

# The occupational hygiene implications of the use of diacetyl in the food flavouring and fragrance industries

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# The occupational hygiene implications of the use of diacetyl in the food flavouring and fragrance industries

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Diacetyl is imported predominantly for use in formulations in the food flavouring industry. There have been reports, mainly in the USA, of damage to the respiratory system caused by inhalation. This report presents the findings of a small study to investigate exposures to diacetyl amongst workers employed in the food flavouring and fragrance industries. The report also includes from previous HSE/HSL visits.

Exposures to diacetyl are generally of short duration, nonetheless, there is potential for workers to be exposed to airborne levels above those recommended by the EU Scientific Committee on Occupational Exposure Limits (SCOEL) of 0.1ppm (8-hour TWA) at the time of the study. Following completion of this report, EU (SCOEL) have tabled new proposed limits of 0.02 ppm (8-hour TWA) and 0.1 ppm (15-minute TWA). There is currently no UK Workplace Exposure Limit (WEL). Personal exposures to diacetyl for this study ranged from 0.01ppm to 1.7 ppm (8hr TWA) and 0.09ppm to 0.33ppm (15min TWA).

Local exhaust ventilation (LEV) was provided during most diacetyl processes. The effectiveness of LEV control was variable. This was mostly due to inappropriate LEV hood design relative to the process.

The respiratory protective equipment (RPE) used was, in most cases, the correct type, however in some cases the assigned protection factors offered (10 & 20) may not reduce exposures to below the SCOEL proposed limits. Deficiencies in the face-fit testing, training and management of RPE were noted. Recommendations refer to hierarchy of control measures including procedural measures, and currently available HSE guidance.

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## EXECUTIVE SUMMARY

### Aim

This work was commissioned as a result of the findings of preliminary research carried out by HSL in 2010, which showed that diacetyl was widely used in the flavouring industry. This earlier research was undertaken in 2010 following reports of ill health to workers exposed to diacetyl in the USA. The aim was to quantify exposure to diacetyl and assess the standard of control in the GB food flavouring industry by visiting 18 sites where diacetyl was said to be used.

As many of the sites originally listed had ceased handling/using diacetyl, the decision was made to summarise the data collected from this study and to include other data from earlier HSL research and Health and Safety Executive (HSE) sector visit reports.

### Main Findings

18 sites were put forward by HSE as potential participants in this research; visits were only possible at three. The reasons for this included:

- Overseas HQ refused permission for the site to participate in this study (three sites);
- Sites had ceased using/handling diacetyl (9 sites);
- Site visited under previous research project (two sites);
- Site enforced by local authority (one site);

At the three sites visited where neat diacetyl is used to make flavours or fragrances, it was reported to HSL that the use of diacetyl was very infrequent (< once a day) and the amount used was typically small (<200g). Customer demand dictated what products were manufactured and the quantities produced at any given time. At all the sites there was capacity to produce much larger quantities of products containing diacetyl than was witnessed during the visits. It was also reported at all three sites visited that the demand for diacetyl products had reduced and that new formulations containing diacetyl were no longer developed.

Results from air monitoring indicated that there is potential for the EU (SCOEL) proposed long-term exposure limit (8-hour TWA) of 0.1ppm\* to be exceeded, even when exposure times are short and quantities handled small. This applied to sites where flavours were produced using neat diacetyl as well as sites using dilute solutions. The highest exposures were measured during the spray drying of liquid formulations to manufacture powdered products.

Exposure control was predominantly reliant on local exhaust ventilation (LEV) with fixed or movable capturing hoods and by using personal protective equipment including tight fitting respirators fitted with organic vapour filters. Limitations in the efficiency of the control measures were found at all the sites visited. With LEV systems, the design

of the hoods did not fully match the process and/or it had not been applied to all the emission sources. It was found that face fitting had not been carried out for tight fitting respiratory protective equipment (RPE) and some deficiencies were found in RPE maintenance/management.

Findings and air monitoring results from previous research visits (four sites) and other HSE visits (two sites) were reviewed and found to be broadly similar to those of this recent work.

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\* At the time of issue the SCOEL proposed exposure limits had been revised to 0.02 ppm (0.07 mg/m<sup>3</sup>) 8-hour TWA and 0.1 ppm (0.36 mg/m<sup>3</sup>) 15-minute STEL. These limits were being considered in a European Consultative Document. <http://www.ser.nl/documents/82310.pdf>

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# 1. INTRODUCTION

Diacetyl is a volatile liquid that occurs naturally in small amounts in butter and fermentation products. It is also called by its chemical name 2,3-butanedione (CAS No 431-03-8). It is manufactured outside Great Britain and imported predominantly for use in formulations by the food flavouring industry, where it is used to impart a buttery creamy taste. There have been reports of damage to the respiratory system caused by inhalation exposure to diacetyl, mainly in the USA. Exposure to high concentrations of diacetyl vapour can have a catastrophic effect on the airways, and the resulting disease is known as ‘bronchiolitis obliterans’.

There is no GB Workplace Exposure Limit (WEL) or US Threshold Limit Value (TLV) for diacetyl; however, the EU Scientific Committee on Occupational Exposure Limits (SCOEL) has recommended an occupational exposure limit of 0.1ppm.\*<sup>[1]</sup> The National Institute for Occupational Safety and Health (NIOSH) in the USA has recommended that exposure should be kept below 0.005ppm (5ppb) averaged over a forty hour week, and a short-term exposure limit (STEL) of 0.025ppm (25ppb).<sup>[2]</sup>

Following the publication of several research reports<sup>[3,4,5,6,7,8]</sup> linking diacetyl to serious lung damage (“bronchiolitis obliterans”) an HSE policy-led working group tasked the relevant HSE sectors and HSL to find out more about the use of diacetyl and the exposures of workers in the industry.

Initial research carried out by HSL in 2010<sup>[9]</sup> as part of an HSE-funded research project called ‘Exploratory work to inform enforcement decisions’ (EXTEND) showed that use of diacetyl was widespread and that exposures were often not as low as the proposed European SCOEL (0.1ppm). It also highlighted that, although controls measures had been implemented, these were not always completely effective.

Literature searches carried out by HSE revealed very little about the extent of use, or the potential exposure risk of workers, in the GB.

The findings from the EXTEND work combined with the lack of available information on the use of diacetyl and the risk posed to the health of people using it, led to the commissioning of this work.

The stated aim of this work was to quantify diacetyl exposure and assess effectiveness of control in the UK food flavour manufacturing industry. This was to be done by measuring diacetyl exposures and assessing the effectiveness of exposure controls at a total of 18 locations.

The sites proposed for inclusion in this research were identified by searching HSE records and by using publically available search facilities. Out of the 18 sites, most had either ceased using/handling diacetyl or moved production abroad.



Only three of the sites were able to participate in this study. This report summarises the findings from the visits and includes the main findings from previous research and results from other HSE visits to companies where diacetyl was used. In total this report includes data from a total of nine separate sites visited between May 2009 and October 2012.

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\*At the time of issue the SCOEL proposed exposure limits had been revised to 0.02 ppm (0.07 mg/m<sup>3</sup>) 8-hour TWA and 0.1 ppm (0.36 mg/m<sup>3</sup>) 15-minute STEL. These were being considered in a European Consultative Document. <http://www.ser.nl/documents/82310.pdf>

## 2. SITE VISITS

Visits were made to three companies that produced formulations containing diacetyl. These were:

- Site 1 - produced liquid and powders for the food, pharmaceutical and animal feed industries;
- Site 2 - produced liquid and powdered flavours for the food industry;
- Site 3 - predominantly produced fragrances for a variety of industries.

### 2.1 Assessment of Controls

A full assessment of controls implemented to prevent exposure to diacetyl was conducted at each site. This involved collecting information on all relevant tasks, and an evaluation of management controls, engineering controls and PPE strategy. Assessment of engineering controls included air velocity measurements and challenge with smoke, where appropriate. Copies of relevant documentation (COSHH assessment, exposure monitoring results, LEV test reports) were obtained. Where the company consented, photographs of relevant work areas and activities were taken.

### 2.2 Air Sampling

Personal air sampling was conducted during activities where diacetyl was used. This included long-term, short-term and task-specific monitoring dictated by the working patterns. Eight-hour time weighted average (TWA) and 15-minute TWA exposures were calculated using the results of the exposure monitoring and information supplied on working practices. Static samples were also taken to determine general background concentrations and, in some cases, to determine the extent of emission from a specific process.

All samples were collected passively onto thermal desorption tubes packed with Chromosorb-106. Personal samples were taken in the breathing zone of the operators (i.e. within 30cm of the mouth).

The samples were analysed for diacetyl using automated thermal desorption (ATD) and gas chromatography with mass spectrometry detection (GC-MS) <sup>[10]</sup>. The method development is described in the Appendix to this report.

## 3. FINDINGS

### 3.1 Site Visits

#### 3.1.1 Material use

It was reported by all the sites visited that the use of diacetyl in formulations was intermittent and reliant on customer demand. This meant that only one or two formulations were prepared using diacetyl during the site visits. At Site 1 the management ensured that all activities where diacetyl was handled were carried out on the day of the visit. At Sites 2 and 3, the only activity monitored was the preparation of one or two liquid formulations.

At all sites neat diacetyl liquid was used, and the method of preparing formulations was very similar. The ingredients were weighed into metal vessels placed on a balance, this process was generally referred to as compounding. After weighing, the ingredients were transferred to a larger container or drum and solvent (typically propylene glycol, ethanol or water) added to give the required strength. The amount of diacetyl weighed out was small (~50-160g) and the activity very infrequent (up to six times per week). The time taken to prepare a formulation was less than 15 minutes. This usage was reported as typical and all the companies reported moving away from using diacetyl where possible, i.e. not developing new formulations containing diacetyl.

Weighing vessels and other small equipment items were cleaned by hand washing in a sink using warm water and detergent. They were then rinsed with water and left to dry. At Site 3 the equipment was also sprayed with isopropyl alcohol prior to drying. At all sites the weighing vessel was rinsed into the finished product, therefore minimising the amount of chemical residue present when cleaning.

Larger equipment such as mixing vessels were typically fitted with 'clean in place' (CIP) equipment, which meant that the operator was more remote from the cleaning process. The cleaning of large vessels was not witnessed, either by using CIP equipment or other techniques.

Quality control samples of raw materials and finished product were routinely taken and analysed by in-house laboratories. The companies also had laboratories where new products were developed by flavour scientists.

#### 3.1.2 Working practices by site

Site 1 manufactured standard and bespoke flavourings for the food, animal feed and pharmaceutical industries. Diacetyl was included in liquid and powdered food flavours. Liquid formulations were weighed out on a balance into a stainless steel beaker prior to being diluted and mixed. Some of the liquid formulations were subsequently spray-dried to produce powdered flavours. The maximum diacetyl concentration in a formulation was 1% and the average annual use of concentrated diacetyl was 10kg.

Including laboratory staff, 13 people were potentially exposed to diacetyl, six of whom worked in production.

Site 2 manufactured a large range of liquid and powder flavours, and vanilla extracts. Diacetyl was included in both liquid and powdered food flavours. Liquid formulations were weighed out on a balance into a stainless steel beaker prior to being diluted and mixed. No products containing diacetyl were put through the spray drying process during the site visit. It was company policy to limit the diacetyl concentration in a formula to less than 1% and the annual usage of concentrated diacetyl was 500kg per year (2011). Including laboratory staff, 20 people were potentially exposed to diacetyl, five of whom worked in production.

Site 3 manufactured a large range of liquid fragrances and flavours. The vast majority of production at the site was of fragrance formulations, the company having moved the production of flavours to an overseas facility. Formulations were weighed out on a balance into an aluminium beaker prior to being diluted and mixed. The average diacetyl concentration in a formulation was 0.5% (max 4.5%) and the approximate annual usage of diacetyl concentrate was 200kg per year (2011/2012). Including laboratory staff, 19 people were potentially exposed to diacetyl, 11 of whom worked in production.

### **3.1.3 Exposure controls**

#### ***Management Controls***

Risk assessments covering exposure to diacetyl had been carried out at two of the sites visited. Site 3 had not carried out formal risk assessments for handling diacetyl, but had issued a series of safety memos that specified the controls to be employed when handling it.

#### ***Engineering Controls***

Site 1 had LEV with both fixed and movable capturing hoods applied to the compounding workstations. The spray-dryer was kept under slight negative pressure, with the exception of the feed tank. There was no forced general ventilation and the compounding was carried out in a large factory unit. The LEV applied to the compounding workstations was subject to an annual examination and test.

Site 2 had LEV with moveable capturing hoods applied to the compounding workstations and a forced general ventilation system with ceiling and low level vents. The LEV was subject to a yearly examination and test.

Site 3 had fixed LEV capturing hoods at seven out of nine compounding workstations. Formulations containing diacetyl were only prepared using workstations fitted with LEV. The compounding was carried out in a large factory unit with no forced ventilation system. The LEV was subject to an annual examination and test; however deficiencies in the quality of the examination were apparent.

In all cases the LEV hoods were not positioned close enough to the weighing process to be fully effective and offered no enclosure of the source.

### ***Personal Protective Equipment***

Production workers at all sites wore suitable overalls, safety shoes, nitrile gloves (powder free latex at Site 3 but they were not seen used) and eye protection in the compounding areas. The cleaning of coveralls and other workwear was also of a good standard. None allowed PPE to be taken off site.

The use of orinasal RPE fitted with combination filters was specified as a control measure when handling diacetyl at two of the sites visited (1&3). The RPE was of suitable type for the short duration of the task and fitted with filters suitable for reducing exposure to diacetyl. RPE was found to be in good condition, however neither site had carried out face fit testing and deficiencies in maintenance were found (i.e. out of date filters, no formal maintenance schedules).

### ***Other Controls***

All sites provided employees with separate eating, hand washing, and changing facilities and those seen were of a good standard. The production areas of most of the sites were operated with strict controls in place to ensure that a high standard of food hygiene was achieved. This meant that breaks were taken in an area segregated from the main work areas and that the operators had to remove coveralls when exiting these areas.

Only Site 2 had carried out health surveillance within the last year; this included lung function testing and a health questionnaire. This was the first time it had been carried out but the latest company policy was to repeat the surveillance every two years.

### 3.1.4 Monitoring results

**Table 1. Summary of airborne diacetyl concentrations**

Site ID and activity	Tasks	Diacetyl Exposure, concentration (ppm)		
		8-hr TWA	15-min TWA	Background measurements
<b>Site 1</b> Preparing liquid flavour compounds and spray-drying	Compounder using diacetyl	0.03	<0.22	-
	Compounder no diacetyl	0.01	-	
	Spray drying	1.7	-	16.9
	Laboratory activities	<0.007 <sup>+</sup>	-	<0.01-0.05 2 samples
<b>Site 2</b> Preparing liquid flavour compounds	Compounder using diacetyl	0.14	0.18	<0.001-0.078 4 samples
	Compounder no diacetyl	0.05	-	
	Other production duties no concentrated diacetyl	0.09 <sup>*</sup>	-	
	Packing diacetyl compound		0.33 <sup>*</sup>	
<b>Site 3</b> Preparing liquid fragrance compounds	Compounder using diacetyl	0.002	0.089	0.014

+ Result based on a 24 minute task-specific exposure result that yielded a non-detected result with a detection limit of less than 0.14ppm. The remaining time the operator was not exposed to diacetyl (background measured as less than 0.01ppm).

\* This result is from a worker who carried out general production duties that did not involve handling concentrated diacetyl. However the 8hr TWA exposure included transferring a dilute diacetyl mixture into drums, for which the 15 minute exposure is shown.

## 3.2 Previous HSE Data

### 3.2.1 EXTEND findings

In 2009/2010 HSL carried out a preliminary investigation into diacetyl use and exposure in the flavouring industry. The work involved visits to four sites where exposure monitoring and an assessment of control measures were carried out.

The main findings were as follows:

**Table 2. Summary of EXTEND exposure results**

Site activity	Diacetyl Exposure, concentration (ppm)		
	8-hr TWA	Maximum task exposure	Background measurements
<b>Site 4 (A)</b> Mixing liquid flavourings	Compounding: 0.27 & 0.67 Mixing: 0.09 – 0.14 Filling: 0.1 – 0.25	Compounding 0.61 (133 mins)	0.12 - 0.74
<b>Site 5 (B)</b> Mixing liquid flavourings	Mixing: <0.01 Adjacent to mixing 0.02	Bottling near diacetyl compounding 0.06 (167 min)	0.03 - 6.5 (highest: at diacetyl booth extract, 8 min)
<b>Site 6 (C)</b> Mixing & drying flavourings	Pouring flavour into carrier 4.4 & 15.8	Pouring flavour into carrier 179 (4 min)	0.9 – 3.4
<b>Site 7 (D)</b> Subdividing flavourings, adding them to dosing tank	Lab work 0.003 Preparing butter 0.006 Preparing flavourings 0.009	Preparing flavourings 0.14 (30 min)	0.001 – 0.13

All the sites visited as part of the EXTEND research employed engineering controls in the form of LEV. Three were systems fitted with capturing hoods with the remaining site using a booth that required operators using RPE to work inside (Site 5). Deficiencies in the design and/or effectiveness of all the LEV systems were reported.

Appropriate RPE was used by operators handling diacetyl at Sites 5 and 6.

### 3.2.2 Other planned visits

Reports were identified from two other planned visits that included exposure measurements. One was from a company that produced powdered foods and beverages using a food flavour containing ~0.3% diacetyl (Site 8); visited in November 2010. The other was from a manufacturer of margarine where flavours containing 2.5-15% diacetyl were used in the process (Site 9); visited in April 2011.

**Table 3. Data from other planned visits**

Site activity	Tasks	Diacetyl Exposure, concentration (ppm)		
		8-hr TWA	15-min TWA	Background measurements
<b>Site 8</b> Powdered food and drink manufacture	Weighing/compounding	0.004 & 0.008*	-	0.009-0.046
<b>Site 9</b> Margarine manufacture	Compounding/Weighing	0.71 <sup>#</sup>	-	0.76 & 0.92
	Decanting compound to mixing vessel.	-	0.14	0.27-1.49 One further result >380 <sup>^</sup>
	QC sampling 'concentrate' tank. Not neat diacetyl	-	1.66	

\* Results are from a 15 and 89 minute sampling period respectively, not 8-hr-TWA.

# Result is a 'worst case' 8-hr TWA assuming that the entire shift was spent working with diacetyl. The analysis was also subject to greater level of uncertainty because the safe sampling volume (7 litres) was exceeded.

<sup>^</sup> This sample was taken on the edge of a vessel containing margarine. The sampler was grossly overloaded with acetic acid which interferes with the diacetyl analysis; therefore the actual diacetyl concentration is very likely to be higher.

No engineering controls or RPE were used at Site 8. Site 9 employed LEV with a movable capturing hood at the weighing operations, and appropriate RPE was used when handling diacetyl-containing materials.

### 3.3 Tabulated Summary of all Available Data

The following table shows a summary of all the data collected from this study and that of other HSE work. It shows the total number of exposures measured and the numbers that exceed the SCOEL recommended limit. It also details the controls measures applied and the adequacy of LEV or RPE used.

The numbers shown for risk (COSHH) assessments and the thorough examination and testing (TEXT) of LEV indicate sites that had carried out these activities but does not mean that they were fully compliant with the relevant regulations.



**Table 4. Summary data collected and previous data**

	<b>This Project</b>	<b>EXTEND</b>	<b>Other</b>	<b>Total</b>
Number of sites visited	3	4	2	9
Number of 8hr TWA exposure results	7	14	2	23
Exposure Over Proposed EU OEL	2	8	1	11
No. of sites with a risk (COSHH) Assessment	2	4	1 (n/k 1)	7(8)*
No. of sites with LEV applied to process	3	4	1	8
No. of sites with fully effective LEV	0	0	0	0
No. of sites with LEV TExT	2	(n/k 4)	0	2(6)*
No. of sites using RPE	2	3	1	6
No. of sites where RPE suitable	2	2	1	5
No. of sites where RPE fit tested or not required	0	1	0	1
No. of sites RPE maintenance issues	2	(n/k 2)	1	3(5)*

n/k = not known because information was not recorded.

\* The figure in brackets shows the total including those where information was not recorded.

**Table 5. Range of exposures for each task**

<b>Process/Activity</b>	<b>Range of Exposure 8hr TWA (ppm)</b>
Weighing/dispensing/compounding	0.002 - 0.71
Charging/mixing	0.003 - 15.8
Decanting/filling/packing	0.09 - 0.25

## 4. DISCUSSION

Of the 18 sites originally identified as being potentially suitable for the purposes of this research only three were able to co-operate. The reasons for this included:

- Overseas HQ refused permission for the site to participate in this study (three sites);
- Sites had ceased using/handling diacetyl (9 sites);
- Site visited under previous research project (two sites);
- Site enforced by local authority (one site);

In addition to data collected during this project, findings from six HSL visits to other companies using diacetyl have been included, four carried out under EXTEND and two other HSE visits.

The UK has not set an exposure limit for exposure to diacetyl. SCOEL has proposed a limit of 0.1ppm and NIOSH recommend that exposure is kept below 0.005ppm (averaged over a 40 hour week) and a STEL of 0.025ppm.

From the three site visits carried out, two out of seven 8-hr TWA exposures exceeded the proposed EU exposure limit (0.1ppm). The highest exposure was measured for a single sample taken on an operator of a spray drying machine that was preparing a powder containing diacetyl. This yielded a task-specific result of 3.7ppm and a calculated 8-hr TWA of 1.7ppm. A static sample placed in the vicinity of the spray-dryer gave a result of 16.9ppm.

Including the data from all the visits identified, of a total of 23 8-hr TWA exposures measured, 11 exceeded the proposed EU exposure limit (0.1ppm). Although not directly comparable to exposure limits, 23 out of the 37 background air samples were over 0.1ppm. A number of the background measurements were taken close to processes and represent a worst case measurement rather than a true background level. The highest exposures measured during the EXTEND study were from a company where spray-drying of diacetyl-containing flavour compounds was carried out. Two calculated 8-hr TWA exposures for process operators were 4.4 and 15.8ppm. The maximum task-specific exposure was 179ppm taken over a 4-minute period where flavour mix was poured into the carrier solution.

All three sites visited had applied LEV fitted with capturing hoods to the compounding weighing workstations. At two sites the position of the hood was adjustable. In all three cases the hoods were not placed close enough to the process and did not enclose the source enough to be fully effective. HSE guidance HSG258 paragraph 146 <sup>[11]</sup> states that capturing hoods are usually only effective when the source is within two hood diameters of the hood face. It also states that the effective capture zone is severely limited, particularly for small hoods. In all cases testing the hoods with smoke showed that the source of diacetyl was outside the effective capturing zone of the hood.

Most of the sites visited under EXTEND and other HSE work (five out of six) used LEV fitted with capturing hoods. One site used a walk-in booth for the weighing operations. Limitations in the efficiency of this LEV were noted at all of the sites.

The highest exposures were measured at two sites where monitoring was carried out during the spray-drying of formulations containing diacetyl. The three calculated 8-hr TWA exposures were 1.7, 4.4 and 17.9 ppm. The two highest exposures included handling of diacetyl and compounding activities without LEV that were carried out in the vicinity of the spray-drying equipment. Therefore the high exposures may not be directly attributable to the spray-drying process. Task specific exposure for an operator packing and sieving powder was 4.5 ppm.

Suitable RPE for reducing exposure to diacetyl during the tasks witnessed would be a minimum of an orinasal respirator fitted with Type A filter, which is appropriate for organic gases and vapours with a boiling point above 65 °C. For RPE of this type to be fully effective the following is required<sup>[12]</sup>:

- Face fit testing for the type of respirator selected
- Maintenance schedule, with particular attention to filter changing
- Operator training in use and maintenance of RPE
- Supervision of staff using RPE

At two of the three sites visited, RPE was used for activities where diacetyl was handled. The RPE and filters were suitable for the tasks, however at both of the sites no face fit testing had been carried out and deficiencies were noted in the maintenance of RPE.

Suitable RPE was specified at two out of four EXTEND sites and one out of two other site visits. Only one of these sites had carried out face fit testing and issues with use and maintenance were reported in some cases.

Other PPE specified at all sites was appropriate to the tasks it was used for and the availability and cleaning arrangements were adequate. Hygiene facilities, storage lockers and eating and drinking arrangements were adequate at all sites visited.

Although there appears to be few sites manufacturing and/or blending diacetyl-containing products, the exposure to operatives can be significant if appropriate controls measures are not present or used correctly.

## 5. CONCLUSIONS

Due to a high fallout in the number of sites able to participate in this project, the amount of data was limited and therefore drawing robust conclusions is difficult. However a number of issues presented themselves during this work that appeared to agree with the data collected under EXTEND and data from other reactive work.

For all of the locations visited, diacetyl exposure was non-continuous and tended to be at intermittent intervals, and for relatively short periods ranging from a few minutes to approximately half a work shift. The exposures were also typically less than one day a week.

There was potential for 8-hr TWA exposures for diacetyl to exceed the EU (SCOEL) proposed long-term exposure limit of 0.1ppm, even when the amount of diacetyl used was small and the exposure time short. The highest exposures were between 15 and 150 times the SCOEL proposed limit and were measured at sites where spray drying was carried out.

Deficiencies in the engineering controls, predominantly LEV, were noted at all the sites included in this report. Although the control effectiveness was variable, in all the cases, the design of the LEV hood did not fully match the process it was controlling. In some cases control had not been applied to all sources of emission.

Workers were using RPE during operations where measured exposures exceeded the EU proposed limit but the protection factors offered (10&20) would not be enough to reduce the highest measured exposures to below this limit.

Where RPE had been specified as a control measure, the type used was suitable in most cases for reducing exposure to diacetyl, however face fit testing had not been carried out in the majority of cases. At both sites where RPE was used during the recent visits, problems with the maintenance and management of RPE were noted.

## 6. RECOMMENDATIONS

In view of the serious health effects associated with diacetyl exposure, the hierarchy of control measures outlined below should be implemented:

- Substitution – replace diacetyl with a less hazardous material
- Process change – use diacetyl in a less volatile form (encapsulated, pre-compounded)
- Process change – keep diacetyl cool/refrigerated and dispense, weigh, charge as the last stage in process
- Enclose – carry out as many stages of the process in closed vessels
- Partial enclosure – weigh & dispense in designated extracted booths
- Local exhaust ventilation (LEV) – apply to enclosed vessels, dispensing booths, charging ports on mixing vessels, discharge chutes etc.
- Procedural measures – limit the time individuals are likely to be exposed to diacetyl-containing materials
- Suitable PPE – including impervious overalls, gloves, eye protection and respiratory protective equipment (RPE). RPE should have the correct filters against diacetyl use.

All control measures must be maintained in an efficient state, in efficient working order, in good repair and in a clean condition.

Where LEV is employed, it should be designed to provide control at source for the tasks being undertaken. Operatives must be trained in proper use of the LEV so the system is used to achieve the desired standard of control.

LEV must be thoroughly examined and tested at intervals not exceeding 14 months. Information relating to all aspects of LEV can be obtained from HSE Guidance booklet HSG258.<sup>[11]</sup>

Where RPE is used, it is necessary to implement an RPE management system, including selection, provision, replacement, inspection, maintenance and testing. All persons using RPE that is reliant on a good seal with the wearer's face should be face-fit tested to ensure the effectiveness of the type provided. Further details of RPE management are provided in HSE Guidance booklet HSG 53.<sup>[12]</sup>

'COSHH Essentials' Guidance sheets published by HSE provide further information on exposure control and LEV systems<sup>[13]</sup>.

All employees carrying out work with diacetyl, either in the neat form or with mixtures containing the material, should be informed of the hazardous nature of the substance and the measures required to minimise their exposure. They should be instructed and trained appropriately in safely carrying out the tasks involving diacetyl-containing materials.

As the clinical effects of diacetyl exposure are recognised, appropriate health surveillance of exposed personnel is recommended. Further details of this can be obtained from an occupational health provider.

## 7. REFERENCES

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- 11) HSE (2011). "Controlling airborne contaminants at work. A guide to local exhaust ventilation (LEV)" (HSG258).
- 12) HSE (2013). "Respiratory protective equipment at work. A practical guide" (HSG53).

### 13) HSE COSHH Essentials Guidance Sheets

- 200 Local exhaust ventilation  
<http://www.coshh-essentials.org.uk/assets/live/G200.pdf>
- 201 Fume cupboard  
<http://www.coshh-essentials.org.uk/assets/live/G201.pdf>
- 202 Laminar flow booth  
<http://www.coshh-essentials.org.uk/assets/live/G202.pdf>
- 206 Sack filling  
<http://www.coshh-essentials.org.uk/assets/live/G206.pdf>
- 210 Charging reactors and mixers from a sack or keg  
<http://www.coshh-essentials.org.uk/assets/live/G210.pdf>
- 212 Drum filling  
<http://www.coshh-essentials.org.uk/assets/live/G212.pdf>
- 213 Drum emptying using a drum pump  
<http://www.coshh-essentials.org.uk/assets/live/G213.pdf>
- 214 Weighing solids  
<http://www.coshh-essentials.org.uk/assets/live/G214.pdf>
- 217 Mixing liquids with other liquids or solids  
<http://www.coshh-essentials.org.uk/assets/live/G217.pdf>



## 8. APPENDIX: MEASUREMENT OF DIACETYL BY THERMAL DESORPTION

### 8.1 Introduction

The United States Occupational Safety and Health Administration (OSHA) published Method PV2118, a partially evaluated method for diacetyl, in January 2003, with a subsequent revision in September 2006 (OSHA, 2006). Sampling is carried out onto two silica gel tubes in series for 60 minutes at a flow rate of 50 ml/min. The sample tubes are then desorbed with ethanol/water (95:5) and analysed by GC and flame ionisation detection (FID). The stated limit of detection (LoD) for this method is 0.3 ppm. The OSHA method is not affected by humidity and is relatively stable under standard sample storage conditions.

Also in 2003, NIOSH published Method 2557, another partially evaluated method for diacetyl (NIOSH, 2003). Sampling is carried out onto Anasorb carbon molecular sieve (CMS) solid sorbent tubes at a flow rate of between 10 and 100 ml/min with subsequent desorption into acetone/methanol (99:1) and analysis by GC FID. A note attached to the method states that there is a suggestion that method is affected by relative humidity with data showing high humidity levels resulting in an underestimation of true concentrations.

During a stakeholder meeting hosted by OSHA in October 2007 (OSHA, 2007) it was stated that NIOSH no longer considers Method 2557 to be the preferred sampling method due to the humidity issues. The point was also made by one stakeholder that a thermal desorption method was a better alternative to both the NIOSH and OSHA methods, providing a lower detection limit. During the meeting it was also suggested that the use of real-time monitors containing a photo-ionisation detector (PID) would have limitations for monitoring diacetyl because of the complex mixtures involved in flavour manufacture leading to interferences.

Due to the increasing concern of workplace exposure to diacetyl, in 2008 OSHA published two new validated sampling and analytical methods for diacetyl, Method 1012 (OSHA, 2008a) and Method 1013 OSHA, 2008b). These two methods allowed longer sampling times and had lower quantitation limits than their previous method (PV2118). The new methods were also validated for acetoin. The difference between the two methods is that Method 1012 is intended for measurement of lower concentrations of diacetyl and acetoin. In both methods sampling is carried out onto two specially dried silica gel tubes in series at a flow rate of 50 ml/min (long-term) or 200 ml/min (short-term). In the case of Method 1012, the sample tubes are desorbed with ethanol/water (95:5) and analysed by GC-FID, whilst for Method 1013 the sample tubes are desorbed with ethanol/water (95:5) containing 2 mg/ml of *O*-(2, 3, 4, 5, 6-pentafluorobenzyl) hydroxylamine hydrochloride (PFBHA) and analysed by GC-ECD. The stated reliable limit of quantitation for Method 1012 is 11 ppb, whilst for Method 1013 it is 1.5 ppb. The method states that samplers must be protected from the light during and after sampling with aluminium foil or opaque tape. Method 1012 also states that, for diacetyl, the derivatisation reaction, may take up to 36 hours to reach completion (acetoin, which only has one carbonyl group, still takes around 3 hours).

Because of these limitations, particularly with regard to reaction time and LoD, it was decided to carry out some method development at HSL to investigate whether existing thermal desorption (ATD) based sampling and analytical procedures, i.e. HSE Method 72 (HSE, 1993) could be adapted for measurement of diacetyl. Experience with ATD methods at HSL has shown them to combine good levels of sensitivity, particularly in combination with mass spectrometry, with an easy to use sampling device and a simple, rapid analysis. This method development is described below.

## 8.2 Preparation of Calibration Standards

To test the suitability of methanol as a solvent for diacetyl calibration a single standard at approximately 0.2 mg/ml diacetyl in methanol was prepared gravimetrically. This standard was subsequently spiked onto four replicate Chromosorb 106 (C-106) thermal desorption (TD) sorbent tubes at a tube loading of approximately 1 µg. For comparison, a gas standard was prepared by addition of 5 µl of diacetyl to a 1-litre air flask (volumetrically). Three different volumes were subsequently withdrawn from this flask and spiked onto C-106 TD sorbent tubes to produce three standards in the approximate range 0.5 to 2.3 µg tube loading. Analysis was by ATD-GC-FID.

There was no evidence from the chromatograms of the formation of other compounds by a reaction of the diacetyl with methanol in the liquid standards. The results from the 2 sets of standards compared well and confirm methanol is a suitable solvent for the preparation of calibration standards for diacetyl. However, it is recommended that standards are freshly prepared for each calibration as methanolic solutions have shown evidence of degradation after only a few days, with the diacetyl being converted to various methoxyether compounds.

## 8.3 Storage Tests

A preliminary storage test of nine C-106 TD tubes was carried out. The tubes were spiked with approximately 1 µg of diacetyl from a gas standard prepared volumetrically in a 1-litre round-bottom flask. After spiking, the tubes were capped and stored in a re-sealable plastic bag in the open lab at room temperature. After approximately 24 hours three of the nine loaded tubes were analysed and compared with three freshly spiked tubes. Analysis of a further three tubes was done after a storage period of a week and the final three tubes after a storage period of two weeks. The results of these analyses are summarised in Table 1.

**Table 1: Recovery of Diacetyl from Stored C-106 ATD Tubes**

<b>Storage Period (days)</b>	<b>1</b>	<b>7</b>	<b>14</b>
<b>% Recovery ± S.D. (n=3)</b>	<b>97 ± 2</b>	<b>95 ± 0</b>	<b>85 ± 4</b>

The results in Table 1 indicate there is a significant loss of diacetyl from the C-106 sorbent after two weeks storage at room temperature. There is no significant loss of diacetyl after one week of storage. The loading method used may have speeded up the loss of analyte from the sorbent tube because the diacetyl will have been placed at the edge of the sorbent bed rather than further along the sorbent bed, which would be the case with actual samples.

## 8.4 Breakthrough Test

The sampling breakthrough volume of diacetyl was assessed for C-106 sorbent. A gas standard was prepared and loaded onto conditioned C-106 sorbent tubes by fitting a tube (primary) to the spiking rig with a second tube (secondary) fitted to the non-sampling end of the primary tube, in series. Both tubes were then exposed to nitrogen at an approximately 50 ml/min flow rate. Two

replicate sets of tubes were loaded in this way for 10, 20 and 60 minutes to give the following sample volumes; 500 ml, 1 litre and 3 litres. The ‘secondary’, backup tube is there to capture any diacetyl that has broken through the first tube due to the volume of nitrogen put through.

The results of these tests, normalised relative to the recovered values for 500 ml (which are assumed to be fully retained), are shown in Table 2. These results suggest no breakthrough for a sample volume of 1 litre. For a sample volume of 3 litres there is a loss of diacetyl from the primary tube, but none found on the secondary tube, suggesting that it is not the result of breakthrough. Further tests would be needed to ascertain the breakthrough volume, however the results indicate that a safe sample volume, at a flow rate of 50 ml/min, of up to 3 litres.

**Table 2: Breakthrough of Diacetyl on C-106 ATD Tubes**

Sample Volume	0.5-litres		1-litre		3-litres	
Tube	1° tube	2° tube	1° tube	2° tube	1° tube	2° tube
Recovery Ratio	1.00	0.00	1.00	0.00	0.90	0.00

Approximate tube loading = 0.9 µg; Results normalised against the mean of the 0.5 litre tube

## 8.5 Limits of Detection

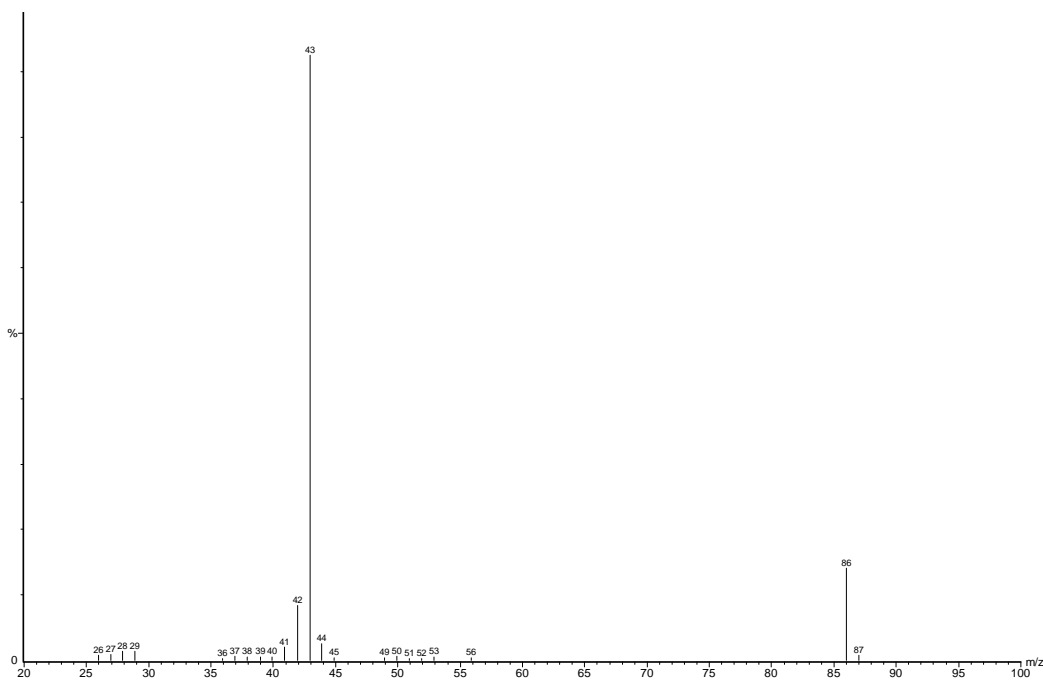
The ATD-GC-FID was set up to carry out analysis of diacetyl with the approximate range of 0 to 80 µg per sample. This produced a limit of detection (LoD) of around 0.02 µg which, for a typical sample volume of around 7.2-litres (240 minutes at 30 ml/min), corresponds to an airborne concentration of approximately 0.001 ppm (1 ppb). This is sufficient for examination of airborne concentrations below 0.05 ppm, which is the concentration range of interest. However, it was anticipated that "real" samples would probably contain a significant number of other components, which might adversely affect the detection limit and so the decision was made to switch to analysis using detection by mass spectrometry (MS) which, as well as offering better sensitivity would also offer improved selectivity. This would also allow the possibility of using diffusive samples and/or short-term (15-minute) sampling.

The ATD-GC-MS instrument conditions for analysis of diacetyl are shown in Table 3. Using these conditions diacetyl elutes with a retention time of 6.7 minutes. Acetoin, a similar compound with one of the carbonyl groups converted to a hydroxy group was also observed, with a retention time of 9.1 minutes. A typical mass spectrum of diacetyl is shown in Figure 1.

Quantitation of diacetyl (and acetoin) was achieved using selected ion monitoring (SIM) using ions 43, 45, 86 and 88 (the 45 and 88 ions are not needed for diacetyl but are present in acetoin). Using this SIM method, the limit of detection was estimated to be less than 0.001 µg, which is 20 times less than obtained using GC-FID, giving a detection limit of around 0.0001 ppm (0.1 ppb) for a 4-hour pumped sample.

**Table 3: Analysis of Diacetyl – ATD-GC-MS Instrument Conditions**

<b>Column Type</b>	VOCOL	<b>Temperature 1</b>	100°C
<b>Column Dimensions</b>	60 m × 0.25 mm × 1.5 µm	<b>Time at Temperature 1</b>	0.50 minutes
<b>Carrier Gas</b>	Helium	<b>Temperature Ramp 1</b>	5°C/minute
<b>Gas Pressure</b>	16 psi	<b>Temperature 2</b>	180°C
<b>Flow Rate (Initial)</b>	2.0 ml/min	<b>Time at Temperature 2</b>	4.5 minutes
<b>Outlet Split</b>	65 ml/min	<b>Temperature Ramp 2</b>	5°C/minute
<b>Inlet Split</b>	Off	<b>Temperature 3</b>	220°C
<b>Desorb Flow</b>	20 ml/min	<b>Time at Temperature 3</b>	1 minute
<b>Desorb Temperature</b>	200°C	<b>Total Run Time</b>	30 minutes
<b>Desorb Time</b>	7 minutes	<b>Quantitation Ions</b>	43; 45; 86; 88



**Figure 1: Mass Spectrum of Diacetyl**



# The occupational hygiene implications of the use of diacetyl in the food flavouring and fragrance industries

Diacetyl is imported predominantly for use in formulations in the food flavouring industry. There have been reports, mainly in the USA, of damage to the respiratory system caused by inhalation. This report presents the findings of a small study to investigate exposures to diacetyl amongst workers employed in the food flavouring and fragrance industries. The report also includes from previous HSE/HSL visits.

Exposures to diacetyl are generally of short duration, nonetheless, there is potential for workers to be exposed to airborne levels above those recommended by the EU Scientific Committee on Occupational Exposure Limits (SCOEL) of 0.1ppm (8-hour TWA) at the time of the study. Following completion of this report, EU (SCOEL) have tabled new proposed limits of 0.02 ppm (8-hour TWA) and 0.1 ppm (15-minute TWA). There is currently no UK Workplace Exposure Limit (WEL). Personal exposures to diacetyl for this study ranged from 0.01ppm to 1.7 ppm (8hr TWA) and 0.09ppm to 0.33ppm (15min TWA).

Local exhaust ventilation (LEV) was provided during most diacetyl processes. The effectiveness of LEV control was variable. This was mostly due to inappropriate LEV hood design relative to the process.

The respiratory protective equipment (RPE) used was, in most cases, the correct type, however in some cases the assigned protection factors offered (10 & 20) may not reduce exposures to below the SCOEL proposed limits. Deficiencies in the face-fit testing, training and management of RPE were noted.

Recommendations refer to hierarchy of control measures including procedural measures, and currently available HSE guidance.

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