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A Preliminary Investigation into the Characterisation of Sooty Soilings on Historic Textiles

by

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ABSTRACT

The research focuses on the characterisation of airborne, atmospheric pollution-derived black carbon-based sooty soilings on historic textiles. It examines current conservation attitudes to sooty soilings and conservation knowledge about soiling identification. An overview is given of historically important sources of sooty soilings which may have affected textiles exhibited in the domestic interior. Methods with potential to aid the characterisation of sooty soilings were researched and put into use during the assessment of the soiling on two domestic interior historic textiles: an early twentieth-century embroidered banner and a nineteenth-century muslin curtain. The thesis focuses on ascertaining of the presence of sooty soiling on the two artefacts. Investigation methods used include physical examination, historical research, and a set of instrumental analytical and imaging techniques such as stereomicroscopy and polarised light microscopy, scanning electron microscopy – energy dispersive X-ray analysis, ion chromatography, Raman spectroscopy, attenuated total reflection infrared spectroscopy, fluorescence spectroscopy and infrared photography. Applicability of the different techniques for the characterisation of sooty textile soilings is evaluated, and recommendations for the future investigation of this novel field of research are provided.

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LIST OF ABBREVIATIONS

ATR- FTIR	Attenuated Total Reflection - Fourier Transform Infrared spectroscopy
cm	Centimetre
c. m. c.	Critical Micelle Concentration
COSHH	Control of Substances Hazardous to Health
CTCTAH	Centre for Textile Conservation and Technical Art History
EDS / EDX	Energy Dispersive X-ray Spectroscopy
EM-CCD	Electron-Multiplying Charge-Coupled Device
ESEM	Environmental Scanning Electron Microscopy
g	Gramme
GC	Gas Chromatography
HPLC	High-Performance Liquid Chromatography
IC	Ion Chromatography
IDA	Industrial Denatured Alcohol
IR	Infrared
l	Litre
LC	Liquid Chromatography
LED	Light-Emitting Diode
mg	Milligramme
ml	Millilitre
mm	Millimetre
µm	Micrometre/ Micron
µS	Microsiemens
MS	Mass Spectrometry
MSDS	Material Safety Data Sheet
nm	Nanometre
pH	Power of Hydrogen
PAH	Polycyclic Aromatic Hydrocarbon / Polyaromatic Hydrocarbon
PLM	Polarised Light Microscopy
PM	Particulate Matter

SD / σ	Standard Deviation
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
TGA	Thermogravimetric Analysis
UV	Ultraviolet light
Vis	Visible light
Wt%	Weight percentage
$\bar{z}$	Mean atomic number

Conservation associations, conferences, and journal titles

AIC	American Institute for Conservation
BCIN	Bibliographic Database of the Conservation Information Network
CCI	Canadian Conservation Institute
ICCROM	International Centre for the Study of the Preservation and Restoration of Cultural Property
ICOM CC	International Council of Museums Conservation Committee
ICON	Institute of Conservation
IIC	International Institute for Conservation
JAIC	Journal of the American Institute for Conservation
JCIC	Journal of the Canadian Institute for Conservation
NATCC	North American Textile Conservation Conference
UKIC	United Kingdom Institute for Conservation

CHAPTER 1: INTRODUCTION

Soiling on historic textiles is composed of a diverse and complex composite, usually containing airborne soils deposited from the objects' varying environments while in use, display or storage. Black carbon-based particles originating from the burning (combustion) of fuels used for domestic heating and lighting, industrial energy generation and transportation, are susceptible to be attracted to textile fibres, thus causing visible soiling. The term 'sooty soiling' will be used in the text to refer to such deposition.

Increased air pollution in Britain since the seventeenth century and throughout the industrial revolution of the nineteenth and early twentieth centuries meant that the concentration of combustion-related particles and associated gaseous pollutants grew considerably in the environment, exacerbating the soiling of clothes and textiles exhibited in the domestic interior.[1] The research presented in this dissertation thesis focuses on this phenomenon in order to gain a better understanding of a historically important type of textile soiling of relevant significance to conservators.

Interestingly, to the present date soot on historic artefacts has been almost exclusively researched in connection with fire damage to museum collections.[2,3,4,5,6,7,8] Other seemingly distant scientific disciplines have always had a considerable interest in combustion particles related to air pollution: medical and environmental science were the first to be concerned about the structure of soot and its impact upon health and climate; and

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[1] P. Brimblecombe, *The Big Smoke: A History of Air Pollution in London Since Medieval Times* (London: Methuen, 1987), 63-68, 96-101.

[2] J.G. Armstrong et al., "A Furnace Puff-Back: A Unique Problem of Soot on Objects and Costumes," *AIC, Pennsylvania Conference, 1981, Preprints,* 10-19 (Washington: AIC, 1981).

[3] B. Roberts et al., "An Account of the Conservation and Preservation Procedures Following a Fire at the Huntington Library and Art Gallery," *JAIC*, 27 (1988):1-31.

[4] S. Spafford and F. Graham, "Fire Recovery at the Saskatchewan Museum of Natural History: Part 2, Post-Disaster Cleanup and Soot Removal," *ICOM CC, 10th Triennial Meeting, 1993, Preprints,* ed. J. Bridgland, 420-426 (Lawrence: Allen Press, 1993).

[5] C.A. Hill, *A Preliminary Investigation Into Soot On Fire-Damaged Painting*s (Canberra: University of Canberra, 1995).

[6] C. Lachelin, "Preliminary Investigation into the Problems of Removing Soot and Smoke Odour from Historic Textiles: Considerations for Conservation," (Diploma work, Textile Conservation Centre, University of London, 1996).

[7] J. Hackett, "Observations on Soot Removal From Textiles," in *AIC Textiles Specialty Group, 1998*, *Postprints*, 63-68 (Washington: AIC, 1998).

[8] F.G. France et al., "Analysis of Soiling and Trace Contaminants of the Star-Spangled Banner," in *Strengthening the Bond: Science and Textiles, NATCC, 2002*, *Preprints,* ed. V.J. Whelan, 153-167 (Philadelphia: NATCC, 2002).

the fuel engineering science has studied the efficiency of combustion and the formation processes of soot. In textile conservation, sooty soilings originating from atmospheric pollution are generally considered grime, their identity is seldom questioned, and their characterisation is approached from the point of view of fire investigations - if approached at all.[9,10]

Research aims and objectives

The aim of this research is four-fold:

1. To unveil the state of present conservation knowledge and attitudes towards sooty soilings caused by atmospheric pollution;
2. To give an overview of historically important sources of sooty soilings which may have affected textiles in the domestic interior;
3. To provide information on the most important physical and chemical characteristics of soiling particles originating from fuel burning;
4. And to evaluate investigative techniques which could be applied to the characterisation of sooty soilings on historic textiles. Characterisation can in the future lead to a better understanding of the potential harmful effects of sooty soilings besides revealing information about chapters in the object's environmental history. Enhanced knowledge of both aspects could become the basis of more informed treatment decision-making.

   As a preliminary step, two textile artefacts with presumably sooty soiling will be examined and the soiling analysed with the help of investigative techniques identified via literature research. The research objectives of the practical project are:

   a. To try out simple techniques during object examination and documentation, and the use of instrumental analytical techniques for the characterisation of the soiling on two chosen artefacts.
   b. To evaluate the information gained by these techniques and how they aid ascertaining whether the textile soiling originates from combustion processes.

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[9] France et al., 164.

[10] K. Francis, "Tapestry Cleaning: Characterisation and Removal of Soils," in *Strengthening the Bond: Science and Textiles, NATCC, 2002*, *Preprints,* ed. V.J. Whelan, 47-54 (Philadelphia: NATCC, 2002).

CHAPTER 2: SOILINGS ON HISTORIC TEXTILES

2.1. A definition of soiling

The terms soil, soiling and dirt are interchangeably used within the textile conservation literature, and their concept has been defined by several authors. In an early work about the 'Principles of Textile Conservation Science', Rice defined soil and dirt as synonyms, referring to some form of 'matter out of place', to '[…] any deposit or discolouration, irrespective of source, which is objectionable. Furthermore, it may not even be visible as long as it can be detected by some other sense, such as odour or touch.' [11]

The widely acknowledged series of 'Science for Conservators' discusses 'The nature of dirt' in a similar manner, as '[…] material which is in the wrong place, rather as a weed is thought of as a plant growing in the wrong place.' [12] Dirt is divided into two categories; to foreign matter originating from external sources and to products of alteration which form from the material of the artefact. The text emphasises that foreign matter may give rise to a product of alteration if chemical reaction takes place between the dirt and the object.

Harrison further elaborates the forms and sources of the soiling matter, arguing that '[…] soiling can be defined as staining, particulates or ingrained matter which may have occurred during a textile's manufacture, its intended use, or when it was no longer fulfilling its original function. It may also consist of degradation products from the textile itself, related components, or be the results of previous repair or conservation treatment.'[13]

This comprehensive definition will be taken as a basis in the context of the present research, and the word soil and soiling will be used to describe the vast array of foreign matter and products of alteration which may be present on historic textiles. Grime, which is a term often used to describe air pollution-derived soiling on built heritage, also has relevance to airborne textile soilings, but due to its biased connotations as a substance which needs to be urgently removed, the term soil and soiling will be used preferentially.

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[11] J.W. Rice, "Principles of Textile Conservation Science No.5: The Characteristics of Soils and Stains Encountered on Historic Textiles," *Textile Museum Journal*, 2 (1964):8.

[12] Museums and Galleries Commission, *Science for Conservators, Vol. 2 Cleaning* (London: Crafts Council, 1992), 13-22.

[13] A. Harrison, "An Investigation into the Degradative Effects of the Retention of Soiling on Historic and Archaeological Textiles and Strategies for Future Preventive Care, with Reference to a Seventeenth-century Doublet" (Diploma work, Textile Conservation Centre, University of London, 1998), 4.

2.2 Sources of soiling on historic textiles

The sources of soiling on historic textiles are divided into three categories by Tímár-Balázsy and Eastop.[14] These include:

- Soiling originating from functional use or handling - particularly often present on garments, costume accessories, household and ceremonial textiles;
- Airborne soiling deposited from the domestic, urban or museum environment - especially noticeable on tapestries, carpets, upholstery and flags on open display;
- And soiling originating from the burial environment in case of archaeological textiles.

As mentioned by Harrison, the manufacturing process and previous repair or conservation treatments may also be sources of textile soiling.

Soils originating from the manufacturing process, functional use, and burial environment have been widely investigated by textile conservators, perhaps due to their acknowledged potential to reveal substantial information about the history of the artefacts.[15] The possible soiling types include food residues, blood, greases, body fluids, human and animal hair, insect parasites and skin particles, amongst others.

Airborne soilings on the other hand have been less studied in relation to historic textiles.[16] Soiling is caused by particles suspended in the atmosphere, collectively referred to as atmospheric particulate matter (PM).[17] Airborne PM originates from natural and anthropogenic sources, with anthropogenic origin prevailing in the urban environment. The composition of particles filtered from the urban air usually contains salts, grit, oils, waxes, clay, black carbon-based particles formed in combustion,[18] spores, with indoor dust usually containing a high proportion of textile fibres and skin fragments.[19] Most of these particles form by means of fragmentation with the exception of combustion products, which are

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[14] Á. Tímár-Balázsy and D. Eastop, *Chemical Principles of Textile Conservation* (Oxford: Butterworth-Heinemann, 1998), 157-162.

[15] D. Eastop and M. Brooks, "To Clean or Not to Clean: The Value of Soils and Creases," in: *ICOM CC, 11th Triennial Meeting, 1996, Preprints,* ed. J. Bridgland, 687-691 (London: James&James Ltd, 1996).

[16] F.G. Thomsen, "MFAH Texas Flags: 1836-1945. Flags as Fine Art?" in *Tales in the Textile: The Conservation of Flags and Other Symbolic Textiles, NATCC, 2003, Preprints,* ed. J. Vuori, 93-98 (New York: NATCC, 2003), 94-95.

[17] J. Grau-Bové and M. Strlič, "Fine Particulate Matter in Indoor Cultural Heritage: A Literature Review," *Heritage Science*, 1 (2013):2-4.

[18] Tímár-Balázsy and Eastop, 157.

[19] The National Trust, *The National Trust Manual of Housekeeping: The Care of Collections in Historic Houses Open to the Public* (Oxford: Butterworth-Heinemann, 2006), 62.

ubiquitous in the air and are considered to be the most important form of anthropogenic atmospheric pollution.

2.3 Preservation of textile - and soiling

2.3.1 Values and risks of textile soiling

Unravelling the sources of soiling on historic artefacts may provide valuable information to historians, art historians and ethnographers.[20] Soiling can contain evidence about the textile's previous use and wear, and its investigation may help dating and attribution problems.[21] For textiles in archaeological, ethnographic and social history collections, soiling can be the main source of contextual information.

In spite of its evidential value, soiling may be regarded objectionable if it impedes the appreciation and interpretation of the artefact by obscuring structural details of the yarns and fabrics, by hiding colours and patterns.[22] Such soiling might prevent the textile to fulfil its role in collections focusing on the history of design and textile technology.

Besides, several types of soiling have the potential to accelerate fibre and dye degradation processes, and the deterioration of non-textile elements of the historic artefact.[23] Even if changes in the material are not perceivable, soils might act as physical and chemical agents of deterioration in the long term.

2.3.2 Preserving soiling and information

In some cases the soiling is considered to have historical value, but also to be a cause of accelerated degradation processes. Furthermore, even if its value is not recognised at the time of the treatment, 'matter out of place' may become 'meaningful matter' in another time, space and institutional context,[24] which underlines the necessity of its preservation in some form. Today, written and photographic documentation ensures that the condition of

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[20] Tímár-Balázsy and Eastop, 157.
[21] Thomsen, 94.
[22] Rice, 8.
[23] Tímár-Balázsy and Eastop, 157.
[24] M. Brooks and D. Eastop, "Matter out of Place: Paradigms for Analysing Textile Cleaning," *JAIC,* 45 (2006):171-181.

the artefact is faithfully recorded before conservation proceeds. Even if a cleaning treatment takes place, the documents are available for future consultation.

Another possibility to reconcile the tension between retaining evidence and removing soils is to undertake routine particle sampling.[25] This has time- as well as storage requirements: while sampling may add to the length of the treatment, sufficient space and administration is required for sample storage. Storing of liquid cleaning samples might be complicated if they require refrigeration.

In many other cases where the evidential value of the soiling is considered primary, soiling may be preserved *in situ*. This is an especially frequent procedure for archaeological textiles.[26]

2.4 Current basis for decisions about conservation cleaning treatments

Cleaning is an irreversible conservation process therefore it is of major importance to justify the need for it and the method of cleaning. Literature research pointed out that there are only a few publications discussing scientific analysis of soils in order to enhance treatment decision-making, and these investigate mainly archaeological textiles.

The scarcity of information is intriguing considering the potential of scientific investigation to aid treatment decision-making in three ways. Firstly, characterizing the source of the soiling can help to establish whether it has historical significance. [27] Secondly, by elucidating its composition it may be determined whether it is a potential source of degradation.[28] Thirdly, by understanding its physical and chemical nature, conservation cleaning treatments can be optimised.[29]

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[25] Thomsen, 94.

[26] M. Brooks, et al., "Artifact or Information? Articulating the Conflicts in Conserving Archaeological Textiles," in *Archaeological Conservation and its Consequences: Contributions to the IIC Copenhagen Congress, 1996, Preprints,* ed. A. Roy and P. Smith, 16-21 (London: IIC, 1996), 16.

[27] F.G. France, "Scientific Analysis in the Identification of Textile Materials," in *Scientific Analysis of Ancient and Historic Textiles: Informing Preservation, Display and Interpretation,* ed. P. Wyeth and R. Janaway, 3-11 (London: Archetype Publications, 2005), 3.

[28] France, 3.

[29] Tímár-Balázsy and Eastop, 157-162.

CHAPTER 3: SOOTY SOILINGS: CONSERVATION ATTITUDES AND APPROACHES

3.1 Conservation attitudes to sooty soilings

3.1.1 The main sources of sooty soilings

Airborne particles produced by combustion are generally referred to as 'smoke' and 'soot' in the conservation literature. Textiles may become covered by sooty soilings due to long term exposure to open fires; vehicle exhaust fumes; industrial pollution from energy generation; cigarette smoke; or rapid pollution created by furnace puff-backs; and accidental or malicious fire events. Soot has been characterised in the conservation literature primarily in the context of rapid soot deposition,[30] while less information is available about textiles soiled by long term exposure to atmospheric pollution.[31] Both scenarios will be examined in order to gain a better understanding of similarities in conservation attitudes.

3.1.2 Attitudes to rapid soot deposition

An accidental fire event or furnace puff-back may have devastating effects on collections, as large amounts of dark soot residue may deposit on the artefacts on display and in storage. [32] Naturally, the historical value of newly deposited sooty soilings is considered low and conservation accounts focus on post-disaster cleanup strategies,[33,34] some including the scientific investigation of the deposit in order to assess its harmful effects.[35,36] Aged sooty deposits have also been analysed on fire damaged paintings in order to better understand the interaction of soot with painted surfaces,[37] and the investigation of a textile with aged sooty soiling originating from a building fire mixed with soiling caused by

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[30] L. Owen, "Fire and Paper: An Examination of the Materials and Techniques of Lee Bontecou's Soot Drawings," *AIC, The Book and Paper Group Annual,* 27 (2008):48.

[31] Literature consulted through: BCIN and ICCROM library catalogue; Reviews in Conservation (IIC), Studies in Conservation (IIC), News in Conservation (IIC), The Conservator (ICON), Conservation News (ICON), JAIC, JCIC, NATCC proceedings, ICOM CC postprints, IIC preprints.

[32] Lachelin, 16.

[33] Spafford and Graham.

[34] Hackett.

[35] Roberts et al.

[36] Armstrong et al.

[37] Hill.

modern urban air pollution was described in order to aid the planning of a cleaning treatment.[38]

The physical and chemical characteristics of sooty depositions have been better understood and cleaning and preventive conservation methods developed due these accounts. It has been noticed that the soiling composition and attributes are largely dependent on the materials burnt and the burning conditions.[39] Common points in all publications are concerns about the disfiguring effect of soot; the possibility of sooty deposits causing chemical damage because of their potentially acidic pH; followed by the concern that an oily, tarry component typically present may embed additional harmful airborne particles. Sooty soilings are generally considered objectionable and are removed by cleaning treatments as soon after the deposition as possible.[40]

3.1.3 Attitudes to sooty soilings from atmospheric pollution

Concerns raised by rapid soot deposition are not new: the same issues surrounded the topic of sooty air pollution since medieval times. Increased attention to the damaging effects of smoke could be attributed to the transition to smoky coal as an industrial, and later also as a domestic fuel. Historic documents describing the damage caused to buildings, clothing and furnishings in England between the thirteenth and twentieth centuries were collected by Brimblecombe. The problem of keeping clothes clean was reported around 1650, and by the eighteenth century thriving businesses were running to refurbish badly 'smoked' items.[41] Damage to interiors including tapestries, paintings, leather chairs and books was also noted, and hangings were reported to be rarely found in city dwellings as they would have been rapidly ruined by smoke.[42] Relation between textile discolouration and the acidity of sooty pollution was observed by the scientist Robert Boyle who advised measuring the acidity of the atmosphere by hanging up dyed cloths and examining their fading rates.[43] In the mid-twentieth-century, soiling caused by smoke was still considered

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[38] France et al.
[39] Hackett, 63.
[40] Hackett, 68.
[41] Brimblecombe, The Big Smoke, 63.
[42] Brimblecombe, The Big Smoke, 64.
[43] Brimblecombe, The Big Smoke, 76.

the major cause of the visible pollution-, and acidic substances in it the cause of tendering of household textiles.[44]

Recent conservation accounts focus on the disfiguring and harmful effects of accumulated soot and on cleaning practices. Case studies discuss the treatment of objects affected by long term exposure to burning candles and oils in places of worship,[45,46] and to coal and wood fires in historic houses. The latter include an account of the cleaning of carpets exposed to coal fires contributing to their particular smell and low pH,[47] and the removal of dark and greasy soiling from tapestries at Doddington Hall.[48] An account of the cleaning of a heavily soiled Flemish tapestry which had been exposed to open fires in the Isabella Stewart Gardner Museum includes the characterisation of soils and is thus a useful reference point for this research. [49]

Preventive conservation literature emphasises the importance of the protection of artefacts from soiling originating from cigarette smoke, working fireplaces, industrial energy generation and diesel exhaust.[50,51,52,53] A useful account describes the characterisation of sooty soiling originating from road traffic on paintings, in order to aid preventive conservation decision-making.[54]

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[44] "Atmospheric Pollution and the Soiling of Textiles," *Journal of the Textile Institute*, 48 (1957):725-726.

[45] J.B.N. Loh, "Decision from Indecision: Conservation of Thangka Significance, Perspectives and Approaches," *Journal of Conservation and Museum Studies*, 8 (2002):6.

[46] K. Seymour and S. Postma, "A Case Study in Laser Cleaning: Soot Removal," in *ICOM CC 14th Triennial Meeting, 2005, Preprints*, ed. I. Verger, 836 (London: James & James, 2005).

[47] J. Tetley, "Observations on Current Usage of Conductivity in Cleaning Carpets from Historic Houses," *News in Conservation*, February 2013, 9-12. http://www.iiconservation.org/node/3505 (accessed May 18, 2013).

[48] "The Holly Room Tapestry Conservation Project," Doddington Hall and Gardens, http://www.doddingtonhall.com/downloads/Flipbook.pdf (accessed July 02, 2013).

[49] Francis.

[50] G. Thomson, *The Museum Environment,* Second Edition (London: Butterworth-Heinemann, 1986), 131-132.

[51] B. Appelbaum, *Guide to the Environmental Protection of Collections* (Connecticut: Sound View Press, 1991), 97-113.

[52] J. Tétreault, *Airborne Pollutants in Museums, Galleries and Archives: Risk Assessment, Control Strategies, and Preservation Management* (Ottawa: CCI, 2003), 7-19.

[53] Grau-Bové and Strlič, 2013.

[54] A. Alebic-Juretic and D. Sekulic-Cikovic, "The Impact of Air Pollution on the Paintings in Storage at the Museum of Modern and Contemporary Art, Rijeka, Croatia," *Studies in Conservation*, 54 (2009):49-57.

3.2 Current conservation approaches to sooty soilings from atmospheric pollution

Unlike in the case of accidental fire events, the exact source of suspected sooty deposition on historic artefacts is unknown. Three publications were found with detailed characterisation of presumably atmospheric pollution-derived soils in order to aid cleaning[55] or preventive conservation decision-making.[56] Current state of knowledge was found to be most revealing in a report by Francis discussing 'soil removal problems and the treatment of an extremely dirty sixteenth-century tapestry'.[57]

The soiling was described as 'Heavy dark oily type soil overall (soot) […]', and as 'black haze' sitting on the uppermost surface of the tapestry. It was observed to '[…] be primarily particulate matter, which could not be lessened by vacuum cleaning but which responded well to crocking type of removal […]', just like a '[…] sooty soil deposited onto textiles by a fire or furnace back up'. Even though coal and wood furnaces and fireplaces in the museum and nearby traffic were the suspected causes of the soiling, to better understand its composition, conservation scientists in the Canadian Conservation Institute were entrusted to perform X-ray microanalysis, X-ray diffraction, and polarised light microscopy of soil samples from several tapestries housed in the museum, which confirmed the presence of combustion particles on the textiles.

This approach shows that even if the likely origin of the soils is known, the current state of understanding of the composition, behaviour and identification of soils originating from combustion processes is far from complete, and soiling which has similar attributes is compared to deposits originating from fire events. The article briefly discusses the results of the scientific analysis, but for detailed information the unpublished analytical report had to be requested from the CCI. [58] The publication may be regarded a discussion-initiator which clarifies uncertainties accounted in this field.

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[55] France et al.

[56] Alebic-Juretic and Sekulic-Cikovic.

[57] Francis, 48.

[58] E. Moffatt, I. Wainwright and M.-C. Corbeil, "Analysis of Soil Samples from Several Tapestries for the Isabella Stewart Gardner Museum," (Unpublished Report, CCI, Report No. ARL 3906, 2000).

3.3 Summary

Sooty soilings originating from atmospheric pollution and depositions caused by fire events have raised similar concerns regarding their disfiguring and damaging attributes. They are generally regarded objectionable and are removed by cleaning treatments.

Soilings caused by accidental fires have been characterised in several cases in order to aid treatment decision-making, whereas sooty soilings from atmospheric pollution have rarely been the subject of analytical investigations. The possible sources, attributes, and characterisation methods of carbonaceous soiling deposited on textiles in the domestic interior will be focused on in the next chapters.

CHAPTER 4: SOOTY SOILINGS ON DOMESTIC INTERIOR HISTORIC TEXTILES

4.1 Sources of sooty soilings in the domestic interior

4.1.1 Interior sources

Burning of fuels for heating, lighting and cooking always played the key role in the generation of sooty pollution indoors.[59]

Initially the main types of fuel used for heating in England were wood and charcoal.[60] With the population growth came frequent wood shortages, and poorer households had to change over to a much smokier fuel: the beginning of the seventeenth century saw the uprise of chimneys and coal, with all its implications. Scotland had already adapted to the new fuel around the fourteenth century due to better quality smokeless coal available in the region.

The chief sources of heating were open fires until the nineteenth century, when enclosed stoves became popular.[61] Open fires emitted considerably more smoke than stoves, however, having an 'open, pokeable, companionable fire' was considered most important.[62] New heating technologies using *coal-gas* and oil were said to have had a sooty flame as early devices did not burn the fuels efficiently.[63] 'Clean' electricity did not compete with other fuels until the 1930s.

Candles made of smoky tallow or cleaner beeswax were the principal sources of artificial lighting until mid-nineteenth-century, when *spermaceti* and *paraffin* candles came into use.[64] Candles were substituted by oil lamps burning animal and vegetable oils until the introduction of *paraffin* lamps in the 1860s.[65] These had a strong smell and a tendency to smoke. Gas lights using *coal-gas* were introduced in the first half of the nineteenth century and 'clean' electric lighting in the second half of the nineteenth century.

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[59] Brimblecombe, The Big Smoke, 3.
[60] Brimblecombe, The Big Smoke, 26-29.
[61] The National Trust, 27-28.
[62] Brimblecombe, The Big Smoke, 116.
[63] L. Wright, *Home Fires Burning: The history of Domestic Heating and Cooking* (London: Routledge & Kegan Paul Ltd, 1964), 149-168.
[64] B. Bowers, *Lengthening the Day: A History of Lighting Technology* (Oxford: Oxford University Press, 1998), 9-26.
[65] Bowers, 27-56.

4.1.2 Exterior sources

Air in the domestic interior was largely influenced by pollution from the surrounding environment. Increase in outdoor air pollution begun in the thirteenth century when coal started to be used for lime burning and smelting,[66] and continued into the seventeenth century with the wide-spread use of coal for domestic heating. The pollution was augmented by the invention of the steam engine during the industrial revolution.[67] Fogs mixed with smoke (today called 'smog') became regular, with an absolute peak in 1890. In cities and the countryside, schemes were devised to filter the air entering buildings: felt seals, cotton wool, jute and hessian filters were fitted to window frames in the hope of blocking out smoke and harmful vapours - with little success.[68] Due to the introduction of smokeless fuels and restricting smoke legislations, the main risk today to artefacts housed in the urban environment is from diesel engine smoke.[69]

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[66] Brimblecombe, The Big Smoke, 11.

[67] Brimblecombe, The Big Smoke, 96-101.

[68] The National Trust, 27-28.

[69] "Atmospheric Pollution and the Soiling of Textiles," *Journal of the Textile Institute*, 48 (1957):725-726.

4.2 Physical and chemical characteristics of sooty soilings

4.2.1 Products of incomplete burning

Burning or combustion is the self-catalysed chemical reaction of a fuel and an oxidiser which produces heat, generally but not necessarily light, and a variety of gases, vapours, particulates, and solid residues.[70] Emissions always depend on the combustion conditions and the chemical composition of the fuel.[71] Although complete combustion of carbon, hydrogen and oxygen in organic materials would lead to the production of carbon dioxide and water, under natural conditions burning tends to be incomplete.[72] Due to insufficiently low temperature and air supply, *reduced carbon species* are produced including gases (carbon monoxide, methane, volatile organic compounds) and carbonaceous particles. As fuels may contain sulphur, nitrogen and metallic elements, these might be incorporated into the combustion products.

'Smoke' and 'soot' are the terms used in the conservation literature to describe visible emissions originating from combustion, without agreement on concise definitions. Environmental science defines 'smoke' as carbonaceous particles smaller than 10µm in diameter, including 'soot' particles generally smaller than 1µm.[73] To provide a comprehensive review for conservation purposes, Winter's categorisation for carbon-based materials will be taken as a basis. [74]

Winter divided combustion particles into three categories: to 'flame carbons or soots' produced in the gas phase and having highly characteristic branched morphology; and carbonised debris including 'cokes' which form from a liquid or plastic precursors and 'chars' which form from solid precursors and often retain the original shape of the fuel. The production of incombustible mineral residues or 'ashes' and incompletely carbonised organic 'tarry material' accompany the burning process, and may be associated with soot.

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[70] B.W. Kuvshinoff (ed.), *Fire Sciences Dictionary* (New York: John Wiley & Sons, 1977), 97.

[71] B.R.T. Simoneit, "Biomass Burning: A Review of Organic Tracers for Smoke from Incomplete Combustion," *Applied Geochemistry*, 17 (2002):131.

[72] H. Cachier, "Carbonaceous Combustion Aerosols," in *Atmospheric Particles, Vol.5*., ed. R.M. Harrison and R.E. van Grieken, 296-348 (Chichester: John Wiley and Sons, 1998).

[73] B.J Alloway and D.C. Ayres, *Chemical Principles of Environmental Pollution* (Glasgow: Blackie Academic & Professional, 1993), 196.

[74] J. Winter, "The Characterisation of Pigments Based on Carbon," *Studies in Conservation*, 28 (1983):49-66.

4.2.2 Soot formation and structure

Soot is formed when partially degraded fuel molecules *polymerise* to produce carbon *nuclei*, which accumulate further material by adsorption.[75] The particles consist of graphite-like sheets composed principally of carbon, oxygen and hydrogen,[76] potentially containing nitrogen and sulphur.[77] The sheets produce spherical forms and the spheres *coagulate* to form branched structures (Figure 1).

Primary soot particles are 0.1-0.2µm in diameter, and aggregates can grow up to a few microns.[78] Sub-micron particles fall into the fine fraction of atmospheric PM that can stay suspended in the air for days or a month.[79] They have a distinctive black colour, and they effectively absorb and scatter light.[80] The appearance and tenacity of carbonaceous soiling may be attributed to the dark colour of the particles and to the fact that the deposits are within the size of the roughness fissures of natural fibres. [81] Soot particles are positively charged whereas textile fibres are negatively charged,[82] which shows the potential for *electrostatic* attraction.

Soot can possess significant internal surface areas reaching tens to hundreds square meters per one gram of soot.[83] Adsorption onto the soot surface is governed by *Van der Waals forces*. Inorganic matter potentially present in soots includes oxides, salts, elemental metals, and adsorbed liquids and gases such as water, sulphuric and hydrochloric acid and nitrogen oxides (Table 1).[84] Organic matter on soot surfaces may include fragments of char and coke, and tarry material which is also called the ‘soluble organic fraction’. This is generally comprised of *aliphatic hydrocarbons* including *carboxylic acids and cycloalkanes*; *phenols*; *polycyclic aromatic hydrocarbons (PAHs)* and their derivatives; and *heterocyclic*

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[75] N.P Levitt et al., “Heterogeneous Chemistry of Organic Acids on Soot Surfaces,” *Journal of Physical Chemistry*, 111 (2007):4804.

[76] Hill, 7.

[77] Winter, 54.

[78] Hill, 8.

[79] Tétreault, 14.

[80] Alloway and Ayres, 196.

[81] Rice, 12-13.

[82] Hill, 9.

[83] V.I. Berezkin et al., “A Comparative Study of the Sorption Capacity of Activated Charcoal, Soot, and Fullerenes for Organochlorine Compounds,” *Technical Physics Letters,* 28 (2002):887.

[84] A.I.Medalia, D. Rivin and D.R. Sanders, “A Comparison of Carbon Black With Soot,” *The Science of the Total Environment,* 31 (1983):3-4.

compounds. The composition of the organic fraction is highly characteristic to the fuel type and the burning conditions.[85]

Figure 1 Soot formation process [86]

4.2.3 The potential harmfulness of soot

Although the black carbon structure of soot is chemically inert, adsorbed materials may be damaging to historic textiles. Fossil fuels containing sulphur and chlorine are potential sources of sulphuric and hydrochloric acids, and high temperature burning

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[85] Simoneit.

[86] J.S. Lighty, J.M. Veranth and A.F. Sarofim, “Combustion Aerosols: Factors Governing Their Size and Composition and Implications To Human Health,” *Journal of the Air and Waste Management Association*, 50 (2000):1570.

processes create nitrogen oxides which can form nitric acid on contact with water,[87] coming from the moisture content of the air or wetting - wet cleaning of the textile. Strong acids promote hydrolysis and oxidation-reduction reactions in fibres, and may cause colour change or bleeding of dyes.[88] Weaker, organic acidic compounds such as *carboxylic acids* and *phenols* may be present in the soluble organic fraction, besides hydrolysable and oxidisable organic components which form acids with ageing.[89]

Common salts associated with soots are ammonium sulphate and ammonium nitrate which form from the reaction of ammonia with sulphur dioxide or nitrogen oxides, all produced during burning. [90] The sharp edges of crystallised salts can cut fibres; in addition they may attract atmospheric moisture and promote the activation of acids.[91] Metallic elements in soot may act as catalysts in fibre degradation processes.

Although information about the potential harmful effects of non-acidic organic compounds has not been found, the organic fraction may indirectly contribute to deterioration processes not only by adhering soot to surfaces by molecular secondary bonding, but also by promoting damaging particulates to adhere to the surface of the textile.[92]

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[87] P. Brimblecombe, *Air Composition and Chemistry* (Cambridge: Cambridge University Press, 1986), 95-98.
[88] Tímár-Balázsy and Eastop, 158.
[89] Roberts, et al., 7.
[90] A. Worobiec et al., “Characterisation of Individual Atmospheric Particles within the Royal Museum of the Wawel Castle in Cracow, Poland,” *e-preservation science,* 3 (2006):63-68.
[91] Tímár-Balázsy and Eastop, 158.
[92] France et al., 159.

Table 1 The composition of smoke from open fires and candle burning [93]

	Bituminous coal	Wood	Beeswax candle
Gases	Reduced carbon gases Sulphur dioxide, Hydrogen chloride, Nitrogen oxides Ammonia	Reduced carbon gases Nitrogen oxides, Sulphur dioxide, Ammonia	Reduced carbon gases Nitrogen oxides
Black carbon	Present	Present	Present
Metallic/ other trace elements	Iron, Sulphur, Chlorine, Arsenic, Lead, Thallium, Antimony, Mercury	Potassium, Calcium, Sulphur, Chlorine, Silicon, Phosphorous, Zinc, Rubidium, Iron	No information found.
Soluble organic fraction	PAHs, alkylated/ oxidised PAHs, n-alkanes, n-alkenes, n-alkan-2-ones, n-alkanals, n-alkanoic acids, n-alkenoic acids, n-alkanols, n-alkylbenzenes, n-alkylnitriles, phenolics, hopanoid-, steroid- and terpenoid *biomarkers*	PAHs, alkylated/ oxidised PAHs, n-alkanes, n-alkenes, n-alkanoic acids and n-alkanols, levoglucosan, guaiacol and syringol, steroid and terpenoid *biomarkers*	PAHs, alkylated/ oxidised PAHs, n-alkanes, n-alkenes, n-alkanoic acids, n-alkenoic acids, n-alkanals, cyclohexylalkanes, wax esters

4.2.4 Interior versus exterior burning sources

The composition of soots largely depends on the burning temperature: the lower the temperature the less complete the burning; the higher the tar and lower the ash content of the soot. Domestic burning produces more tarry material than industrial processes,[94] which might increase the tackiness of particles affecting the soiling of domestic interiors. Moreover, the amount of tars, ashes, and larger char and coke fragments which tend to

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[93] Brimblecombe, Air Composition and Chemistry, 89-101.
J.A. Rau, "Composition and Size Distribution of Residential Wood Smoke Particles," *Aerosol Science and Technology*, 10 (1989):181-192.
P.M. Fine, G.R. Cass and B.R.T. Simoneit, "Characterisation of Fine Particle Emissions from Burning Church Candles," *Environmental Science and Technology*, 33 (1999):2352-2362.
D. R. Oros, & B.R.T. Simoneit, "Identification and Emission Rates of Molecular Tracers in Coal Smoke Particulate Matter," *Fuel*, 79 (2000):515-536.
B.R.T. Simoneit, "Biomass Burning: A Review of Organic Tracers for Smoke from Incomplete Combustion," *Applied Geochemistry*, 17 (2002):129-162.
T.G. Spiro and W.M. Stigliani, *Chemistry of the Environment, Second Edition* (New Jersey: Prentice-Hall Inc, 2003), 216-227.

[94] J.B. Cohen and A.G. Ruston, *Smoke: A Study of Town Air* (London: Edward Arnold, 1912), 4-6.

deposit near the combustion source due to gravity settling, may be increased in the proximity of flames (Figure 2).[95] Artefacts positioned near the fireplace may be exposed to this debris in addition to the fine particles of soot.

Fragments of unburned solid fuel may also be present in the air. Dust generated by coal is said to have had an accumulative and adverse effect on home furnishings.[96] Soot brought back to the interior by chimney draughts and ash distributed in the air by stirring up of the fire, were everyday hazards.[97]

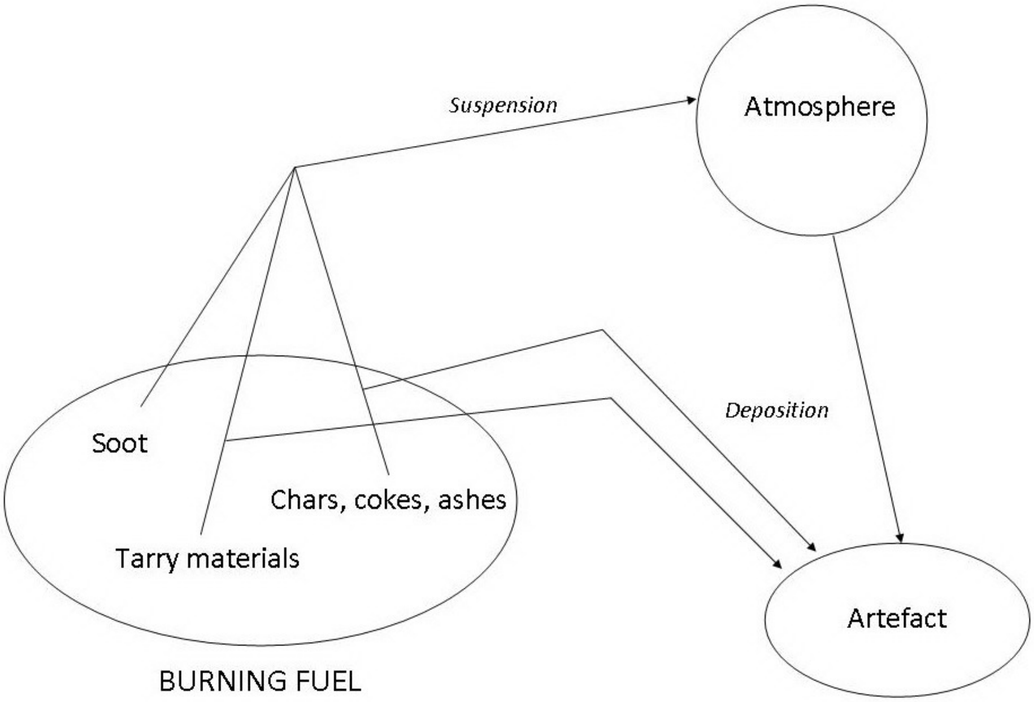

Figure 2 Products of combustion: suspension and deposition [98]

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[95] Winter, 55-56.

[96] J. Miller, *Period Fireplaces: A Practical Guide to Period-Style Decorating* (London: Reed International Books Ltd, 1995), 9-10.

[97] The National Trust, 27-28.

[98] Based on Cohen and Ruston; Winter.

4.3 Summary

Historically important soiling sources and the characteristics of combustion particles and the effect of fuels and burning conditions on the composition of sooty soilings were investigated. Findings show the importance of compositional investigations due to the overt potential of soot to cause damage to textiles. As the composition of sooty soilings can be highly typical of the burning source, the future possibility of exact source appointment by analytical methods in order to reveal relevant historical information is intriguing.

For the present research, morphological and compositional attributes of combustion-related soiling were used to help the selection of investigative techniques which may be applied to ascertain the presence of sooty soilings on historic textiles.

CHAPTER 5: ASSESSMENT METHODS FOR SOOTY SOILINGS ON HISTORIC TEXTILES

5.1 Characterisation of sooty soilings: a preliminary research

5.1.1 Literature research methodology

The aim of this section is to introduce a variety of assessment methods – simple and instrumental – with potential to be used for the characterisation of sooty soilings on historic textiles. Conservation and conservation science literature was consulted in order to clarify recent advances and the state of science and practice in this specific field. A limited number of case studies discussing the examination and analysis of soot were found within different conservation disciplines, primarily paintings, textile and preventive conservation. The topic is just becoming increasingly popular amongst museum professionals but environmental- forensic- and fuel engineering sciences have long been pioneering in the scientific analysis of combustion products, so the literature from these disciplines has also been included where information about specific techniques in conservation literature is scarce. This was done with the aim to provide a better overview of possibilities for future analytical applications for conservation.

5.1.2 A methodology for the characterisation of textile soilings

A scheme for the full characterisation of an object devised for conservators starting a new conservation treatment is included in the work of Appelbaum (Table 2).[99] By taking textile soiling as the only feature to be explored, the characterisation grid can be taken as a basis for the determination of tools necessary for its full characterisation.

For the characterisation of textile soilings, object specific information about the material aspects of the textile gained via physical examination, analysis, imaging and testing are of primary importance (1). If the history of the textile can be reconstructed with the help of institutional records and custodian interviews this may help narrow down the range of references for interpretation of the characterisation results (2). If such information is not

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[99] B. Appelbaum, *Conservation Treatment Methodology* (Oxford: Butterworth-Heinemann, 2007), 10-21.

available, general information collected about the use and display methods for the type of textile and about the level of environmental pollution throughout the textile's lifetime (both having an immediate effect on soiling deposition) may help the interpretation of results gained from physical examination, imaging, testing and analysis (4). Research into the deterioration of materials present aids the understanding of inter-relations between the artefact and the soiling (3).

On the basis of this methodology, a grouping of characterisation methods and terminology is proposed *in section 5.2* (Figure 3).

Table 2 Information to be included in a full characterisation of an object [100]

	Material aspects	**Non-material aspects**
Object specific information	*Information*: Observed phenomena and their interpretation, materials identification, determination of structure. *Source*: Object. *Strategy*: Physical examination, analysis, imaging, testing. 1	*Information*: History of the object, current values, projected future. *Source*: Custodian, others. *Strategy*: Interview, consulting institutional records. 2
Non-object specific information	*Information*: Methods of manufacture, material properties, deterioration studies. *Source*: History of technology, materials science, conservator's knowledge of similar objects. *Strategy*: Consult conservation literature. 3	*Information*: Information about related objects, art history, general cultural information. *Source*: Allied professions, conservator's prior knowledge. *Strategy*: Review literature, consult allied professionals. 4

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[100] Appelbaum, Conservation Treatment Methodology, 10.

5.2 From examination towards scientific investigation

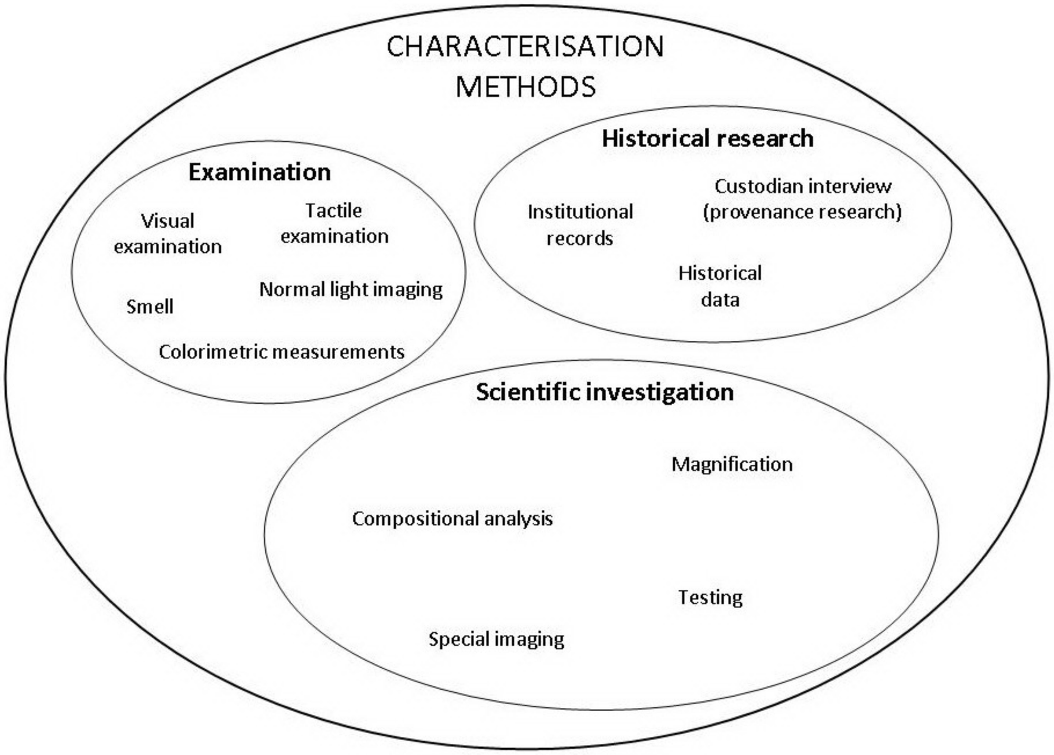

Figure 3 Proposed terminology for the characterisation of textile soilings

5.2.1 Examination

The basis of all conservation examinations is the initial physical examination of the object, when the first general observations about the soiling are made. With the help of visual and tactile examination the colour, touch and smell of the soiling can be assessed. Conservators working with textiles affected by sooty deposition from fire damage, open fires and furnaces and urban pollution described the soilings with the following terms: the soiling is of dark grey to black colour due to its black carbon content;[101,102,103] sometimes accompanied by a yellow-brown discolouration;[104,105,106] greasy touch;[107,108,109] and differing smell due to tarry components potentially present in the soiling.[110,111,112]

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[101] France et al.,159.
[102] Francis, 48.
[103] Tímár-Balázsy and Eastop, 206.
[104] Spafford and Graham, 420-421.

The soiling pattern can also provide information about the direction of the deposition and the mean of delivery. Airborne soiling collects on surfaces most exposed to it during use or storage, whereas contact soiling becomes deposited where the textile is most often touched or exposed to localised liquid or solid soiling agents. As sooty soilings originate from airborne carbonaceous particles, clues in soiling pattern underpinning airborne origin might be of help. Such clues were identified by Francis, 'We assumed that the particulates were very small since the soils were carried up to the top of the tapestry […].'[113]

5.2.2 Historical research

Naturally, one of the most reliable clues in the identification of sooty soilings is knowledge about the textiles' past environments. This is most apparent in case of artefacts affected by accidental fires and furnace puff-backs. Institutional records and original heating devices preserved in museums and historic houses may provide valuable information.[114,115] However, as France put it '[…] Since this [the history of the textile in terms of its lifetime usage, display and storage environments] is often not documented, a variety of analytical techniques are essential to provide the missing information.'[116]

5.2.3 Testing

Testing of pH, conductivity, solubility and ease of mechanical removal are discussed separately as they only provide a general assessment – of acidity/alkalinity, ion content, polarity and strength of adherence – of the soiling. Conservation literature suggests that no generalisations should be made about the acidity of sooty soilings as pH may range from

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[105] Hackett, 63.
[106] Armstrong et al., 11.
[107] Armstrong et al., 11.
[108] Roberts et al., 2.
[109] Francis, 48.
[110] Hackett, 63.
[111] Lachelin.
[112] Tetley, 10.
[113] Francis, 49.
[114] Francis, 49.
[115] The National Trust, 661-667.
[116] France, 3.

neutral to highly acidic.[117] Conductivity might increase in the presence of salts deposited with combustion products, but salts may originate from other airborne sources. To date no reference has been found linking increased conductivity with the presence of sooty soilings. Solubility tests may lead to a general categorisation of the soiling as polar or non-polar, or as soiling which needs to be emulsified. [118] However, as useful as it is in the planning of a cleaning treatment, solubility tells little about the source and exact composition of the soiling. Although the ease of crocking type of removal has been mentioned by conservators,[119] it is not yet an established fact that the ease of removal can be attributed to the presence of combustion-related soiling.

5.2.4 Invasive and non-invasive scientific investigation techniques

Scientific techniques used for the investigation of works of art are generally divided into two categories: non-invasive and invasive.[120] Non-invasive techniques are used directly on artefacts without removing or altering the material of the object, whereas invasive techniques require sampling. If the sample is destroyed during the analytical process, the method is called destructive. Non-destructive methods which leave the sample intact can maximise the information gained from the sample by enabling its reuse for subsequent analyses.

As conservation aims at preserving as much of the original material as possible, the possibility to use non-invasive techniques is generally explored before invasive ones. A categorisation of investigation techniques discussed in this chapter, according to their invasive/non-invasive character is provided in Table 3.

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[117] Acidic pH measured by Armstrong et al., Spafford and Graham, Hill, Hackett. Neutral pH measured by Roberts et al., France et al.

[118] Tímár-Balázsy and Eastop, 159-162.

[119] Francis, 48.

[120] "New Analytical Technologies and Protocols," Getty Conservation Institute, http://www.getty.edu/conservation/about/science/analytical_tp.html (accessed June 15, 2013).

Table 3 Scientific investigation techniques for the characterisation of sooty soilings

Scientific investigation techniques		
Non-Invasive	**Invasive**	
	Non-destructive	**Destructive**
▪ Stereomicroscopy (5.3.1.1) ▪ Infrared photography (5.4.2.1) ▪ Raman spectroscopy using fibre optics (5.4.2.3) ▪ Attenuated total reflection infrared spectroscopy (ATR-FTIR) (5.4.3.3) ▪ Fluorescence spectroscopy using fibre optics (5.4.3.4)	▪ Polarised light microscopy (PLM) (5.3.1.2) ▪ Scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDS/EDX) (5.3.2.1, 5.4.1) ▪ Raman spectroscopy and micro-spectroscopy using a microscope (5.4.2.3) ▪ Infrared spectroscopy and micro-spectroscopy using a microscope (5.4.3.3) ▪ Fluorescence spectroscopy (5.4.3.4)	▪ Transmission electron microscopy (TEM) coupled with energy dispersive X-ray spectroscopy (EDS/EDX) (5.3.2.2, 5.4.1) ▪ Thermogravimetry (TGA) (5.4.2.2) ▪ Gas chromatography (GC) (5.4.3.1) ▪ Liquid chromatography (LC) (5.4.3.1) ▪ Mass spectrometry (MS) (5.4.3.1) ▪ Ultraviolet-Visible spectroscopy (UV-Vis) (5.4.3.1) ▪ Ion chromatography (IC) (5.4.3.2)

5.3 Techniques for the morphological characterisation of sooty soilings

5.3.1 Optical microscopy

Optical microscopy is used to visualize fine details in the structure of an object, and to provide information of the physical characteristics of removed samples.[121] It involves the interaction of visible light from approximately 350nm to 700nm and the object under study.[122]

5.3.1.1 Stereomicroscopy

Stereomicroscopes use reflected light to obtain a three-dimensional magnified image of the object with full perception of depth.[123] The long working distance makes stereomicroscopes especially suitable for the *in situ*, non-invasive examination of soiling on artefacts. The *magnification* of stereomicroscopes is commonly below ×100.

Hill examined the surface of fire-damaged paintings which appeared to the naked eye to be entirely covered with a 'black, rough layer', by stereomicroscopy under ×64 magnification.[124] This allowed for the examination of larger ashes and chars, but the fine particles of soot were not discernible under low magnification. Hill emphasised that no stereomicroscopy images of soot deposition on paintings were available for comparison.

Fusco used stereomicroscopy at ×25 magnification for the examination of a 'black, crystalline soiling' on a Peruvian burial shroud which appeared as 'granules embedded in the interstices of the weave',[125] and was identified as black carbon by Raman spectroscopy. The photomicrographs included in her work were the only images found within the textile conservation literature showing black carbon-based soiling, which points out the lack of reference materials which could aid simple identification by stereomicroscopy.

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[121] B.H. Stuart, *Analytical Techniques in Materials Conservation* (Chichester: John Wiley & Sons Ltd, 2007), 80-87.

[122] S. Bradbury, *An Introduction to the Optical Microscope* (Oxford: Oxford University Press, 1989), 1-8.

[123] Bradbury, 10-11.

[124] Hill, 10-30.

[125] M. Fusco, "The Evolvement of a Treatment Plan for a Peruvian Burial Shroud and its Relation to Wider Treatment and Display Considerations for Funerary Textiles in Foreign and Descendant Cultures," (MA dissertation, Textile Conservation Centre, University of Southampton, 2007), 25-32.

5.3.1.2 Polarised light microscopy (PLM)

Polarised light microscopy can produce enlarged images of fibre and soiling samples up to approximately ×1000 magnification, and enables the identification of particles by examining them between polarising filters.[126]

Passing light through a polariser restricts its vibration to one plane. The shape, colour and size of the particles can be described under normal and plane polarised light, and compared to published reference materials such as those found in the McCrone Particle Atlas.[127,128] Combustion particles appear black and opaque, but they have differing shapes and sizes.

By examining particles between crossed polars, particles having *amorphous* and *crystalline* internal structure may be distinguished. [129] *Amorphous* materials such as soot, char and ash do not alter the vibration of the light and light extinction occurs in the microscope (Figure 4), whereas most *crystalline* materials with the exception of *cubic crystals* cause changes in the vibration and exhibit colours.

Although PLM is a potent tool in the identification of combustion products, it has the disadvantage that it requires a great deal of training and experience.[130] Removal of samples is necessary, which can be mounted in a temporary or permanent medium, the former enabling sample recovery and the latter allowing future examination of the specimen.

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[126] W.C. McCrone, “Polarised Light Microscopy in Conservation: A Personal Perspective,” *JAIC*, 33 (1994):101-114.

[127] W.C. McCrone, *The Particle Atlas, Vol.1-5*, (Michigan: Ann Arber Publishing Inc., 1973).

[128] “McCrone Atlas of Microscopic Particles,” McCrone Group, http://www.mccroneatlas.com/ (accessed July 15, 2013).

[129] “Polarised Light Microscopy,” University of Cambridge, http://www.unalmed.edu.co/rrodriguez/MENAS/DoITPoMS%20TLP%20-%20Optical%20Microscopy%20and%20Specimen%20Preparation%204.htm (accessed June 08, 2013).

[130] McCrone, “Polarised Light Microscopy in Conservation: A Personal Perspective,” 104.

Figure 4 Amorphous (isotropic) materials under crossed polars[131]

PLM was used for the characterisation of combustion particles by conservation scientists Moffatt, Wainwright and Corbeil.[132] Samples of vacuumed-off soiling and soiling collected onto clean cotton flannel by rubbing the surface of the soiled textiles were dispersed in a thermoplastic mounting medium for examination. Loose soiling contained black particles of various shapes, some resembling fireplace ash, some bituminous coal, and some insect parts when compared to images in McCrone's Particle Atlas. The fibres were covered with sub-micron spherical, black particles, on some fibres these were distributed in patches of brown-yellow medium. The authors concluded that the particles were suggestive of atmospheric pollution, but without appropriate reference materials a more precise characterisation could not be undertaken.

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[131] University of Cambridge.

[132] Moffatt, Wainwright and Corbeil.

5.3.2 Electron microscopy

Electron microscopy involves the interaction of an electron beam and the material under examination. It provides higher magnifications than optical microscopy and can be coupled with special detectors to reveal information about the elemental composition of the sample. Electron microscopy can be a powerful method in the morphological and elemental characterisation of textile soilings.

5.3.2.1 Scanning electron microscopy (SEM)

Scanning electron microscopy involves the scanning of the sample by a beam of electrons, which causes secondary and backscattered electrons to be dislodged from the atoms on its surface.[133] These electrons are detected and amplified to create a three-dimensional image at magnifications reaching the order of ×100 000. While traditional SEM requires that the samples be coated with a good conducting material, environmental scanning electron microscopes (ESEM) enable the analysis of uncoated samples and facilitate sample recovery.

The morphology and composition of combustion particles have been extensively researched by SEM within the field of environmental,[134] fuel engineering,[135] and forensic sciences.[136] A case study in art conservation using SEM for the morphological examination of sooty deposits will be included here, while SEM coupled with elemental analysis will be discussed in *section 5.4.1*.

Soot deposited on a fibre on the surface of a fire-damaged painting, and soot embedded in the paint layer has been investigated by Hill.[137] Soot on the fibre formed clumped masses with outer regions made up of three-dimensional branched forms (Figure 5). Experimenting with different sampling methods prompted the observation that soot may become easily deformed, therefore sampling techniques causing least disturbance in the

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[133] Stuart, 91-100.

[134] Cachier.

[135] M.C. Paul (ed.), *Soot: Sources, Formation and Health Effects* (New York: Nova Science Publishers, 2012).

[136] M.T. Pinorini et al., "Soot as an Indicator in Fire Investigations: Physical and Chemical Analyses," *Journal of Forensic Sciences*, 39 (1994): 933-973.

[137] Hill, 51-67.

soiling morphology need to be found. Hill emphasised that extensive experience is needed for the interpretation of identifying features of soot deposits.

Figure 5 Soot deposition on a fibre by SEM [138]

5.3.2.2 Transmission electron microscopy (TEM)

In transmission electron microscopy, accelerated electrons are transmitted through a thin section of the sample, and are projected on a detector.[139] Magnification of TEM reaches ×200 000.

TEM is a preferred analytical technique for the imaging of soot within the fuel engineering,[140] environmental[141,142] and forensic sciences,[143] probably due to its high magnification and to its suitability for studying the internal structure of particles. No reference has been found to the use of TEM in art conservation for the analysis of combustion particles, perhaps due to the difficulty of sample preparation and destructive manner of the analysis.

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[138] Hill, 53.
[139] Stuart, 87-91.
[140] Paul.
[141] Medalia, Rivin and Sanders.
[142] Cachier.
[143] Pinorini.

5.4 Techniques for the compositional analysis of sooty soilings

5.4.1 Elemental composition: Energy dispersive X-ray spectroscopy (EDS/EDX)

Energy dispersive X-ray spectroscopy involves the analysis of characteristic X-rays emitted by the atoms of the sample due to excitation by the electron beam of the scanning electron microscope or transmission electron microscope.[144] SEM-EDS, also called SEM-EDX has been used by conservators for the investigation of the elemental composition of sooty soilings.

Moffatt, Wainwright and Corbeil included in their report the results of the elemental analysis of loose particulate soiling removed from the textiles by vacuuming.[145] ESEM-EDS detected carbon in almost every sample, which might have been either black carbon or carbon contained in the fibre fragments and other organic materials in the soiling.

France et al. utilised SEM-EDS for the investigation of soiling particles on the fibres of the Star-Spangled Banner, which were found to be in the sub-micron to micron region indicating airborne origin.[146] The only element identified on all fibres was carbon, although it was not specified how the carbon content of the soiling and the organic carbon of the fibre polymer were differentiated.

5.4.2 Black carbon content

5.4.2.1 Infrared photography

Infrared photography involves the irradiation of an object with light containing a high proportion of infrared radiation with wavelengths of 700nm to 1mm, and the detection of infrared light coming from the object with a camera equipped with a visible light filter.[147] Infrared photography and reflectography are widely used techniques in painting conservation due to the ability of infrared light to penetrate varnish and paint layers, and to

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[144] Stuart, 92.

[145] Moffatt, Wainwright and Corbeil.

[146] France et al., 159.

[147] F. Mairinger, "UV-, IR- and X-ray imaging," in *Non-Destructive Microanalysis of Cultural Heritage Materials*, eds. S.K. Janssens and R. Van Grieken, 15-71 (Amsterdam: Elsevier, 2004).

detect carbon-based graphite, charcoal and inks used by artists to create underdrawings.[148] Underdrawings which absorb infrared light appear dark on the pictures whereas surrounding areas of, for example highly reflective gesso appear contrasting white.[149] This phenomenon is of interest for the present research as soot on textiles containing a high proportion of black carbon may behave in a similar manner to the carbon-based underdrawings.

Previous applications which might have involved the detection of carbonaceous substances on textile substrates, such as making worn stencil marks visible on American civil war canteens, and documenting preparatory sketches on South East Asian cloth paintings and thangkas were reported.[150] No publications have been found with reference to the use of infrared photography for detecting soot on historic textiles.

5.4.2.2 Thermogravimetric analysis (TGA)

Thermogravimetric analysis involves the measurement of weight loss as a function of temperature or time, providing characteristic curves for any given material.[151] The use of ignition for the study of black carbon in soot was described in 1912.[152] In the conservation literature a study by Alebic-Juretic and Sekulic-Cikovic included assessment of the soot content of soiling deposits on paintings by TGA.[153] Cotton wool swabs used for cleaning the surface of the paintings were burned at 550° and ignition loss of the remaining ash was achieved at 800°, thus the weight loss of the sample gave the quantity of soot in the soiling.

TGA is an invasive technique as an area needs to be cleaned in order to have sample for analysis. However, the sample needed is small and the technique has potential for the identification of sooty soilings on historic textiles.

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[148] "Fine Art Conservation," D. Lizun, http://fineartconservation.ie/damian-lizun-fine-art-conservation-4-4-44.html (accessed June 06, 2013).

[149] C. Dietz, G. Catanzariti and A.J. Martínez, "Infrared Reflectography Using 3-D Laser Scanning," *e-conservation magazine*, 18 (2011): 32-42, http://www.e-conservationline.com/content/view/982 (accessed July 12, 2013).

[150] S.R. Andrew and D. Eastop, "Using Ultra-Violet and Infra-Red Techniques in the Examination and Documentation of Historic Textiles," *The Conservator*, 18 (1994):50.

[151] Stuart, 341-347.

[152] Cohen and Ruston, Appendix B.

[153] Alebic-Juretic and Sekulic-Cikovic.

5.4.2.3 Raman spectroscopy

Raman spectroscopy is a powerful technique for the identification of black carbon containing materials. It is based on the interaction of the sample and the incident laser light radiating in the *near-ultraviolet,* visible or *near-infrared* region.[154] Rotational and vibrational changes in the sample molecules cause slight increase or decrease in the wavelength of the scattered light, detected by the spectrometer. While using low laser powers enables non-destructive analysis, Raman spectroscopy can also be used in a non-invasive manner with the help of *fibre optics*.

Raman spectroscopy has been increasingly employed in art conservation for the detection of black carbon. Edwards and Chalmers provided information about the spectral characteristics of lampblack pigment - soot derived from burning oil lamps - to aid its identification on objects of art and archaeology.[155] Fusco identified black carbon on a sampled fibre from the Peruvian burial shroud using a Raman microscope.[156] Raman microscopy has been applied by Worobiec et al. for the molecular fingerprinting of atmospheric particulates: the authors argued that the extremely Raman-active, high carbon content of the examined particles masked other components.[157]

5.4.3 Molecular characterisation by chromatography and molecular spectroscopy

Chromatography and molecular spectroscopy techniques have been applied in the characterisation of *polycyclic aromatic hydrocarbons* (PAHs), acidic compounds and anions originating from combustion processes, both within the field of conservation and other scientific disciplines.

Chromatography is an excellent technique for separating a mixture of compounds and identifying its components.[158] Very small amounts of a component can be detected which makes it especially suitable for use in conservation. Chromatography is based on the affinity of the components to the mobile phase (gas or liquid) passing through the column

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[154] Stuart, 136-157.

[155] H.G.M. Edwards and J.M. Chalmers (ed.), *Raman Microscopy in Archaeology and Art History* (Cambridge: The Royal Society of Chemistry, 2005), 92.

[156] Fusco, 28-29.

[157] Worobiec et al., 63-68.

[158] Stuart, 296-297.

and the stationary phase packed in the column. Components are separated according to their differing retention times and are observed after elution with special detectors. Chromatography techniques need careful sample preparation, and reference compounds have to be run in the system to calibrate retention times and detection responses, and to enable quantitative measurements.

Molecular spectroscopy is the analysis of electromagnetic radiation absorbed, emitted or scattered by molecules in the sample which undergo characteristic transitions between energy levels.[159] Although sample preparation is generally less complicated than for chromatography techniques, the sensitivity of the measurements may be lower and the interpretation of results may pose difficulties due to the complexity of spectra gained from samples of heritage materials.

5.4.3.1 Gas and liquid chromatography (GC, LC)

Gas and liquid chromatography have been widely used for the identification of PAHs and other organic molecules related to fuel burning. They require the removal and solubilisation of soiling samples.

In Hill's study of soiling on fire damaged paintings gas chromatography-mass spectrometry (GC-MS) was employed in order to identify PAHs present in the soiling. [160] Samples were gained by swabbing the surface of the artefacts and extracting the organic fraction in an organic solvent. Hill argued that PAHs may undergo degradation processes due to aging and thus may become unrecognisable for the chromatography technique.

Alebic- Juretic and Sekulic-Cikovic also chose PAHs as an indicator that the soiling on the examined paintings had originated from combustion.[161] They removed the soiling with cotton swabs and analysed the extracted organic fraction by high performance liquid chromatography (HPLC) - ultraviolet-visible (UV-Vis) detection.

In environmental science, a wide array of organic molecules in smoke from biomass and coal burning were identified by GC-MS technique,[162,163] showing the possibility for exact source appointment of smoke particles. As the experiments included the analysis of young,

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[159] Stuart, 109-110.
[160] Hill, 68-82.
[161] Alebic-Juretic and Sekulic-Cikovic.
[162] Oros and Simoneit.
[163] Simoneit.

artificially generated particles, the amount and composition of organic components in them might not be comparable with those found on historic artefacts.

5.4.3.2 Ion chromatography (IC)

Ion chromatography is used for identifying ions in a complex mixture.[164] The chromatograph operates with a column containing ion-exchange resin as a stationary phase. IC requires the solubilisation of a soiling sample.

Alebic- Juretic and Sekulic-Cikovic demonstrated the use of IC for the identification of anions in removed soiling samples. [165] Sulphates were found to be the dominant ionic species on paintings which had been exposed to higher levels of urban air pollution and sulphur dioxide levels during the 1980s. Nitrate content which was found to be higher in the soiling of paintings stored next to a busy road might indicate the impact of traffic-generated nitrogen oxides.

5.4.3.3 Infrared spectroscopy

Infrared spectroscopy is a technique based on the vibrations of the atoms of a molecule.[166] The spectrum is produced by passing infrared radiation of wavelength 2.5µm to 20µm through a sample and determining what fraction of the radiation is absorbed at a particular energy. Spectra interpretation is attained by comparing the spectral pattern of a known substance to the unknown substance.[167] Additionally, functional groups producing absorptions near the same frequency may be identified. Although transmission infrared spectroscopy requires sampling, attenuated total reflection (ATR-FTIR) spectroscopy can be carried out *in situ*.

Black carbon highly absorbs infrared radiation and does not lend itself to analysis by infrared spectroscopy. Infrared spectroscopy has been applied by Roberts et al. for the assessment of acidity in soot deposits on drapery contaminated by smoke from a building

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[164] Stuart, 323-328.

[165] Alebic-Juretic and Sekulic-Cikovic.

[166] Stuart, 110-136.

[167] M.R. Derrick, D. Stulik and J.M. Landry, *Infrared Spectroscopy in Conservation Science* (Los Angeles: Getty Conservation Institute, 1999), 14.

fire.[168] In the field of environmental science, infrared spectroscopy has been used for the examination of adsorbed organic molecules on *nascent* soot surfaces,[169] and of PAHs produced by burning vegetation.[170]

5.4.3.4 Fluorescence spectroscopy

Fluorescence occurs when the molecules of a material absorb an incident radiation, and their atoms on their return to the ground state loose energy through the emission of visible radiation which ceases after the removal of the excitation source.[171] Lasers are used for the excitation of the sample molecules, and artefacts can be examined in a non-invasive manner with *fibre optics*. As polycyclic aromatic compounds produce characteristic fluorescence emission spectra,[172] fluorescence spectroscopy shows potential for the study of PAHs in sooty soilings.

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[168] Roberts et al., 7.

[169] J.P. Cain et al., "Micro-FTIR study of Soot Chemical Composition: Evidence of Aliphatic Hydrocarbons on Nascent Soot Surfaces," *Journal of Physical Chemistry and Chemical Physics*, 12 (2010):5206-5218.

[170] R.A. Sutherland, R. K. Khanna & M. J. Ospina, **"**Infrared Properties of Atmospheric Aerosol Constituents: Polyaromatic Hydrocarbons and Terpenes," *Aerosol Science and Technology Online,* 20 (1994):62-70.

[171] Stuart, 163-168.

[172] A. Matuszewska and M. Czaja, "Aromatic Compounds in Molecular Phase of Baltic Amber: Synchronous Luminescence Analysis," *Talanta*, 56, (2002):1049-1059.

CHAPTER 6: CASE STUDIES

6.1 Introduction

6.1.1 Selection of objects

Two domestic interior historic textiles, an early twentieth-century embroidered banner and a nineteenth-century muslin curtain were chosen for soiling assessment within the framework of the dissertation project. The dominant type of soiling on both textiles was dark grey in colour: a colour often associated with soils containing black carbon. Initial examination of the objects under low magnification allowed black particles to be seen adhered to the textile fibres, which prompted further investigation.

Having been displayed and stored in the domestic interior, it is likely that the two textiles were exposed to combustion particles at some point during their lifetime. Soiling might have originated from open fires, or the polluted outdoor atmosphere. However, this stays an assumption until the character of the soiling is revealed.

6.1.2 Introduction to the objects

The William Morris Action Song Challenge Banner

The subject of the first case study is an early twentieth-century embroidered banner which was given to the Centre for Textile Conservation and Technical Art History (CTCTAH) for conservation from a private client. The author worked on the conservation of the banner between February and April, 2013. The banner had a dark grey, greasy soiling unevenly covering the front and reverse sides. Cleaning of the textile was requested by the client, therefore the treatment focused on the examination and documentation of the soiling and the planning and implementation of a progressive cleaning treatment. Treatment time was limited, and invasive sampling for soiling investigation purposes was kept to minimum due to general ethical considerations. However, soiling samples at different cleaning stages were retained and with the client’s agreement they were used for further analysis.

The Muslin Curtain

The subject of the second case study is a nineteenth-century muslin curtain, chosen from the Karen Finch Reference Collection at the CTCTAH. The textile was examined subsequently to the banner, between May and July, 2013. The white muslin curtain had a marked dark grey soiling. The soiling was assessed for identification purposes only, without the aim of carrying out a complete cleaning treatment. Although a historic textile, the muslin curtain belongs to the study collection of the CTCTAH therefore in agreement with the MPhil Textile Conservation course tutors, information gained by the investigation techniques was prioritised and invasive analytical techniques were also considered.

6.1.3 Complementing case studies

The purpose of the two case studies was to show the applicability of different examination and scientific investigation techniques identified in Chapter 5 for the assessment of sooty soilings. Different techniques were applied in the two cases when they were felt to have a better potential for soiling identification. The different materials and construction, and the effect of these attributes on the nature of soiling deposition also influenced choices.

6.1.4 From examination to analysis

Research of the soiling included the use of simple and advanced instrumental analytical techniques. Examination of the objects and the use of readily available scientific techniques took place in the CTCTAH, while others depended on external equipment and expertise availability. Most of the instrumental analytical techniques were available via the Scottish Crucible funded ‘Dirty Stories’ research project.[173] Operational support and advice on sample preparation were provided by experienced analytical researchers. All sample preparation, instrument operation where practically possible, and input to result

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[173] The ‘Dirty Stories’ project runs in 2012 and 2013, in collaboration between analytical scientist Dr Johannes Kiefer, University of Aberdeen and Dr Anita Quye, University of Glasgow. The project aim is to develop analytical protocols for the investigation of dark soiling seen on nineteenth and early twentieth-century domestic textiles, suspected to have arisen from polluted domestic and industrial environments of the period.

interpretation was in every case undertaken by the author. Where possible, samples were prepared to allow additional future investigation.

Being a new area of research for the CTCAH, existing comparative data and reference materials were limited. For techniques requiring the use of reference materials to help the interpretation process, references were collected or prepared by the author. These will be discussed under each sub-chapter and details included in the Appendices.

6.1.5 Soiling characterisation methods

A brief description of the objects and their general condition will be followed by the characterisation of the textile soiling according to the scheme introduced in Chapter 5 (Figure 6-7).

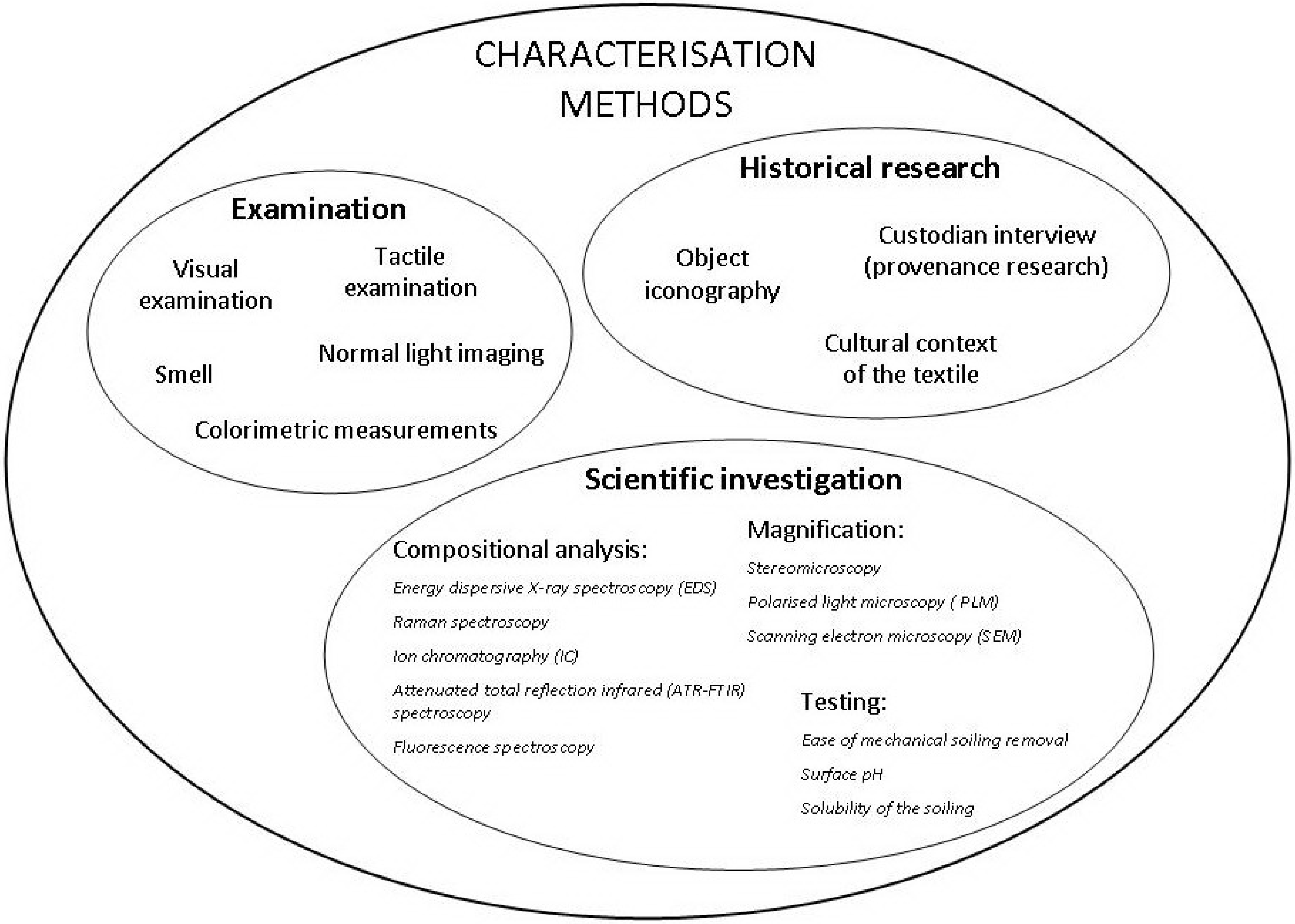

Figure 6 Soiling characterisation: William Morris Action Song Challenge Banner

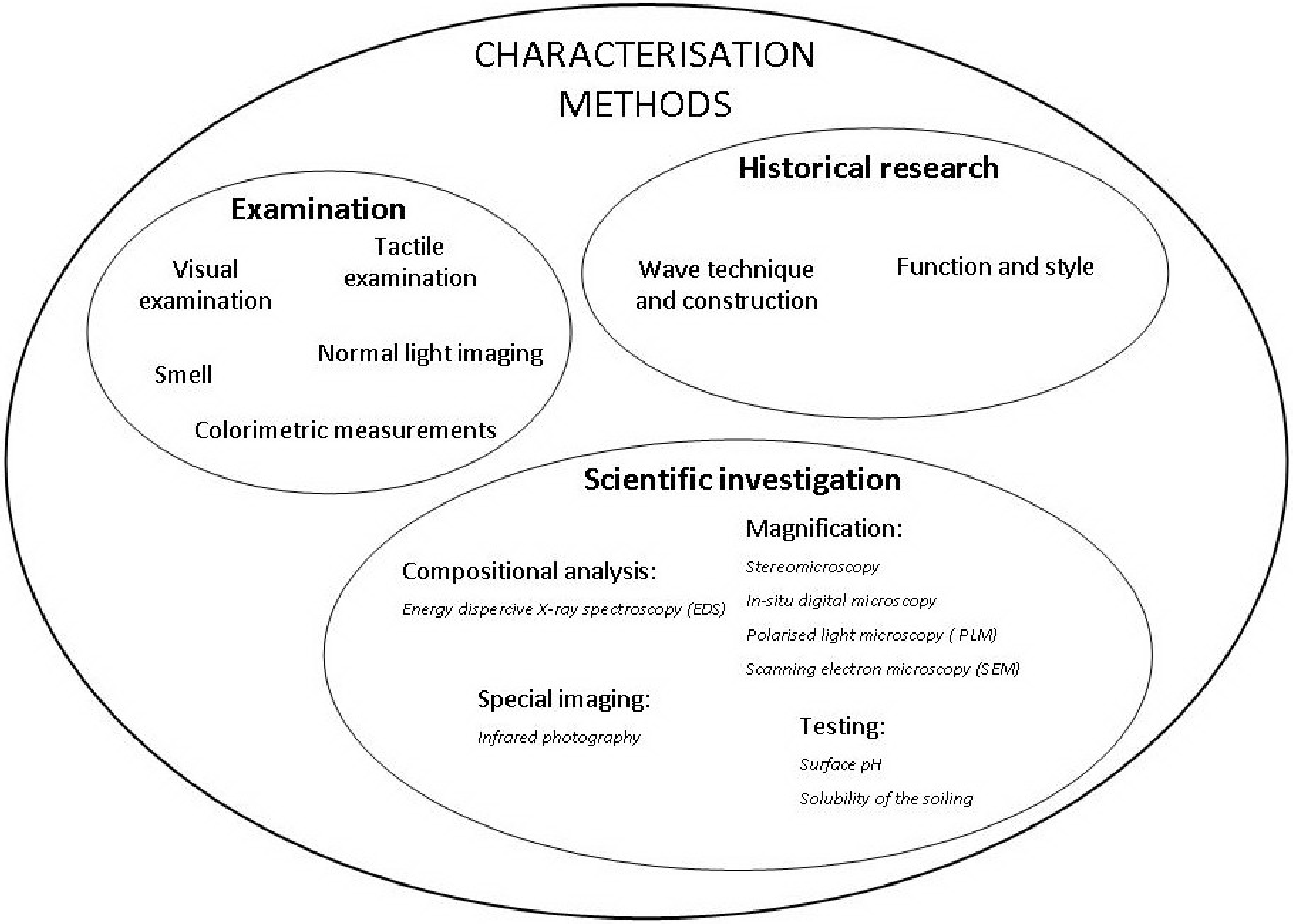

Figure 7 Soiling characterisation: muslin curtain from the Karen Finch Reference Collection

6.2 Case Study 1: The 'William Morris Action Song Challenge Banner'

6.2.1 Object description

The banner (Figure 8-9) was made of undyed plain weave linen top and lining fabrics, a stiffened dark brown plain weave cellulosic interlining, and a light brown and green twisted cotton fringe along the lower edge. The top fabric was decorated with colourful silk embroidery, depicting a garland of flowers at the top with an inscription: 'William Morris Action Song Challenge Banner', and a group of playing children at the bottom. The fabrics and fringe were attached by hand-stitching and the embroidery was also hand-made. The banner could be attached to a wooden rod, which was covered with three metal plates bearing engravings. A twisted undyed linen cord was tied to the ends of the rod from which the banner could be hung. (For details on materials and construction, see Appendix 2.)

Figure 8 Front of the banner before treatment (©CTCTAH)

Figure 9 Reverse of the banner before treatment (©CTCTAH)

6.2.2 Historical background

The wording of the embroidery and the engravings indicated that the banner was associated with an ‘action song’ youth competition which comprised of singing and acting, held regularly in the first decades of the twentieth century. Personal communication with the client clarified that the banner was a prize for the competition. It was presented by the William Morris Choir, one of the dozen socialist choirs operating in Glasgow from the late 1890s.[174]

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[174] A. Lees and L. H. Lees, *Cities and the Making of Modern Europe, 1750-1914* (Cambridge: Cambridge University Press, 2007), 231.

As a competition prize, it is likely that the banner was handed from one custodian to the other, being in an ever-changing environment. In the care of its current custodian, the textile was hung on a wall in an unheated room with no obvious soiling source. It is likely that the grey soiling originated from previous exposure to airborne contaminants.

6.2.3 Examination

The condition of the textile with special regard to the characteristics of the soiling was assessed by visual and tactile examination and recorded by normal light photography according to CTCTAH guidelines. [175,176] Photography was carried out with a Nikon D50 SLR camera fitted with a standard 18-55mm zoom lens. As colour vision is subjective, colour in heavily soiled and cleaner areas was recorded by a Minolta Chroma Meter CR-210 colorimeter.

6.2.3.1 Visual examination

The banner was found to be in a structurally sound condition, with overall grey soiling and localised stains being the dominant condition features. Sharp creasing and areas of damage in the embroidery were due to previous rolled storage and to mechanical action.

The grey soiling especially heavily deposited around the edges of the top fabric and on the bottom half of the lining (Figure 10-11). The original colour of the fringe and the vivid colours of the embroidery were masked by the soiling. Its distribution pattern on the lining indicated a mainly airborne source. Additionally, greasy components deposited due to handling could have embedded more airborne particulates around the edges.

General yellowing of the top fabric particularly around the soiled edges and of the soiled bottom half of the lining was observed. It was unclear whether yellowing was caused by a coloured component of the soiling, or by chemically or light-induced cellulose degradation processes or perhaps both.

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[175] University of Glasgow, “Guide to Documentation,” 2012.
[176] University of Glasgow, “Photography Manual,” 2011.

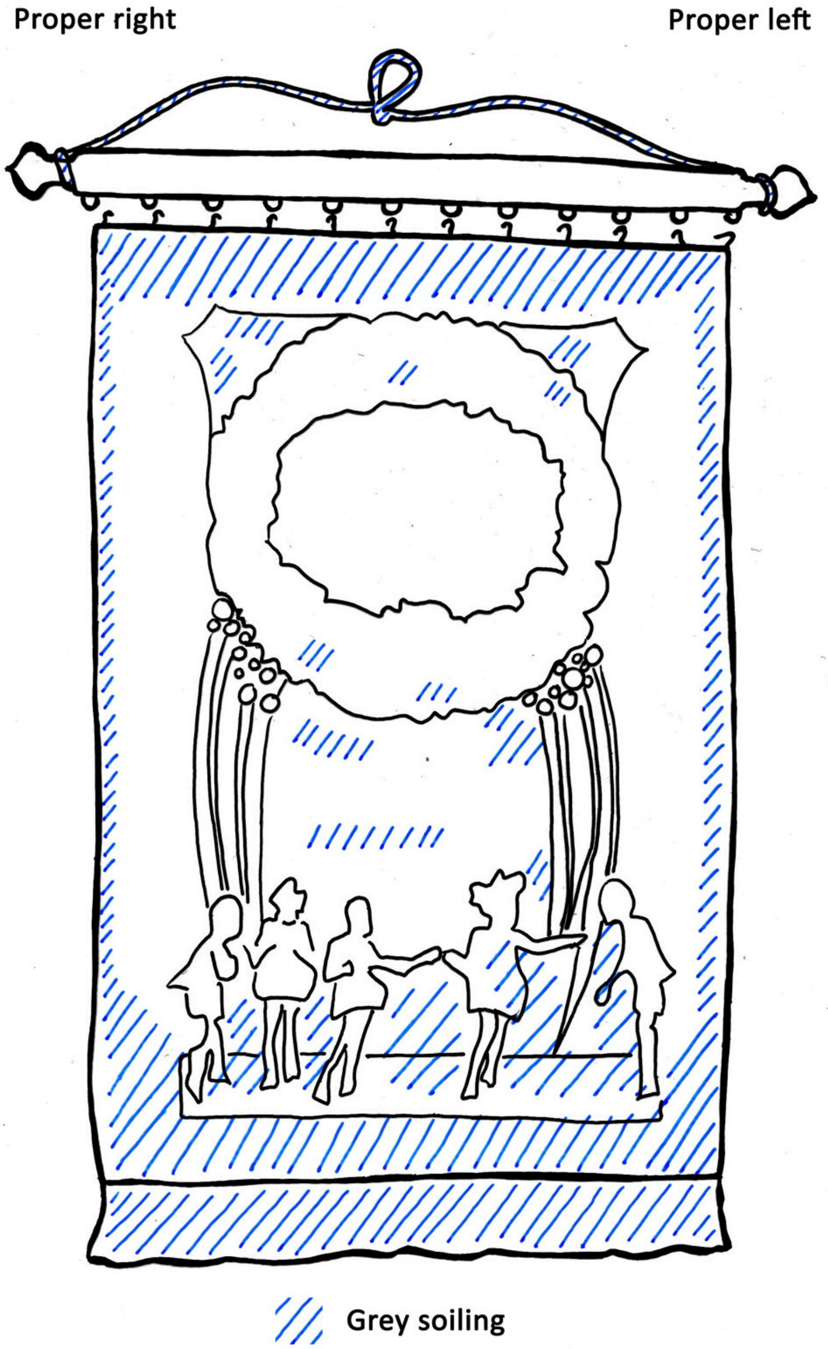

Figure 10 Grey soiling on the front

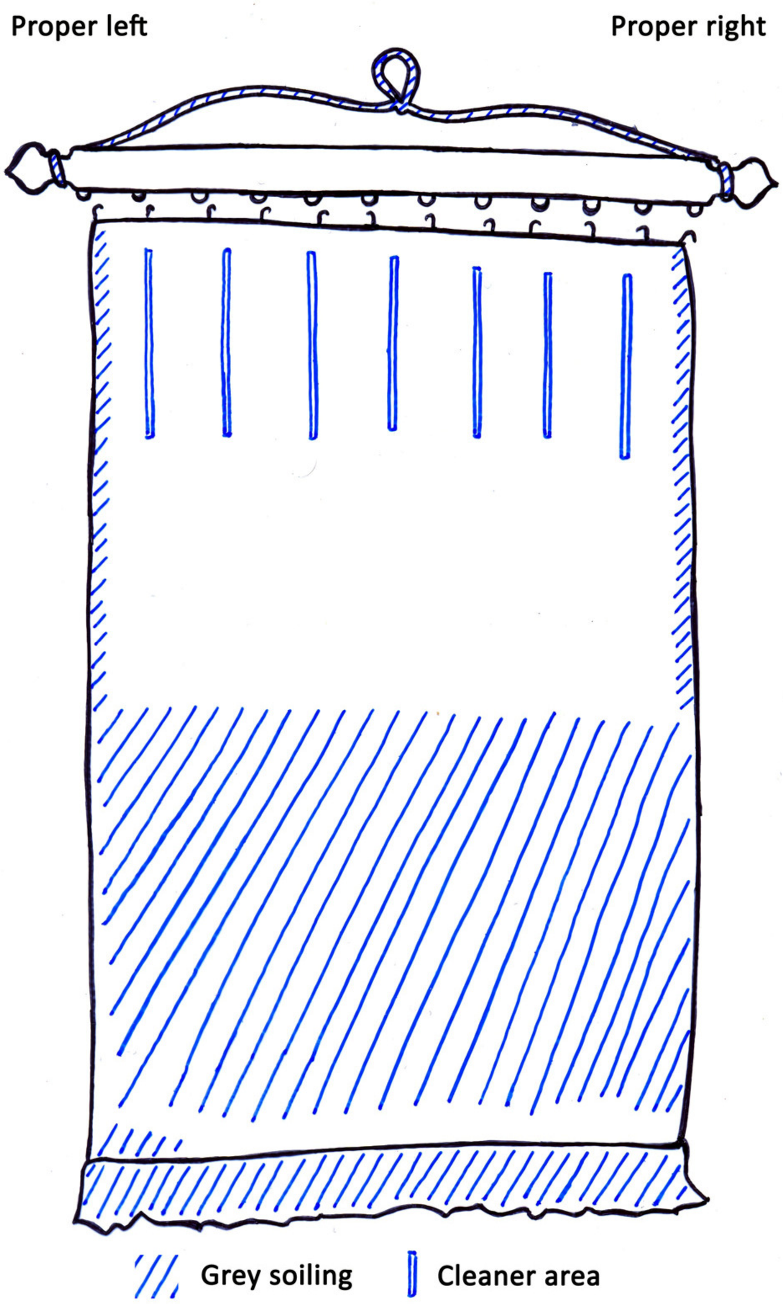

Figure 11 Grey soiling on the reverse

6.2.3.2. Tactile examination

During manipulation of the textile the soiling left a greasy-feeling dark residue on the fingers. The residue had a strong smell, which was not possible to describe objectively.[177] The soiling did not affect the flexibility of the fabrics, but stiffened the yarns of the fringe and caused them to stick together.

6.2.3.3 Colorimetric measurements

The level of greying and yellowing was recorded by colorimeter for future reference (for measurements see Appendix 2). Due to the fact that greying might be caused by non-carbonaceous soiling,[178] colour measurement in itself - similarly to the determination of 'smoke shades' in environmental science,[179] was not feasible for soiling identification purposes.

6.2.4 Testing

6.2.4.1 Ease of mechanical removal

Vulcanised natural rubber sponge - also called Smoke sponge - was tested for mechanical cleaning.[180] Smoke sponges had been used in several cases for the removal of sooty soilings. [181] The sponge effectively lifted off the grey soiling from the linen fabrics, and became itself almost black. In comparison, it did not remove any other types of localised soiling.

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[177] Lachelin pointed out difficulties in the description of smells.

[178] W.C. Powe, "The Nature of Tenaciously Bound Soil on Cotton", *Textile Research Journal*, 29 (1959):879-884.

[179] Brimblecombe, The Big Smoke, 152-153.

[180] "Smoke Sponges for Clean up in Fire Restoration," Preservation Equipment Ltd, http://www.preservationequipment.com/Store/Products/Disaster-$4-Cleaning/Just$9In$9Case/Smoke-Sponges-for-Clean-up-in-Fire-Restoration (accessed July 02, 2013).

[181] Hackett.

6.2.4.2 Solubility and pH of the soiling

Solubility tests were carried out with deionised water, anionic and non-ionic detergents, and organic solvents (for details see Appendix 2). Tests showed that the soiling and yellowing were most soluble in aqueous, especially in anionic detergent solutions followed by their solubility in industrial denatured alcohol (IDA), indicating that the soiling responded to emulsification and to polar solvents. Soiling removed with aqueous solutions was tested with pH strips, and showed a slightly acidic pH 6.1-6.4. Acidity might have arisen from the soiling, but also from the aged cellulosic fibres.

6.2.5 Scientific investigation

The following section describes the analytical methods used to examine the soiling (Table 4).

Table 4 Summary of samples for scientific investigation of the banner

Subject (sample)	Technique
Textile	Stereomicroscopy
Soiled Smoke sponges from mechanical cleaning	SEM-EDS ATR-FTIR spectroscopy
Soiled thread sample (5mm) removed from fringe	SEM-EDS Raman spectroscopy
Thee washbath samples (100-100ml) from the three subsequent cleaning immersions of the textile in IDA	ATR-FTIR spectroscopy Fluorescence spectroscopy
Dry residue gained from the second washbath by evaporation (of 50ml)	PLM
Aqueous solution of the dry residue of the second washbath (5ml)	Ion chromatography

6.2.5.1 Magnification by stereomicroscopy

In situ examination of the grey soiling was undertaken at the CTCAH with a Stemi 2000-C stereomicroscope. Illumination was provided by a KL 1500 LCD light box. The stereomicroscope could be operated between ×6.5 and ×50 magnifications. Photographs were taken with a Canon PowerShot A75 compact camera through the eyepiece in lack of a camera attachment.

The lining (Figure 12) and the top fabric (Figure 13) showed similar microscopic appearance in areas of grey soiling: black particulates smaller in diameter than individual linen fibres were visible (fibre diameter is approximately 15-20µm but fibre bundles are thicker);[182] loose fibres emerging from the threads were coated with black substance; and white salt-like crystals were apparent in the interstices of the weave. Black coating on loose fibres was particularly visible on the yarns of the fringe (Figure 14). Furthermore, brown solid residue was adhered to the embroidery threads (Figure 15).

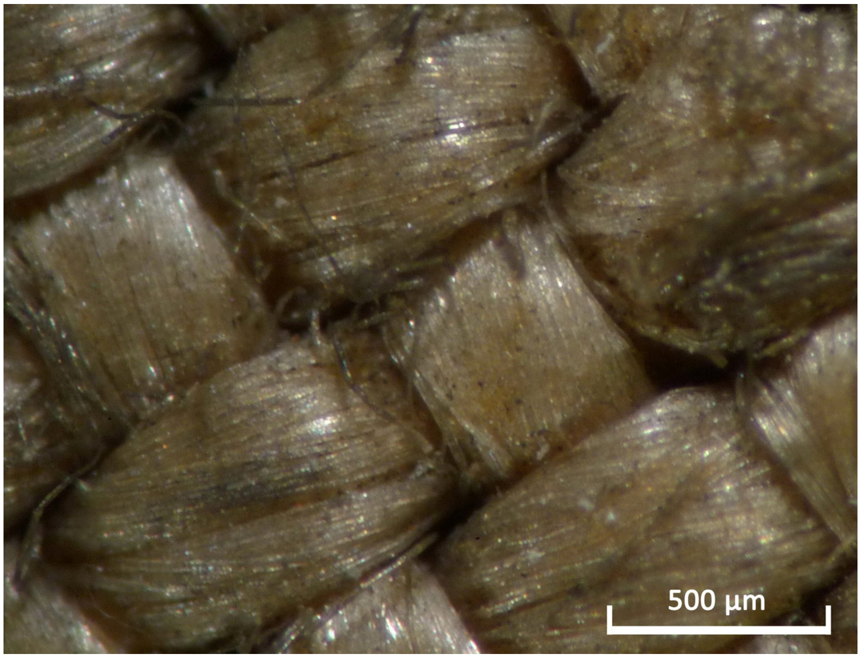

Figure 12 Soiling on the lining by stereomicroscopy

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[182] M. Sawbridge and J.E. Ford, *Textile Fibres under the Microscope* (Manchester: Shirley Institute, 1987), 17.

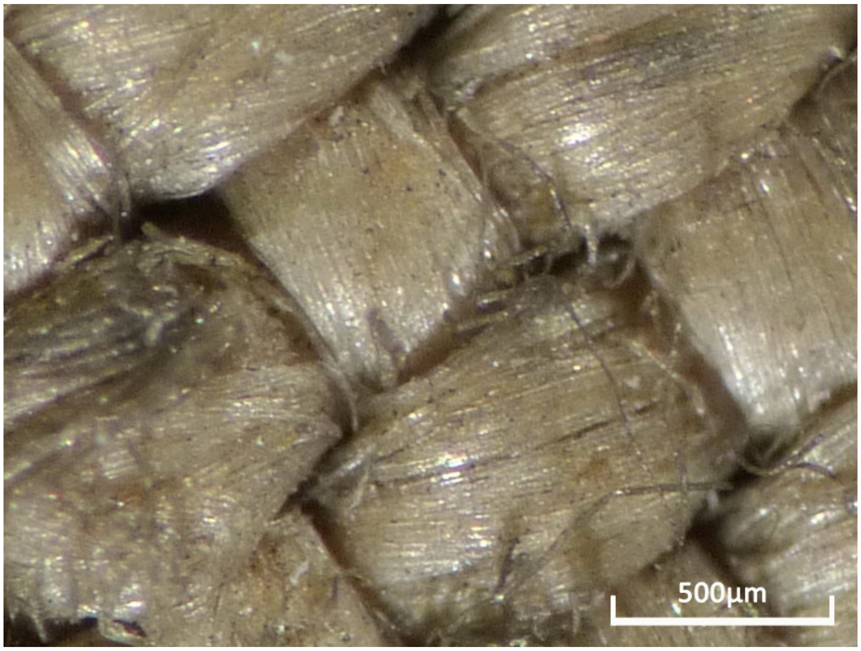

Figure 13 Soiling on the top fabric by stereomicroscopy

Figure 14 Soiling on the fringe by stereomicroscopy

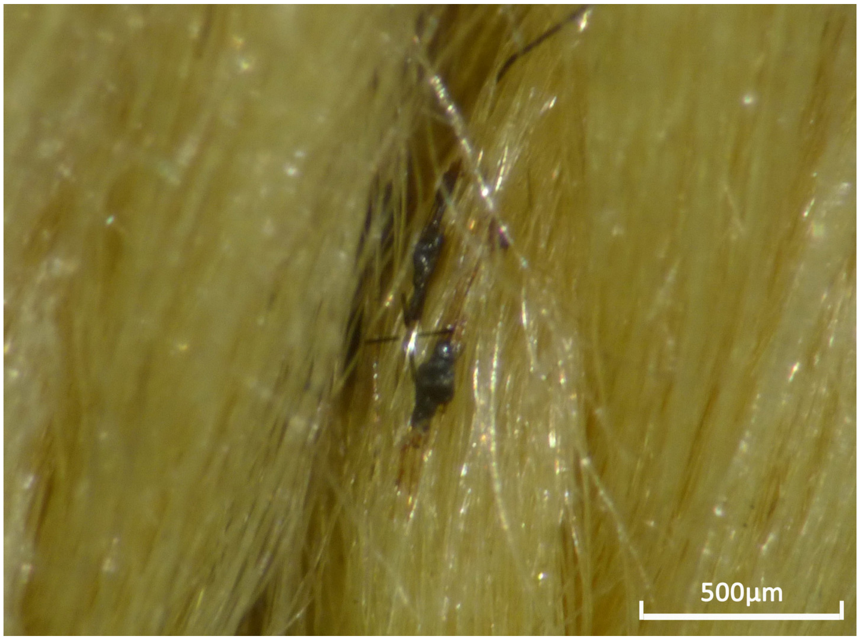

Figure 15 Soiling on the embroidery by stereomicroscopy

6.2.5.2 Magnification and elemental analysis by SEM-EDS

The soiled surface of a Smoke sponge, and a soiled fibre sample removed from the cotton fringe were analysed by SEM-EDS (Figure 16). It was acknowledged that as the sponge and the fibre were made of carbon-based polymers - natural rubber and cellulose, respectively, there would be no ‘elemental contrast’ between the organic carbon content of the substrates and the black carbon content of the soiling if soot was present. Information was confined to morphological features and trace elements in the soiling which might indicate combustion processes.

SEM-EDS was carried out by Peter Chung at ISAAC, School of Geographical and Earth Sciences, University of Glasgow, on a Carl Zeiss Sigma Variable Pressure Analytical SEM operated with SmartSEM software. The Oxford Microanalysis X-Max Silicon Drift Detector was capable of detecting low atomic number elements including carbon. Point analysis in environmental pressure mode collected information about elemental composition from a larger area around a selected point.

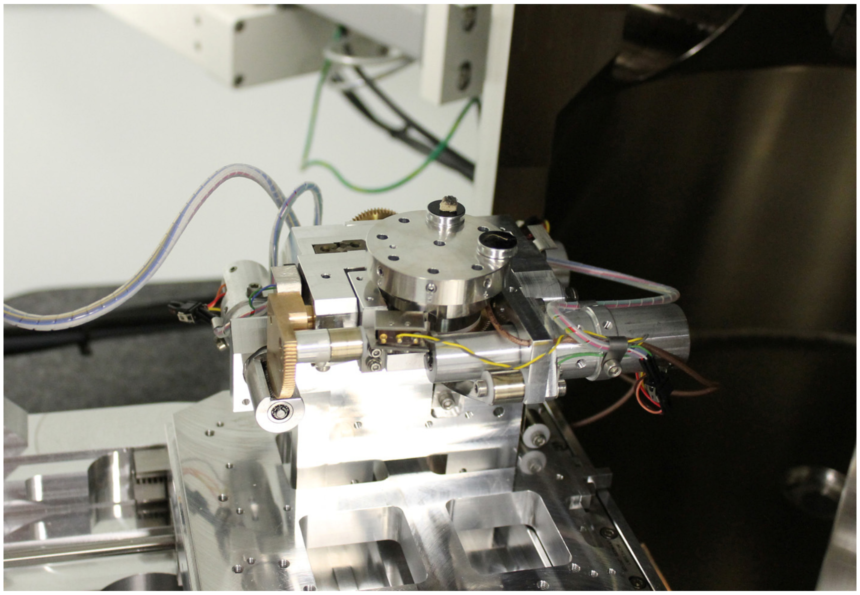

Figure 16 Smoke sponge and thread samples on the sample wheel of the SEM

The Smoke sponge was made of vulcanised natural rubber with calcium carbonate filler.[183] Sulphur, traces of aluminium, silicon, iron, zinc, sodium, and oily components could have been added during the manufacturing process.[184] SEM imaging did not show a distinctive soiling morphology. Dominant morphological features were the angular particles of calcium carbonate and fibre fragments caught on the sponge surface (Figure 17). EDS analysis showed a high percentage of carbon in all areas which might have partially originated from the soiling. Point analysis of angular particles showed calcium, zinc, sulphur, silicon, and aluminium (Figure 18), and any of these might be present in the sponge. A particle with high mean atomic number ($\bar{z}$) contained lead (Figure 19), which might have originated from a paint pigment, leaded petroleum,[185] or fossil fuel combustion.[186]

As the soiling most likely became deformed by the sponging action and elements which could be indicators of the source of the soiling might be present in the sponge in trace amounts, SEM-EDS analysis did not reveal conclusive information for soiling identification.

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[183] Preservation Equipment Ltd.

[184] Hackett, 67.

[185] J.M. Horner, "Lead in House Paints: Still a Health Risk that Should Not Be Overlooked," *Journal of Environmental Health Research*, 3 (2004). http://www.cieh.org/JEHR/lead_house_paints.html (accessed July 16, 2013).

[186] See *section 4.2.3*, Table 1.

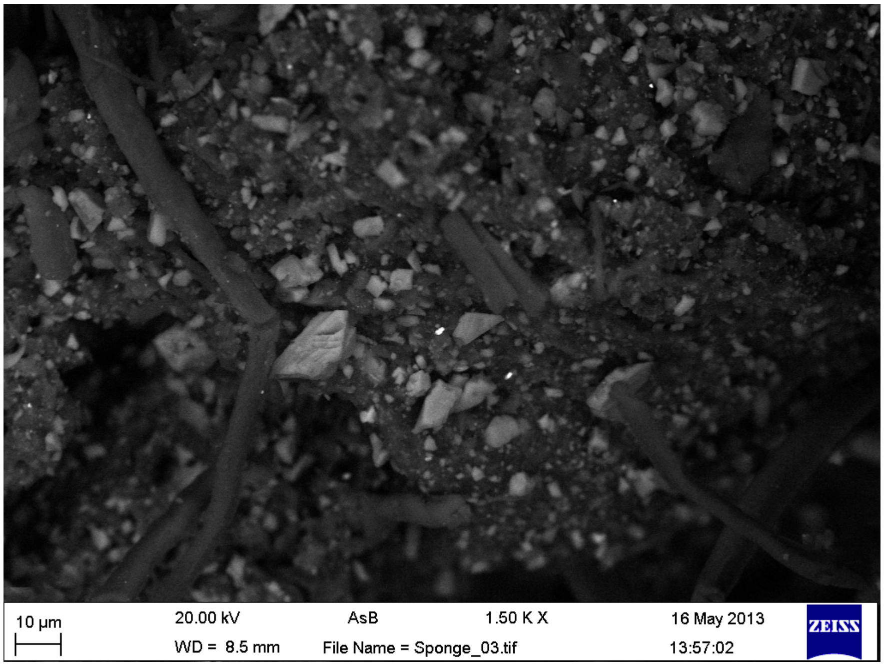
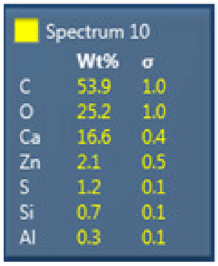


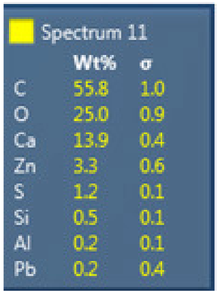
Figure 17 Surface of the Smoke sponge, SEM 1500×



Spectrum 10

	Wt%	σ
C	53.9	1.0
O	25.2	1.0
Ca	16.6	0.4
Zn	2.1	0.5
S	1.2	0.1
Si	0.7	0.1
Al	0.3	0.1

Figure 18 Angular particles, EDS



Spectrum 11

	Wt%	σ
C	55.8	1.0
O	25.0	0.9
Ca	13.9	0.4
Zn	3.3	0.6
S	1.2	0.1
Si	0.5	0.1
Al	0.2	0.1
Pb	0.2	0.4

Figure 19 Particle with high z̄, EDS

Wt% ~ weight percentage
Ϭ ~ standard deviation

SEM imaging of the thread sample revealed that the soiling particles were in the micrometer to sub-micrometer region in size (Figure 20). SEM-EDS point analysis showed that particles with high z̄ (Figure 21) contained a high percentage of carbon from the fibre polymer and possibly the soiling, oxygen and iron, lesser amounts of sulphur and sodium, and traces of phosphorous, calcium, potassium, chlorine, aluminium, and silicon. Analysis in areas of lower z̄ (Figure 22) did not show the presence of iron and silicon. Iron and sulphur may possibly be indicators of fossil fuel burning.[187] Other metallic elements may indicate the

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[187] See *section 4.2.3*, Table 1.

effect of general urban air pollution.[188] Elemental analysis of the soiling on the fibre sample was thus revealing.

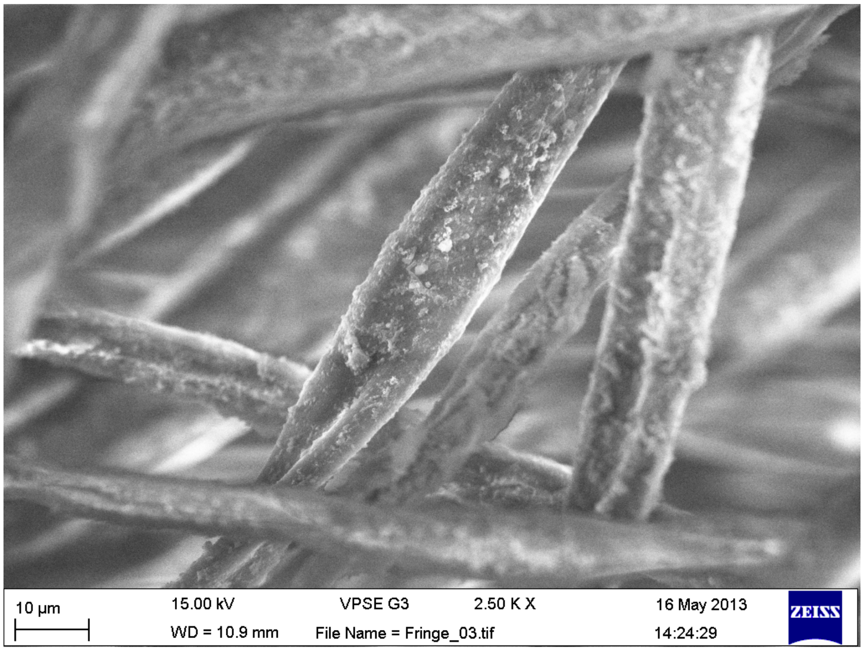
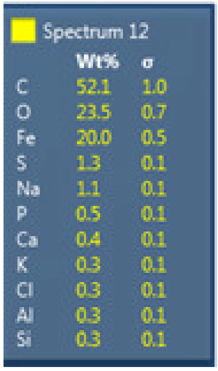


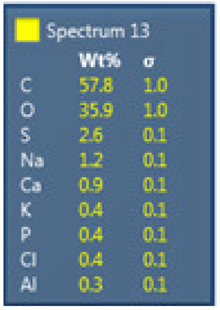
Figure 20 Surface of the fibre, SEM 2500×



Spectrum 12

	Wt%	σ
C	52.1	1.0
O	23.5	0.7
Fe	20.0	0.5
S	1.3	0.1
Na	1.1	0.1
P	0.5	0.1
Ca	0.4	0.1
K	0.3	0.1
Cl	0.3	0.1
Al	0.3	0.1
Si	0.3	0.1

Figure 21 Particle with high z̄, EDS



Spectrum 13

	Wt%	σ
C	57.8	1.0
O	35.9	1.0
S	2.6	0.1
Na	1.2	0.1
Ca	0.9	0.1
K	0.4	0.1
P	0.4	0.1
Cl	0.4	0.1
Al	0.3	0.1

Figure 22 Particle with lower z̄, EDS

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[188] France et al., 159.

6.2.5.3 Raman spectroscopy of a thread sample

The soiled thread sample was examined using a Renishaw® inVia Raman Microscope at the ISAAC, University of Glasgow. Raman spectrum for black carbon was available for consultation.

A small black particulate on the surface of a single fibre needed to be focused on under the ×20 objective lens, but unsuccessful trials to achieve this prevented recording the spectrum. Perhaps larger soiling aggregates such as the ones mentioned by Fusco could have been more easily analysed with the instrument.[189] As the analysis of black carbon on fibres has been tried successfully in the past, the technique would worth further investigation.

6.2.5.4 Polarised light microscopy of soiling removed by immersion cleaning

Each of the three subsequent IDA washbaths was sampled (Figure 23). Dry residue was obtained by evaporating the solvent from 50ml of the second washbath sample (Figure 24). A small amount of residue was scraped off with a clean needle, and mounted on a microscope slide with Eukitt® permanent mounting medium.

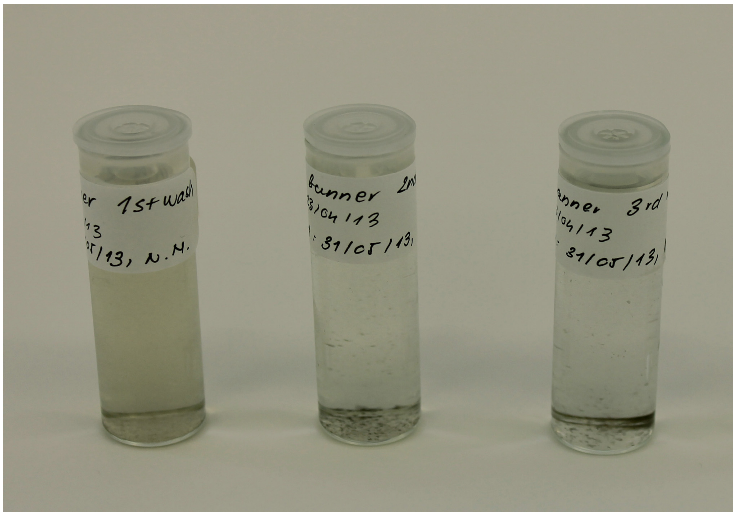

Figure 23 Three IDA washbath samples

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[189] Fusco, 28-29.

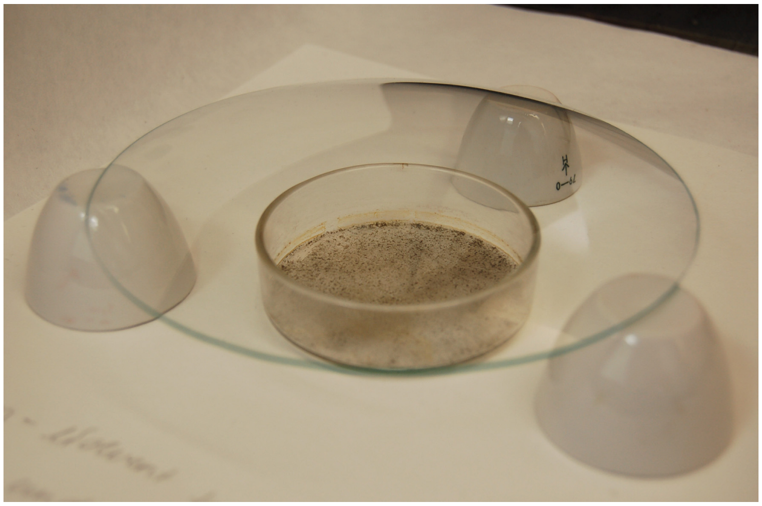

Figure 24 Preparation of dry washbath residue

The slide was examined on an Olympus BH2 polarising transmitted light optical microscope with a rotating stage at ×400 magnification, with William Revie at Construction Materials Consultants, Stirling. Photomicrographs were taken with a Nikon Coolpix 950 camera. Particle sizes were measured with the ocular scale.

The sample was composed of silk and linen fibre fragments and black opaque particles of various shapes having diameters between 7.5-40µm (Figure 25). Black particles smaller than 2.5µm were held together as aggregates in a greyish-greenish film, probably due to the solubilisation of substances in the washbath and evaporation of the solvent. Photomicrographs were compared to images of combustion particles in the Particle Atlas.[190] Three images were found with comparable morphological features: that of coke from a gas producer (Figure 26), of bituminous coal (Figure 27) and diesel exhaust (Figure 28). No further conclusions were drawn from morphological similarities.

To examine the sample under crossed polars, the stage was rotated with 45° and again with 45° degrees. While fibre fragments which have a partially *crystalline* structure changed colour with the rotation, the opaque black particles remained invisible and were declared to be *amorphous* structured (Figure 25: 1.2-5.2, 1.3-5.3). The three particle characteristics - black colour, opaque appearance, and *amorphous* internal structure - point in the direction that the particles could belong to the group of combustion products.

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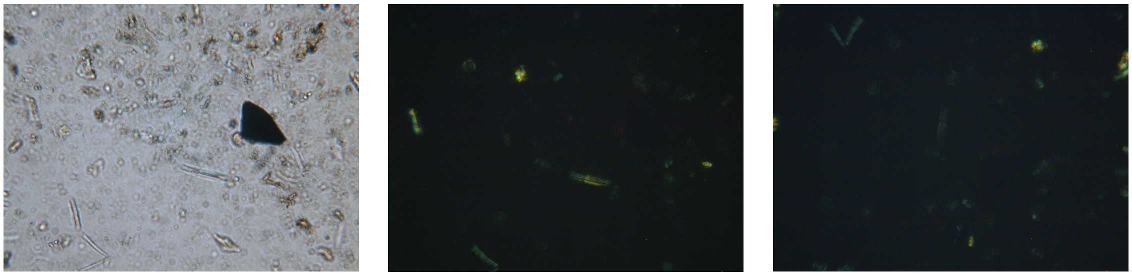
[190] McCrone, The Particle Atlas.

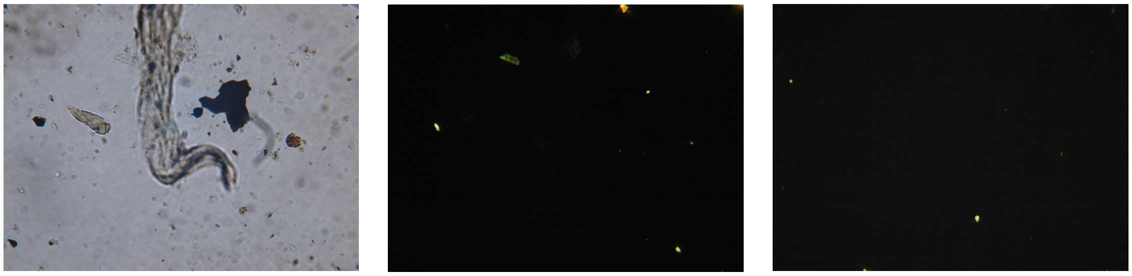
Plane polarised light | *Cross polarised light 90°* | *Cross polarised light 45°*

1. Angular equant black particle



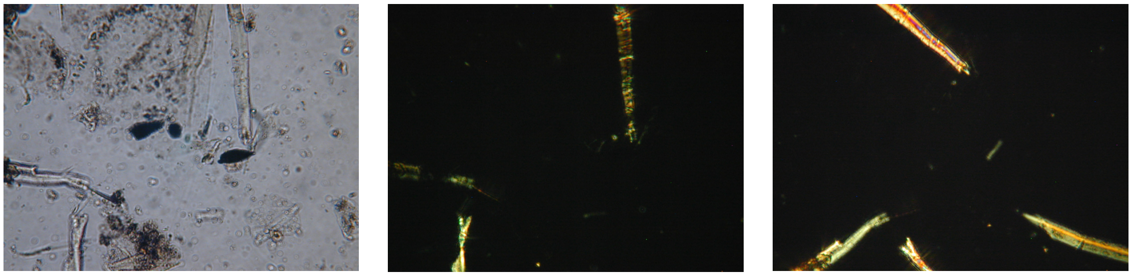
1.1 1.2 1.3

2. Irregularly equant and aggregated black particles



2.1 2.2 2.3

3. Acicular black particles and fibre fragments

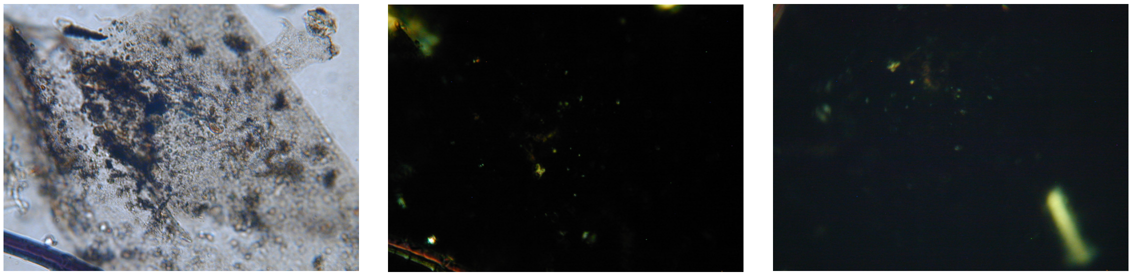


3.1 3.2 3.3

Figure 25 Polarised light microscopy photomicrographs of the washbath residue, ×400

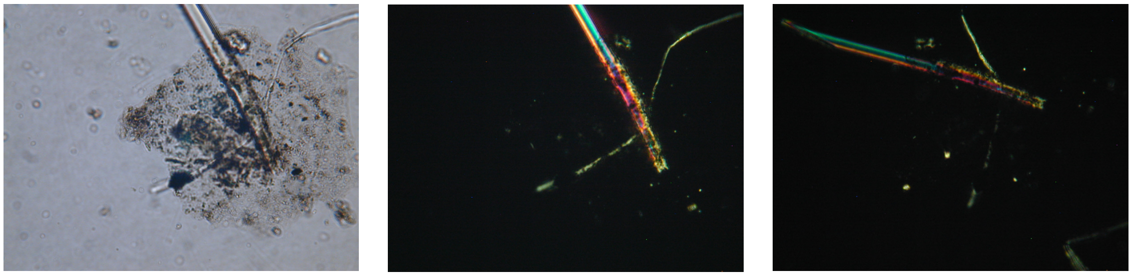
Plane polarised light *Cross polarised light 90°* *Cross polarised light 45°*

4. Aggregated black particles



4.1 4.2 4.3

5. Aggregated black particles and fibre fragments



5.1 5.2 5.3

Figure 25 Continued: Polarised light microscopy photomicrographs of the washbath residue, ×400

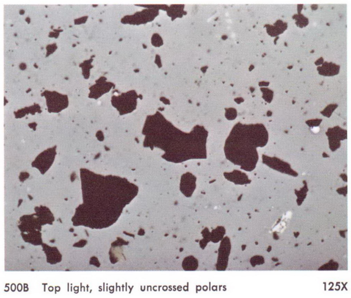


Figure 26 Coke[191]

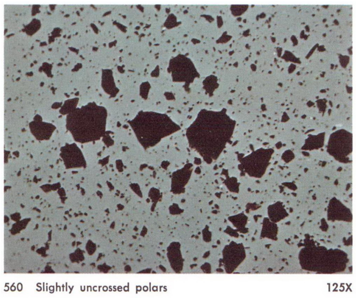


Figure 27 Bituminous coal[192]

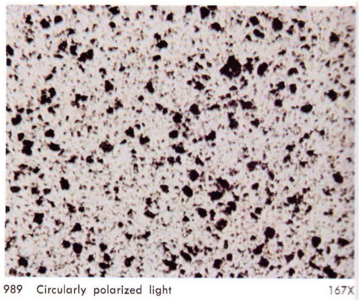


Figure 28 Diesel exhaust[193]

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[191] McCrone, The Particle Atlas, 519.
[192] McCrone, The Particle Atlas, 544.
[193] McCrone, The Particle Atlas, 1334.

6.2.5.5 Ion chromatography: detecting sulphate and nitrate anions

Sample for ion chromatography was prepared by solubilising the dry residue of the second washbath (see *section 6.2.5.4*) in 5ml deionised water. As a reference, 50ml IDA was evaporated from a beaker which was rinsed with 5ml deionised water.

Ion chromatography was carried out with the help of Moira McMenemy and Matthew Palmer, Department of Pure and Applied Chemistry, University of Strathclyde, on a DIONEX DX-100 Ion Chromatograph operating with a Dionex Ion Pac AS22 Anion-Exchange column (Figure 29). The eluent was sodium-carbonate in 0.68 millimolar concentration. Calibration was done with deionised water theoretically containing no nitrates and sulphates, and four standard solutions containing 1, 2, 3 and 4 mg/l sulphate and nitrate anions, respectively. Chromatograms showed the conductivity in microsiemens on the vertical and retention time on the horizontal axis.

The reference solution gave small peaks for nitrate (NO_3^-), sulphate (SO_4^{2-}) and chloride (Cl^-) (Table 5). Particulates from the sample solution were removed with a 0.2µm syringe filter, and the sample was diluted by ×50 in distilled water to achieve a chromatogram with values within the calibration range of the measurement. As chloride content was beyond the scope of the project, only one calibration solution containing 3mg/l chloride anions was run to calibrate its retention time. From the peak size an estimate for chloride concentration was given.

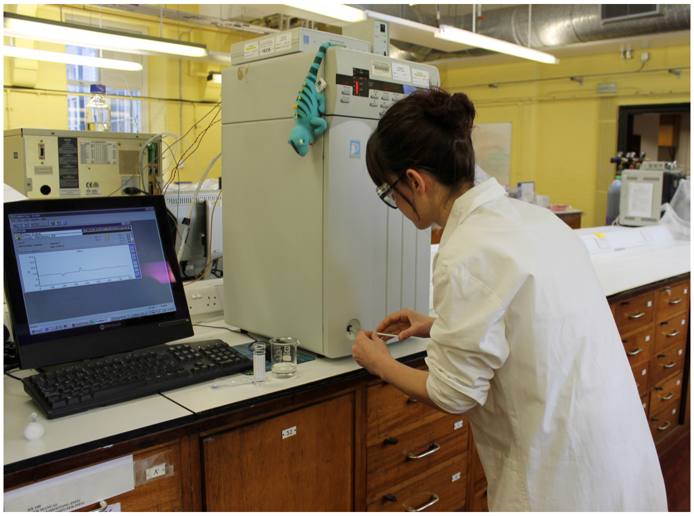

Figure 29 Sample injection

Table 5 Ion chromatography results

	Concentration (mg/l)			
Ion type	**Reference solution**	**Diluted sample ×50**	**Non-diluted sample**	**Original second washbath**
Nitrate	0.70	1.75	87.46	8.75
Sulphate	0.28	1.23	61.28	6.13
Chloride	Not quantified.	Approx. 3.5	Approx. 175	Approx. 17.5

Chloride anions were in the highest concentration, followed by nitrate and sulphate anions. Chlorides are ubiquitous in the air and their most likely source is sea-spray.[194] Sulphates and nitrates might be indicators of a combustion-related soiling source. Other sources of sulphates may be plaster soiling, [195] and sources of nitrates the decomposition of protein based materials such as silk.[196] The silk embroidery of the banner is, however, in good condition therefore silk deterioration is not expected to be a source of nitrates.

6.2.5.6 ATR-FTIR spectroscopy for molecular analysis

Attenuated total reflection infrared spectroscopy was undertaken with Dr Johannes Kiefer at the University of Aberdeen, School of Engineering, with a Bruker Vertex 70 FTIR Spectrometer equipped with a Platinum ATR single reflection diamond ATR accessory, operated with Opus software.[197] Spectra were recorded at 2cm^{-1} resolution between 4000cm^{-1} and 500cm^{-1} wavenumbers, the samples were scanned sixteen times. Dry samples were held in contact with the ATR crystal by the tightened clamp (Figure 30), while the crystal was covered with a few drops of the liquid samples (Figure 32). Dry samples were measured three times to avoid misinterpretation of spectral features arising from faulty contact between sample and diamond cell. Findings will be illustrated with the help of one-one selected spectra.

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[194] Museums and Galleries Commission, 14.

[195] Alebic-Juretic and Seculic-Cikovic, 53-54.

[196] S. Conti and M. Lorenzini, “The Veil of S Marina from the Church of S Maria Formosa in Venice,” in *Recovering the Past: the Conservation of Archaeological and Ethnographic Textiles, NATCC, 2005, Preprints,* eds. E. Cortes, and S. Thomassen-Kraus, 277-79, (Mexico City: NATCC, 2005), 277.

[197] Data interpretation with Dr Johannes Kiefer, Senior Lecturer at the University of Aberdeen and Dr Anita Quye, Lecturer in Conservation Science at the University of Glasgow.

Figure 30 Recording ATR-FTIR spectra of Smoke sponges

Spectra of soiled Smoke sponges were compared with the spectra of clean sponges (Figure 31). To aid the result interpretation, spectra of coal soot collected from a domestic chimney flue which might show similar absorbance pattern to a soot-containing soiling were also taken. To evaluate the contribution of linen fibre fragments on the soiled Smoke sponges to the spectral features, a plain weave linen fabric was sponged and the spectra of these sponges were recorded as a comparison.

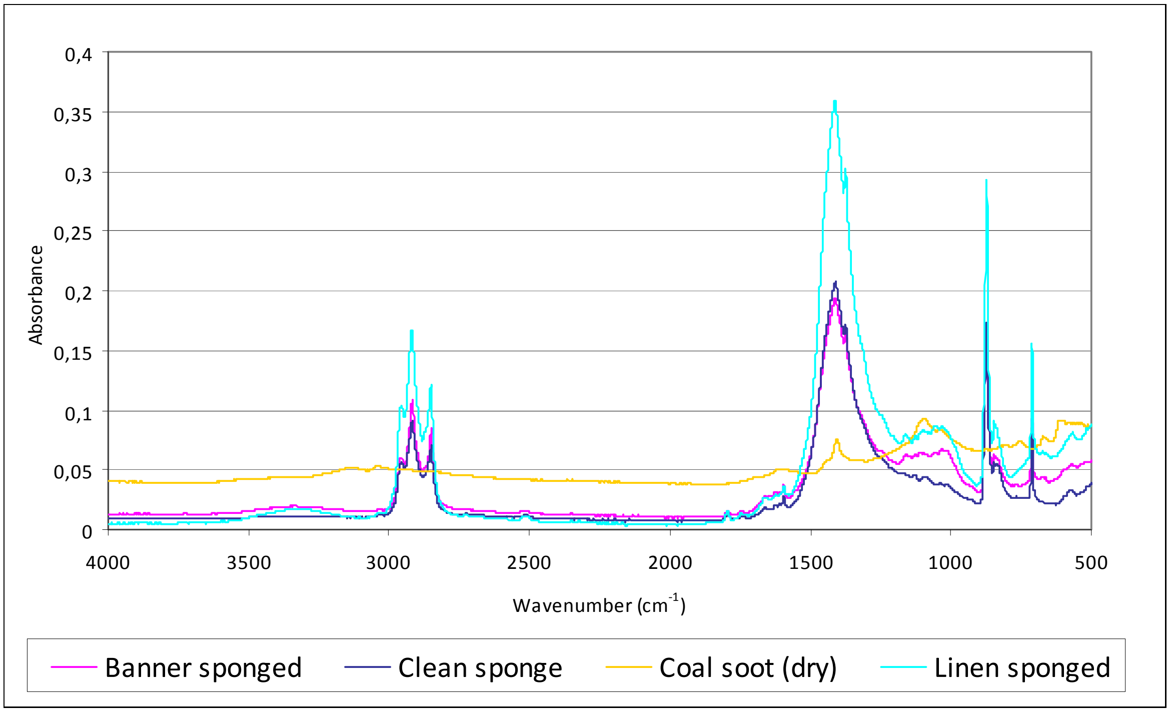

Figure 31 ATR-FTIR spectra of Smoke sponges

Peaks at 1800-, 1410-, 880- and 710cm^{-1} were attributed to the calcium carbonate filler of the Smoke sponge,[198] and peaks between 3050 cm^{-1} and 2800cm^{-1} to the *aliphatic* carbon-hydrogen stretching of the rubber polymer.[199] The only prevailing difference between soiled and clean sponges was around 1030cm^{-1}, most likely caused by linen fibre fragments and in particular by the carbon-oxygen ether stretching of cellulose.[200] Thus, analysis of the sponges did not shed light on the composition of the soiling.

Figure 32 Recording ATR-FTIR spectra of liquid samples

Spectra of the IDA washbath samples were compared to the spectrum of the IDA used for cleaning (Figure 33). The IDA contained ethanol with a few percents methanol and water. As the solvent dominated the absorbance spectra, difference spectra were created by subtracting the spectrum of IDA from the spectra of the washbath samples.

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[198] "Spectral Database: Calcium Carbonate," Infrared and Raman Users Group, http://www.irug.org/ed2k/spectra.asp?file=IMP00117.DX (accessed July 30, 2013).
[199] Stuart, 118.
[200] Stuart, 120.

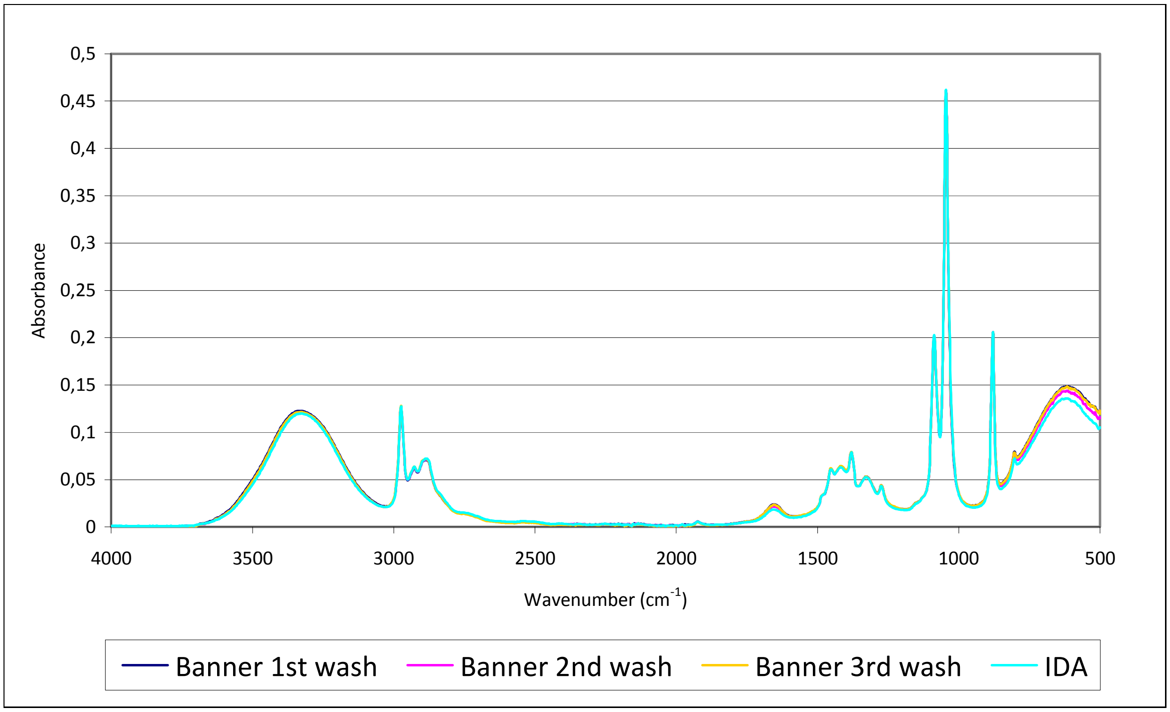

Figure 33 ATR-FTIR spectra of washbath samples

The band around 1030cm^{-1} which corresponds to the carbon-oxygen stretching of ethanol, showed a frequency shift and absorbance change (Figure 34). The shift towards higher wavenumbers could be interpreted as a sign of enhanced non-polar interactions of ethanol with its molecular environment;[201] the weakening of its hydrogen bonding network by hydrocarbon or carbon-based materials. These materials are likely to be present in a soiling which originates from combustion processes. The decreasing absorbance of the subsequent washing stages indicated that at each stage less and less quantity of these materials was removed from the textile. The spectral features between 3050 cm^{-1} and 2800cm^{-1} which shows the *aliphatic* carbon-hydrogen stretching of molecules supported these conclusions: decreasing absorbance of subsequent wash samples indicated that less and less hydrocarbon or carbon-based material was removed from the textile at each cleaning stage (Figure 35).

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[201] A. Ellis, F.M. Zehentbauer and J. Kiefer, "Probing the Balance of Attraction and Repulsion in Binary Mixtures of Dimethyl Sulfoxide and n-alcohols," *Physical Chemistry and Chemical Physics*, 15 (2013):1093-1096.

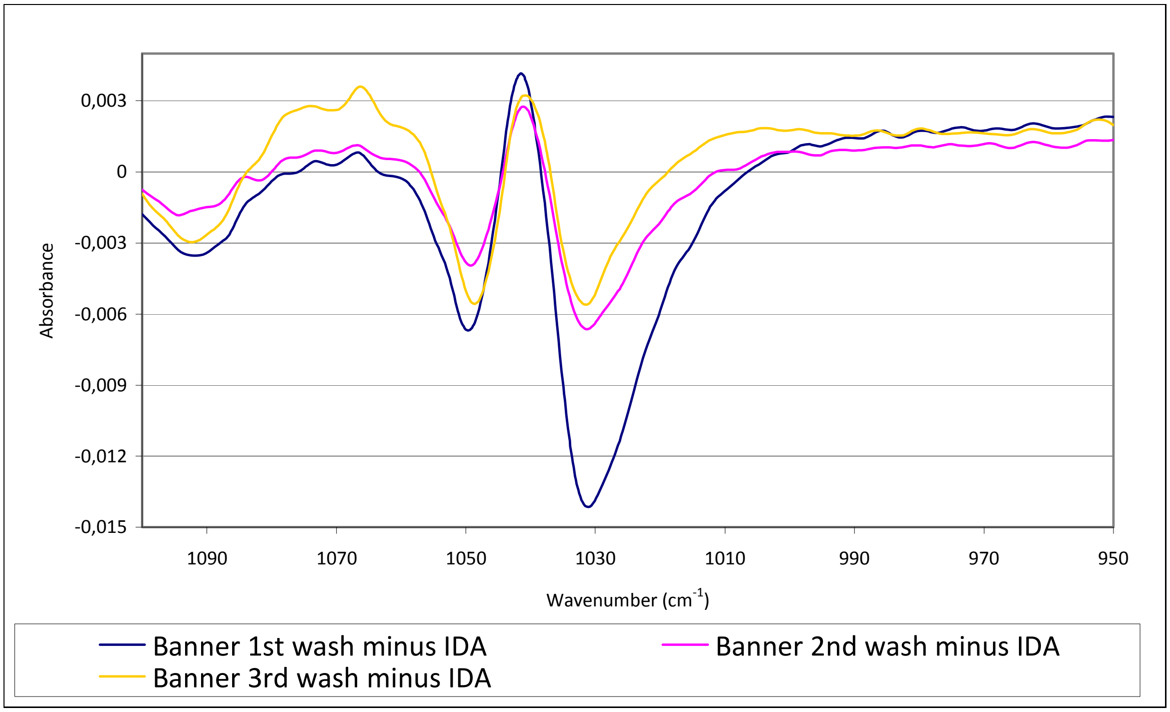

Figure 34 Difference ATR-FTIR spectra of washbath samples and IDA, 1100-950cm^{-1}

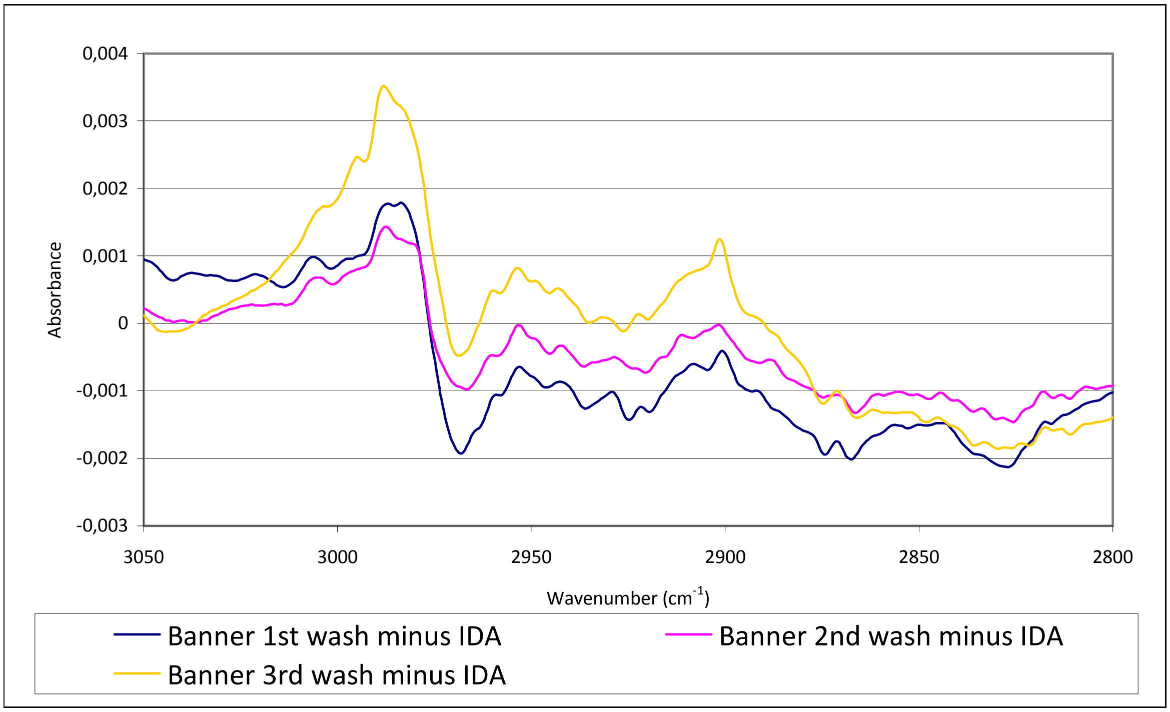

Figure 35 Difference ATR-FTIR spectra of washbath samples and IDA, 3050-2800cm^{-1}

6.2.5.7 Fluorescence spectroscopy for molecular analysis

Fluorescence measurements were taken at the University of Aberdeen, with a setup comprising a LED ultraviolet light source emitting at 360nm to 370nm; a lens system focusing the light on the sample; and a fibre-coupled Andor Shamrock spectrograph (600 lines/mm grating, 163mm focal length) equipped with an EM-CCD Andor Newton detector (Figure 36).[202] Data were imported with the Andor software. The samples were irradiated with the incident light in a cylindrical glass sample container.

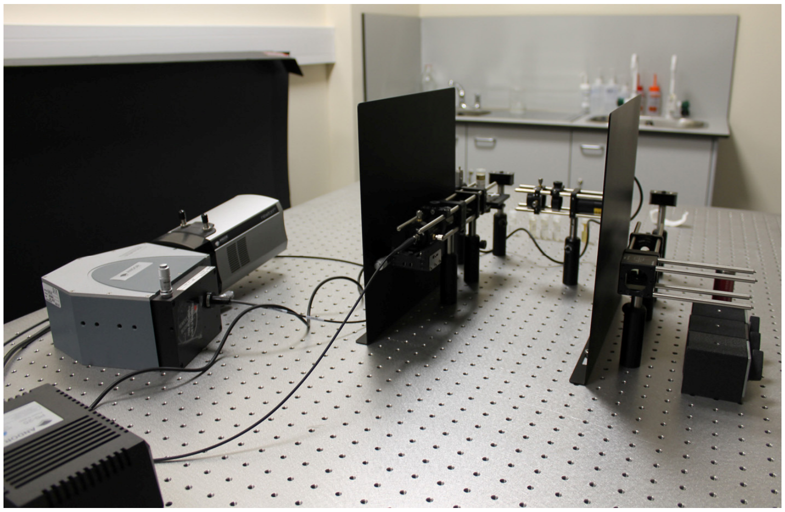

Figure 36 Fluorescence spectroscopy setup

Spectra of the IDA cleaning samples were compared with the spectrum of IDA, and the IDA extract of coal soot collected from a chimney flue (Figure 37). (For the extraction procedure, see Appendix 6).

All washbath sample spectra showed the presence of fluorescent aromatic hydrocarbon or carbon-based materials. It was not possible to identify specific aromatic molecules from the spectra. The signal was strongly decreasing at the subsequent wash stages, indicating that less and less of these materials was removed. The bandwidth of the signals was also decreasing, meaning that the variety of aromatic hydrocarbons removed from the textile decreased with the subsequent washes.[203] The bandwidths of the washbath

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[202] Data interpretation with Dr Johannes Kiefer.

[203] Bandwidth is observed at half maximum of the intensity (Stuart, 164).

samples were similar to the bandwidth of the coal soot extract, indicating that the hydrocarbons in both cases may be similar in structure.

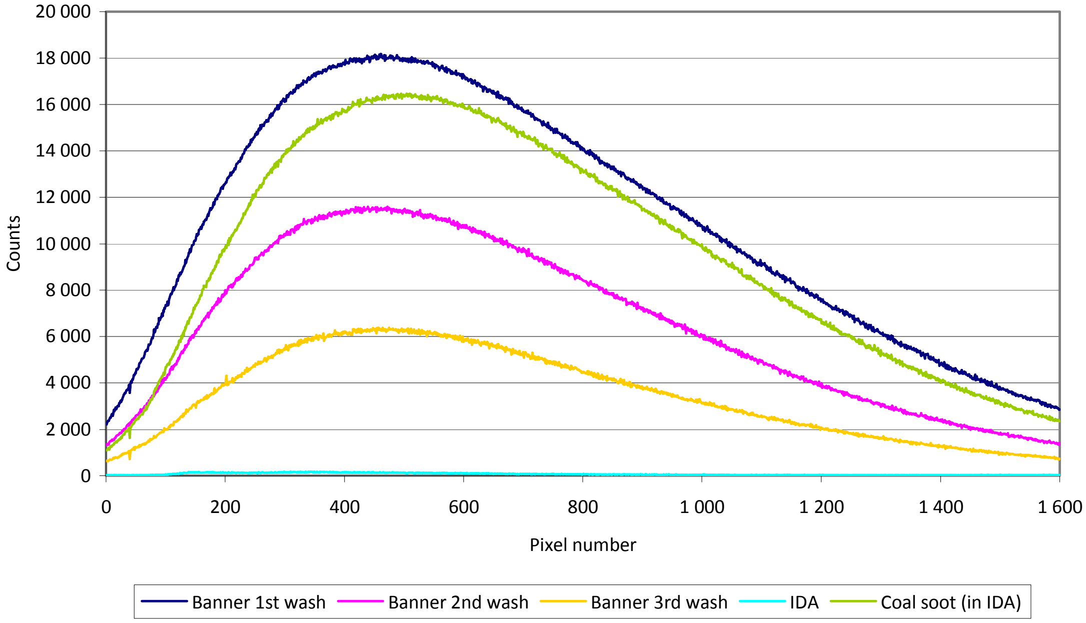

Figure 37 Fluorescence spectra of washbath samples

6.2.6 Evaluation of the characterisation results

Historical investigation revealed that the textile had probably been handed from one custodian to the other, exposed to an ever-changing environment. During macroscopic examination the soiling pattern indicated an airborne origin, while the presence of a greasy component, strong smell, and ease of removal by Smoke sponges showed similarities with textiles affected by rapid soot deposition. Stereomicrosopic examination revealed particulate black soiling, dark coating on fibres and salt crystals, indicative of combustion-related deposition but in lack of reference micrographs comparative evaluation was not possible. SEM-EDS of the thread sample did not show characteristic soot morphology but trace elements in the soiling could be indicative of fossil fuel burning. Analysis of the immersion cleaning solutions was revealing: PLM clarified that the black particulates in the washbath were *amorphous* structured, similarly to combustion particles. Ion chromatography ascertained the presence of sulphates and nitrates, which might be indicators of fuel burning (besides being indicative of the acid potential of the soiling). Spectroscopic analysis gave insight into the hydrocarbon content of the soiling and to the removal-dynamics. On the basis of the analytical results, it is likely that the soiling on the banner originated from combustion processes.

6.3 Case Study 2: Muslin curtain from the Karen Finch Reference Collection, CTCTAH

6.3.1 Object description

The muslin curtain was made of a white lightweight plain weave cotton fabric decorated with a repeated ribbon and polka dot pattern, created by a thicker white cotton supplementary weft (Figure 38-39). The textile was sewn together from three pieces of the same fabric: a large rectangular central piece and two stripes of decorative trimming attached to the central piece along its longer edges. It is likely that early machine stitching was used for attaching the three pieces, for gathering the trimming and finishing the longer edges of the fabrics, while the shorter edges were finished by hand-stitching. The textile was starched. (For details on materials and construction, see Appendix 3.)

Figure 38 Front of the muslin curtain (©CTCTAH) [204]

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[204] Due to its large size the textile was photographed in three parts and the photographs were joined in Adobe Photoshop CS3®.

Figure 39 Reverse of the muslin curtain (©CTCTAH)

6.3.2 Historical background

The textiles in the Karen Finch Reference Collection were collected as donations and in most cases little information had been recorded about their provenance. The short description on the object label read: ‘Curtain for the Central Horizontal Rod of a Bassinet’. The following technical research aimed at establishing the date when the textile was made and used in order to have a better understanding about the object’s past environments.

6.3.2.1 Patterned muslins

“Muslin is an open-textured cloth, thin and sheer, woven to varying degrees of fineness depending on the quality of yarn used and the skills of both the spinner and the weaver. The surface, particularly of hand-woven muslin, has a softness to the touch that has been described as ‘mossiness’.”[205]

Muslin production originated from India, where it was manufactured from extremely fine yarns spun from short-staple cotton fibres, accounting for the softness of the textile. It began to be imported to Britain around 1670, and by the end of the eighteenth century Bolton, Glasgow and Paisley became important production centres within Europe.[206]

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[205] S. Ashmore, *Muslin* (London: V&A Publishing, 2012), 8.
[206] Ashmore, 34-35.

The curator of textiles in Paisley Museum and Art Galleries was consulted for advice on the weave structure. The textile was identified as a typical example of figured muslin fabric of the late eighteenth and early nineteenth centuries.[207] This type of figuring called 'spotting' was achieved by the insertion of heavier supplementary 'spotting' wefts in the figured area and clipping the floating wefts on the reverse.[208] The probable use of early machine stitching in the construction supported the early nineteenth-century origin.

6.3.2.3 Function and style

Two parallel lines of stitching holes preserved in the central area (Figure 42) showed that the textile could have once been folded in two and hung from a rod over the bassinet. Experts in the Museum of Childhood, London were consulted about bassinet styles. A photograph showing the bassinet of Prince Charles is a twentieth-century example of an earlier, nineteenth-century style (Figure 40).[209] It is likely that the muslin curtain was once used in a similar manner.

Figure 40 Prince Charles's bassinet (©Museum of Childhood)

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[207] E-mail communication with Dan Coughlan, Curator of Textiles, Paisley Museum and Art Gallery on June 11, 2013.

[208] J. Murphy, *A Treatise on the Art of Weaving, Illustrated by Engravings*, Second Edition (Glasgow: Blackie, Fullarton & Co., 1827), 196-251.

[209] E-mail communication with Catherine Howell, Collections Manager, Museum of Childhood on June 13, 2013.

6.3.3 Examination

The condition of the textile with special regard to the soiling characteristics was assessed as described for the William Morris Action Song Challenge Banner in *section 6.2.3*.

6.3.3.1 Visual examination

The muslin curtain was in a structurally sound condition. Grey soiling near the foldlines, spots of yellow-brown staining and discolouration throughout the textile, and particulate soiling on the reverse were the dominant condition features. Sharp creasing and foldlines had been created by folded storage. Structural damages in the weave and the stitching were confined to small areas.

The distribution of the grey soiling suggested that it occurred at a time when the textile was folded up (Figure 41). Three evidences indicated airborne origin: firstly, folds were protruding areas physically most exposed to an airborne pollutant; secondly, the soiling was evenly distributed along the folds and soiled areas had an undefined edge; and thirdly, the symmetry in the heaviness of the soiling suggested that the outer layers acted as a ‘filter’ towards the inner layers. Protection from atmospheric pollutants in the CTCAH is well controlled, so it is probable that the soiling occurred before the textile’s accession.

Yellow-brown discolouration on cellulosic artefacts is often referred to as iron stain or foxing.[210] Staining made the fibres thinner and brittle, however, the soiling distribution patterns showed that most of the small holes were caused by handling and not by soiling (Figure 42). Inactive moth balls, large fibre fragments and a small leaf fragment on the reverse indicated that the textile had been stored in an uncontrolled environment.

6.3.3.2 Tactile examination

The grey soiling did not change the flexibility of the fibres, nor did it have a greasy touch or a differing smell from the rest of the textile. It is possible that the textile has been laundered following the soiling deposition, which might have changed the soiling attributes.

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[210] J.M. Carter, “Iron Stains on Textiles: A Study to Determine Their Nature and to Evaluate Current Treatments,” in *ICOM CC 7th Triennial Meeting*, *1984, Preprints*, 84.9.11-84.9.14 (Paris: ICOM, 1984).

Figure 41 Grey soiling and foldlines on the front

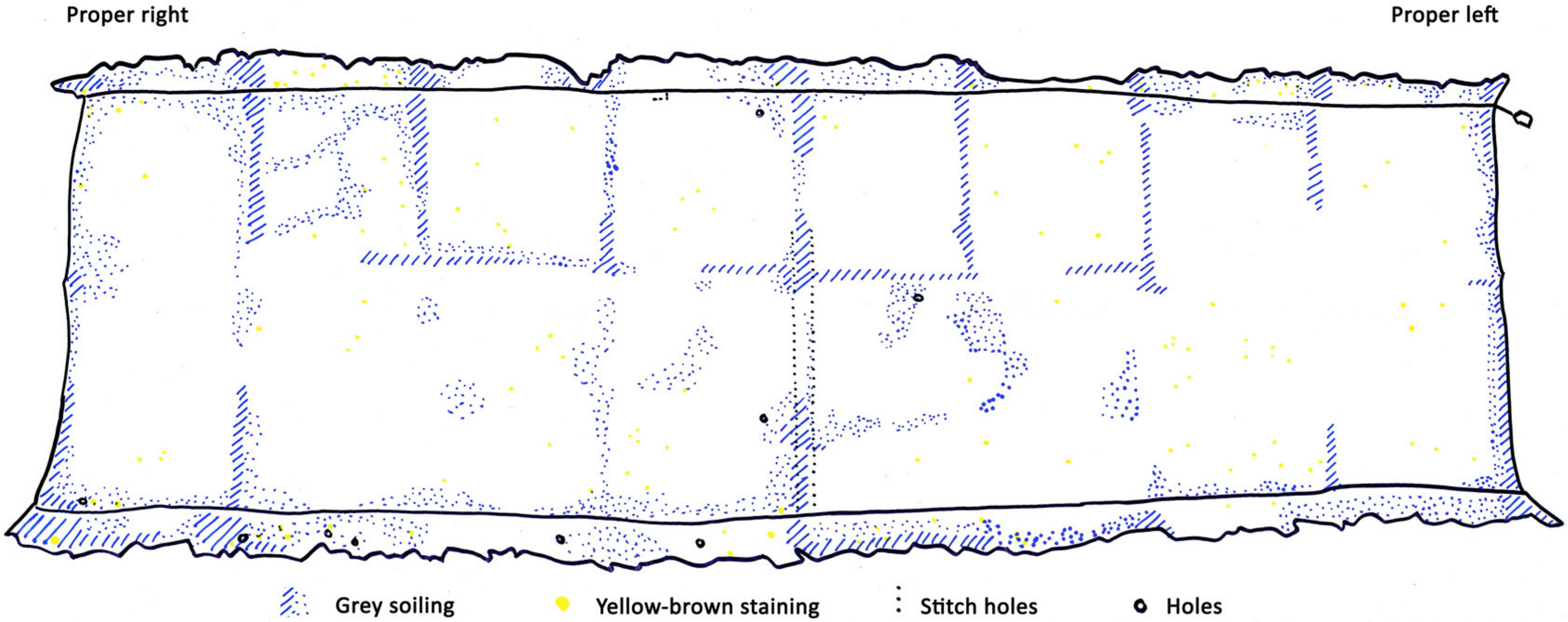


Figure 42 Grey soiling, staining and structural damage on the front

Due to the large size of the textile, four 40mm×40mm areas (A-D) were selected for further study (Figure 43). The areas were chosen for their differing intensity of grey soiling and position within the textile. Colour of the four areas was recorded for future reference as described in *section 6.2.3* (for measurements see Appendix 3).

Figure 43 Areas for further study

6.3.4 Testing

Surface pH was measured with a Fisherbrand FB68797 Flat Surface pH probe connected to a HANNA instruments, HI 9024 Microcomputer pH meter. The instrument was calibrated with neutral and acidic solutions (Fischer Scientific). A neutral pH blotting paper was placed underneath the area, the textile was wetted with a drop of deionised water and the probe was held onto the textile (Figure 44). The pH of the deionised water was measured as 5.02.

The values on the screen were changing rapidly, dropping from 5.02 presumably with the solubilisation of acidic substances in the textile, and further decreased with the drying of the probe. When a certain value stabilised for a few seconds, the reading was taken (Table 6). Rapid changes in pH value and the resulting high standard deviation were most likely caused by the openness of the weave.

Figure 44 Surface pH measurement

Table 6 Surface pH measurements

	1st	**2nd**	**3rd**	***Max***	***Min***	***Mean***	***SD***
Deionised water	5.02						
Area A	3.70	3.90	4.41	4.41	3.70	4.00	0.30
Area B	4.28	4.19	4.57	4.57	4.19	4.35	0.16
Area C	4.01	4.59	4.46	4.59	4.01	4.35	0.25
Area D	3.85	4.21	4.46	4.46	3.85	4.17	0.26

The solubility of the soiling was tested with a non-ionic detergent solution and IDA (for details see Appendix 3). The detergent was slightly more effective in the removal of the soiling, indicating that the soiling responded to emulsification.

6.3.5 Scientific investigation

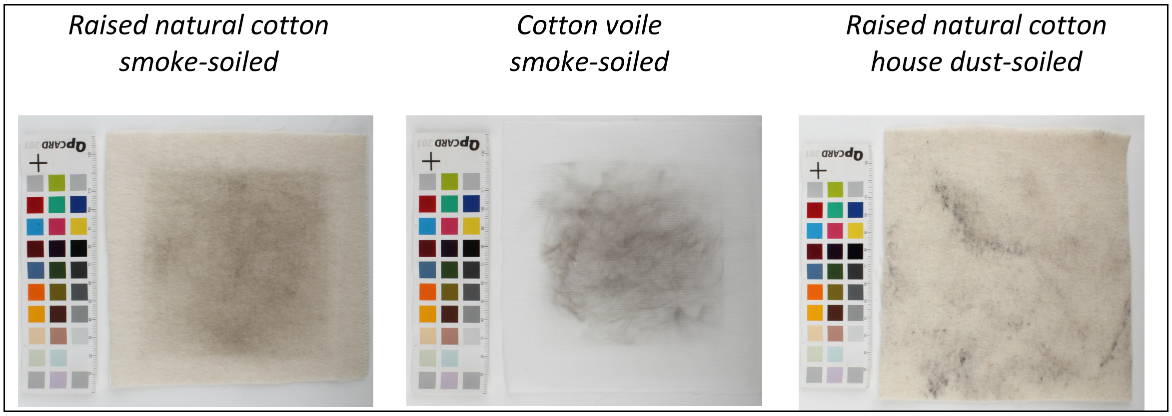
The following section describes the analytical methods used to examine the textile (Table 7).

Table 7 Summary of samples for scientific investigation of the muslin curtain

Subject (sample)	**Technique**
Textile in area A, B, C, D	Digital microscopy Stereomicroscopy
Textile in area A, D	IR Photography
Soiled thread samples (5mm) removed from area A, C, D	PLM
Soiled thread samples (5mm) removed from area A, C	SEM-EDS

6.3.5.1 Creating soiled fabrics as reference for microscopic examinations

Initial stereomicroscopic examination of the textile showed the presence of black particles adhered to the fibre surfaces. It was felt that more information could be gained from the microscopic examinations if reference soiled fabrics were available for comparison (Figure 45). Soiled fabrics were created by exposing cotton fabrics to candle smoke and collecting sooty particulates on the fibre surfaces (for procedure see Appendix 7). Raised natural cotton and white cotton voile were chosen, the former due to its loose fibres capable to easily catch soiling; the latter due to its similarity in open weave to the muslin curtain. It was acknowledged that the soiling would not be representative to all types of sooty soiling. A third reference fabric was created by collecting house dust on the raised natural cotton, to see if particles in modern dust attach to cotton fibres in a similar manner to soiling from smoke.



Raised natural cotton smoke-soiled

Cotton voile smoke-soiled

Raised natural cotton house dust-soiled

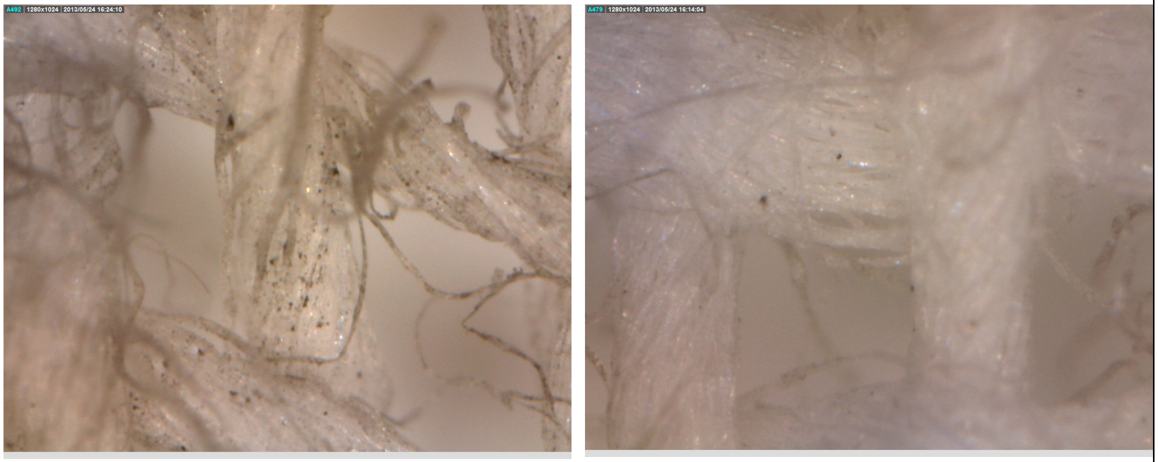
Figure 45 Reference soiled fabrics

6.3.5.2 Digital microscopy and stereomicroscopy

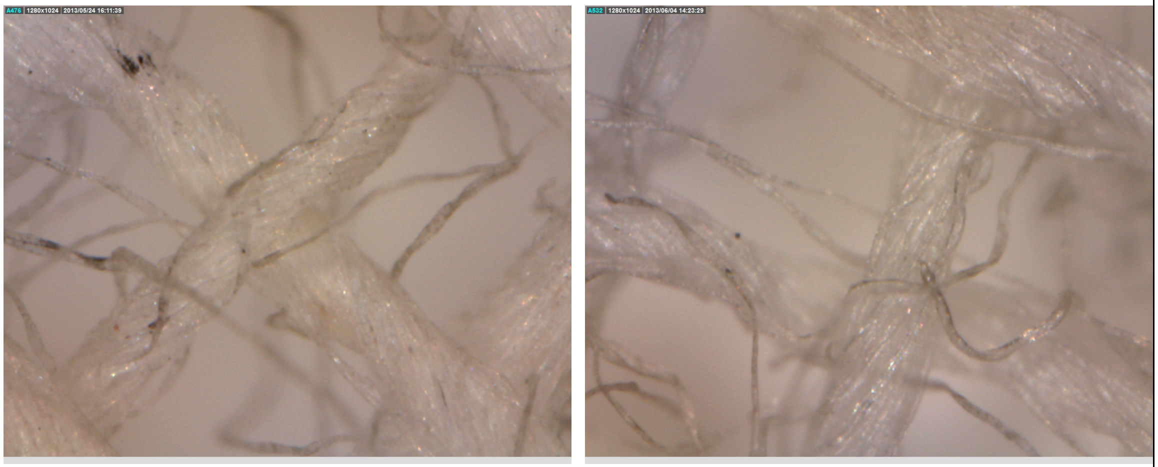
Portable Dino-Lite Premier Digital Microscope AM-3013T (×500 magnification) was utilised for the examination of the grey soiling and recording of images at the CTCTAH (Figure 46). Digital microscopes are easy to use and they can provide a higher magnification than stereomicroscopes, but just a slight movement of the microscope head causes small particles on the fibre surface to move out of focus.

To complement the imaging, the soiling was also examined with a stereomicroscope at the CTCTAH, as described for the William Morris Action Song Challenge Banner in *section 6.2.5.1*. The advantage of the stereomicroscope was the ease of focusing on the three-dimensional surface of the cotton fibres. Observations were recorded in writing as no camera attachment was available for use (for detailed observations see Appendix 3).

Area A *Area B*



Area C *Area D*



Raised natural cotton smoke-soiled *Cotton voile smoke-soiled*

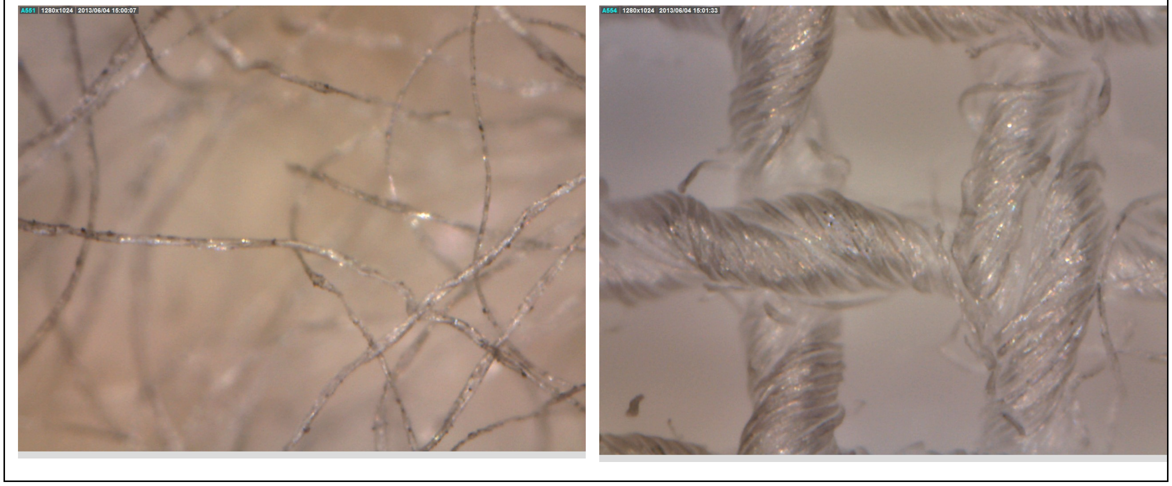


Figure 46 Grey soiling on the muslin curtain and reference soiled fabrics, Dino-Lite, ×500


Raised natural cotton house dust-soiled

Figure 46 Continued: Grey soiling on the muslin curtain and reference soiled fabrics, Dino-Lite, ×500

The appearance of heavy soiling on the muslin curtain in area A and C was created mainly by the dark coating on loose fibres emerging from the threads of the ground weave. Under stereomagnification this coating was seen to be composed of small black particles. Aggregates of black particles smaller than the cotton fibre diameter (approximately 15-22µm)[211] were seen in all examined areas and were most abundant in area A, B and C. These aggregates were seen to be in the process of further fragmentation. Black particles larger than the fibre diameter were loosely adhered to the threads and had various shapes, sizes, surface texture and reflectivity.

While the house dust-soiled reference fabric showed the presence of loosely adhered black particles slightly smaller and larger than the fibre diameter, together with coloured fibre fragments and white crystals, its fibres did not have a dark coating. The smoke-soiled raised natural cotton collected black particles smaller than the fibre diameter and a dark coating which was seen to be composed of small black particles, while the cotton voile threads were evenly covered with the grey particulate substance. Soiling on the raised natural cotton showed most similarity with the soiling on the muslin curtain.

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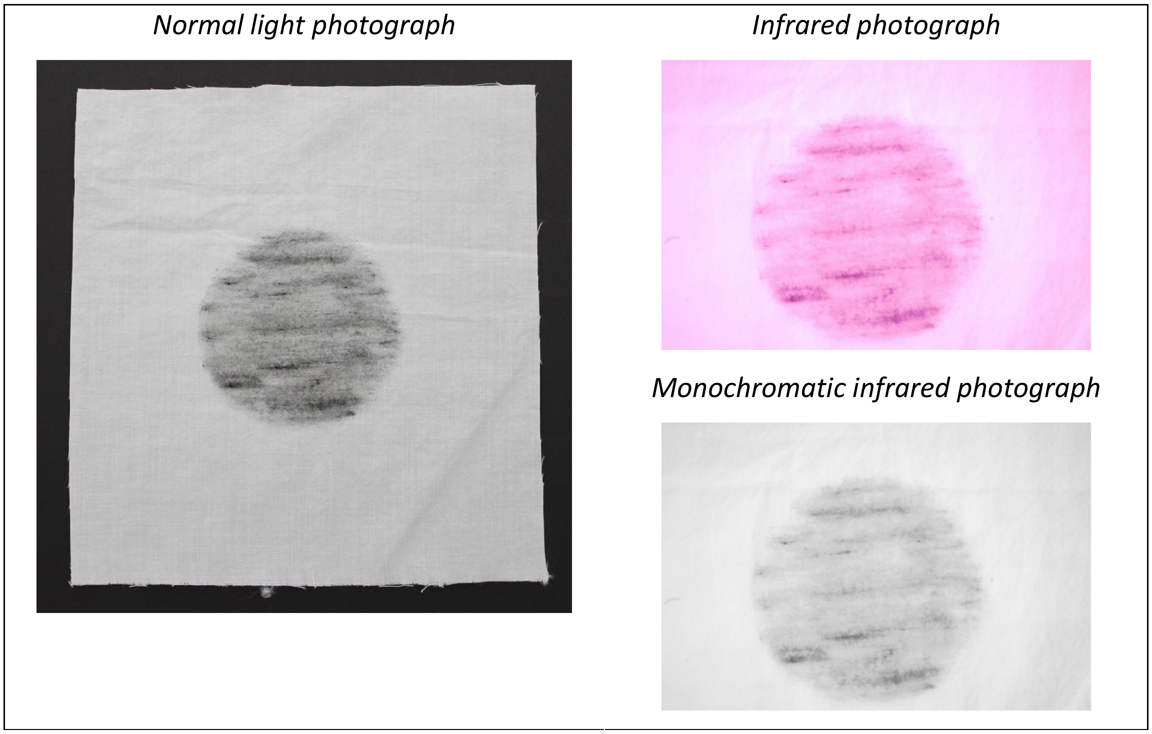
[211] Sawbridge and Ford, 13.

6.3.5.3 Infrared photography: detecting black carbon

Infrared photography was undertaken at the CTCTAH to try its application for the detection of black carbon in the grey soiling of the muslin curtain. A Nikon D60 SLR camera adapted to photograph in the infrared region was fitted with Nikon DX AF-S Nikkor 18-55mm zoom lens. Hoya 52mm Infrared [RM90] filter which passes only infrared rays above 900nm, and [RM72] filter which passes rays above 720nm were utilised. The light source was natural daylight which has a high proportion of infrared radiation and is therefore often used for infrared photography.[212]

Trials were carried out on two artificially soiled cotton fabrics specially prepared for the experiment: candle soot was applied to white cotton lawn fabrics at a darker and a lighter shade (for procedure see Appendix 7). On the infrared photographs soot was well distinguishable from the surrounding clean fabric on both textiles (Figure 47-48). Even small differences in the thickness of soot application showed up on the infrared images.



Normal light photograph

Infrared photograph

Monochromatic infrared photograph

Figure 47 Infrared photography of candle soot-soiled cotton, darker shade

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[212] A. Davidhazy, "Infrared Photography," http://people.rit.edu/andpph/text-infrared-basics.html (accessed June 2, 2013).

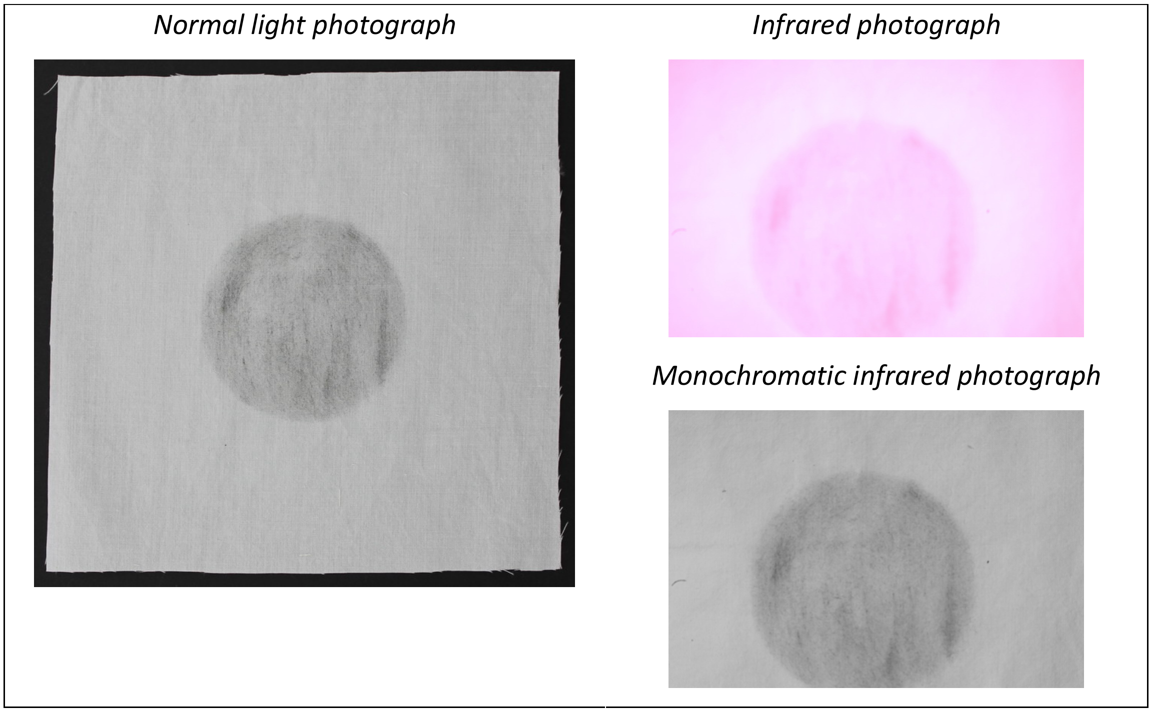

Figure 48 Infrared photography of candle soot-soiled cotton, lighter shade

The muslin curtain was positioned so that shadows caused by creasing would not be emphasised over the soiling features (Figure 50). Two areas (Figure 49) were photographed with the Hoya [RM72] filter.

Figure 49 Selected areas for infrared photography

Figure 50 Infrared photography in progress

Only small shade differences were visible between the cleaner and soiled areas, when compared to the normal light images taken in the same position. After changing the exposure and contrast on the infrared images in Adobe Photoshop CS3®, shade differences in the areas of grey soiling became more pronounced (Figure 51-52). In comparison, the yellow-brown staining did not appear on the infrared photographs, showing that the stain was not absorbent of infrared light.

Due to the small shade differences and the influence of creases in the textile, results were considered inconclusive. Unfortunately, infrared photography could not be further tested within the framework of the project due to equipment availability.

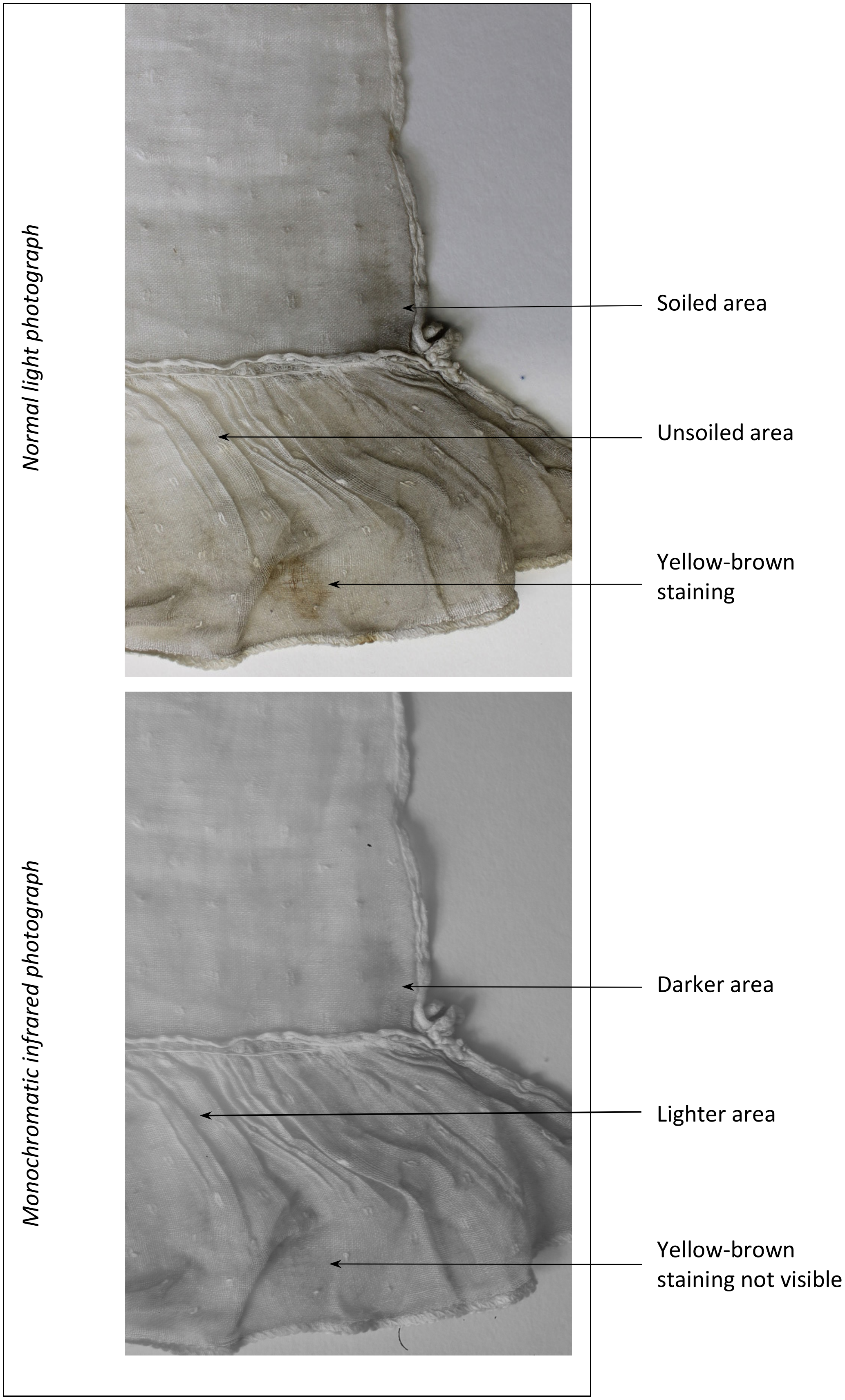

Figure 51 Infrared photography of the muslin curtain 1

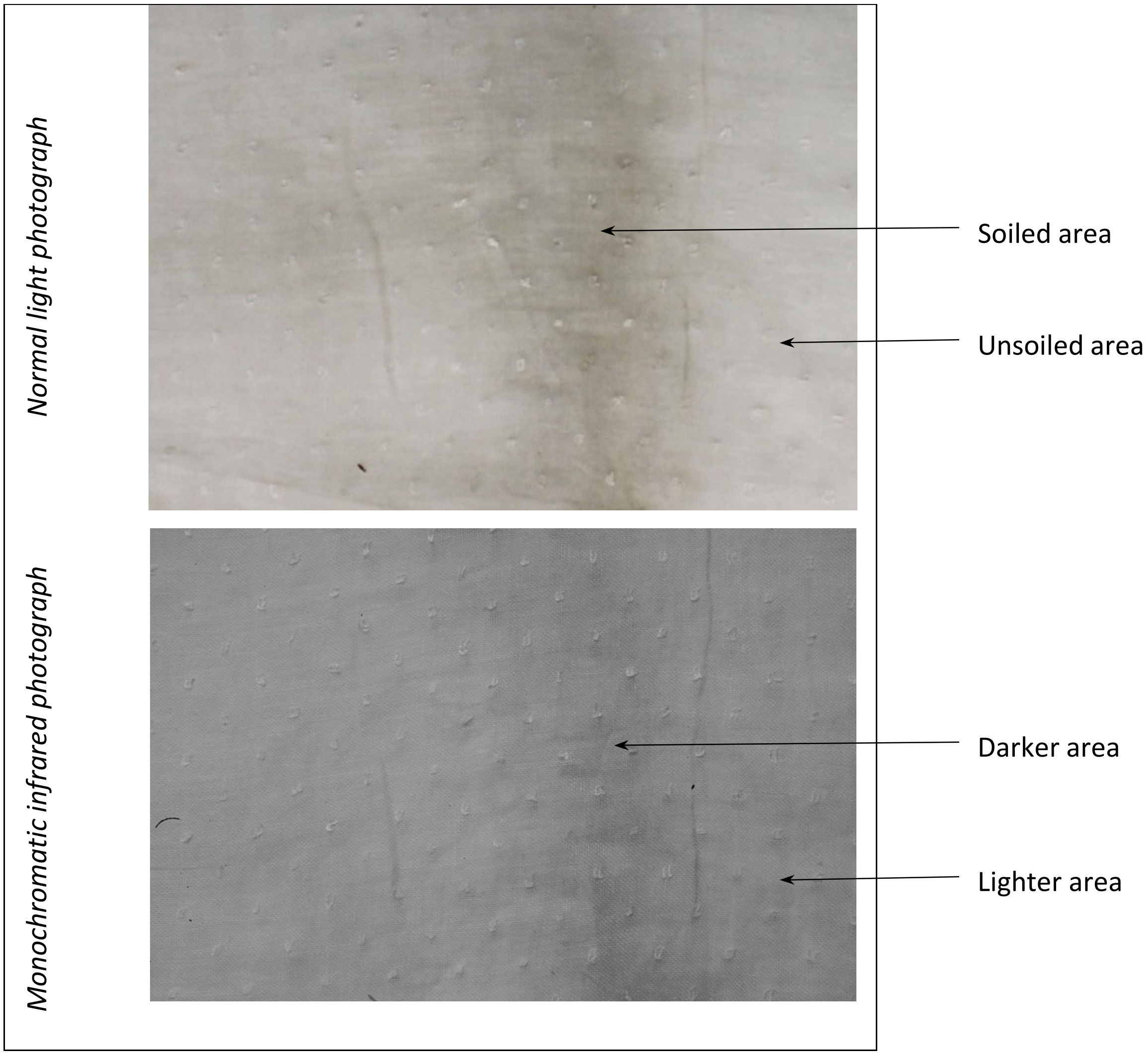

Figure 52 Infrared photography of the muslin curtain 2

6.3.5.4 Polarised light microscopy: the morphology of soiling on the fibres

During *in situ* examination the dark soiling was seen to be closely adhered to the fibres. Fibre samples from area A, C, and D and from a clean but damaged area on the trimming were removed in order to undertake a polarised light microscopy examination similarly to Moffatt, Wainwright and Corbeil.[213] Sampling in area A, C and D was done with the least disturbance of the weave by cutting the weft thread in between the same warps in 5mm distance from each other and removing the sample with tweezers. Invasive sampling was only considered as the muslin curtain was designated for study purposes. The samples were macerated and mounted with Eukitt® permanent mounting medium. For comparison, fibre samples of smoke-soiled raised natural cotton – on which the soiling was most similar in appearance, see *section 6.3.5.2* – and clean cotton were prepared in the same way. Zeiss Axiolab polarising transmitted light microscope was used for the examination at ×400 magnification at the CTCTAH. Particle sizes were measured with the ocular scale. The results of the examination are summarised in Table 8.

Table 8 Polarised light microscopy of thread samples

Studied area on the muslin curtain / Reference textiles	**Description**
Area A	Fibres unevenly covered with black particles smaller than 2.5µm. Numerous 2.5-5µm black particles of various shapes trapped between the fibres.
Area C	Same appearance as in area A.
Area D	Fibres had a particularly heavy, uneven coverage of black particles smaller than 2.5µm. Numerous 2.5-5µm black particles of various shapes trapped between the fibres.
Clean fibre from trimming	Few black particles smaller than 2.5µm and few 2.5-5µm black particles of various shapes visible.
Raised natural cotton smoke-soiled	Fibres evenly covered with black particles smaller than 2.5µm.
Raised natural cotton clean	No particles visible.

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[213] Moffatt, Wainwright and Corbeil, 2000.

6.3.5.5 SEM-EDS analysis: morphology and elemental composition of the soiling

For SEM-EDS analysis a heavily soiled spotting weft was sampled from area A referred to as ‘Thread sample 1’, and a ground weft from area C referred to as ‘Thread sample 2’. Smoke-soiled and clean areas of the raised natural cotton were cut off for comparative purposes (Figure 53). The analysis was undertaken as described for the William Morris Action Song Challenge Banner in *section 6.2.5.2*.

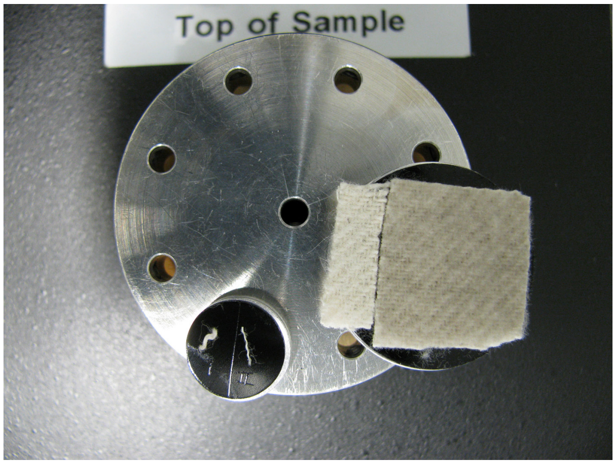

Figure 53 Thread and fabric samples on the sample wheel of the SEM

The morphology of the soilings was compared at ×1250 magnification (Figure 54-57) and above ×30 000 magnification (Figure 58-60). Soiling on the artificially smoked cotton was composed of particle aggregates forming branched structures, of which the elementary particles were smaller than 0.1µm in diameter. Images of ‘Thread sample 1’ and ‘2’ showed angular and spherical particles evenly distributed on the fibre surface, their dimensions varied from below 0.1µm to 1µm, indicating airborne or re-deposited origin.

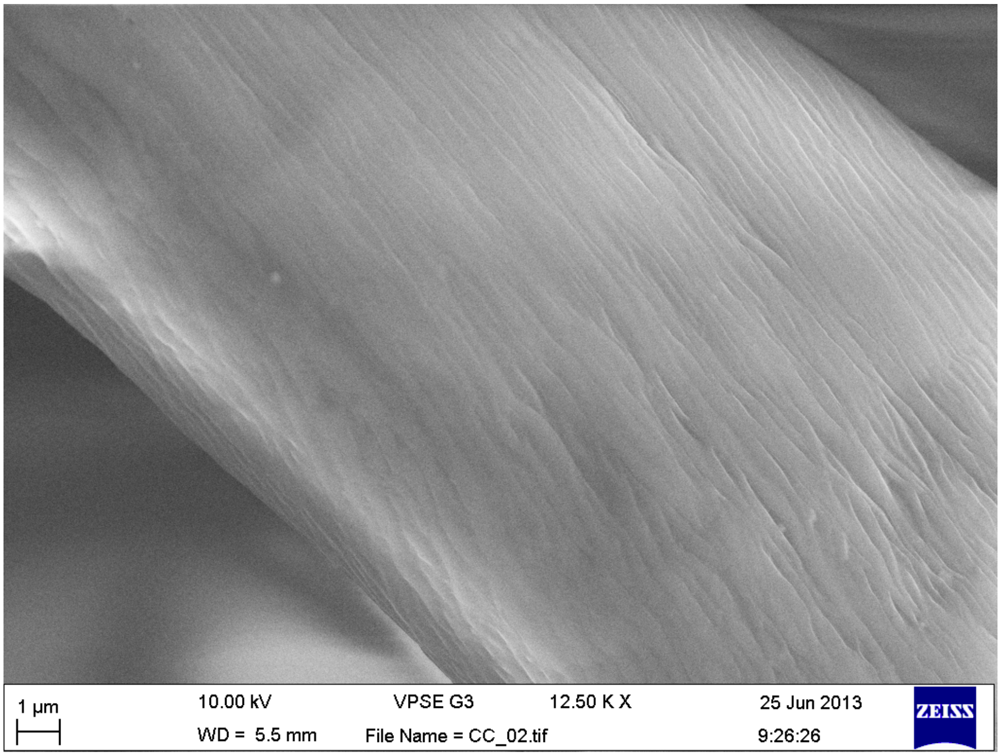

Figure 54 Raised natural cotton clean, SEM ×1250

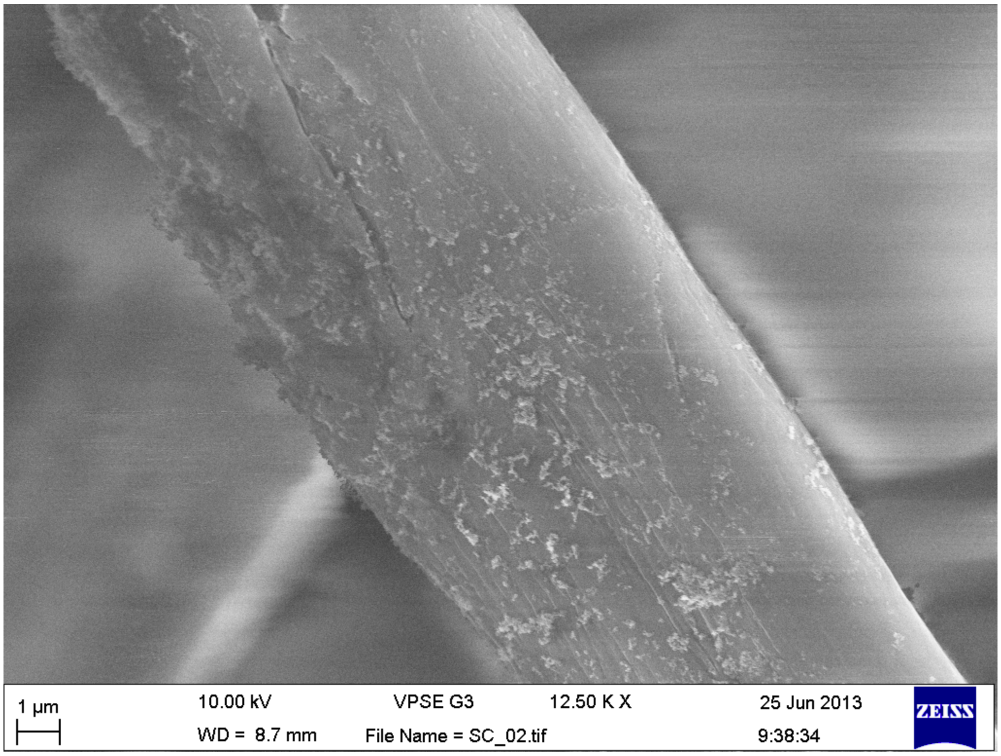

Figure 55 Raised natural cotton smoke-soiled, SEM ×1250

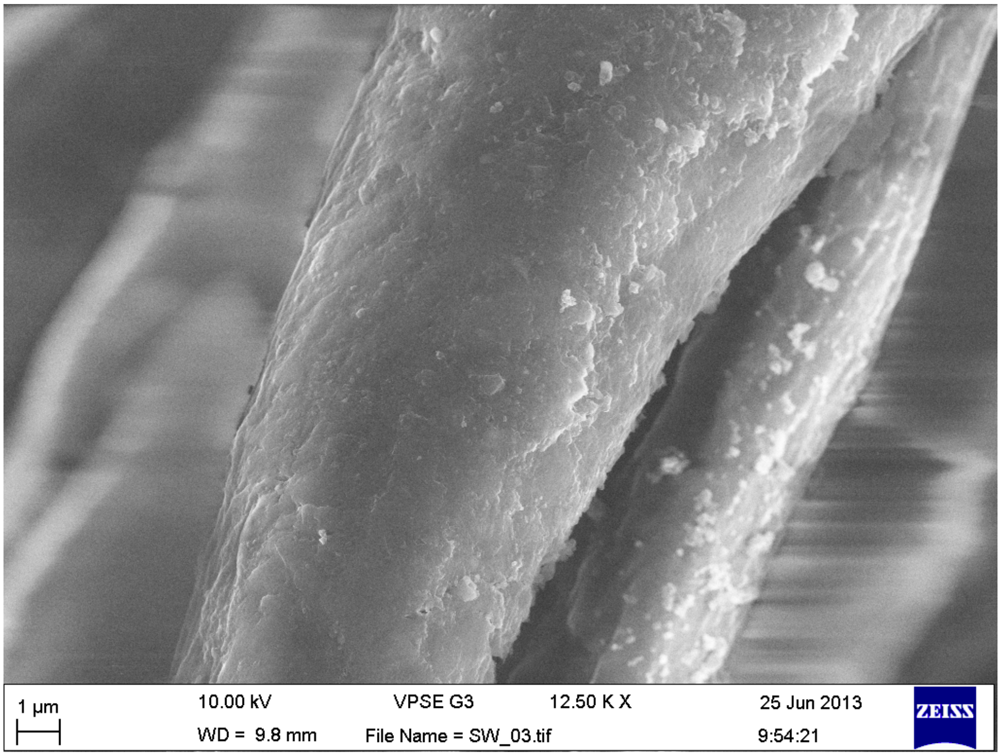

Figure 56 Thread sample 1, SEM ×1250

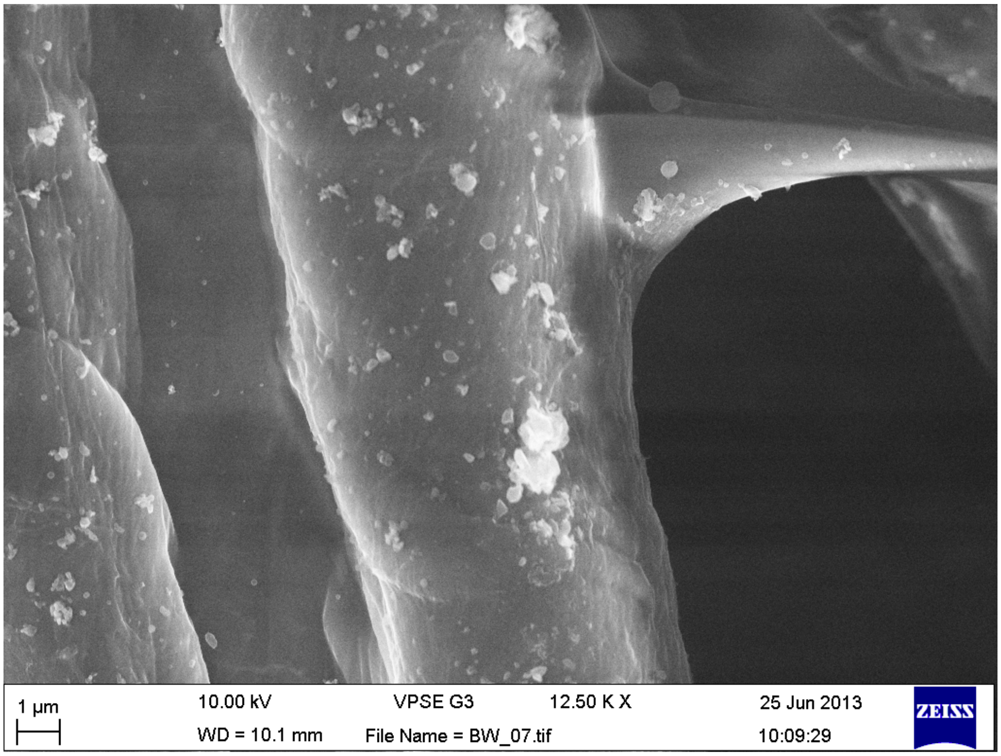

Figure 57 Thread sample 2, SEM ×1250

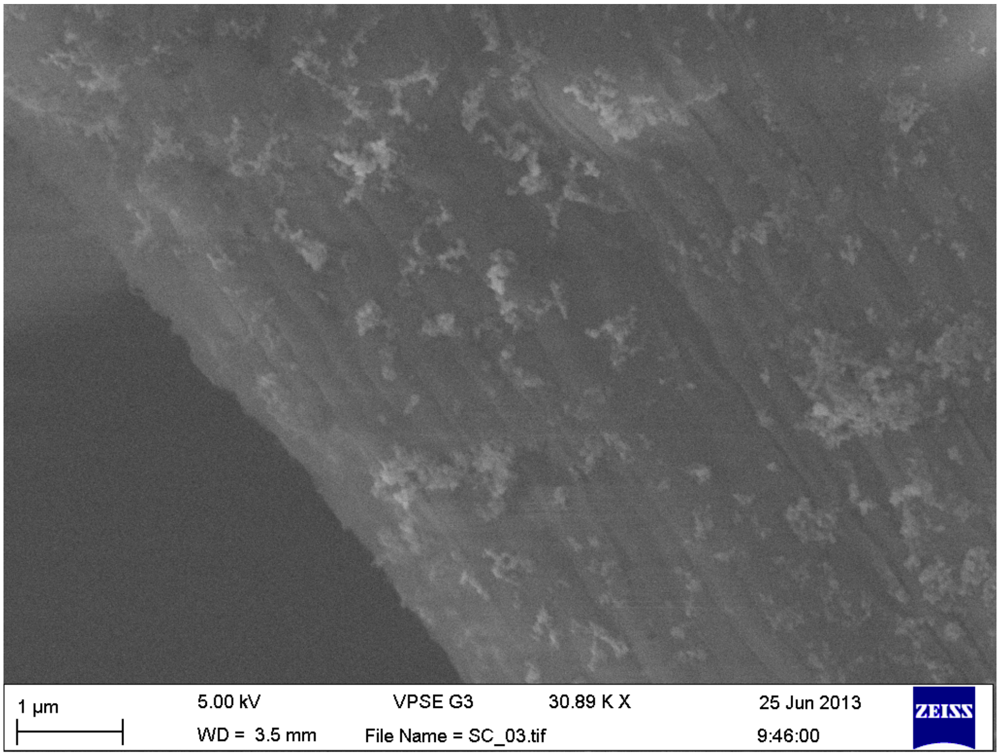

Figure 58 Raised natural cotton smoke-soiled, SEM ×30890

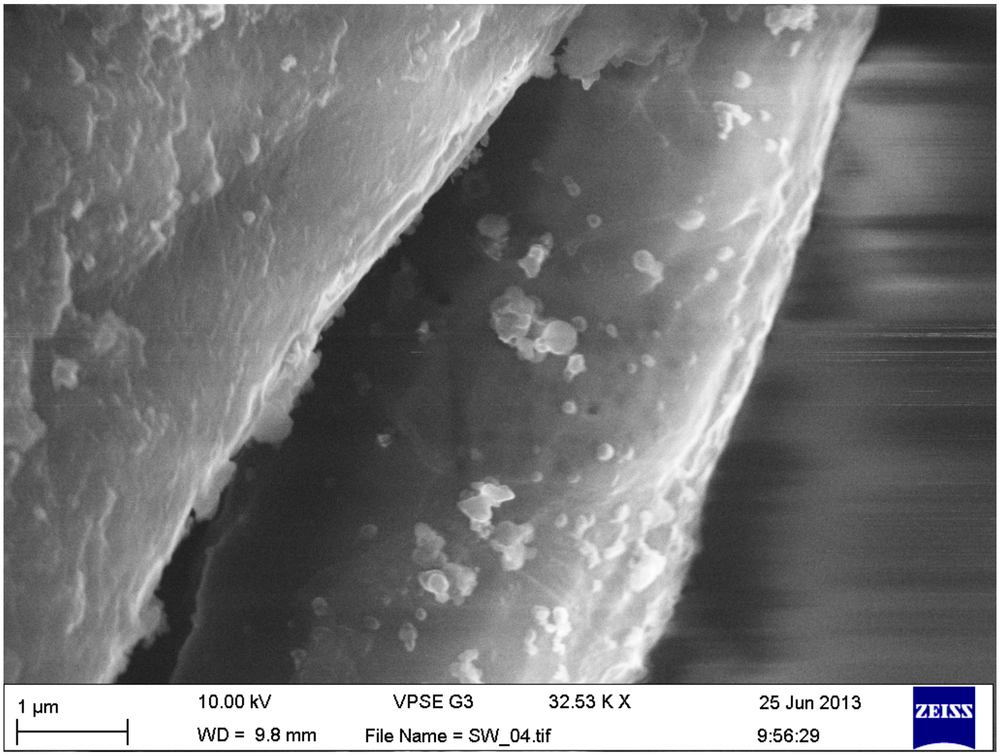

Figure 59 Thread sample 1, SEM ×32530

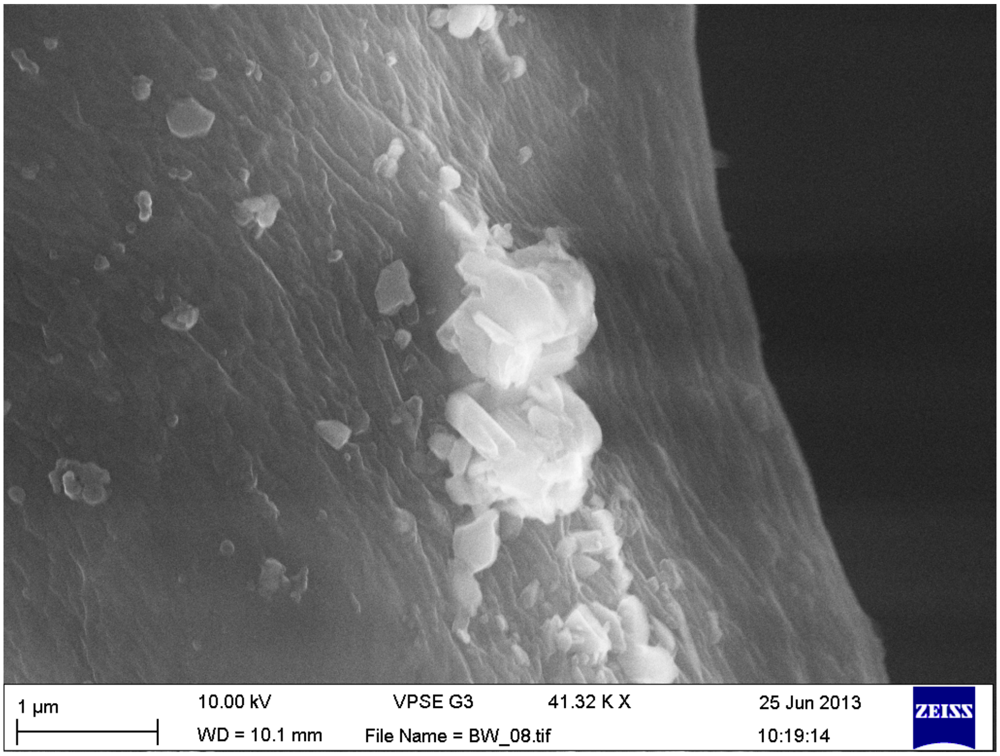

Figure 60 Thread sample 2, SEM ×41320

The artificially smoke-soiled cotton fibres were covered with particles containing carbon and oxygen (Figure 61-62). Carbon and oxygen were the only elements identified on the thread samples taken from the muslin curtain in unsoiled areas (Figure 63) and areas where soiling consisted of small spherical particles (Figure 64). A larger globular particle was likely to have a biological origin (Figure 65).[214] The angular crystals contained calcium and sulphur (Figure 66), so were identified as calcium sulphate. Their origin may be the deterioration of plaster walls or the reaction of combustion-related sulphur dioxide and calcium carbonate in the indoor atmosphere.[215] Calcium sulphate may also adsorb soot onto its surface.[216]

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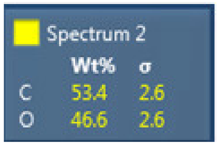
[214] A. Phenix and A. Burnstock, "The Deposition of Dirt: A Review of Literature, With Scanning Electron Microscope Stuies of Dirt on Selected Paintings," in *Dirt and Pictures Separated*, ed. S. Hackney, J. Townsend and N. Eastaugh, 11-18 (London: UKIC, 1990), 17.

[215] Alebic-Juretic and Sekulic-Cikovic, 49-50.

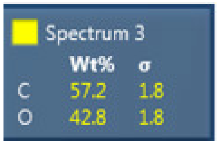
[216] Worobiec et al., 64.



Spectrum 2

	Wt%	σ
C	53.4	2.6
O	46.6	2.6

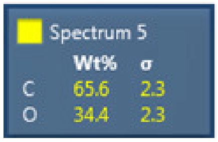
Figure 61 Raised natural cotton clean, EDS



Spectrum 3

	Wt%	σ
C	57.2	1.8
O	42.8	1.8

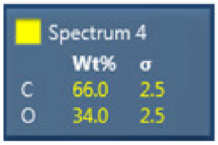
Figure 62 Raised natural cotton smoke-soiled, EDS



Spectrum 5

	Wt%	σ
C	65.6	2.3
O	34.4	2.3

Figure 63 Thread sample 1 unsoiled point, EDS



Spectrum 4

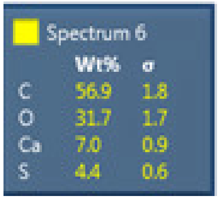
	Wt%	σ
C	66.0	2.5
O	34.0	2.5

Figure 64 Thread sample 1 spherical particles, EDS

Spectrum 7

	Wt%	σ
C	69.1	1.9
O	30.9	1.9

Figure 65 Thread sample 2 globular particle, EDS



Spectrum 6

	Wt%	σ
C	56.9	1.8
O	31.7	1.7
Ca	7.0	0.9
S	4.4	0.6

Figure 66 Thread sample 2 angular particle, EDS

б ~ standard deviation
Wt% ~ weight percentage

6.3.5.6 Liquid sampling

Solubilised soiling samples were prepared in order to aid future spectroscopy and chromatography analysis within the 'Dirty Stories' research project. Details of sampling can be found in Appendix 3.

6.3.6 Evaluation of the characterisation results

Technical-historical investigation revealed that the muslin curtain dates to the nineteenth century, so the soiling might have originated from the polluted environment of that period. The soiling pattern indicated airborne origin but no differing smell or greasy touch was observed, unlike in the case of the banner. There was, however the possibility that the soiling attributes changed due to laundering of the textile. Infrared photography was successful in the detection of soot on reference cotton fabrics, but the results of the photography of the muslin curtain were considered inconclusive. Microscopic examination utilised artificially smoke-soiled fabrics to aid characterisation by comparison: stereomicroscopy and PLM pointed out similarities but it was acknowledged that the artificial soiling did not cover the different soiling scenarios and that there is a need for representative reference materials. SEM-EDS revealed that the soiling did not have a branched structure unlike the soiling on the smoke-soiled reference fabric, but was composed of sub-micron particles indicating airborne or re-deposited origin. Spherical particles containing only carbon and oxygen might be combustion-related and calcium sulphate crystals present might have adsorbed soot on their surfaces. On the basis of the characterisation, it is possible that the soiling on the muslin curtain originated from combustion processes. Future spectroscopic and chromatographic analysis of the liquid samples has the potential to reveal more about the soiling origin and composition.

CHAPTER 7: CONCLUSIONS

The dissertation research aimed at outlining conservation attitudes to atmospheric pollution-derived sooty soilings on historic textiles, and unveiling current conservation knowledge about the soiling attributes and identification. The characteristics of sooty depositions were introduced in the context of historically important domestic and outdoor burning sources affecting textiles exhibited in the domestic interior.

Several methods with potential for the characterisation of sooty soilings were found with the help of literature research in conservation, environmental, fuel engineering and forensic sciences. Physical examination, historical research and scientific investigation including testing, special imaging, the use of magnification and compositional analysis were identified as the most powerful characterisation techniques.

The practical project focused on the applicability of relevant techniques to ascertain of the presence of sooty soilings on two domestic interior historic textiles. Investigation of the soilings started from historical research and readily available examination methods, and proceeded towards the utilisation of instrumental analytical techniques.

The research pointed out the need for reference materials and data in this new field, which could aid characterisation by comparison and would allow stepping towards the exact source appointment and harmfulness assessment of sooty soilings. References could include artificially soiled fabrics; normal and infrared light, optical and scanning electron microscope images, elemental and molecular composition surveys of these reference soilings.

Simple non-invasive techniques including visual and tactile examination, stereomicroscopy and infrared photography were found to be potent tools for soiling characterisation, the efficacy of which could be increased by availability of reference materials. Polarised light microscopy could be applied to the characterisation of mechanically removed soiling before more invasive sampling takes place.

Scanning electron microscopy of mechanically removed soiling and soiled fibre samples could be useful if reference materials and elemental analytical data were available, and could complement molecular analytical techniques such as Raman and infrared micro-spectroscopy. Infrared and fluorescence spectroscopy require more invasive sampling if the soiling is analysed in liquid solutions. Both techniques may be used for the detection of combustion-related substances, and fluorescence spectroscopy has shown a great potential for quantitative analysis.

Even though chromatography and mass spectrometry require destructive analysis of soiling samples, they could be applied to the full profiling of sooty soilings if reference compounds were available. Ion chromatography performed within the dissertation project showed its utility in the analysis of anions with potential to cause damage to historic textiles. The destructive method of thermogravimetry could be effective in showing the presence of black carbon in small samples of textile soilings.

A future task would be to create a protocol for the assessment of presumably sooty soilings starting from non-invasive methods through micro-sampling, and more invasive dry and liquid sampling. Emphasis could be on establishing a set of quick, simple and cost-effective techniques available to every textile conservator, and on developing analytical techniques for compositional fingerprinting. In the meantime, an advice to conservators is to preserve removed historic soiling samples if possible - and to collaborate with analytical researchers who are interested in what ‘dirty stories’ of historic textiles may tell.

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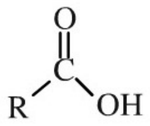
APPENDICES

Appendix 1: Glossary

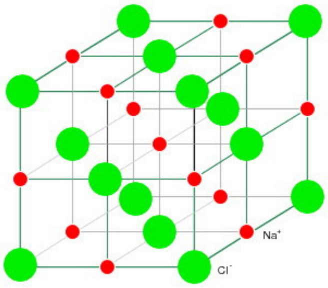
Aliphatic hydrocarbon	Hydrocarbons containing carbon and hydrogen joined together in straight chain, branched trains, or non-aromatic rings.
Amorphous	In amorphous materials, there is no three-dimensional order between the constituent atoms or molecules.
Anisotropic	Being directionally dependent. In crystallography, anisotropic substances refer to materials which have more than one refractive index, including crystals in the hexagonal, tetragonal, orthorhombic, monoclinic, or triclinic system, oriented polymers, or isotropic substances that are mechanically or thermally strained. Anisotropic substances can be seen between fully-crossed polarisers, if they are not in an extinction position. (*See: Isotropic.)*
Biomarker	A biomarker or biological marker is a substance used as an indicator of a biological state, for example, an indicator of normal biological processes.
Carboxylic acid	Organic molecules containing at least one carboxyl (-COOH) functional group, comprising of a carbonyl (C=O) and a hydroxyl (-OH) group. Carboxylic acids are weak acids which only partially ionise in water.



Formula of carboxylic acids

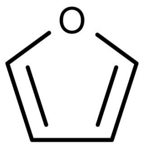
Coagulation	Destabilisation of colloids by neutralising the forces that keep them apart, resulting in the collision of particles which form larger particle agglomerates.

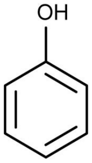
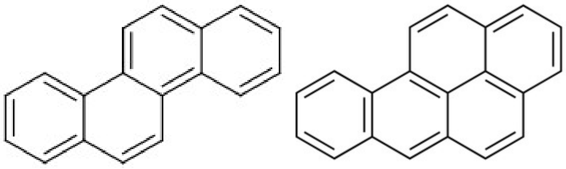
Coal-gas	Coal-gas is a flammable gaseous fuel produced by the destructive distillation of coal. It is comprised of a mixture of gases, mainly hydrogen and carbon monoxide.
Crystalline	Crystalline materials are characterised by repetition of their constituent atoms or molecules in a three-dimensional array.
Cubic crystal	Also called isometric crystal, relating to a crystal system characterised by three mutually perpendicular axes of the same length. Crystals in the isometric cubic system have only one refractive index, designated n.



Sodium chloride has a cubic structure

Cycloalkanes	Saturated hydrocarbons which form rings with two hydrogen atoms per carbon (their formula: C_nH_{2n}).
Electrostatic forces	Attraction between a negatively charged surface (which possesses an extra electron) and a positively charged surface (which had lost an electron).
Fibre optics	Optical fibres have a hair-thin transparent core made of extruded glass or plastic, which is surrounded by a transparent cladding material. In fibre-optics, light travels by total internal reflection to great distances with remarkably little attenuation, or reduction in intensity.
In situ (lat.)	In position

Heterocyclic compounds	Cyclic organic compounds in which at least one carbon atom is substituted by another element, most often by oxygen, nitrogen or sulphur.  Furan is a heterocyclic compound
Isotropic	A material with identical properties in all directions. In crystallography isotropic substances have only one refractive index, including gases, glasses, liquids (except liquid crystals), unoriented polymers, and crystals in the cubic system. Isotropic substances cannot be seen between fully-crossed polarisers, regardless of their orientation. (*See: Anisotropic.*)
Magnification	The ratio of the size of the image to the size of the object in microscopy. Useful magnification is the range of magnifications in which enlarging the size of the image allows detail resolved by the objective lens to be detected more easily by the eye (*see: Resolution*).
Nascent (lat.)	Having recently come into existence.
Near-infrared	Infrared light with wavelengths between 800 and 2500 nm ('near' the visible light wavelengths).
Near-ultraviolet	Ultraviolet light with wavelengths between 300nm and 400nm ('near' the visible light wavelengths).
Nuclei (lat.)	Core.
Paraffin	Petroleum distillate composed of saturated hydrocarbons. Its solid form, paraffin wax is comprised of a mixture of high molecular weight hydrocarbons with carbon numbers between 20 and 40 (their formula: $C_{20}H_{42}$ – $C_{40}H_{82}$) and has a melting point above approximately 37°C.

Phenols	Organic compounds in which a hydroxyl group is bonded directly to an aromatic hydrocarbon group.  Phenol is part of the wider group ‘phenols’
Polycyclic aromatic hydrocarbons (PAHs)	Organic molecules containing four or more fused aromatic benzene rings.  Chrysene and benzo-[a]-pyrene are examples of PAHs
Polymerise	To make up larger molecules by addition of small repeating units, so-called monomers.
Reduced carbon	Carbon species being in a higher energy state which can be further oxidised.
Resolution	The smallest distance between two points in an object which can just be distinguished as separate points in microscopy.
Spermaceti	Oil derived from the head of the sperm whale.
Van der Waals forces	Weak attractive force acting between all atoms and molecules, induced by one atom distorting the electron cloud of a neighbouring atom, thus causing a temporary imbalance of charge and mutual attraction.

Appendix 2: William Morris Action Song Challenge Banner - supplementary information

2/1 Details on materials and construction of the textile elements of the banner

All fibres were identified with a Zeiss Axiolab polarising transmitted light optical microscope. The fibre samples were macerated and mounted on a microscope slide with deionised water.

Starch test was undertaken with starch indicator solution (0.1g iodine plus 1g potassium iodide in 100ml water). One drop of the indicator solution was pipetted onto a fibre sample: if the sample turned dark blue, this indicated that the fibre had been starched.

	Material	**Thread construction**	**Weave and thread count or technique**
Top fabric	Undyed linen	Z-spun (diameter: approx. 0.4mm).	Plain weave, thread count: 17warps/10mm, 20wefts/10mm.
Lining	Undyed linen	Z-spun (diameter: approx. 0.4mm).	Plain weave, thread count: 16warps/10mm, 20wefts/10mm
Interlining	Dark brown linen/hemp/ramie, starched	Z-spun.	Plain weave, thread count: 13warps/10mm, 14wefts/10mm.
Embroidery	Dyed silk	Loosely Z-spun, 2 ply, S-twisted.	Satin stitches with preparatory drawing underneath.
Fringe	Dyed cotton	Loosely Z-spun 2 ply, S-twisted.	Several threads Z-twisted.
Cord	Undyed linen	Z-spun 2 ply, S-twisted.	Several threads Z-twisted.
Stitching thread	White cotton	Z-spun, S-twisted.	Slip stitches attaching top fabric and lining; running- and herringbone stitches attaching interlining and top fabric.

2/2 Colorimetric measurements on the banner

Colour measurements were taken on the heavily soiled and yellowed proper right bottom corner of the lining, and the less heavily soiled and yellowed proper right top corner of the lining. Readings were taken in L*a*b mode after calibration of the instrument. The colorimeter took the measurements within a 50mm diameter circle.

Already visible differences were quantified by the colorimeter. The largest difference was in the lightness (L value) of the areas – the more soiled area being significantly darker (lower L value), and in yellowness – the more soiled area being more yellow (higher +b value) than the less soiled area. Simple comparison of these colour values to other soiled textiles is not possible due to fundamental differences between the weave type and densities, and the colour of the textiles which would all influence colour measurements.

Lining:	**Soiled proper right bottom corner**			**Less soiled proper right top corner**		
	L	A	b	L	a	b
1	40.37	+3.45	+12.70	50.94	+2.62	+8.60
2	40.09	+3.56	+12.68	50.94	+2.56	+8.55
3	40.21	+3.58	+12.68	50.96	+2.58	+8.60
Max	40.37	+3.58	+12.70	50.96	+2.62	+8.60
Min	40.09	+3.45	+12.68	50.94	+2.56	+8.55
Mean	**40.22**	**+3.53**	**+12.69**	**50.95**	**+2.59**	**+8.58**
SD	*0.14*	*0.07*	*0.01*	*0.01*	*0.03*	*0.03*

2/3 Solubility tests on the grey soiling on the banner

Solubility tests were carried out on 10mm×10mm areas on the proper right bottom edge of the lining. The areas were swabbed with cotton swabs moistened with the cleaning agents and the areas were blotted from both sides. Areas treated with detergent solutions were rinsed with deionised water.

Cleaning agent	**Removal of greying**	**Removal of yellowing**
Deionised water	Effective	Effective
Anionic detergent solution: Orvus WA Paste® 4×c.m.c.* with 0.5g SCMC** in softened water	Most effective	Most effective
Anionic detergent solution: Hostapon TPHC® 4×c.m.c.* with 0.5g SCMC** in softened water	Most effective	Most effective
Non-ionic detergent solution: Dehypon LS45® 4×c.m.c.* with 0.5g SCMC** in softened water	Effective	Effective
Industrial denatured alcohol (IDA)	Effective	Less effective
Acetone	Not effective	Not effective
White spirit	Not effective	Not effective

*c.m.c – critical micelle concentration (indicator of cleaning strength/concentration)

**SCMC – sodium carboxymethyl cellulose (anti soil re-deposition agent)

Appendix 3: Muslin curtain - supplementary information

3/1 Details on materials and construction of the muslin curtain

All fibres were identified with a Zeiss Axiolab polarising transmitted light optical microscope. The fibre samples were macerated and mounted on a microscope slide with deionised water.

Starch test was undertaken with starch indicator solution (0.1g iodine plus 1g potassium iodide in 100ml water). One drop of the indicator solution was pipetted onto a fibre sample: if the sample turned dark blue, this idicated that the fibre had been starched.

	Material	**Thread construction**	**Weave and thread count or technique**
Muslin ground warps and wefts	White cotton, starched	Z-spun (diameter: approx. 0.15mm).	Plain weave, thread count: 20 warps/10mm, 17 wefts/10mm.
Supplementary weft	White cotton, starched	S-spun (diameter: approx. 0.3mm).	Spotting technique.
Stitching thread	White cotton	Z-spun, 2-ply and S-twisted.	The three pieces of the textile were probably attached by early machine stitching using lock stitches, the gathering created by simple chain stitches, and the longer edges finished with double locking chain stitches.[217] The shorter edges were finished with hand-made running stitches.

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[217] „How Stuff Works? How Sewing Machines Work," T. Harris, http://home.howstuffworks.com/sewing-machine1.htm (accessed July 18, 2013).

3/2 Colorimetric measurements on the muslin curtain

Colour of the four examined areas was recorded in L*a*b mode after calibration of the instrument. The colorimeter took the measurements within a 50mm diameter circle. Measurements were taken with the textile placed on white blotting paper - due to the openness of the weave this probably caused an increase in lightness (increased L value). All measurements showed red and green values (±a values) near to zero. In the darker areas (lower L values) a shift towards yellow (higher +b value) was observable.

	Blotting paper background			**Area A**			**Area B**			**Area C**			**Area D**		
	L	a	b	L	a	b	L	a	b	L	a	b	L	a	b
1	94.69	-0.44	+3.01	64.55	+0.68	+10.84	91.67	-0.41	+4.62	82.10	+0.05	+7.70	82.02	-0.17	+8.49
2	94.67	-0.44	+3.00	64.29	+0.64	+10.82	91.64	-0.41	+4.63	82.11	+0.02	+7.73	81.95	-0.19	+8.53
3	94.66	-0.40	+3.02	64.27	+0.69	+10.84	91.68	-0.45	+4.64	82.15	+0.04	+7.70	81.80	-0.16	+8.58
Max	94.69	-0.40	+3.02	64.55	+0.69	+10.84	91.68	-0.41	+4.64	82.15	+0.05	+7.73	82.02	-0.16	+8.58
Min	94.66	-0.44	+3.00	64.27	+0.64	+10.82	91.64	-0.45	+4.62	82.10	+0.02	+7.70	81.80	-0.19	+8.49
Mean	**94.67**	**-0.44**	**+3.02**	**64.37**	**+0.67**	**+10.83**	**91.66**	**-0.42**	**+4.63**	**82.12**	**+0.04**	**+7.71**	**81.92**	**-0.17**	**+8.53**
SD	*0.01*	*0.02*	*0.01*	*0.15*	*0.02*	*0.01*	*0.02*	*0.02*	*0.01*	*0.02*	*0.01*	*0.01*	*0.11*	*0.01*	*0.04*

3/3 Stereomicroscopic observations of the soiling on the muslin curtain and reference artificially soiled fabrics

Criteria for examination were established in order to record the most information about the black particulate soiling according to easily distinguishable particle sizes: particles small enough to coat the fibres; particles adhered to the fibres which were smaller than the cotton fibre diameter; and particles adhered to the threads which were larger than a fibre diameter. The presence of other coloured particles and the condition of the fibres in each examined area were also recorded.

Size measurements were calculated as a product of multiplications of the fibre diameter (indicated: *) and as ratios of the thread diameter (indicated: **) in lack of a measuring stereomicroscope ocular scale. The largest dimension of particles is given in the table, e.g. in case of elongated particles this is the length of the particle. Cotton fibres of the ground warp, weft and the spotting weft were measured with the ocular scale of the Zeiss Axiolab polarising microscope and had a mean diameter of 15µm. Ground warp and weft threads were approximately 0.15mm = 150µm in diameter according to the scanning electron microscope images.

All magnification at ×50 unless otherwise indicated.

a. Muslin curtain in area A

Criteria	**Soiling coverage and comments**
Dark coating on loose fibres	Loose fibres emerging from the threads are fully coated, while fibres on the surface of the threads are partly coated. Coating consists of small black particles.
Black particles smaller than the fibre diameter adhered to fibres	Very abundant irregular shaped black particle aggregates throughout.
Black particles larger than the fibre diameter loosely adhered to threads	Few reflective acicular black particles approx. 15-30µm*. (Magnification at ×20-×50)
Other coloured particulate soiling	-
Condition of threads in examined area	Threads are in a stable condition. Loose fibres are seen to be accentuated by the dark coating. Slight brownish-yellow discolouration of the threads is visible.

b. Muslin curtain in area B

Criteria	**Soiling coverage and comments**
Dark coating on loose fibres	Uneven coating on some of the loose fibres emerging from the threads and on some of the fibres on the surface of the threads. Coating consists of small black particles.
Black particles smaller than the fibre diameter adhered to fibres	Aggregates of irregular shaped black particles found on the surface of the threads closely adhered to them – these are in the process of further fragmentation.
Black particles larger than fibre diameter loosely adhered to threads	Few reflective acicular, needle-like and irregular shaped black particles approx. 15-30µm*. (Magnification at ×20-×50)
Other coloured particulate soiling	Few light brown and green particles approx. 75µm**. A brown fibre fragment coated with small black particles and a few blue fibre fragments.
Condition of threads in examined area	Threads are in a good condition. Yellowing in spots on the ground weave is independent from the black soiling.

c. Muslin curtain, area C

Criteria	**Soiling coverage and comments**
Dark coating on loose fibres	Loose fibres emerging from the threads are fully coated. Coating consists of small black particles.
Black particles smaller than the fibre diameter adhered to fibres	Aggregates of irregular shaped black particles found on the surface of the threads closely adhered to them – these are in the process of further fragmentation.
Black particles larger than the fibre diameter loosely adhered to threads	Reflective acicular black particles approx.15-30µm* throughout. Two reflective black flakes with cerrated edge on the supplementary weft approx. 75µm**. Two reflective irregular shaped black particles with a rough surface approx. 150µm**. A dull needle-like black particle approx. 150µm**. (Magnification at ×20-×50)
Other coloured particulate soiling	Light brown, light blue particles and few blue fibre fragments.
Condition of threads in examined area	Threads are in good condition. Loose fibres are seen to be accentuated by the dark coating. Yellowing in spots on the ground weave is independent from the black soiling.

d. Muslin curtain, area D

Criteria	Soiling coverage and comments
Dark coating on loose fibres	Uneven coating on some of the loose fibres emerging from the threads and on some of the fibres on the surface of the threads. Coating consists of small black particles.
Black particles smaller than the fibre diameter adhered to fibres	Individual dull spherical black particles throughout. Few aggregates of irregular shaped black particles found on the surface of the threads closely adhered to them – these are in the process of further fragmentation.
Black particles larger than the fibre diameter loosely adhered to threads	Reflective acicular black particles approx.15-30µm* throughout. Few reflective elongated black plates approx. 50µm**. (Magnification at ×20-×50)
Other coloured particulate soiling	Blue fibres and a brown fibre fragment covered with a black coating.
Condition of threads in examined area	Threads are in good condition. Loose fibres are seen to be accentuated but not damaged by the dark coating. Yellowing in spots on the ground weave is independent from the black soiling.

e. Raised natural cotton smoke-soiled

Criteria	Soiling coverage and comments
Dark coating on loose fibres	The loose surface fibres have a dark coating consisting of small black particles. These fibres protected the rest of the weave from the soiling.
Black particles smaller than the fibre diameter adhered to fibres	Individual black particles on the fibres are most likely aggregates of the previously mentioned smaller particles.
Black particles larger than the fibre diameter loosely adhered to threads	-
Other coloured particulate soiling	Large dark brown crystalline particles probably collected during storage.
Condition of threads in examined area	Very good.

f. Cotton voile smoke-soiled

Criteria	Soiling coverage and comments
Dark coating on loose fibres	Even dark coating on fibres on the surface of threads which were most exposed from the smoking direction. The few fibres emerging from the threads have a dark coating consisting of small black particles.
Black particles smaller than the fibre diameter adhered to fibres	-
Black particles larger than the fibre diameter loosely adhered to threads	-
Other coloured particulate soiling	-
Condition of threads in examined area	Very good.

g. Raised natural cotton house dust-soiled

Criteria	Soiling coverage and comments
Dark coating on loose fibres	-
Black particles smaller than the fibre diameter adhered to fibres	Black particles individually adhered to the fibres.
Black particles larger than the fibre diameter loosely adhered to threads	Dull spherical and elongated black particles approx. 15-30µm in diameter.** (Magnification at ×20-×50)
Other coloured particulate soiling	Several types of coloured debris and fibre fragments.
Condition of threads in examined area	Very good.

3/4 Solubility tests on the grey soiling on the muslin curtain

Solubility tests were carried out on 10mm×10mm areas on the proper left bottom corner of the trimming. Two areas were swabbed with cotton swabs moistened with the cleaning agents and the areas blotted from both sides. Solubility test were carried out under fume extraction. Areas treated with detergent solutions were rinsed with deionised water.

Cleaning agents were selected for sampling in consultation with Dr Anita Quye, taking the future aims of the 'Dirty Stories' project into consideration.

Cleaning agent	**Removal of greying**
Industrial denatured alcohol	Effective
Non-ionic detergent solution: Dehypon LS45® 5×c.m.c.* in softened water	Most effective

*c.m.c. – critical micelle concentration (indicator of cleaning strength/concentration)

3/5 Liquid sampling of the muslin curtain

Industrial denatured alcohol and the non-ionic detergent solution Dehypon LS45® in five times its critical micelle concentration (3g/l) were selected for sampling in consultation with Dr Anita Quye, taking the future aims of the 'Dirty Stories' project into consideration. The heavily soiled proper right bottom corner (containing 'area A') and proper left bottom corner (containing 'area D') of the muslin curtain were locally immersed in glass beakers filled with IDA and the detergent solution, respectively. Sampling was carried out in the fume hood. The liquid samples were transferred to glass sample containers. The area treated with detergent solution was rinsed with softened and deionised water. The sampled areas were blotted and dried.

Appendix 4: List of suppliers

- **White cotton lawn**
- **White cotton voile**
- **Raised natural cotton**
- **Plain weave natural linen**

Company name: Whaleys (Bradford) Ltd

Address: Harris Court
Great Horton
Bradford
West Yorkshire
BD7 4EQ
UK

Web: http://www.whaleys-bradford.ltd.uk/

- **Rolled natural beeswax candles**

Company name: Candle Cavern

Address: Unit 6
Buriton Business Park
Mapledurham Lane
Petersfield
Hampshire
GU32 3NJ
UK

Web: http://www.candlecavern.co.uk/

- **Cleaning Sponge (76Wx152Lx22mmH, Type: 961-100)**

Company name: Preservation Equipment Ltd

Address: Vinces Road
Diss
Norfolk
IP22 4HQ
UK

Web: http://www.preservationequipment.com

- **Eukitt® quick-hardening mounting medium**

Company name: Sigma-Aldrich Company Ltd

Address: The Old Brickyard
New Road
Gillingham
Dorset
SP8 4XT
UK

Web: http://www.sigmaaldrich.com/united-kingdom.html

- **Industrial denatured alcohol**
- **Acetone**

Company name: Fischer Scientific UK Ltd

Address: Bishop Meadow Road
Loughborough
Leicestershire
LE11 5RG
UK

Web: http://www.fisher.co.uk/

- **Dehypon LS45®**

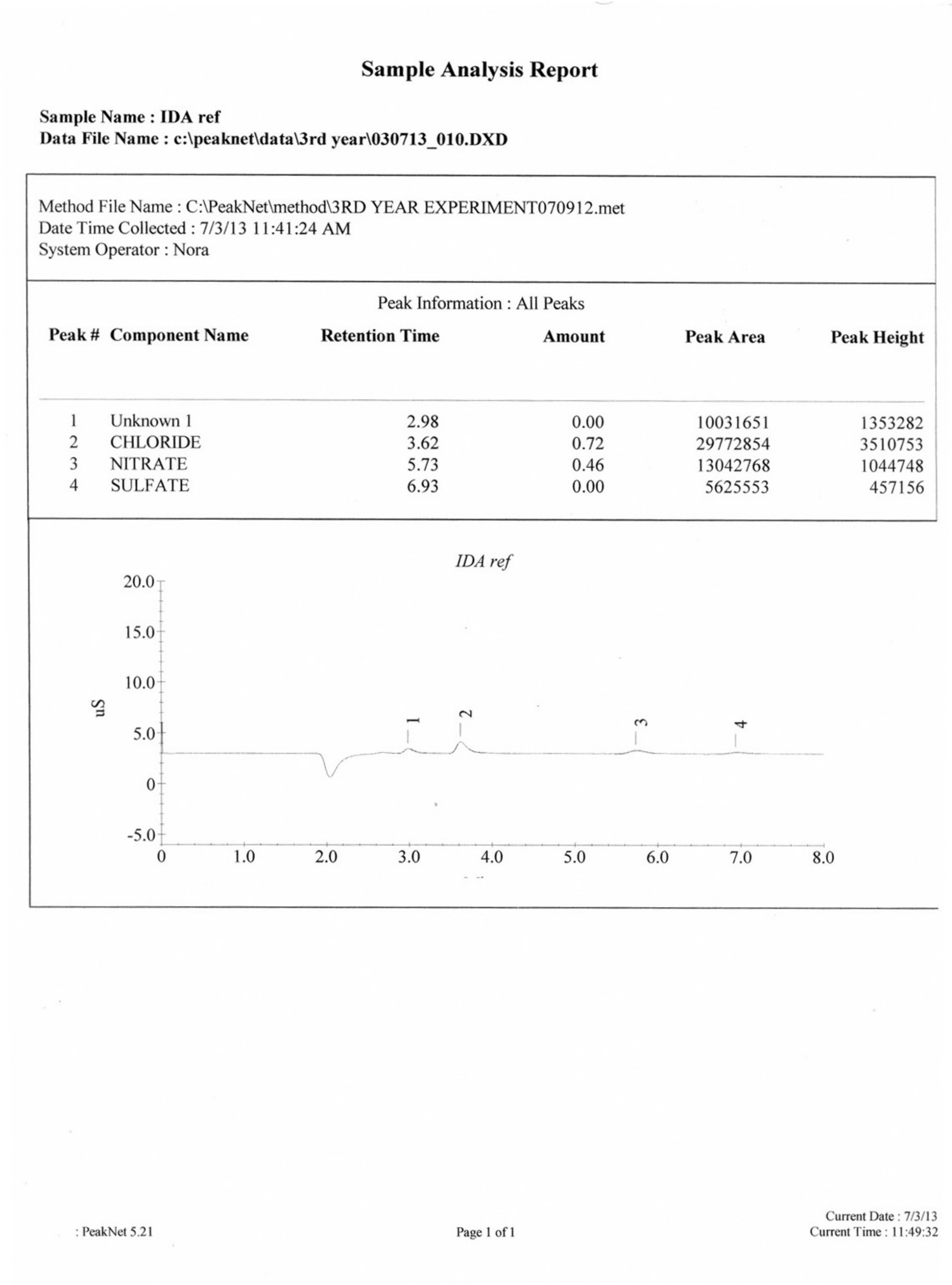
Company name: Conservation By Design Ltd

Address: 5 Singer Way
Woburn Rd Ind. Estate
Kempston
Bedford
MK42 7AW
UK

Web: http://www.conservation-by-design.co.uk

Appendix 5: Ion chromatography results and column details

5/1 Chromatogram of the reference solution



Sample Analysis Report

Sample Name : IDA ref
Data File Name : c:\peaknet\data\3rd year\030713_010.DXD

Method File Name : C:\PeakNet\method\3RD YEAR EXPERIMENT070912.met
Date Time Collected : 7/3/13 11:41:24 AM
System Operator : Nora

Peak Information : All Peaks

Peak #	Component Name	Retention Time	Amount	Peak Area	Peak Height
1	Unknown 1	2.98	0.00	10031651	1353282
2	CHLORIDE	3.62	0.72	29772854	3510753
3	NITRATE	5.73	0.46	13042768	1044748
4	SULFATE	6.93	0.00	5625553	457156

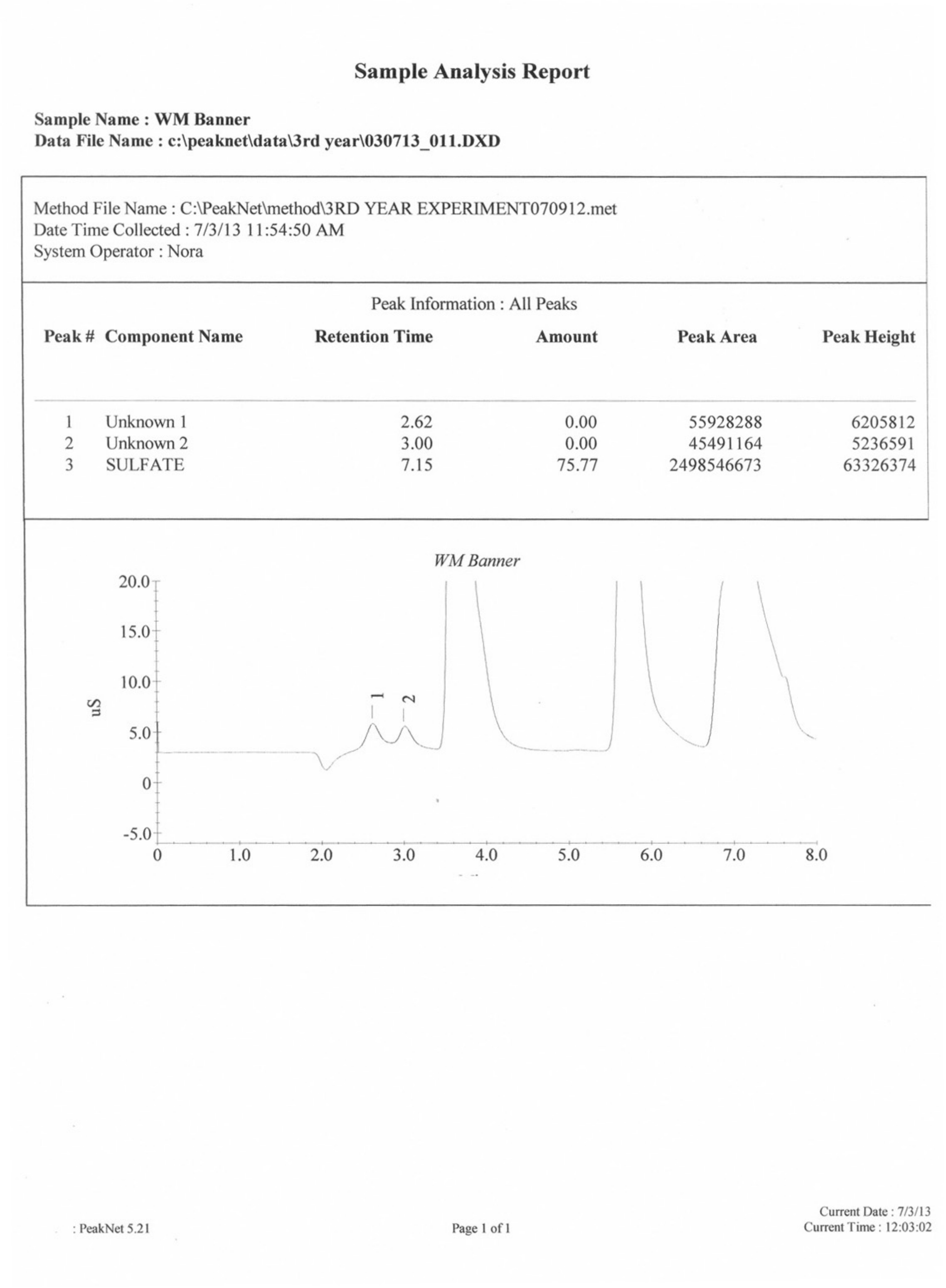
IDA ref

: PeakNet 5.21

Page 1 of 1

Current Date : 7/3/13
Current Time : 11:49:32

5/2 Chromatogram of the undiluted sample solution



Sample Analysis Report

Sample Name : WM Banner
Data File Name : c:\peaknet\data\3rd year\030713_011.DXD

Method File Name : C:\PeakNet\method\3RD YEAR EXPERIMENT070912.met
Date Time Collected : 7/3/13 11:54:50 AM
System Operator : Nora

Peak Information : All Peaks

Peak #	Component Name	Retention Time	Amount	Peak Area	Peak Height
1	Unknown 1	2.62	0.00	55928288	6205812
2	Unknown 2	3.00	0.00	45491164	5236591
3	SULFATE	7.15	75.77	2498546673	63326374

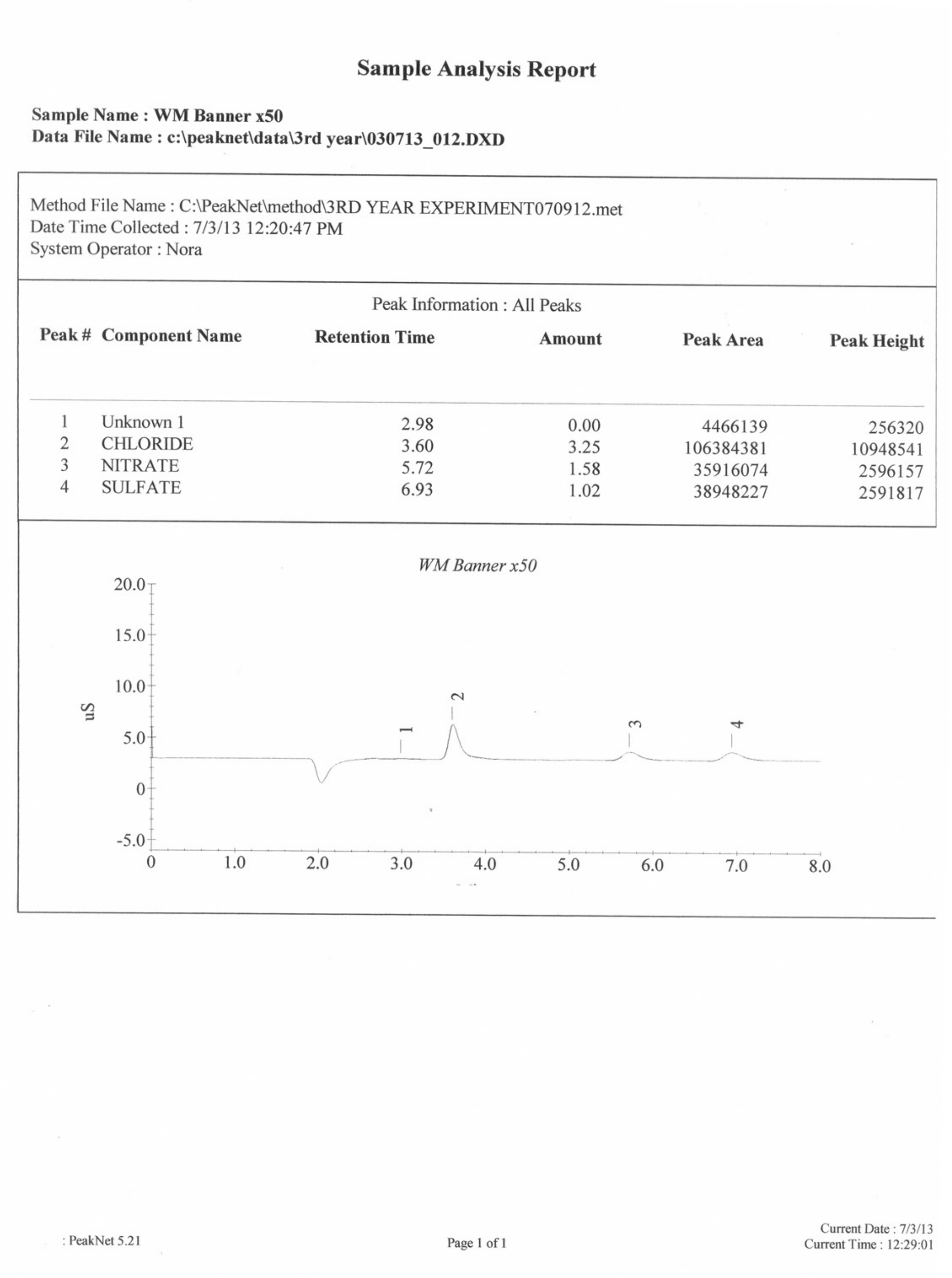
WM Banner

: PeakNet 5.21

Page 1 of 1

Current Date : 7/3/13
Current Time : 12:03:02

5/3 Chromatogram of the ×50 diluted sample solution



Sample Analysis Report

Sample Name : WM Banner x50
Data File Name : c:\peaknet\data\3rd year\030713_012.DXD

Method File Name : C:\PeakNet\method\3RD YEAR EXPERIMENT070912.met
Date Time Collected : 7/3/13 12:20:47 PM
System Operator : Nora

Peak Information : All Peaks

Peak #	Component Name	Retention Time	Amount	Peak Area	Peak Height
1	Unknown 1	2.98	0.00	4466139	256320
2	CHLORIDE	3.60	3.25	106384381	10948541
3	NITRATE	5.72	1.58	35916074	2596157
4	SULFATE	6.93	1.02	38948227	2591817

WM Banner x50

: PeakNet 5.21

Page 1 of 1

Current Date : 7/3/13
Current Time : 12:29:01

5/4 Regression analysis of the sulphate anions

Regression Analysis

Name Sulphate
Date: 2013.07.03

Sample description:
A linear fit using the equation $Y = B^*(X-X_0)$.

Best Fit Values of the Parameters

Symbol	*Value*	*Standard Deviation*	*Name*
X_0	0,121112116	0,098078538	Intercept on the x-axis
B	35262323,5	1470664,863	Linear co-efficient
S_E	4604604,595	n/a	The standard error

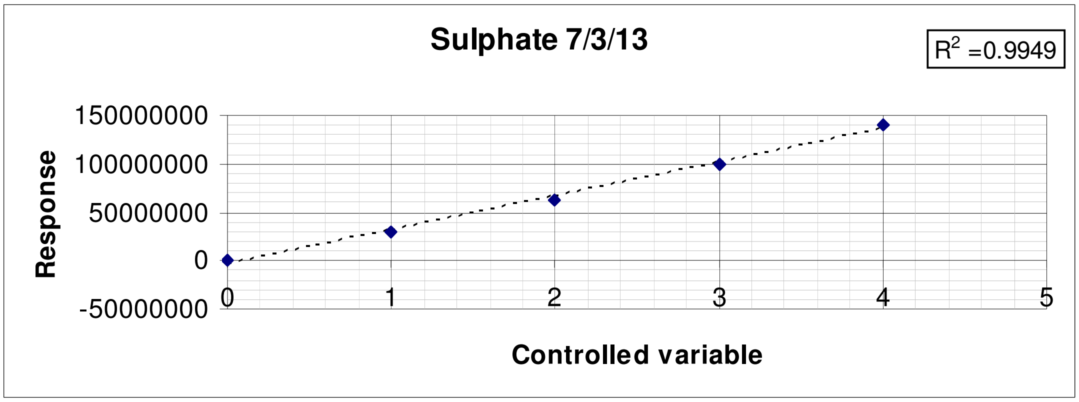
Fit of data to equation

X	*Y, observed*	*Y, calculated*
0	0	-4270694,6000
1	28689531	30991628,9000
2	62415055	66253952,4000
3	99017586	101516275,9000
4	141147590	136778599,4000

Results

Response	*Concentration*	*S_X*	*S_C*	*S_R*
5625553	0,280646498	0,160255598	0,092899661	0,130581429
38948227	1,225640211	0,146979737	0,067465054	0,130581429

S_C = Standard deviation of X due to the bad fit of the calibration graph
S_R = Standard deviation of X due to the replication error of Y
S_X = Standard deviation of X total, $S_X^2 = S_C^2 + S_R^2$



5/5 Regression analysis of the nitrate anions

Regression Analysis

Name Nitrate
Date: 2013.07.03

Sample description:
A linear fit using the equation $Y = B*(X-X_0)$.

Best Fit Values of the Parameters

Symbol	*Value*	*Standard Deviation*	*Name*
X_0	0,109238209	0,102085478	Intercept on the x-axis
B	21899302,6	946831,2584	Linear co-efficient
S_E	2964498,353	n/a	The standard error

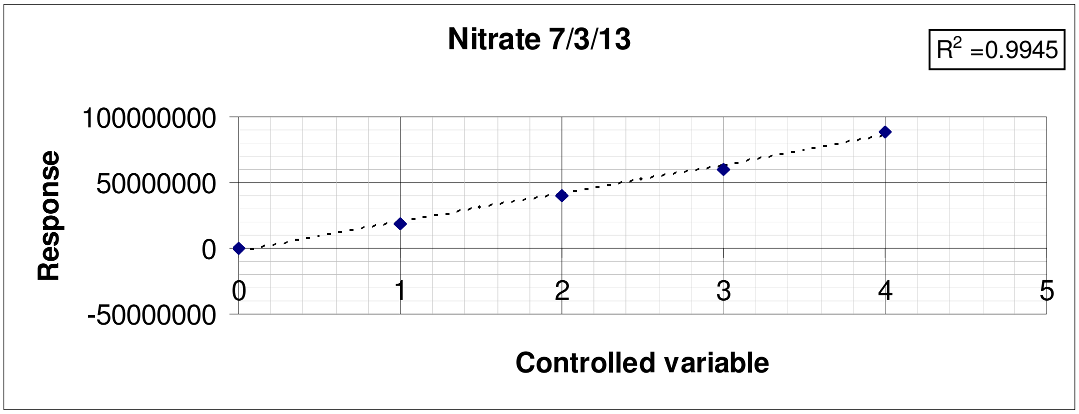
Fit of data to equation

X	*Y, observed*	*Y, calculated*
0	0	-2392240,6000
1	17901858	19507062,0000
2	40451402	41406364,6000
3	60462242	63305667,2000
4	88216321	85204969,8000

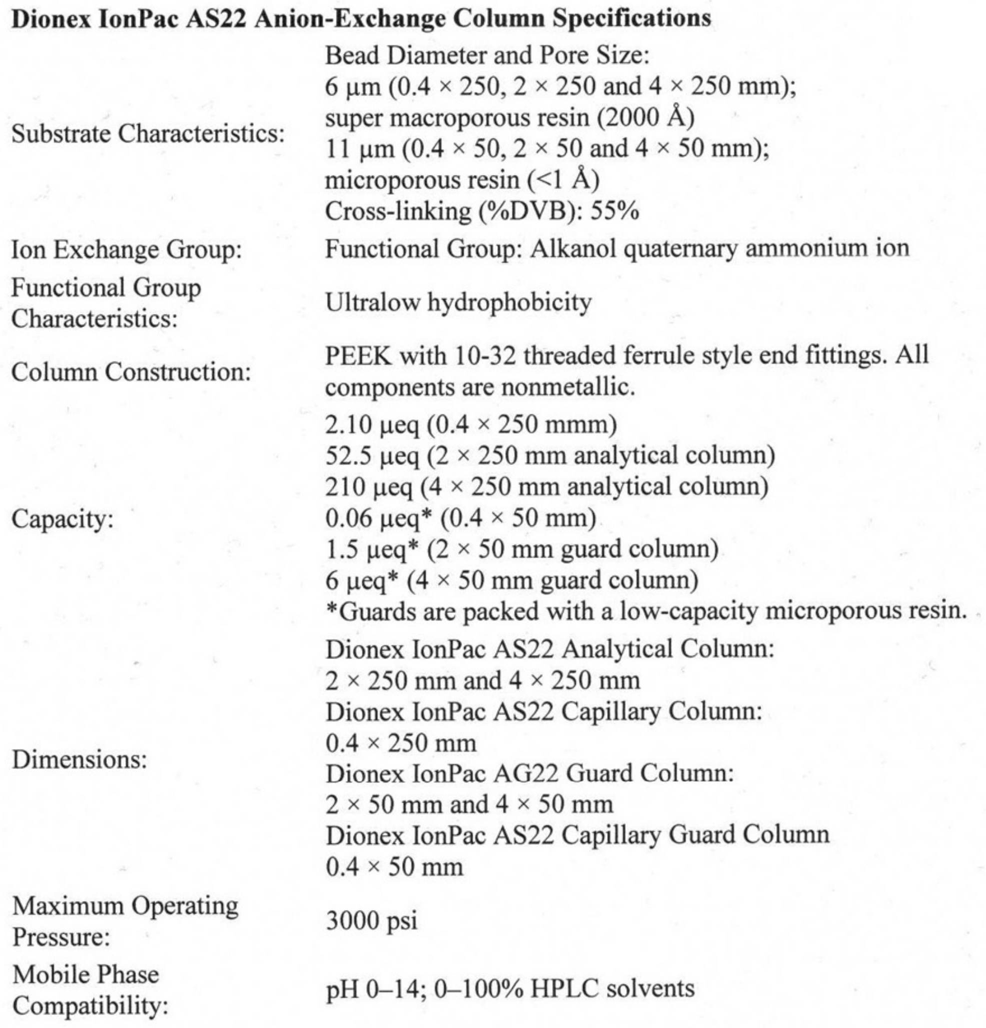
Results

Response	*Concentration*	*S_X*	*S_C*	*S_R*
13042768	0,704817358	0,158833135	0,083084623	0,135369532
35916074	1,749293815	0,148985789	0,062224231	0,135369532

S_C = Standard deviation of X due to the bad fit of the calibration graph
S_R = Standard deviation of X due to the replication error of Y
S_X = Standard deviation of X total, $S_X^2 = S_C^2 + S_R^2$



5/6 Ion chromatograph column specifications



Dionex IonPac AS22 Anion-Exchange Column Specifications

Substrate Characteristics:	Bead Diameter and Pore Size: 6 µm (0.4 × 250, 2 × 250 and 4 × 250 mm); super macroporous resin (2000 Å) 11 µm (0.4 × 50, 2 × 50 and 4 × 50 mm); microporous resin (<1 Å) Cross-linking (%DVB): 55%
Ion Exchange Group:	Functional Group: Alkanol quaternary ammonium ion
Functional Group Characteristics:	Ultralow hydrophobicity
Column Construction:	PEEK with 10-32 threaded ferrule style end fittings. All components are nonmetallic.
Capacity:	2.10 µeq (0.4 × 250 mmm) 52.5 µeq (2 × 250 mm analytical column) 210 µeq (4 × 250 mm analytical column) 0.06 µeq* (0.4 × 50 mm) 1.5 µeq* (2 × 50 mm guard column) 6 µeq* (4 × 50 mm guard column) *Guards are packed with a low-capacity microporous resin.
Dimensions:	Dionex IonPac AS22 Analytical Column: 2 × 250 mm and 4 × 250 mm Dionex IonPac AS22 Capillary Column: 0.4 × 250 mm Dionex IonPac AG22 Guard Column: 2 × 50 mm and 4 × 50 mm Dionex IonPac AS22 Capillary Guard Column 0.4 × 50 mm
Maximum Operating Pressure:	3000 psi
Mobile Phase Compatibility:	pH 0–14; 0–100% HPLC solvents

Appendix 6: Preparation of the coal soot industrial denatured alcohol extract

- 0.015 g coal soot collected from a domestic chimney flue was measured into a volumetric flask.
- 20ml industrial denatured alcohol was added to the flask from a volumetric pipette.
- The bottle was covered with a glass stopper, placed in an ultrasonic bath filled with deionised water and the ultrasonic bath turned on for 30 minutes.
- The liquid was poured into clean glass sample containers, leaving the coal soot residue at the bottom of the volumetric flask.

Appendix 7: Preparation of artificially soiled reference fabrics

7/1 Preparation of artificially smoke-soiled reference fabrics for microscopy examination

- The cotton voile and raised natural cotton fabrics were ironed and cut to approximately 150×150mm squares.
- A frame was cut from corrugated polypropylene sheet with a 100×100mm square opening, and the textiles were attached to it around the four edges with masking tape.
- The candle was lit and a stainless steel rod was held into the flame to produce a sooting flame.
- The frame was held 150mm above the top of the flame to avoid heat damage, parallelly to the ground, for 2 minutes to collect the smoke particles.
- The samples were stored in acid-free tissue paper until use.

7/2 Preparation of artificially candle soot-soiled fabrics for infrared photography

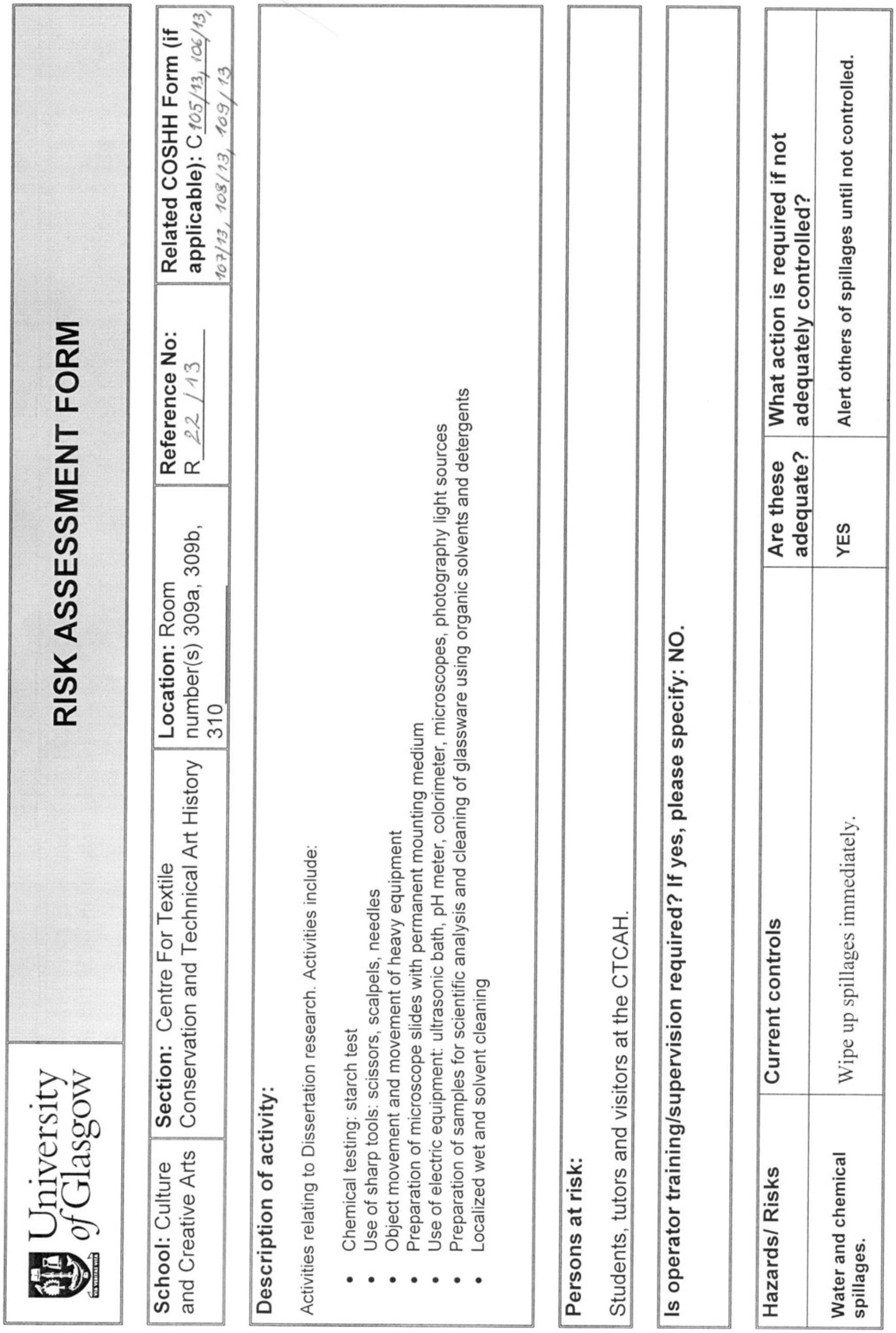
- White cotton lawn fabric was ironed and cut to approximately 150×150mm squares.
- A template made of acid-free blotting paper with a hole of 60mm diameter was laid on top.
- Candle soot was created by holding a glass sheet above a flame thus collecting the soot, and scraping it off with a stainless steel rod into a jar lined with tinfoil.
- Sample with darker shade of soot was created by applying the candle soot onto the fabric with a cotton wool swab.
- Sample with lighter shade of soot was created by applying the candle soot onto the fabric with a soft brush.
- The samples were stored in acid-free blotting paper folder until use.

Appendix 8: Infrared photography caption details

	Aperture	**Shutter speed (seconds)**	**ISO**
Candle soot on white cotton lawn, darker shade	4.8	1/13	400
Candle soot on white cotton lawn, lighter shade	4.8	1/15	400
Infrared photography 1 **(Proper right bottom corner)**	18	2	100
Infrared photography 2 **(Proper right top middle)**	18	1	100

Appendix 9: Risk Assessment, COSHH Assessment and MSDS forms

9/1 Risk Assessment form, page 1



University of Glasgow

RISK ASSESSMENT FORM

School: Culture and Creative Arts	**Section:** Centre For Textile Conservation and Technical Art History	**Location:** Room number(s) 309a, 309b, 310	**Reference No:** R 22 / 13

Related COSHH Form (if applicable): C 105/13, 106/13, 107/13, 108/13, 109/13

Description of activity:

Activities relating to Dissertation research. Activities include:

- Chemical testing: starch test
- Use of sharp tools: scissors, scalpels, needles
- Object movement and movement of heavy equipment
- Preparation of microscope slides with permanent mounting medium
- Use of electric equipment: ultrasonic bath, pH meter, colorimeter, microscopes, photography light sources
- Preparation of samples for scientific analysis and cleaning of glassware using organic solvents and detergents
- Localized wet and solvent cleaning

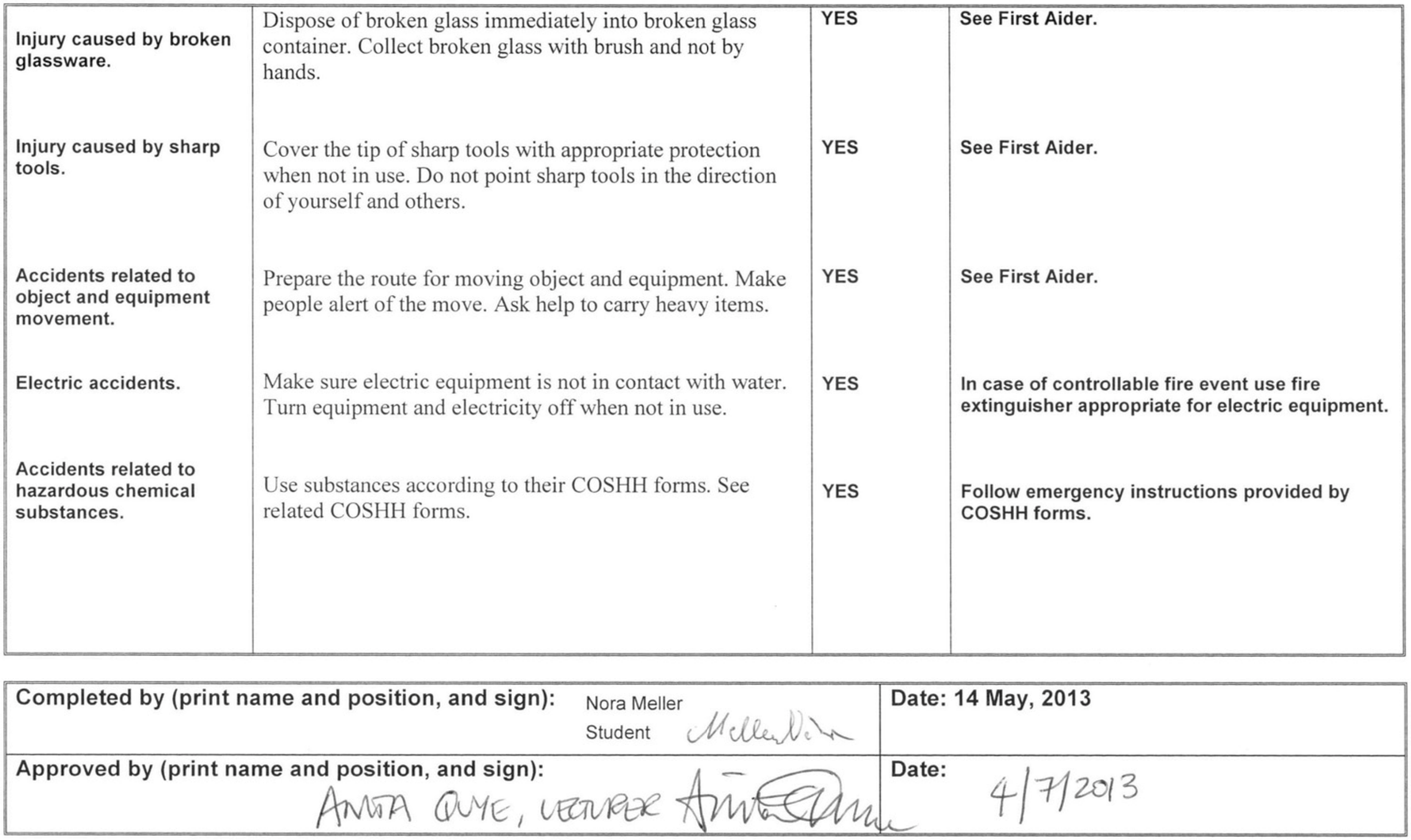
Persons at risk:

Students, tutors and visitors at the CTCAH.

Is operator training/supervision required? If yes, please specify: NO.

Hazards/ Risks	Current controls	Are these adequate?	What action is required if not adequately controlled?
Water and chemical spillages.	Wipe up spillages immediately.	YES	Alert others of spillages until not controlled.

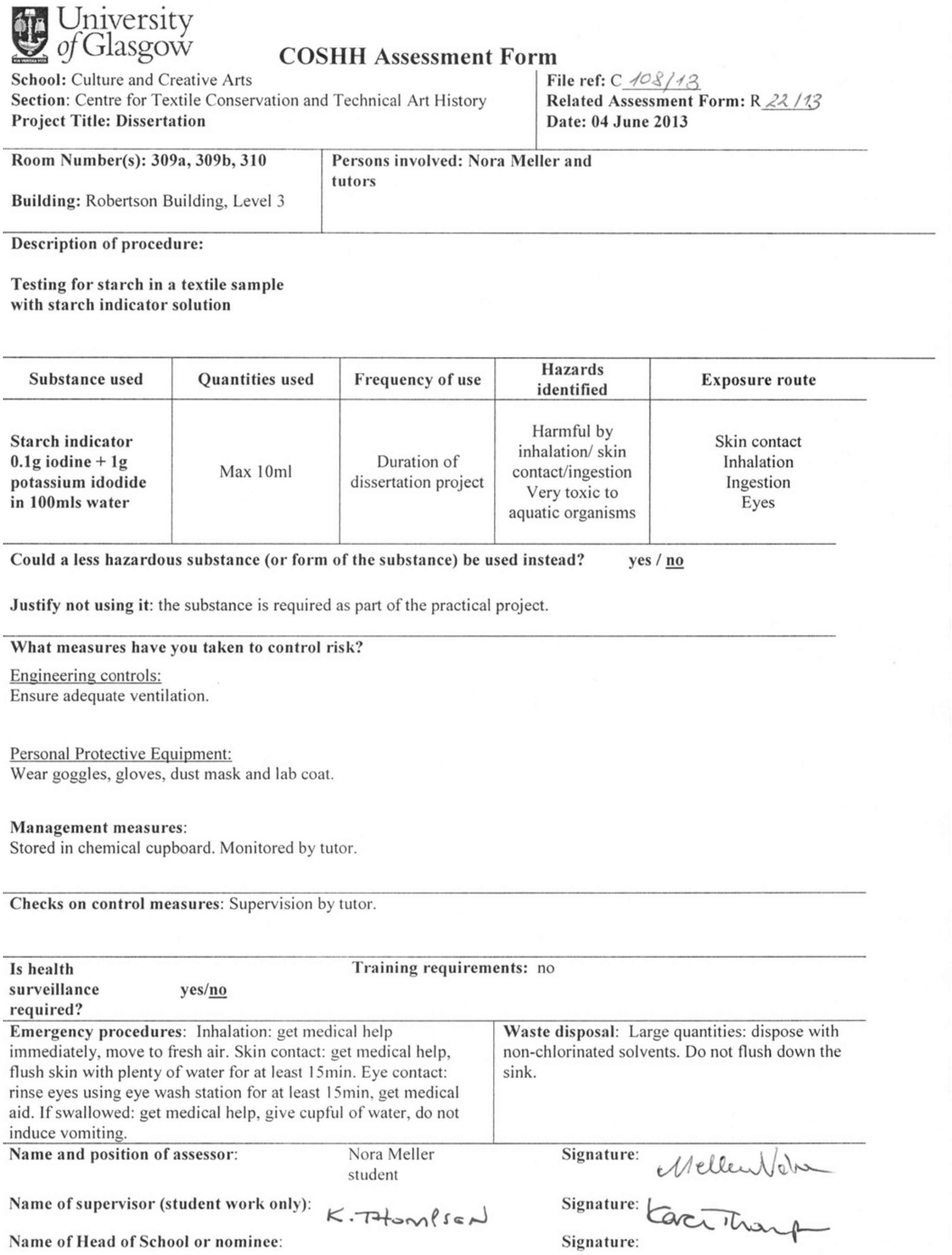
9/2 Risk Assessment form, page 2



Injury caused by broken glassware.	Dispose of broken glass immediately into broken glass container. Collect broken glass with brush and not by hands.	**YES**	**See First Aider.**
Injury caused by sharp tools.	Cover the tip of sharp tools with appropriate protection when not in use. Do not point sharp tools in the direction of yourself and others.	**YES**	**See First Aider.**
Accidents related to object and equipment movement.	Prepare the route for moving object and equipment. Make people alert of the move. Ask help to carry heavy items.	**YES**	**See First Aider.**
Electric accidents.	Make sure electric equipment is not in contact with water. Turn equipment and electricity off when not in use.	**YES**	**In case of controllable fire event use fire extinguisher appropriate for electric equipment.**
Accidents related to hazardous chemical substances.	Use substances according to their COSHH forms. See related COSHH forms.	**YES**	**Follow emergency instructions provided by COSHH forms.**

Completed by (print name and position, and sign): Nora Meller Student	**Date: 14 May, 2013**
Approved by (print name and position, and sign): ANITA QUYE, LECTURER	**Date:** 4/7/2013

9/3 COSHH Assessment Form: Starch indicator solution



University of Glasgow

COSHH Assessment Form

School: Culture and Creative Arts
Section: Centre for Textile Conservation and Technical Art History
Project Title: Dissertation

File ref: C 108/13
Related Assessment Form: R 22/13
Date: 04 June 2013

Room Number(s): 309a, 309b, 310

Building: Robertson Building, Level 3

Persons involved: Nora Meller and tutors

Description of procedure:

Testing for starch in a textile sample with starch indicator solution

Substance used	Quantities used	Frequency of use	Hazards identified	Exposure route
Starch indicator 0.1g iodine + 1g potassium idodide in 100mls water	Max 10ml	Duration of dissertation project	Harmful by inhalation/ skin contact/ingestion Very toxic to aquatic organisms	Skin contact Inhalation Ingestion Eyes

Could a less hazardous substance (or form of the substance) be used instead? **yes / [✓ no]**

Justify not using it: the substance is required as part of the practical project.

What measures have you taken to control risk?

Engineering controls:
Ensure adequate ventilation.

Personal Protective Equipment:
Wear goggles, gloves, dust mask and lab coat.

Management measures:
Stored in chemical cupboard. Monitored by tutor.

Checks on control measures: Supervision by tutor.

Is health surveillance required? **yes/[✓ no]**

Training requirements: no

Emergency procedures: Inhalation: get medical help immediately, move to fresh air. Skin contact: get medical help, flush skin with plenty of water for at least 15min. Eye contact: rinse eyes using eye wash station for at least 15min, get medical aid. If swallowed: get medical help, give cupful of water, do not induce vomiting.

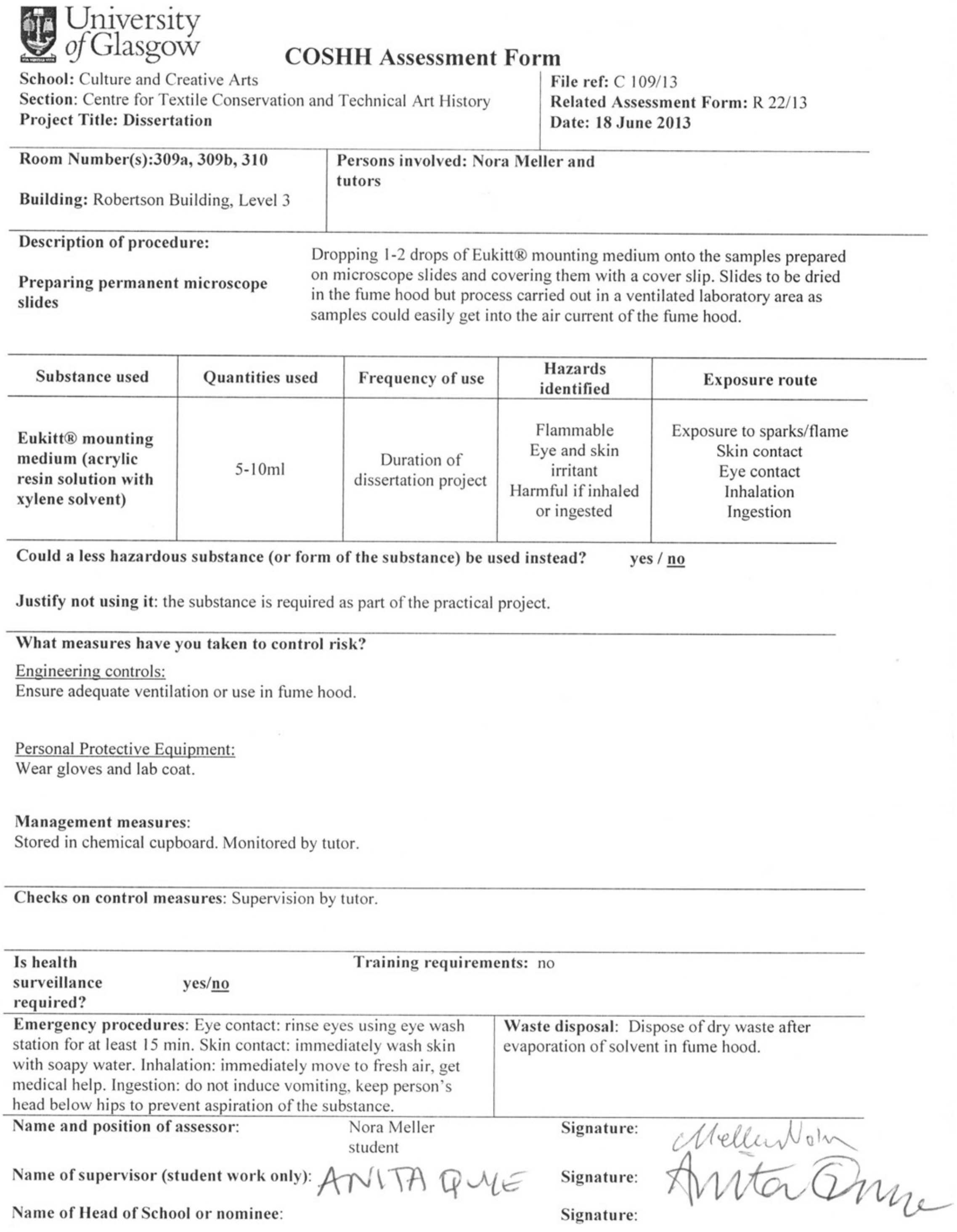
Waste disposal: Large quantities: dispose with non-chlorinated solvents. Do not flush down the sink.

Name and position of assessor: Nora Meller student **Signature**: Meller Nora

Name of supervisor (student work only): K. Thompson **Signature**: Karen Thompson

Name of Head of School or nominee: **Signature**:

9/4 COSHH Assessment Form: Eukitt® quick-hardening mounting medium



University of Glasgow

COSHH Assessment Form

School: Culture and Creative Arts
Section: Centre for Textile Conservation and Technical Art History
Project Title: Dissertation

File ref: C 109/13
Related Assessment Form: R 22/13
Date: 18 June 2013

Room Number(s):309a, 309b, 310

Building: Robertson Building, Level 3

Persons involved: Nora Meller and tutors

Description of procedure:

Preparing permanent microscope slides

Dropping 1-2 drops of Eukitt® mounting medium onto the samples prepared on microscope slides and covering them with a cover slip. Slides to be dried in the fume hood but process carried out in a ventilated laboratory area as samples could easily get into the air current of the fume hood.

Substance used	Quantities used	Frequency of use	Hazards identified	Exposure route
Eukitt® mounting medium (acrylic resin solution with xylene solvent)	5-10ml	Duration of dissertation project	Flammable Eye and skin irritant Harmful if inhaled or ingested	Exposure to sparks/flame Skin contact Eye contact Inhalation Ingestion

Could a less hazardous substance (or form of the substance) be used instead? yes / [✓ no]

Justify not using it: the substance is required as part of the practical project.

What measures have you taken to control risk?

Engineering controls:
Ensure adequate ventilation or use in fume hood.

Personal Protective Equipment:
Wear gloves and lab coat.

Management measures:
Stored in chemical cupboard. Monitored by tutor.

Checks on control measures: Supervision by tutor.

Is health surveillance required? yes/[✓ no]

Training requirements: no

Emergency procedures: Eye contact: rinse eyes using eye wash station for at least 15 min. Skin contact: immediately wash skin with soapy water. Inhalation: immediately move to fresh air, get medical help. Ingestion: do not induce vomiting, keep person's head below hips to prevent aspiration of the substance.

Waste disposal: Dispose of dry waste after evaporation of solvent in fume hood.

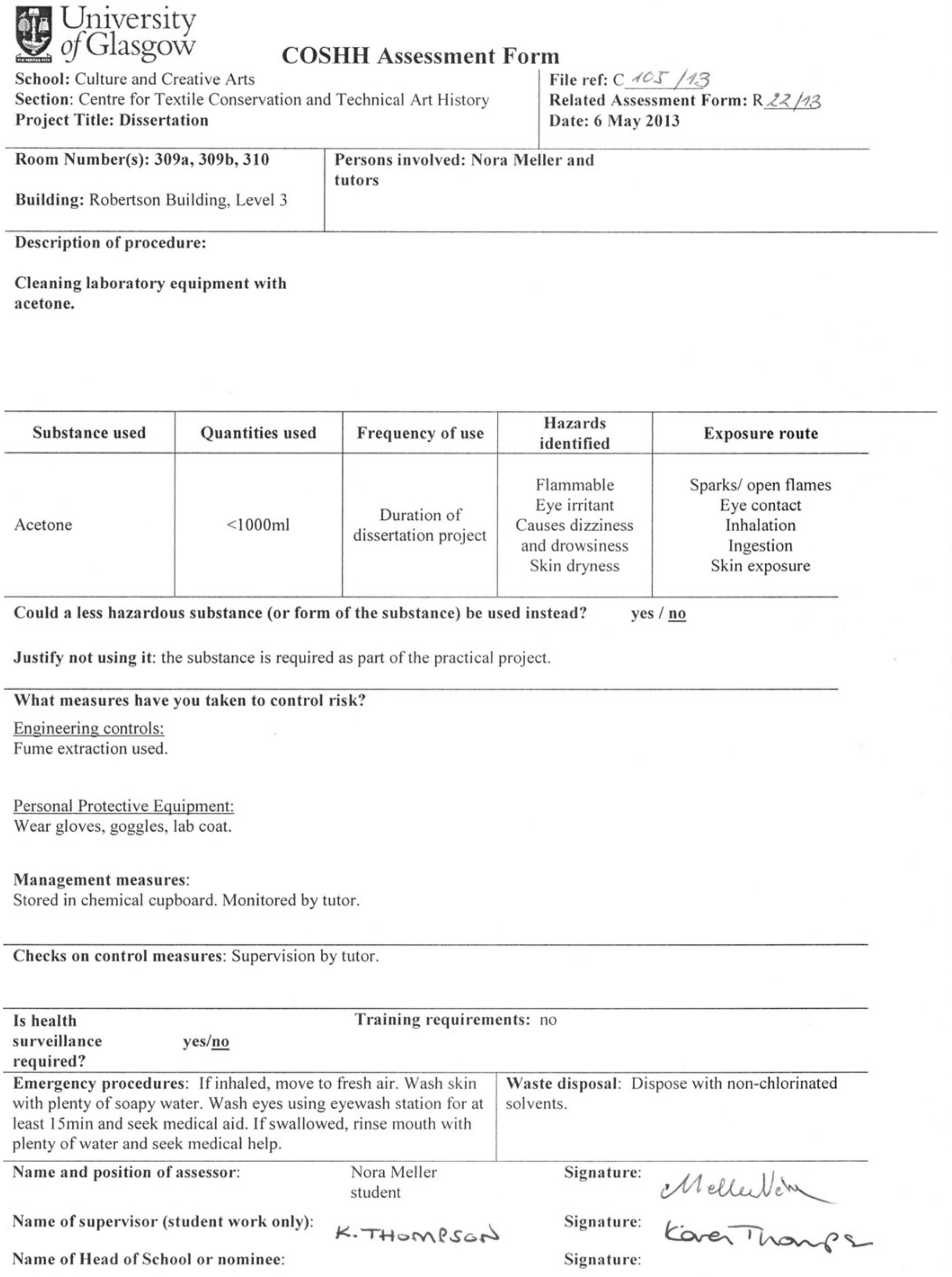
Name and position of assessor: Nora Meller student **Signature:** Meller Nora

Name of supervisor (student work only): ANITA QUYE **Signature:** Anita Quye

Name of Head of School or nominee: **Signature:**

MSDS form (Eukitt®):
http://www.sigmaaldrich.com/catalog/product/fluka/03989?lang=en&region=GB

9/5 COSHH Assessment Form: Acetone



University of Glasgow

COSHH Assessment Form

School: Culture and Creative Arts
Section: Centre for Textile Conservation and Technical Art History
Project Title: Dissertation

File ref: C 105/13
Related Assessment Form: R 22/13
Date: 6 May 2013

Room Number(s): 309a, 309b, 310

Building: Robertson Building, Level 3

Persons involved: Nora Meller and tutors

Description of procedure:

Cleaning laboratory equipment with acetone.

Substance used	Quantities used	Frequency of use	Hazards identified	Exposure route
Acetone	<1000ml	Duration of dissertation project	Flammable Eye irritant Causes dizziness and drowsiness Skin dryness	Sparks/ open flames Eye contact Inhalation Ingestion Skin exposure

Could a less hazardous substance (or form of the substance) be used instead? yes / [✓ no]

Justify not using it: the substance is required as part of the practical project.

What measures have you taken to control risk?

Engineering controls:
Fume extraction used.

Personal Protective Equipment:
Wear gloves, goggles, lab coat.

Management measures:
Stored in chemical cupboard. Monitored by tutor.

Checks on control measures: Supervision by tutor.

Is health surveillance required? yes/[✓ no]

Training requirements: no

Emergency procedures: If inhaled, move to fresh air. Wash skin with plenty of soapy water. Wash eyes using eyewash station for at least 15min and seek medical aid. If swallowed, rinse mouth with plenty of water and seek medical help.

Waste disposal: Dispose with non-chlorinated solvents.

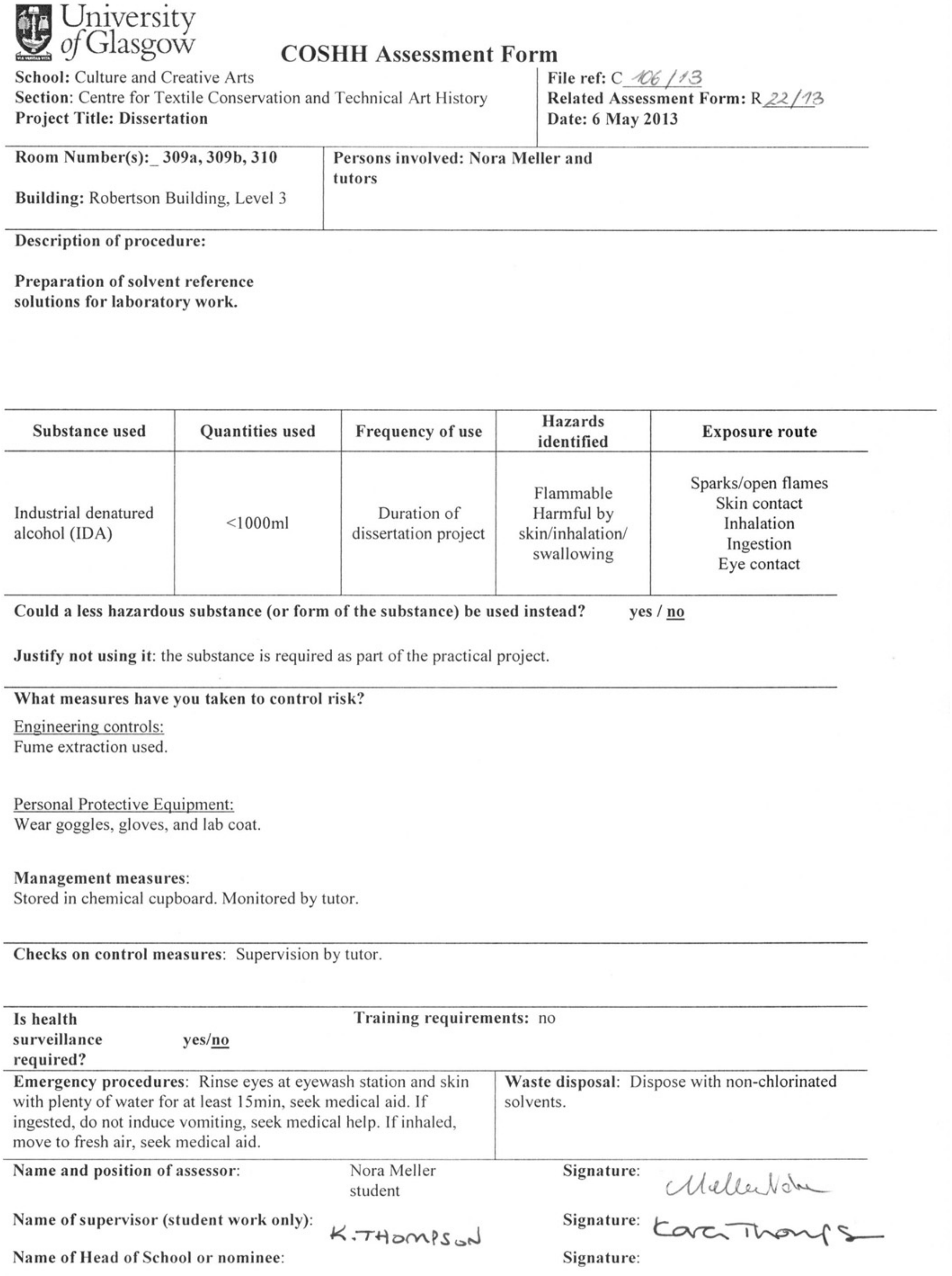
Name and position of assessor: Nora Meller student **Signature:** Meller Nora

Name of supervisor (student work only): K. THOMPSON **Signature:** Karen Thompson

Name of Head of School or nominee: **Signature:**

MSDS form (Acetone):
https://extranet.fisher.co.uk/chemicalProductData_uk/wercs?itemCode=A%2F0520&lang=EN

9/6 COSHH Assessment Form: Industrial denatured alcohol



University of Glasgow

COSHH Assessment Form

School: Culture and Creative Arts
Section: Centre for Textile Conservation and Technical Art History
Project Title: Dissertation

File ref: C 106/13
Related Assessment Form: R 22/13
Date: 6 May 2013

Room Number(s):_ 309a, 309b, 310

Building: Robertson Building, Level 3

Persons involved: Nora Meller and tutors

Description of procedure:

Preparation of solvent reference solutions for laboratory work.

Substance used	Quantities used	Frequency of use	Hazards identified	Exposure route
Industrial denatured alcohol (IDA)	<1000ml	Duration of dissertation project	Flammable Harmful by skin/inhalation/ swallowing	Sparks/open flames Skin contact Inhalation Ingestion Eye contact

Could a less hazardous substance (or form of the substance) be used instead? **yes / [✓ no]**

Justify not using it: the substance is required as part of the practical project.

What measures have you taken to control risk?

Engineering controls:
Fume extraction used.

Personal Protective Equipment:
Wear goggles, gloves, and lab coat.

Management measures:
Stored in chemical cupboard. Monitored by tutor.

Checks on control measures: Supervision by tutor.

Is health surveillance required? **yes/[✓ no]**

Training requirements: no

Emergency procedures: Rinse eyes at eyewash station and skin with plenty of water for at least 15min, seek medical aid. If ingested, do not induce vomiting, seek medical help. If inhaled, move to fresh air, seek medical aid.

Waste disposal: Dispose with non-chlorinated solvents.

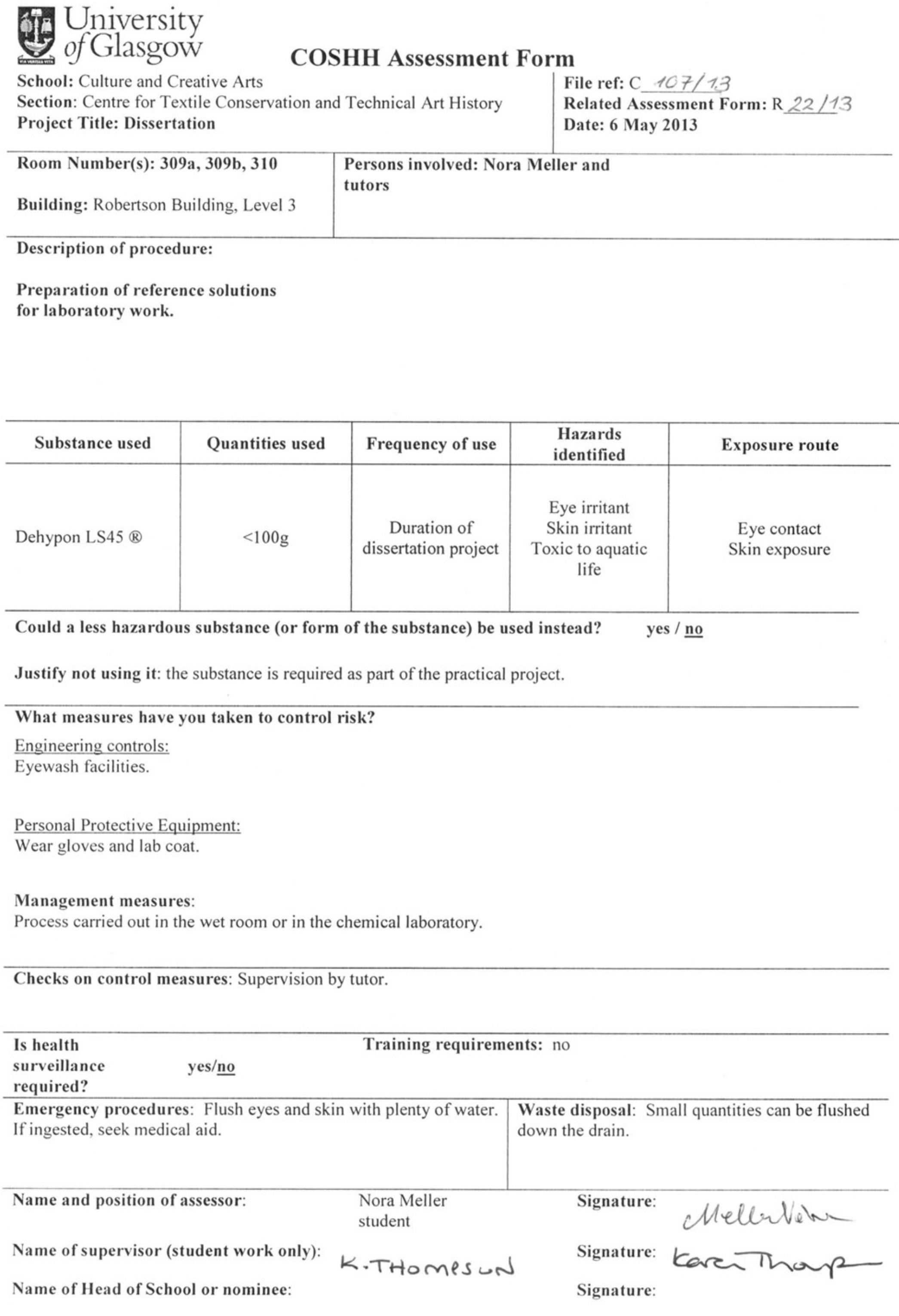
Name and position of assessor: Nora Meller student **Signature**: Meller Nora

Name of supervisor (student work only): K.THOMPSON **Signature**: Kara Thomps

Name of Head of School or nominee: **Signature**:

MSDS form (Industrial denatured alcohol):
https://extranet.fisher.co.uk/chemicalProductData_uk/wercs?itemCode=M%2F4400&lang=EN

9/7 COSHH Assessment Form: Dehypon LS45®



University of Glasgow

COSHH Assessment Form

School: Culture and Creative Arts
Section: Centre for Textile Conservation and Technical Art History
Project Title: Dissertation

File ref: C 107/13
Related Assessment Form: R 22/13
Date: 6 May 2013

Room Number(s): 309a, 309b, 310

Building: Robertson Building, Level 3

Persons involved: Nora Meller and tutors

Description of procedure:

Preparation of reference solutions for laboratory work.

Substance used	Quantities used	Frequency of use	Hazards identified	Exposure route
Dehypon LS45 ®	<100g	Duration of dissertation project	Eye irritant Skin irritant Toxic to aquatic life	Eye contact Skin exposure

Could a less hazardous substance (or form of the substance) be used instead? yes / [✓ no]

Justify not using it: the substance is required as part of the practical project.

What measures have you taken to control risk?

Engineering controls:
Eyewash facilities.

Personal Protective Equipment:
Wear gloves and lab coat.

Management measures:
Process carried out in the wet room or in the chemical laboratory.

Checks on control measures: Supervision by tutor.

Is health surveillance required? yes/ [✓ no]

Training requirements: no

Emergency procedures: Flush eyes and skin with plenty of water. If ingested, seek medical aid.

Waste disposal: Small quantities can be flushed down the drain.

Name and position of assessor: Nora Meller student **Signature**:

Name of supervisor (student work only): K.THOMPSON **Signature**:

Name of Head of School or nominee: **Signature**:

MSDS form (Dehypon LS45®):
http://www.conservation-by-design.co.uk/pdf/datasheets/Dehyphon%20LS45%20Technical%20Data%20Sheet.pdf