ levels are more similar to the levels of contamination found on contaminated OH samplers in the workplace (see Tables 4 and 5) than the low spikes.

Fig. 4. Mass spectrum of TGIC (THF extract of bulk powder).
All of the long-term storage results using the GC/MS method, except the high swab spike, are unacceptable because of poor recovery, poor precision or both.

The suit results are in the acceptable range for routine analysis. No long-term storage was carried out on the suits because storage of the OH samples had already been found to cause problems. The bulk powder coatings were found to contain: red 2.28% TGIC, % RSD 7.4; white 0.89% TGIC, % RSD 3.1.

The GC/MS method was found to be robust. For example, for the workplace sample sequence, check samples (150 μg/ml) were run every seven injections. The sequence consisted of ~70 samples comprising six standards, run at the front and back of the sequence, 42 samples, seven check samples and blanks and had a total run time of ~35 h. For the check samples, a ‘recovery’ of 100% of the ‘actual’ value, with a relative standard deviation of 3.8, was found, suggesting that there is little instrumental drift throughout the

Table 2. Recovery results for TGIC using GC/MS

<table>
<thead>
<tr>
<th>Device</th>
<th>Level</th>
<th>Amount of spike (μg)</th>
<th>Time to desorption (days)</th>
<th>Recovery (%)</th>
<th>% RSD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glove</td>
<td>High</td>
<td>3.4</td>
<td>0</td>
<td>114</td>
<td>1.9</td>
</tr>
<tr>
<td>Glove</td>
<td>Low</td>
<td>0.17</td>
<td>0</td>
<td>54</td>
<td>36.8</td>
</tr>
<tr>
<td>Glove</td>
<td>High</td>
<td>3.4</td>
<td>20</td>
<td>48</td>
<td>31.6</td>
</tr>
<tr>
<td>Glove</td>
<td>Low</td>
<td>0.17</td>
<td>20</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>Swab</td>
<td>High</td>
<td>6.7</td>
<td>0</td>
<td>73</td>
<td>8.9</td>
</tr>
<tr>
<td>Swab</td>
<td>Low</td>
<td>0.07</td>
<td>0</td>
<td>112</td>
<td>21.1</td>
</tr>
<tr>
<td>Swab</td>
<td>High</td>
<td>6.7</td>
<td>20</td>
<td>68</td>
<td>3.5</td>
</tr>
<tr>
<td>Swab</td>
<td>Low</td>
<td>0.07</td>
<td>20</td>
<td>41^</td>
<td>31.3</td>
</tr>
<tr>
<td>Suit</td>
<td>White powder</td>
<td>~1000</td>
<td>0</td>
<td>125</td>
<td>16.3</td>
</tr>
<tr>
<td>Suit</td>
<td>Red powder</td>
<td>~30</td>
<td>0</td>
<td>112^</td>
<td>10.7</td>
</tr>
<tr>
<td>Filter</td>
<td>High</td>
<td>1.4</td>
<td>0</td>
<td>79</td>
<td>8.5</td>
</tr>
<tr>
<td>Filter</td>
<td>Low</td>
<td>0.14</td>
<td>0</td>
<td>74</td>
<td>23.9</td>
</tr>
<tr>
<td>Filter</td>
<td>High</td>
<td>1.4</td>
<td>31</td>
<td>59^</td>
<td>22.3</td>
</tr>
<tr>
<td>Filter</td>
<td>Low</td>
<td>0.14</td>
<td>31</td>
<td>72</td>
<td>59.4</td>
</tr>
</tbody>
</table>

All of Glove, Low, 20 days were not detected. % RSD is percent relative standard deviation.

Each result is the average of six data points (n = 6) unless indicated otherwise. For Swab, Low, 20 days (n = 5) one sample was not detected, possibly a ‘missed’ spike. For Filter, High, 31 days (n = 4) two samples were split. These results were all excluded from the Recovery (%) and % RSD calculations.

Suit spike recoveries are calculated with respect to a TGIC (%) determination on the bulk powder by the GC/MS method. For Red Powder, Suit spikes, one result gave ~300% recovery, probably due to a mistake in spiking, i.e. a ‘double spike’, and these results were calculated on that assumption.

Table 3. Recovery results using MDHS 85

<table>
<thead>
<tr>
<th>Device</th>
<th>Level</th>
<th>Amount of spike (μg)</th>
<th>Time to desorption (days)</th>
<th>Recovery (%)</th>
<th>% RSD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glove</td>
<td>High</td>
<td>16.84</td>
<td>0</td>
<td>52</td>
<td>6.4</td>
</tr>
<tr>
<td>Glove</td>
<td>Low</td>
<td>1.68</td>
<td>0</td>
<td>55</td>
<td>12.7</td>
</tr>
<tr>
<td>Swab</td>
<td>High</td>
<td>33.6</td>
<td>0</td>
<td>66</td>
<td>12.7</td>
</tr>
<tr>
<td>Swab</td>
<td>Low^</td>
<td>3.4</td>
<td>0</td>
<td>68</td>
<td>22.6</td>
</tr>
</tbody>
</table>

Each result is the average of six data points (n = 6), % RSD is % relative standard deviation. MDHS 85, paragraph 19, gives the following recoveries for filter spikes: 2–40 μg, desorbed after 1 day, recovery 100%, standard deviation 4%; 20–40 μg, desorbed after 7 days, recovery 94%, standard deviation 6%; 2 μg, desorbed after 7 days, recovery 68%, standard deviation 11%.

For Swab, Low, 0 days, one sample gave 250% recovery, this is probably due to a mistake in spiking, i.e. a ‘double spike’, and these results were calculated on that assumption.

All of the long-term storage results using the GC/MS method, except the high swab spike, are unacceptable because of poor recovery, poor precision or both.

The suit results are in the acceptable range for routine analysis. No long-term storage was carried out on the suits because storage of the OH samples had already been found to cause problems. The bulk powder coatings were found to contain: red 2.28% TGIC, % RSD 7.4; white 0.89% TGIC, % RSD 3.1.
sequence. No appreciable retention time drift was noted during the run. MDHS 85 gives low recoveries for the glove and swab spikes for the immediate desorption experiments. This is presumably because of adsorption to the sampler and losses during the extra sample work-up steps required in comparison with the GC/MS method. The low level spikes have poorer precision than the high spikes.

The major finding is that the samples should be sent to the laboratory and desorbed as quickly as possible after sampling because TGIC decomposes on storage. This agrees with observations made on a TGIC standard and TGIC solutions (see Chemicals and reagents above). The filter results quoted in MDHS 85 also suggest that there is a problem with storage, especially at low TGIC levels (see MDHS 85, paragraph 19). Alternative storage conditions were not explored here because of time constraints. If extended storage is unavoidable then use of a TGIC standard, similarly stored, to allow a correction for storage losses is suggested. Another option in these circumstances would be the development of a derivatization method to give a stable TGIC derivative.

A comparison of the GC/MS and MDHS 85 methods finds that the GC/MS method gives better extraction recoveries, is easier to use and is less time consuming. The GC/MS method in SIM mode was found to give better sensitivity; an estimated limit of detection (signal to noise ratio = 3) of ~0.002 µg/ml was determined. MDHS 85 (paragraph 14) states an estimated limit of detection for TGIC of 0.18 µg/ml.

Comparison of the MDHS 85 and GC/MS methods for workplace TGIC samples

Samples (gloves and swabs) collected during workplace monitoring for TGIC were analysed using both the GC/MS method and MDHS 85 to allow a comparison to be carried out. The results are given in Table 5. Both sets of results were corrected for recoveries. The results are in reasonable agreement, with the GC/MS data typically giving slightly higher exposures than MDHS 85. A paired t-test showed that there was a significant bias ($P = 0.01$) between the two sets of results (GC/MS and MDHS 85). A plot of the MDHS 85 results ($x$-axis) versus the GC/MS results ($y$-axis) gave a straight line, $y = 1.11x + 75.1$, with a correlation coefficient ($r$) of 0.9831. This data shows that the MDHS 85 and GC/MS data are correlated. The bias of the GC/MS method relative to MDHS 85 is therefore +11% (Miller and Miller, 1993). The differences between the two sets of results are larger at the

<table>
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<tr>
<td>0–500</td>
<td>3</td>
<td>0</td>
</tr>
<tr>
<td>500–1500</td>
<td>2</td>
<td>0</td>
</tr>
<tr>
<td>1500–4000</td>
<td>3</td>
<td>1</td>
</tr>
<tr>
<td>4000–8000</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>8000–12 000</td>
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<td>0</td>
</tr>
<tr>
<td>12 000–20 000</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>&gt;20 000</td>
<td>2</td>
<td>1</td>
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</table>
lower concentrations of TGIC (<100 µg) (see Table 5). This may be due to the lower extraction efficiencies of the LC method or because the GC/MS SIM method misidentifies small co-eluting peaks from the powder coating matrix. The spiking experiments suggest that the GC/MS method performs better for the OH samplers at these lower levels (see Tables 2 and 3). These differences at low concentrations are not significant in comparison with the total potential dermal exposure. It is suggested that both methods are best considered semi-quantitative at these low levels (<100 µg/OH sampler).

Scan mode GC/MS analysis of THF extracts of the powder coating bulks identified many peaks in addition to TGIC, but these peaks did not affect the quantification of TGIC (SIM mode) in the work presented here. For this reason it is suggested that each bulk powder extract is analysed qualitatively, using the GC/MS in scan mode, prior to quantification of the samples. This ensures correct identification of the TGIC peak and that there are no peaks in the extracts that could interfere with the SIM analysis. Modification of the GC timetable may be necessary to remove the TGIC peak from any co-eluting substances.

Rapid confirmation of the presence of TGIC in a bulk powder has been carried out at the Health and Safety Laboratory using diffuse reflectance Fourier transform infrared (DRIFT FT-IR) of a powder coating disc sampled onto the DRIFT matrix and compared with a TGIC disc. This is useful for forensic work when a confirmatory analysis may be required.

Both sets of results show the expected pattern of exposure, with the gloves being highly contaminated. The difference between the TGIC concentration found on the right and left gloves is presumably dependant on whether the worker was right or left handed. Swabs 4 and 5 (see Dermal sampling above) were the most contaminated swab samples.

In conclusion, these results suggest that there is a small bias (+11%) of the GC/MS method in comparison with MDHS 85 for the analysis of TGIC in workplace samples. The GC/MS method is easier to use and has a better limit of detection.

**Workplace measurement of potential dermal exposure using whole suit desorption and swab sampling**

Results were obtained for workplace TGIC exposure after whole suit and swab sampling in powder coating factories. The results are shown in Table 4. The potential dermal exposure (PDE) was estimated, from the swab samples, by relating the amount of TGIC on a sampling swab to the exposed area of the body, based on the WHO protocol (WHO, 1982) and as used in previous work (Garrod et al., 1998, 1999, 2000). A multiplication factor is applied to each of the six externally worn swab results giving a body part equivalent. The six body part equivalents are then added together and divided by two to account for areas of the body which are not exposed during work operations, for example in the creases in overalls and areas under the arms. The multiplication factors and the use of PDE as an exposure modeling tool are discussed in EH 74/3 (HSE, 1999).

The seventh swab, swab number 3, is intended to measure penetration of the operators work clothing. Therefore, it does not contribute to potential dermal exposure. In this study no significant penetration of the clothing was observed, suggesting very low skin exposure.

For the whole suit samples, analysis gives a result which is the PDE of the worker.

It can be seen from Table 4 that the exposure to TGIC can vary widely and is known to be dependent on the job the worker was carrying out (i.e. bagging, weighing, mixing, cleaning, etc.). There are insufficient results for the swab method to carry out a detailed statistical comparison of the swab and whole suit methods.

**CONCLUSIONS**

The existing HSL method for TGIC in air (MDHS 85) has been extended to OH sampling devices. A GC/MS method for TGIC in powder coatings has been described which has been developed to include OH samplers and applied to workplace samples. The new GC/MS method has comparable performance to MDHS 85 but is much simpler to use. The method has a lower estimated limit of detection than MDHS 85.

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