Appendix 2 - Report on the characterisation of a particulate matter sample collected using an ACCU located in the vicinity of a poultry unit. Advance Environmental Ref: ae/dddc/pm/accu/01/09/v2.

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# REPORT ON THE CHARACTERISATION OF A PARTICULATE MATTER SAMPLE COLLECTED USING AN ACCU LOCATED IN THE VICINITY OF A POULTRY UNIT FOR DERBYSHIRE DALES DISTRICT COUNCIL



Report on the Characterisation of a Particulate Matter Sample Collected Using an ACCU Located in the Vicinity of a Poultry Unit for Derbyshire Dales District Council

#### **Report submitted to:-**

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1<sup>st</sup> September 2009



#### **EXECUTIVE SUMMARY**

Advance Environmental was contracted by Derbyshire Dales District Council to provide particle characterisation on particulate matter sample collected using an Automatic Cartridge Collection Unit (ACCU) used in conjunction with a Tapered Element Oscillating Microbalance (TEOM) that utilised a  $PM_{10}$  sampling head. The samples, collected by the Environment Agency on behalf of Derbyshire Dales District Council, were reported to have been collected in the vicinity of a poultry unit. Two samples were selected for characterisation using Scanning Electron Microscopy with Energy Dispersive x-ray Spectrometry (SEM/EDS) for particle type, size and relative frequency. This methodology allows discrimination between different types of particulate matter and can be used to apportion likely sources.

Unfortunately, the filters were loaded up-side-down in the ACCU cassettes. This resulted in the collection of particulate matter being restricted to limited areas of the filter, between the filter backing structure.

The samples as presented were dominated by 2 particle species: sulphates and carbon-based materials. Whilst the former were probably representative regional or trans-boundary secondary particles, the carbon-based material included large flakes of skin. This latter material was unusual within ambient particulate matter samples and was possibly associated with poultry litter. However, the sample that represented the wind direction away from the farm recorded a greater proportion of skin. This could be due to the dilution effects caused by sulphates and other aqueous soluble material in the characterised samples along with the effects from the re-suspension of poultry dust.

Therefore, a portion of the samples may be attributed to the poultry units. However, due to sampling issues, the characterisation data was compromised. If information from the characterisation of samples is to be optimised, it is recommended that a standard gravimetric sequential sampler, such as a Partisol 2025, be utilised. This should be configured to collect daily samples in accordance with the requirements of the National Air Quality Strategy. Appropriate samples could be selected based upon daily wind direction and particle loading.



#### **INTRODUCTION**

Advance Environmental was contracted by Derbyshire Dales District Council to provide particle characterisation on particulate matter sample collected using an Automatic Cartridge Collection Unit (ACCU) used in conjunction with a Tapered Element Oscillating Microbalance (TEOM) that utilised a  $PM_{10}$  sampling head. The samples, collected by the Environment Agency on behalf of Derbyshire Dales District Council, were reported to have been collected in the vicinity of a poultry unit.

Six samples were collected onto 47mm Emfab<sup>®</sup> glass fibre filters. The filters had been labelled, conditioned and weighed by Advance Environmental prior to being dispatched to the Environment Agency for use within the ACCU. After exposure the filters were unloaded and returned to Advance Environmental for weighing. Two of the resultant samples were subsequently selected for characterisation. The compositional and morphological characteristics of these samples are presented in this report and consideration given to the potential provenance of the particulate matter.

Unfortunately, the filters were loaded up-side-down in the ACCU cassettes. This resulted in the collection of particulate matter being restricted to limited areas of the filter, between the filter backing structure.



#### METHODOLOGY

A portion of each filter was mounted onto an aluminium stub using a carbon disc and gold coated. The sample was characterised using Scanning Electron Microscopy with Energy Dispersive x-ray Spectrometry (SEM/EDS) for particle type, size and relative frequency. This included both insoluble material such as mineral particles and aqueous soluble salts such as sodium chloride and calcium sulphate. Approximately 100 individual particles were analysed for the sample. This semi-quantitative methodology allows discrimination between different types of particulate matter and thereby apportions likely sources (Merefield et al, 1999).



#### RESULTS

These samples would usually be subjected to an aqueous extraction with the quantitative analysis of resultant ions, however, it was felt that they were too compromised having been loaded up-side-down. Notwithstanding, it was considered that characterisation could be used to examine if there was a substantial influence of particles associated with the poultry units. As such, samples DDDC 1 and DDDC 2 were selected, after examination by optical microscopy indicated that they represented the samples with the greatest covering of particulate matter. It was purported that sample DDDC 1 accorded with sampling when the wind direction encompassed the poultry unit, whilst DDDC 2 represented the wind direction away from the farm during the period of 10<sup>th</sup> March to 8<sup>th</sup> April 2009.

#### Sample DDDC 1

The composition of the characterised sample is presented in table 1 and graphically in appendix 1. The composition may be divided into 7 categories:

- 1. **Carbon**-based material accounted for 25% relative frequency of particles within the sample. These ranged in size from 0.5 to 50µm with the smaller particles being generally sub-rounded to rounded morphologies and the larger material being increasingly angular and flat (see appendix 2, photomicrographs 1 and 3). The larger particles were probably flakes of skin, their dimensions exceeding the 10µm size selective cut-off due to both the low specific gravity of carbon and the low sphericity (see appendix 2, photomicrograph 3). The smaller carbon particles may be derived from a combustion source.
- 2 Potassium-iron alumina-silicates were present as 2 to 5µm particles, with a mean of 3.3µm and a mode of 3µm. These particles occurred as discrete platelets probably representing clays minerals. They accounted for 8% relative frequency.



- 3 Occasional **quartz / silica** were present.
- 4 **Spherical flyash** particles accounted for 10% relative frequency of the sample and occurred within the size range of 0.5 to 3μm. These products of combustion were predominantly carbon-based with alumina-silicate also present.
- 5 **Angular iron** occurred as 1% relative frequency.
- **Sulphate-based** particles dominated the sample and accounted for 54% relative frequency with a size range of 0.5 to 12µm skewed towards the smaller size. These particles, which typically represent secondary material, are usually aqueous extracted as part of the characterisation process. Within this sample, the sulphates were associated with three measurably cations, namely: magnesium, potassium, calcium and sodium. The presence of the ammonium cation could not be readily determined using Energy Dispersive X-ray Spectrometry.
- A light to medium covering of **soot** particles was observed within the sample (see appendix 2, photomicrograph 3). However, soot was not included in the particle count due to the physical constraints of counting individual soot particles. A feature of such material is that it is typically present as a vast number of ultra-fine particles, 0.05 to 0.2µm in diameter. These can occur either as discrete particles or agglomerates of chains and clusters. Within filter-based particle samples that are collected for equal time periods, such as the 24-hour metric required within the Air Quality Strategy, it is possible to report soot as a relative intensity in order to develop time series-based patterns. However, within the ACCU-based system, with their varying periods of sampling, this is not possible. Notwithstanding, the sample included a light loading of soot.

#### Sample DDDC 2



The composition of the characterised sample is presented in table 2 and graphically in appendix 1. The composition may be divided into 7 categories:

- Carbon-based material accounted for 45% relative frequency of particles within the sample. These ranged in size from 1 to 40µm with the larger particles probably being flakes of skin (see appendix 2, photomicrograph 4). The smaller material included both sub-rounded to sub-angular morphologies.
- Potassium-iron alumina-silicates (clay) accounted for 8% relative frequency of particles within the sample. These were present as 3 to 6µm particles, with a mean of 4.3µm and a mode of 4µm.
- 3. **Quartz / silica** were present as 6% relative frequency with a size range of 4 to 8µm.
- 4. A single alumina-silicate **spherical flyash** was observed in the characterised portion of the sample.
- 5. **Angular iron** (see appendix 2, photomicrograph 4) occurred as 5% relative frequency within a size range of 3 to  $4\mu$ m.
- 6. A couple of particles of **sodium chloride** (sea salt) were observed.
- 8 **Sulphate-based** (see appendix 2, photomicrograph 4) particles accounted for 33% relative frequency with a size range of 0.5 to 5μm. Within this sample, the sulphates were associated with three measurably caions, namely: magnesium, potassium, calcium and sodium.
- 9 A light loading of **soot** particles was observed within the sample.



#### DISCUSSION

The samples as presented were dominated by 2 particle species: sulphates and carbon-based materials. Whilst the former were probably representative of secondary particles derived from the nucleation of atmospheric gases and have a tendency to represent regional or transboundary phenomena, the carbon-based material included large flakes of skin. This latter material was unusual for ambient particulate matter samples and was possibly associated with poultry litter. However, sample reference DDDC 2 recorded a greater proportion of skin. This sample was purported to represent the wind direction away from the farm. Two possible explanations for this anomalous situation include the dilution effect from including sulphates and other aqueous soluble material in the characterised samples and re-suspension of poultry dust.

The larger flakes of skin greatly exceeded the  $10\mu$ m size selective cut-off for PM<sub>10</sub>. This was due to PM<sub>10</sub> representing the median aerodynamic equivalent diameter, whereby sampling has a 50% efficiency cut off below 10µm and with specific gravity and sphericity included in the calculation. Therefore, material that possesses a light specific gravity, such as carbon-based particles, and have a low sphericity, can be substantially larger than 10µm in diameter.

Soot particles were more prevalent in the sample facing the poultry units.

Therefore, a portion of the sample may be derived from the poultry units. However, if information from the characterisation of samples is to be maximised, it is recommended that a standard 'gravimetric' sequential sampler, such as a Partisol 2025, be utilised. This should be configured to collect daily samples in accordance with the requirements of the National Air Quality Strategy. Samples could be selected based upon daily wind direction and particle loading.



### REFERENCES

J R Merefield, I Stone, J. Barron and J Jones

'Techniques for tracing fugitive mineral dusts for nuisance control and health risk'. *Transactions of the Institution of Mining and Metallurgy, 1999* 



# TABLES

Particle type	Relative	Size range	Mean size	Modal size	
	frequency	(µm)	(µm)	(µm)	
	(%)				
Carbon	25	0.5 - 50	7.6	2	
Clay, shale, soil lithoclasts - KAlSi	8	2-5	3.3	3	
Quartz / silica	1	4	4.0	4	
Spherical flyash	10	0.5 – 3	2.0	2	
Angular iron	1	1	1.0	1	
Sodium chloride	0	-	-	-	
Magnesium sulphate	23	3 – 12	5.0	4	
Potassium sulphate	9	2-5	3.0	3	
Calcium sulphate	9	1 – 6	3.2	2	
Sodium sulphate	14	0.5 - 8	3.4	2	
Soot loading	Light to medium – discrete and clumps				

# Table 1. Composition of ACCU filter sample reference DDDC 1

 Table 21. Composition of ACCU filter sample reference DDDC 2

Particle type	Relative	Size range	Mean size	Modal size	
	frequency	(µm)	(µm)	(µm)	
	(%)				
Carbon	45	1 - 40	8.9	2	
Clay, shale, soil lithoclasts - KAISi	8	3 - 6	4.3	4	
Quartz / silica	6	4 - 8	5.7	4	
Spherical flyash	1	2	2.0	2	
Angular iron	5	3 - 4	3.4	3	
Sodium chloride	2	2-3	2.5	*	
Magnesium sulphate	3	3 - 4	3.7	4	
Potassium sulphate	0	-	-	-	
Calcium sulphate	25	0.5 – 4	2.3	2	
Sodium sulphate	5	2-5	3.2	3	
Soot loading	Light (minimal)				

\* Multiple modes



# APPENDIX 1. PARTICLE TYPE AND SIZE DISTRIBUTION GRAPHS.







## APPENDIX 2 PHOTOMICROGRAPHS

Photomicrograph 1 Sample reference: DDDC 1 Description: Magnesium sulphates, clay and carbon (near mid-top)



Photomicrograph 2 Sample reference: DDDC 1 Description: Magnesium sulphate crystal and spherical carbon





Photomicrograph 3 Sample reference: DDDC 1 Description: Large flake of skin plus soot and sulphates



## Photomicrograph 4 Sample reference: DDDC 2 Description: Large flakes of skin plus small angular iron and sulphates



30µm