

Residential Dust Analysis Report Number: U508

25th August 2021

LPD Lab Services Limited

Suite 1, D Building
Glenfield Business Park
Philips Road
Blackburn
Lancashire, BB1 5RZ
United Kingdom
www.lpdlabservices.co.uk
enquiries@lpdlabservices.co.uk
Tel 01254-676074
Fax 01254-278845

Suzanne McLaughlin - West Berkshire Council

Council Offices Market Street Newbury RG14 5LD

<u>Disclaimer</u>

This report shall not be 'published' or 'extracts reproduced' without written permission from 'LPD Lab Services Limited', in accordance with the <u>laboratory's terms and conditions of sale</u>.

LPD Lab Services Limited Suite 1, D Building Glenfield Business Park Philips Road, Blackburn. BB1 5RZ www.pdlabservices.co.uk Tel 01254 676074 FAX 01254 278845

1 Introduction

LPD Lab Services received four dust samples from West Berkshire Council for analysis. The dust was selected at the windows (on the sills) inside three residential properties and from the window sill outside of a nearby commercial property.

Table 1 r	roconte the	identification	of the	rocoived	camples
Table 1 L	resents the	Huenuncation	or the	received	Samples.

LPD ID	Reference	Description	
U508-1	LC1	Flat 3 ground floor. LHS living/dining room window sill - openable window.	
U508-2	LC2	Flat 4 1 st floor. LHS living/dining area window sill - openable window.	
U508-3	LC3	Flat 9 2 nd floor. LHS window sill - openable window.	
U508-4	LC4	External window sill RHS of opening to warehouse, far LHS edge.	

Table 1. Identification of the received samples.

This report discusses the results of light optical microscopy (LOM), scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) performed on the dust samples. The purpose of this work was to understand whether the black particles in the dust could have been tyre particles that originated from the factory.

2 Sample Preparation/Method Details

Low magnification optical examinations were performed on the received dust samples, using a Leica MZ12 binocular zoom microscope. Subsamples of the dust were transferred to sticky SEM stubs and then sputter coated with a thin layer of gold prior to analysis. The coating of samples in this way is standard practice for electron microscopy of non-conductors. The gold film creates a conductive path for the electrons and helps prevent localised sample charging and beam distortions, giving superior images compared to a non-coated sample.

The SEM examination was carried out using a Philips XL30 SEM equipped with a tungsten filament and an EDX analyser. Backscattered electron (BSE) imaging, where contrast differences are generally signs of different average elemental compositions, was employed. Generally, heavy elements have a lighter BSE contrast and light elements have a darker BSE contrast.

EDX analysis is a semi-quantitative technique based on the detection of X-rays emitted from the top few micrometres of the sample surface after interaction with the electron beam. X-rays are characteristic for each element present. Even though EDX does not indicate the chemical states of elements, it can sometimes provide a degree of compound information from relative atomic

proportions. Because gold coating is part of the sample preparation and not part of the sample it must be discounted when interpreting the EDX data.

The optical examinations were done on the 6^{th} of August and the SEM and EDX analyses were performed on the 20^{th} of August 2021. The results relate to the samples after suitable sample preparation.

3 Results and Comments

3.1 Light Optical Microscopy

All of the samples contained numerous particle types, such as fibres from clothes, cellulose from paper and wood, particulates from building materials and sand. These are not uncommon in residential dust samples. However, all samples also contained numerous optically-black particles that were immediately noted as more unusual for this type of sample. The particulate matter originating from plants and also from fibres common from clothing, etc., were not deemed relevant and were not analysed further during this examination.

The following groups of images show, for each sample, representative dust particles at lower and higher magnification in their as-received state - on first opening the aluminium foil. Images are also shown at higher magnification in the aluminium foil, and at lower and higher magnifications on the SEM stub, prior to loading into the SEM.

3.1.1 Sample LC1

The dust collected in the foil showed a range of materials. There were many wood fibre fragments and some man-made fibres (white/blue and red) as shown in Figure 1 and Figure 2. There was also some moss like material (green) and many irregular shaped black particles.

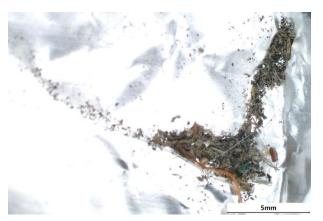


Figure 1. Lower magnification optical image of sample LC1 as received in the aluminium foil.



Figure 2. Higher magnification optical image of sample LC1 as received in the aluminium foil.



Figure 3. Lower magnification optical image of sample LC1 on the SEM stub.

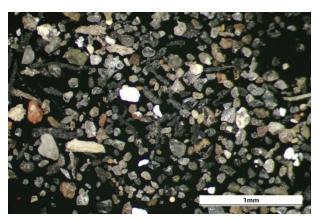


Figure 4. Higher magnification optical image of sample LC1 on the SEM stub.

The sample LC1 also had angular inorganic particles of varying colour suggesting a mineral origin (Figure 4). The particles showed a range of colours from light grey, light brown and red - typical of building materials. Dispersed amongst these particles were black particles of around the same size.

3.1.2 Sample LC2

The LC2 sample was composed of many brown wood fibre particles (Figure 5 and Figure 6) and also many black particulates and some angular particles that appeared like inorganic mineral. There were bright white irregular shaped particles probably of man-made material (paint/plaster?).



Figure 5. Lower magnification optical image of sample LC2 as received in the aluminium foil.



Figure 6. Higher magnification optical image of sample LC2 as received in the aluminium foil.

The large brown twisted 'fibres' evident in Figure 7 and Figure 8 (samples removed to SEM stubs) were natural in origin (possibly plant material).



Figure 7. Lower magnification optical image of sample LC2 on the SEM stub.

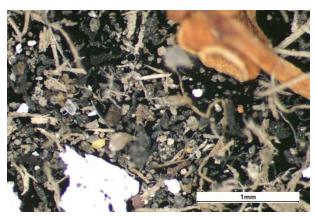


Figure 8. Higher magnification optical image of sample LC2 on the SEM stub.

3.1.3 Sample LC3

The sample was composed of large off-white mineral particles (Figure 10), typically of the same size as sand particles from building materials. Within the foil, many irregular and 'stringy' black particles were also observed.

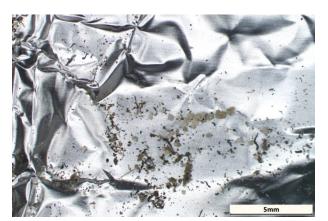


Figure 9. Lower magnification optical image of sample LC3 as received in the aluminium foil.



Figure 10. Higher magnification optical image of sample LC3 as received in the aluminium foil.



Figure 11. Lower magnification optical image of sample LC3 on the SEM stub.

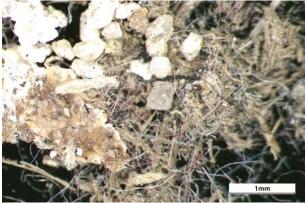


Figure 12. Higher magnification optical image of sample LC3 on the SEM stub.

Concentrating the dust onto the SEM stub showed also that there were many fibres, cotton and man-made (Figure 12). The fibres had a range of colours from white, blue and red - typical of clothing. There were also very large flat

white particles seen which are indicative of white paint flakes or white painted plaster. Some angular particles on the SEM stub were also typical of building materials, being mineral in character.

3.1.4 Sample LC4

Dust sample LC4 showed an overall brown appearance, as seen in the low magnification optical image of Figure 13 and at higher magnification in Figure 14, showing many brown wood fibre fragments. Concentrating on the inorganic particles shown dispersed on the right-hand side of the SEM stub (Figure 15) appeared typical of particles generated from building materials (brick-work, cement/concrete), having a predominance of inorganic angular particles. There were black irregular particles dispersed throughout the sample.

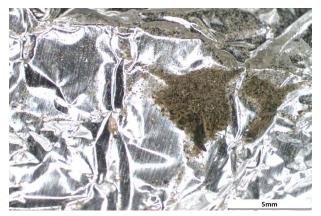


Figure 13. Lower magnification optical image of sample LC4 as received in the aluminium foil.



Figure 14. Higher magnification optical image of sample LC4 as received in the aluminium foil.



Figure 15. Lower magnification optical image of sample LC4 on the SEM stub.



Figure 16. Higher magnification optical image of sample LC4 on the SEM stub.

Large wood fibres were observed as well as some man-made fibres (blue and white, as shown in Figure 15, typical of clothing). The number of clothing-type fibres in this sample was very low compared to the other samples.

3.2 Summary Overview of Optical Inspection

The common trends of the samples LC1 to LC4 were relatively large angular and rounded mineral particles typical of building materials such as sand particles and particles from concrete/cement.

All of the samples showed brown fragments and degraded particles of wood. These too can be generated from wooden objects. As wood is somewhat ubiquitous, the source could be from anything found internally such as window frames, furniture and externally - pallets, wood fencing etc.

Many of the samples showed fibres from clothing and man-made fabrics - LC3, particularly, but also very significant amounts in LC1 and LC2. These man-made fibre types were far less evident in sample LC4.

All of the samples showed black particles dispersed throughout the dust. With the variation in levels of other materials (fibres, wood & plant, minerals), the relative amount of the black particles is difficult to gauge.

The sample form, the "commercial area" – LC4 – appears quite similar, in overview, to sample LC2 (albeit with far less clothing-type fibre).

3.3 SEM Examination and EDX Analysis

3.3.1 Sample LC1

Figure 17 shows a low magnification BSE image of some of the particles from sample LC1. Four particles selected for EDX analysis are indicated in the image.

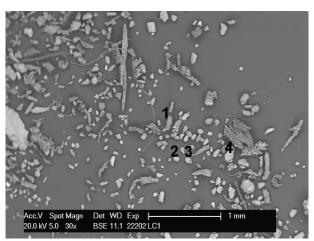


Figure 17. Lower magnification BSE image of some particles on sample LC1.

Figure 18 shows a higher magnification BSE image of particle 1. Figure 19 shows the EDX spectrum and composition of the analysed area on particle 1. The data indicates that these particle types are an agglomerate of fine particles which consist mainly of carbon, oxygen and calcium with some silicon, iron, aluminium and magnesium and trace levels of other elements. It is most likely an agglomerate particle of building materials; probably limestone (calcium carbonate), sand (silicon dioxide) and also cement (calcium alumino-silicate),

minor amounts of gypsum (calcium sulphate, e.g. building plaster) and also a large amount of carbon (i.e. likely organic material), which has bound the particles together.

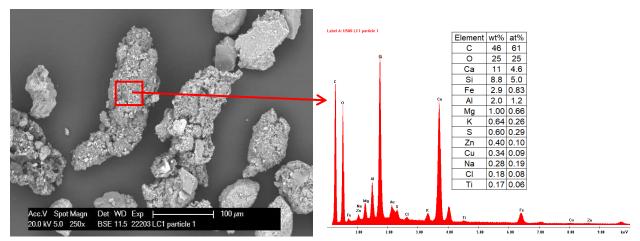
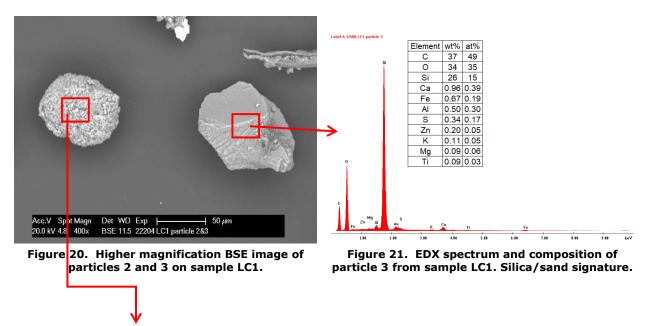


Figure 18. Higher magnification BSE image of particle 1 on sample LC1.

Figure 19. EDX spectrum and composition of particle 1 from sample LC1. Silica/sand, calcium carbonate/cement signatures.

Figure 20 shows a higher magnification BSE image of particles 2 and 3 and Figure 21 and Figure 22, respectively, shows the EDX spectra and compositions of the analysed areas on the particles. Particle 2 consisted mainly of oxygen, calcium, sulphur and carbon and trace levels of other elements; this particle has a signature of mainly calcium sulphate (gypsum/plaster) with possibly some organic material (or possibly even calcium carbonate (limestone)). Gypsum or plaster particles are naturally very small (sub 10 μ m in size) and so this particle represented a relatively large chunk of gypsum (plaster) particles.

Particle 3 consisted of mainly carbon, oxygen and silicon with trace levels of other elements. The angular mineral shape and EDX composition suggest that it was likely to be sand from building materials. Natural sand often has a silicon dioxide signature but with trace levels of other elements, but characteristically iron which gives sand its orange/brown colouration.



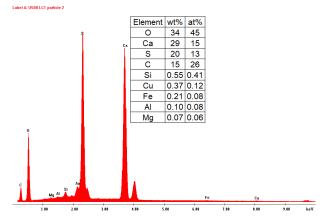


Figure 22. EDX spectrum and composition of particle 2 from sample LC1. Calcium sulphate/gypsum/plaster signature.

Figure 23 shows a higher magnification BSE image of particle 4, an angular particle of uniform composition, and Figure 24 shows the EDX spectrum and composition of the analysed area. The composition was mainly silicon and oxygen with trace levels of other elements. This particle was also indicative of a silica or sand particle (as was particle 3 from this sample).

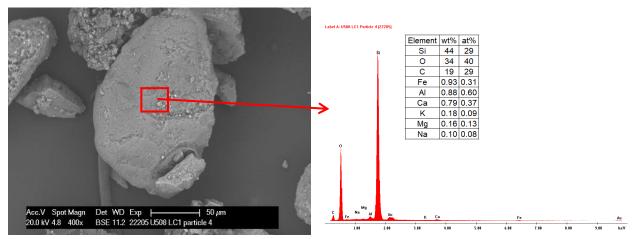


Figure 23. Higher magnification BSE image of particle 4 on sample LC1.

Figure 24. EDX spectrum and composition of particle 4 from sample LC1. Silica/sand signature.

3.3.2 Sample LC2

Figure 25 shows a low magnification BSE image of the particles on sample LC2. Six particles were selected for EDX analysis and these are indicated in the image. There were many fibres in the sample from cotton fibres to man-made fibres and also natural fibres.

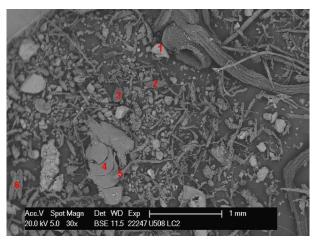


Figure 25. Lower magnification BSE image of some particles on sample LC2.

Figure 26 shows a higher magnification BSE image of particle 1, and Figure 27 shows the EDX spectrum/ composition of the analysed area. The angular mineral appearance of the particle and predominantly silicon and oxygen peaks indicated a sand/silica particle.

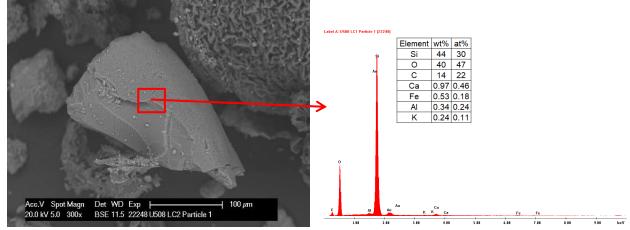


Figure 26. Higher magnification BSE image of particle 1 on sample LC2.

Figure 27. EDX spectrum and composition of particle 1 from sample LC2. Silica/sand signature.

Figure 28 shows a higher magnification BSE image of particle 2, and Figure 29 shows the EDX spectrum and composition of the analysed area. It consisted of carbon and oxygen with nitrogen, potassium, silicon, phosphorus and sulphur and trace levels of other elements. The particle appeared rounded and more natural in shape compared to the angular mineral particles nearby. signature was predominantly carbon and a natural organic particle due to the nitrogen, phosphorous and sulphur indicating proteinaceous or complex organic particle. The particle was too large for a skin flake (of the order of 20 µm in size) and so could be from plant material.

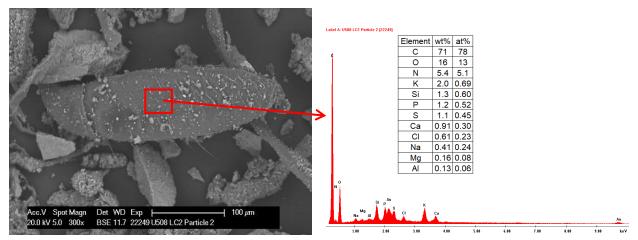


Figure 28. Higher magnification BSE image of particle 2 on sample LC2.

Figure 29. EDX spectrum and composition of particle 2 from sample LC2. Natural organic/proteinaceous signature.

Figure 30 shows a higher magnification BSE image of particle 3 and Figure 31 shows the EDX spectrum and composition of the analysed area. It consisted of carbon and oxygen with nitrogen, calcium, silicon and zinc and trace levels of other elements. The shape showed an organic looking particle rounded and 'pill' shaped as though it had been 'rolled' during its lifetime. A definitive identity could not be given for this particle. The particle had 'natural' organic signatures of carbon, low nitrogen and sulphur, but could also be man-made. Some man-made organic materials can contain nitrogen and also sulphur and zinc could be from cross-linking agents used in rubbers. The surface 'decoration' of the particle could also contain zinc oxide which is a compound found in cosmetics/ paints and many other applications. As the trace elements and majority signals can be interpreted in many different ways, a definitive identification cannot be made.

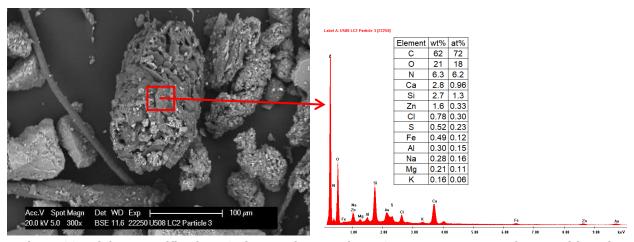
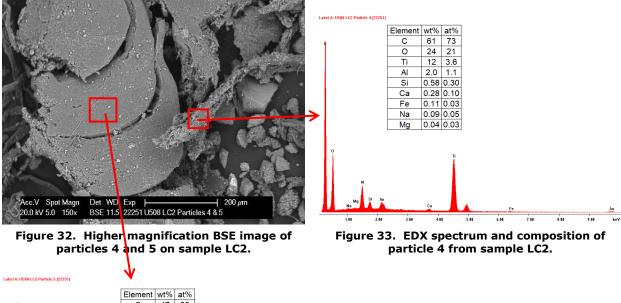


Figure 30. Higher magnification BSE image of particle 3 on sample LC2.

Figure 31. EDX spectrum and composition of particle 3 from sample LC2.

Figure 32 shows a higher magnification BSE image of particles 4 and 5. The EDX spectra and compositions of the analysed areas are shown in Figure 33 and Figure 34, respectively. Particle 4 consisted of carbon and oxygen with nitrogen, silicon, calcium, aluminium, iron, nitrogen and sodium and trace levels of other elements. The particle appeared as a flat scalloped surface with regular cut marks along its length. The particle was bright white and was likely to be a

silicone fragment with a decoration on its surface of organic material and elements associated with building materials (calcium alumina-silicate). Particle 4 consisted of carbon, oxygen and titanium with aluminium and trace levels of other elements. The high levels of titanium indicate titanium dioxide - a widely used whitener for paints and many man-made objects. The fibrous nature of the particle and presence of titanium would indicate a particle from a white-painted wood source.



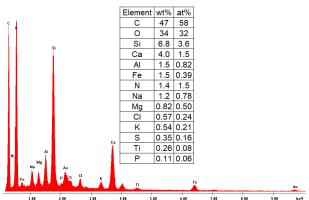


Figure 34. EDX spectrum and composition of particle 5 from sample LC2.

Figure 35 shows a higher magnification BSE image of particle 6 and Figure 36 shows the EDX spectrum and composition of the analysed area. It consisted of carbon and oxygen with calcium, silicon, iron and nitrogen and trace levels of other elements. It was likely to be a decorated wood fibre fragment having many small particulates on its surface. The spectrum showed a majority carbon peak, nitrogen, oxygen, phosphorous and sulphur (from the natural wood) and probably calcium silicate / silica / calcium carbonate particulates decorating the surface.

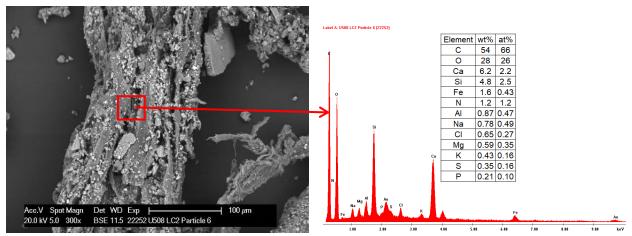


Figure 35. Higher magnification BSE image of particle 6 on sample LC21.

Figure 36. EDX spectrum and composition of particle 6 from sample LC2.

3.3.3 Sample LC3

Figure 37 shows a low magnification BSE image of the particles on sample LC3. Three particles were selected for EDX analysis and these are indicated in the image.

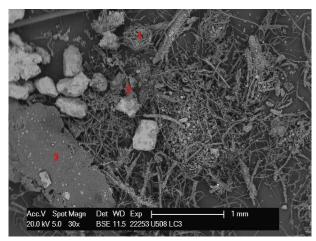


Figure 37. Lower magnification BSE image of some particles on sample LC3.

Figure 38 shows a higher magnification BSE image of particle 1 and Figure 39 shows the EDX spectrum and composition of the analysed area on particle 1. It was an agglomerate of particles within a twisted ball of man-made fibre (uniform and of thin diameter) and it consisted of carbon and oxygen with silicon, calcium, nitrogen and iron and trace levels of other elements. The carbon, nitrogen and oxygen could arise from the fibre and the smaller inorganic particles could be from building materials (calcium silicate/silica/calcium carbonate).

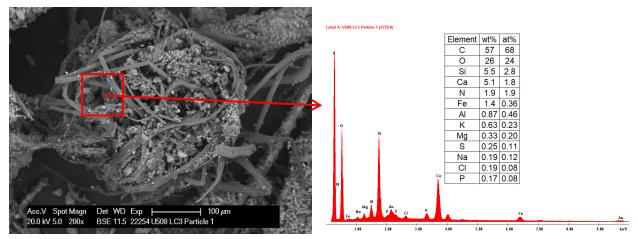


Figure 38. Higher magnification BSE image of particle 1 on sample LC3. Ball of man-made fibres with agglomerated inorganic particles.

Figure 39. EDX spectrum and composition of particle 1 from sample LC3.

Figure 40 shows a higher magnification BSE image of particle 2 and Figure 41 shows the EDX spectrum and composition of the analysed area. It consisted of carbon and oxygen with silicon, calcium, nitrogen and zinc and trace levels of other elements.

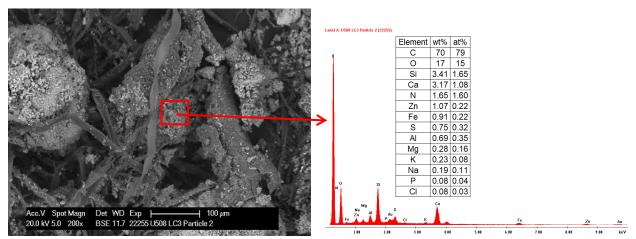


Figure 40. Higher magnification BSE image of particle 2 on sample LC3.

Figure 41. EDX spectrum and composition of particle 2 from sample LC3.

Figure 42 shows a higher magnification BSE image of particle 2 and Figure 43 shows the EDX spectrum and composition of the analysed area. It consisted of carbon, oxygen and nitrogen with calcium and zinc and trace levels of other elements. It was likely to be from a man-made material, as the particle had a relatively flat sheet-like appearance with an irregular (or torn) edge. particle was organic in nature and could have been from a man-made material.

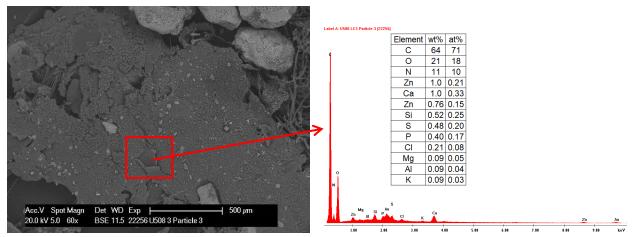


Figure 42. Higher magnification BSE image of particle 3 on sample LC3.

Figure 43. EDX spectrum and composition of particle 3 from sample LC3.

3.3.4 Sample LC4

Figure 44 shows a low magnification BSE image of the particles on the righthand side of the sample LC4 stub (shown optically in Figure 15). Four particles were selected for EDX analysis and these are indicated in the image. inorganic particles had the same appearance and looked the same as the inorganic particles from the residential property samples.

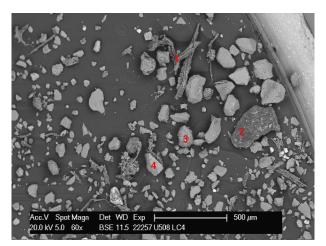


Figure 44. Lower magnification BSE image of some particles on sample LC4. Mainly inorganic mineral particles.

Figure 45 shows a higher magnification BSE image of particle 1 and Figure 46 shows the EDX spectrum and composition of the analysed area. It consisted of carbon and oxygen with silicon, calcium, nitrogen and iron and trace levels of other elements. It was most likely a degraded wood fibre decorated with smaller inorganic particles. The inorganic particles were probably from compounds associated with building materials - i.e. silica, calcium silicate, calcium carbonate.

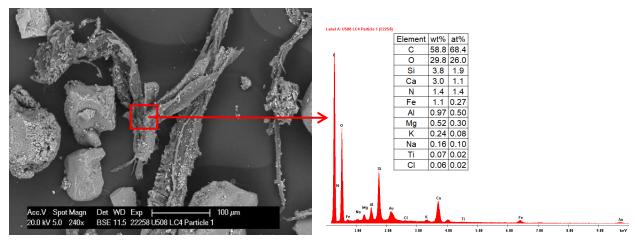


Figure 45. Higher magnification BSE image of particle 1 on sample LC4.

Figure 46. EDX spectrum and composition of particle 1 from sample LC4.

Figure 47 shows a higher magnification BSE image of particle 2 and Figure 48 shows the EDX spectrum and composition of the analysed area. It consisted of carbon and oxygen with nitrogen, silicon, and calcium and trace levels of other It was most likely a man-made organic particle decorated with smaller particles, again likely to be associated with building materials. cannot identify organic particles and other techniques would be required to identify this particle.

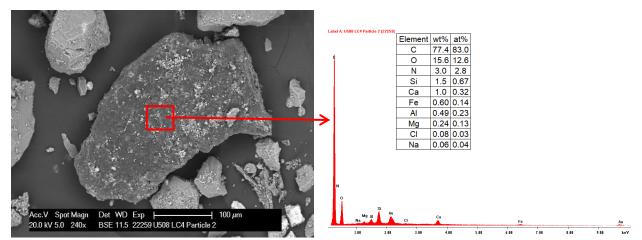


Figure 47. Higher magnification BSE image of particle 2 on sample LC4.

Figure 48. EDX spectrum and composition of particle 2 from sample LC4.

Figure 49 shows a higher magnification BSE image of particle 3 and Figure 50 shows the EDX spectrum and composition of the analysed area. It consisted of oxygen, carbon and silicon with calcium, iron, aluminium and magnesium and trace levels of other elements. It had a signature indicating a sand or silica particle.

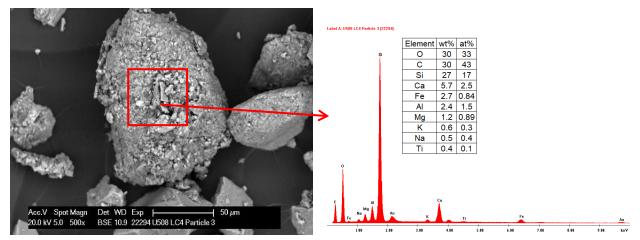


Figure 49. Higher magnification BSE image of particle 3 on sample LC4.

Figure 50. EDX spectrum and composition of particle 3 from sample LC4.

Figure 51 shows a higher magnification BSE image of particle 4 and Figure 52 shows the EDX spectrum and composition of the analysed area. The particle was an angular mineral particle. It consisted of oxygen, carbon and silicon with potassium, aluminium and nitrogen and trace levels of other elements. It was most likely a potassium alumina-silicate material. Typical examples are from feldspar.

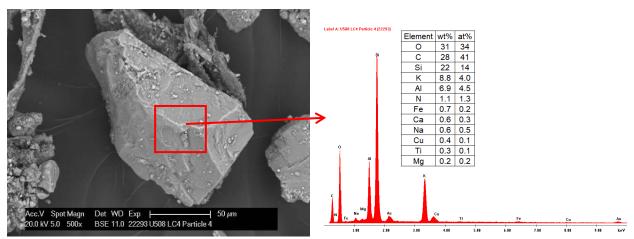


Figure 51. Higher magnification BSE image of particle 4 on sample LC4. Angular mineral particle.

Figure 52. EDX spectrum and composition of particle 4 from sample LC4.

4 Conclusions

The angular mineral particles observed in all of the samples appeared to be from building materials including materials such as calcium alumino-silicates / calcium silicates (cement/concrete), plus calcium carbonate (limestone/concrete), silica (sand) and gypsum (calcium sulphate - plaster). Many of these particles were relatively large (100's of microns in size) and so would not be airborne for long and/or would not be carried for any substantial distance by air currents. The building materials were common to most buildings and rooms and do not have a unique elemental composition (EDX signature). Therefore, the inorganic mineral fraction of the dust could not uniquely identify LC4 as the source of these particles.

Residential Dust Analysis U508 Page 18 of 20

All of the samples (LC1 to LC4) contained degraded wood fibres and also some decorated with smaller inorganic particles. The inorganic particles had similar elemental signatures from building materials. Occasionally they showed titanium dioxide decoration, suggesting materials such as white painted wood or plaster.

The fibres from the internal sills of the residential areas all showed some fibres from clothing such as cotton and man-made fibres. Some of these were coloured as would be expected from fabric material (red/blue/white). The LC4 sample had very few such man-made fibres present.

Black particles present in all samples were not "normal" in terms of historical analysis of residential dusts. These are likely to be indicative of organically-bound agglomerations, but generally heavily decorated – potentially materials like concrete bound in rubber, that can be generated in areas with significant traffic such as lorries and/or fork-lift trucks, driven on open concreted areas.

Although these particles commonly showed organic signatures, sometimes with low levels of sulphur and zinc, it is difficult to unequivocally identify them as from rubbers without additional confirmation from organic analytical methods, such as Fourier Transform InfraRed spectroscopy (FTIR).

The amount of wood and plant-type fibre was also higher than generally seen in residential areas. This would also be consistent with commercial operations involving movement of large numbers of palleted products, as well as wood processing operations. The latter is not known to be present within the commercial units around the area where the samples were collected, but the nearest business, from which the LC4 sample was collected, is "Delivered (UK) Ltd", which are a delivery company specialising in haulage and distribution of parcels and pallets. Another company "BDT Ltd" also operates a presumably similar haulage business on a neighbouring site.

4.1 Overview Summary

As none of the samples had a particle type that was not present in the others, it is difficult to state the likely individual sources of the materials. Gauging differences in the relative amounts of the different material types is also difficult without a more extended study than the small sampling undertaken within the current work. However, some comments regarding the sample set overall may prove useful:

- Overall, the materials present include common building materials, wood, clothing fibre, but an optically-black particle type that is not considered "normal" in residential properties was observed in all samples.
- The amounts of wood also seem quite high (but can be from internal materials, as well as outside)
- The variations between the samples does not show any stand-out materials in any one sample that are not present in the others. While this is unusual, it is probably due to the proximity of the residential properties.

- Although based on a low number of samples, a cursory inspection suggests some variations in relative proportions:
 - LC4 (commercial site entrance) had the common wood fibres and black particles, but significantly less clothingtype fibres. This might be expected – as the residential properties would be expected to show more of these (unless the commercial operations involved clothing manufacture/transport, which they do not).
 - LC1 has significant wood fibre present, plus clothing-type fibres and mineral particles, as well as the black particle type.
 - LC2 has more wood (relatively) than LC1, plus large plant-based fibres and some off-white lumps/flakes probably paint/plaster related. This unit is closer to the LC4 sample location than the other 2 residential units, suggesting the wood may be related to the commercial activity. It remains difficult to be unequivocal in this statement, though, without a larger sampling activity (i.e. extending the brief testing done thus far)
 - LC3 (smaller original sampling of dust than others in foil) had more clothing-type fibres than the other samples and more off-white lump/flake (paint/plaster)
- Although the amount of wood is somewhat higher than you'd expect from inside residential properties, the particle "of-note" was the optically black particle type, that has not been seen historically in residential properties.

The nature of the black particle suggests it may be an agglomeration of mineral type particles, but stuck together in a probable organic matrix, which could include rubber-type materials. This (in the past) has been linked to warehousing activities – rubber from lorry and fork-lift tyres, with minerals from concrete and other road materials and sometimes raised levels of wood if a lot of pallets are being moved around. This seems consistent with the commercial property that LC4 was collected from ("Delivered UK Ltd") – as they are a distribution company handling/transporting a lot of products, however, there are other companies also appearing to operate lorries (so probably also fork-lift activities too) in the vicinity (such as BDT Transport).

5 Further Work

Gaining further insight is not possible from this small sampling and optical/SEM methods alone. To draw more conclusions on the sources of the different materials present (and common to all samples) would require increased sampling, in particular from residential properties further from the LC4 sampling position and the commercial activities in general.

Some work using organic materials analysis techniques, such as Fourier Transform InfraRed spectroscopy (FTIR) might provide some clearer link to the possible culprit materials – such as tyre rubbers – that may contribute to the black particle type, as this is the less-common material found historically within residential properties. However, this would ideally need samples of the culprit materials (e.g. tyre rubber samples from lorry and fork lift tyres) to give a more reliable assignment.

Authors:					
Function:	Senior Metallurgist	Senior Applications Scientist			
Signature:	(hard copy only)	(hard copy only)			
Additional Text and checking: (Technical Director					
End of repo	rt.				