

Non destructive techniques for analysis of Diesel engine exhaust emissions (DEEE) on filters

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Non destructive techniques for analysis of Diesel engine exhaust emissions (DEEE) on filters

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Diesel engine exhaust emissions represent a hazard to worker health. The amount of elemental carbon (EC) is the routine measure of DEEE. Historically, an instrument, the Bosch meter, has been used in underground mines in the UK. The meter measures 'blackness' of a filter paper as a proxy for EC and HSL showed that there was a relationship between blackness and EC.

The Bosch meter is no longer available and this report details the investigations into a replacement system. The instruments tested were those that were commercially available at the time the research was undertaken: a Magee Scientific OT21 transmissometer, a DR-Lange Micro colour II and a system where a filter is scanned using a normal office scanner and then measured using a tool in the Adobe Photoshop software program.

Each has been assessed against the Bosch meter and the amount of EC as determined by two-stage combustion on an Analytik Jena elemental analyser.

The DR-Lange Micro colour II was found to be the closest like for like replacement for the Bosch meter.

When compared with EC, the Magee Scientific OT21 transmissometer was found to be the most effective instrument, having the best precision and range of analysis. The DR-Lange Micro Colour II and scanner/ photoshop were found to be acceptable for normal use and each was an improvement on the Bosch meter.

The scanner/photoshop method is considerably less expensive to purchase

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KEY MESSAGES

Three instruments have been tested to assess their suitability for replacement of the Bosch meter for the measurement of diesel engine exhaust emissions (DEEE) in workplace environments. These were a Magee Scientific OT21 transmissometer, a DR-Lange Micro colour II and a system where a filter is scanned using a normal office scanner and then measured using a tool in the Adobe Photoshop software program.

Each has been assessed against the Bosch meter (the original non-destructive technique) and measurement of elemental carbon (the accepted quantity when measuring DEEE), as determined by two-stage combustion on an Analytik Jena elemental analyser.

The DR-Lange Micro colour II was found to be the closest like for like replacement for the Bosch meter.

When compared with elemental carbon determined using an Analytik Jena elemental analyser, the Magee Scientific OT21 Transmissometer was found to be the most effective instrument, having the best precision and range of analysis. The DR-Lange Micro Colour II and scanner/photoshop were found to be acceptable for normal use and each was an improvement on the Bosch meter.

The scanner/photoshop method is considerably less expensive to purchase.

EXECUTIVE SUMMARY

Diesel engine exhaust emissions (DEEE) are recognised to have a link with respiratory disease, are known to cause irritation of the respiratory tract and have possible links to lung cancer. DEEE are of particular concern where engines are run in enclosed environments, for example warehouses or mines.

DEEE is a complex mixture of substances that can be split into two groups: organic carbon (OC) and elemental carbon (EC). OC represents all the molecular species that are either combustion products or unburned fuel constituents, for instance benzene and polyaromatic hydrocarbons (PAHs). EC is the carbon soot left over from combustion and has been identified as the primary issue of concern for respiratory illness (D Dabill, 2005).

Historically, the Health and Safety Laboratory (D. Dabill, 2005) developed a method for determination of EC DEEE on filters using the Bosch meter instrument. This instrument was simple to use, robust, non-destructive and quick and therefore it was useful for workplaces to do their own monitoring without the expense or delay of laboratory analysis. The instrument was capable of measuring filters from any source and came complete with a separate pump for sampling vehicle exhaust. Unfortunately the instrument is now obsolete. This project was commissioned to investigate which instruments and methods would be suitable replacements.

Three instruments/methods were investigated: a DR-Lange Micro colour II, a Magee Scientific OT21 transmissometer and using an office scanner and Adobe Photoshop software. None of these include a pump for sampling vehicle exhaust and as such only replace the instrument part of the Bosch system. No practical commercially available system for replacing the Bosch pump was found although Wohler equipment is documented in this report. There are many systems for monitoring vehicle exhaust which are primarily designed for MOT testing and have not been investigated in this study.

All three techniques were found to be suitable replacements for the Bosch meter with the DR-Lange Micro colour II being the closest direct replacement.

The Bosch meter did not measure EC instead it used the 'blackness' of the filter as a surrogate. Therefore the fact that the DR-Lange Micro colour II is the closest to the Bosch did not mean it was also the best at measuring EC. In fact the Bosch meter is quite poor at measuring EC and all three techniques are comparable with it. The Magee Scientific OT21 transmissometer and DR-Lange are both improvements on the Bosch meter. The scanner is also an improvement although it does not quite have the range or precision of the other two.

The Magee Scientific and DR-Lange instruments cost in the region of £8000 each, in contrast the scanner/photoshop method uses equipment commonly available in office environments and a ubiquitous software program.

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1. INTRODUCTION

Diesel engine exhaust emissions (DEEE) have been linked with respiratory disease in workers. DEEE is a complex mixture of substances that can be split into two groups: organic carbon (OC) and elemental carbon (EC). OC represents all the molecular species that are either combustion products or unburned fuel constituents, for instance benzene and polyaromatic hydrocarbons (PAHs). EC is the carbon soot left over from combustion and has been identified as the primary issue of concern for respiratory illness (D Dabill, 2005). The United Kingdom does not currently have a workplace exposure limit for DEEE. However, under the Control of Substances Hazardous to Health Regulations (COSHH), DEEEs are classed as a substance hazardous to health and as such these require that exposure be prevented or, where this is not reasonably practicable, adequately controlled. Germany has imposed a limit on exposure to DEEEs of $100 \mu\text{g}/\text{m}^3$ (TRGS 900, 1996).

Historically, the Health and Safety Laboratory (D. Dabill, 2005) developed a method for determination of EC DEEE on filters using the Bosch meter instrument. This instrument has since become obsolete. This project was commissioned to investigate what instruments and methods could be developed as replacements.

The Bosch meter technique is non destructive and it was relatively inexpensive to purchase the instrument. Also analysis does not require great expertise or training. These characteristics facilitated its use in the field by employers to routinely monitor their vehicles and staff. The instrument determines the absorption of light by the sample filter, i.e. its 'blackness'. This is achieved by measuring the light reflected by the sample and the resulting absorbance is given a number between 0.0 and 9.9. Staining is a crude measure of the contamination of the filter by exhaust fumes but it has been shown (Dabill, 2005) that the Bosch no. has a strong relationship with EC contamination. The disadvantage of the instrument is that it has a limited analytical range because staining of the filter reaches a point where the instrument can no longer determine a change in blackness despite further loading of diesel fume. In practice this occurs at a Bosch no. of approximately 9.0, which is equivalent to an EC loading of approximately $80 \mu\text{g}$. For a typical sampling regime of 2 litres/min for 4 hours this is equivalent to $160 \mu\text{g}/\text{m}^3$.

The Bosch meter was designed primarily to monitor exhaust as it exits the vehicle to check engine condition. As such, calibration with respect to EC concentration is not strictly required as the measurement is relative to a base level for that vehicle. Provided that separate samplers and pumps were available, the instrument could also be used for personal and general body air sampling. However, in this case EC calibration is essential.

Several options were identified as alternatives to the Bosch meter. Like the Bosch meter they all use light to measure the amount of exhaust emission on a filter without destroying it. These options, which are described individually below, were a Wohler exhaust pump, a DR-Lange colour meter, an electronic scanner and Adobe Photoshop software and the aethalometer technology produced by Magee Scientific.

By developing one or more alternative protocols to replace the Bosch meter, the aim of this project was to facilitate the continuation of monitoring of DEEE emissions by employers. If the procedure could be made more accurate, cost effective or simpler then this would also be of benefit. Publication of a replacement procedure could also

highlight the issue for employers and perhaps lead to an increased focus on what is a real issue for workers respiratory health.

2. IMPLICATIONS

This study demonstrates that three different light absorption techniques are each a suitable replacement for the Bosch meter for the non-destructive analysis of (DEEEs on filters).

As the Bosch meter is also a technique that relies upon light absorption it is likely that any light absorption technique will represent an acceptable alternative to the Bosch meter.

No alternative to the Bosch exhaust pump has been identified but this likely to outlast the Bosch meter and so, if an organisation already has one available, it can continue to be used and the analysis transferred to one of the new techniques described.

The results from the DR-Lange Micro colour II and scanner/photoshop methods can be converted to Bosch no. and so, if a calibration exists between elemental carbon and Bosch no. for a site, this can continue to be used. There is an increase in uncertainty if this approach is taken and, as such, it should be considered a temporary solution and the filters stored until a sufficient range of loadings are available for a new direct calibration with EC by combustion to be performed.

Filter samples can now be measured quickly with very low instrument costs using the scanner/photoshop method although the results should be calibrated for the site using the combustion method for the best accuracy to be attained.

3. METHODOLOGY

3.1 BACKGROUND

DEEE comprises soot particles, the EC, with many different organic molecules, the OC, adsorbed onto them. The OC species are more volatile and therefore a lower temperature and lower oxygen concentration are sufficient to oxidise them to CO₂. The EC soot core is considered to be almost pure carbon and a higher temperature and higher oxygen concentration are necessary to oxidise this to CO₂.

Adverse health effects that have been observed from exposure to DEEE are irritation of the eyes and respiratory tract from short-term exposure and a possible slight increase in the risk of lung cancer (Dabill, 2005).

As EC is the primary species of interest, it is necessary to ensure that the measurement of EC is free from interferences. The reference method for EC determination is thermal analysis by controlled combustion at a specific temperature. At HSL, a temperature of 950°C is used. In order to avoid positive interference by OC species, the OC is determined in an initial step by combustion at the lower temperature of 650°C, so that only the EC soot remains when the temperature is raised to 950°C.

The optical techniques are based on the assumption that the staining of the filter operates as a surrogate for the captured soot particles (EC), as it is the EC that results in the majority of the colour change.

The techniques that rely on total reflected light cannot measure OC although, as stated above, EC is the primary species of interest. The Magee transmissometer has a protocol for measurement of OC by the absorption of UV light at a wavelength of 370 nm and this is compared to the OC determined by thermal analysis in this work.

3.2 INSTRUMENT DESCRIPTIONS

Several instruments were obtained for assessment and each is described below. Each reports a result in arbitrary units specific to that instrument.

3.2.1 Wohler

The Wohler pump is a stirrup type pump (Figure 1), which incorporates a diluter system and is cycled a set number of times to load a small spot of exhaust onto a paper filter. This is then compared to a card with 10 progressively darker shaded circles printed on it. Each of these circles has a central hole punched in it and viewing the filter through this hole allows the spot to be assigned the number of the circle to which it is closest.

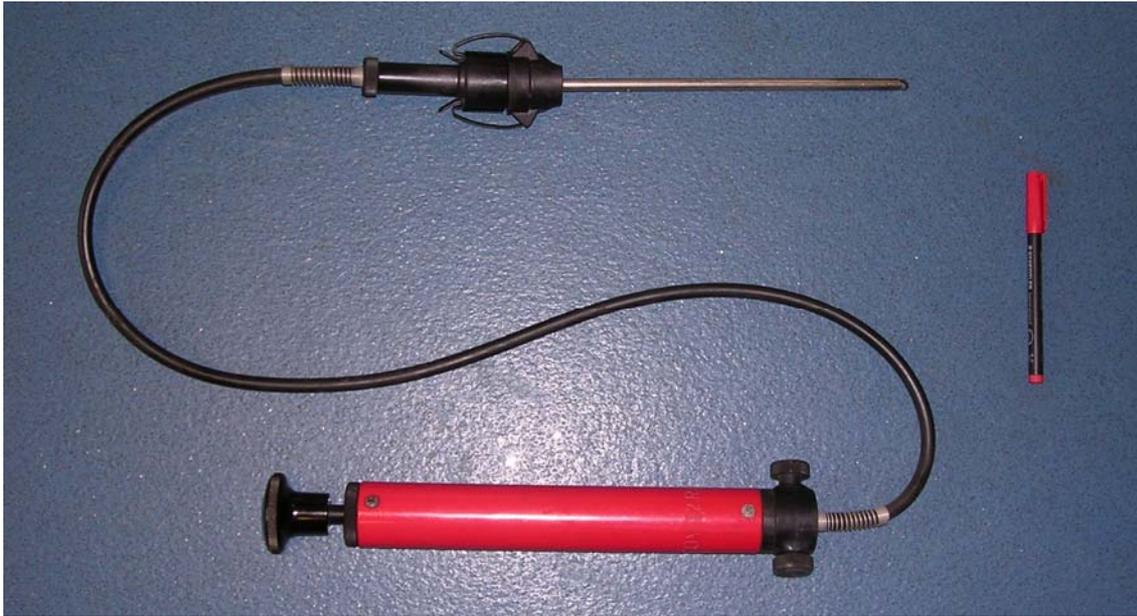


Figure 1 Photograph of a Wohler pump

The filter supplied with the equipment is not compatible with the combustion analysis technique as it is made from a carbon-based material. The nature of the filter clamping system means that it is not practicable to replace the supplied filter with a quartz fibre filter suitable for combustion analysis as quartz filters are too friable.

The Wohler equipment represented a significant reduction in precision compared with the Bosch meter. This, combined with the potential for human error/variability in the measurement and the impracticality of calibration against a combustion technique, meant it was decided not to investigate it further. Despite this the low cost of the apparatus and its lack of any electronic parts could make it useful for monitoring of vehicles only.

3.2.2 DR-Lange Micro Colour II

This instrument is similar to the Bosch meter in that it measures reflected light from the sample. It is a more sophisticated instrument that measures two colour ranges, blue to yellow and red to green, as well as blackness. The L scale for blackness alone was used in this study.

3.2.3 Scanner/Photoshop

Images of filters were obtained with a photocopier/scanner using greyscale and colour photo modes. The images were then viewed using Adobe Photoshop Elements 6 software, the histogram application was opened and the stained area of the filter selected using the magnetic lasso tool. The mean peak of the histogram of this area of the image was then recorded.

The filters were placed upside down onto a card with a series of 25 mm diameter holes punched into it. This was then covered with an acetate sheet and another piece of card and the arrangement secured with paper clips to provide rigidity whilst being transported to the scanner. In this way 35 filters could be scanned at once.

The method is quite labour intensive but has the advantage that it requires no equipment other than that generally available in an office environment.

3.2.4 Magee Aethalometer OT21 Transmissometer

This is the most sophisticated of the techniques investigated. It is based upon light absorption of a single wavelength (880 nm) that the manufacturer has identified as specific to black carbon, this is reported as attenuation or reduction in light reaching the detector relative to a blank filter.

The instrument will also measure at 370 nm and the manufacturer states this can be interpreted as an indicator of the presence of aromatic organic compounds. This is not representative of the OC species determined in the combustion test and as such has not been investigated in this study.

The filters were placed directly into the instrument slide holder.

3.2.5 Analytik Jena multiEA3100

This served as the reference instrument and determined OC and EC by thermal analysis. The sample was heated in a controlled atmosphere of argon and oxygen at two temperatures (650°C and 950°C). The carbon dioxide produced at each stage was measured by infrared absorption and this gave the amount of each carbon classification present in the sample.

3.3 ANALYTICAL PROCEDURE

3.3.1 Initial calibration

In order to test each analysis method a set of 94 filters loaded with varying levels of diesel fume were prepared. This was done by sampling the exhaust of HSL's mobile crane using a vacuum pump and 5-way splitter device, incorporating needle valves, in order that each filter (contained in the black cowl) had the same volume of gas drawn through it (Figure 2). Thus 5 replicate samples were prepared at each loading. The crane vehicle was chosen for its similarities with the type of heavy plant used underground.

4. RESULTS

4.1 COMPARISON WITH BOSCH NO.

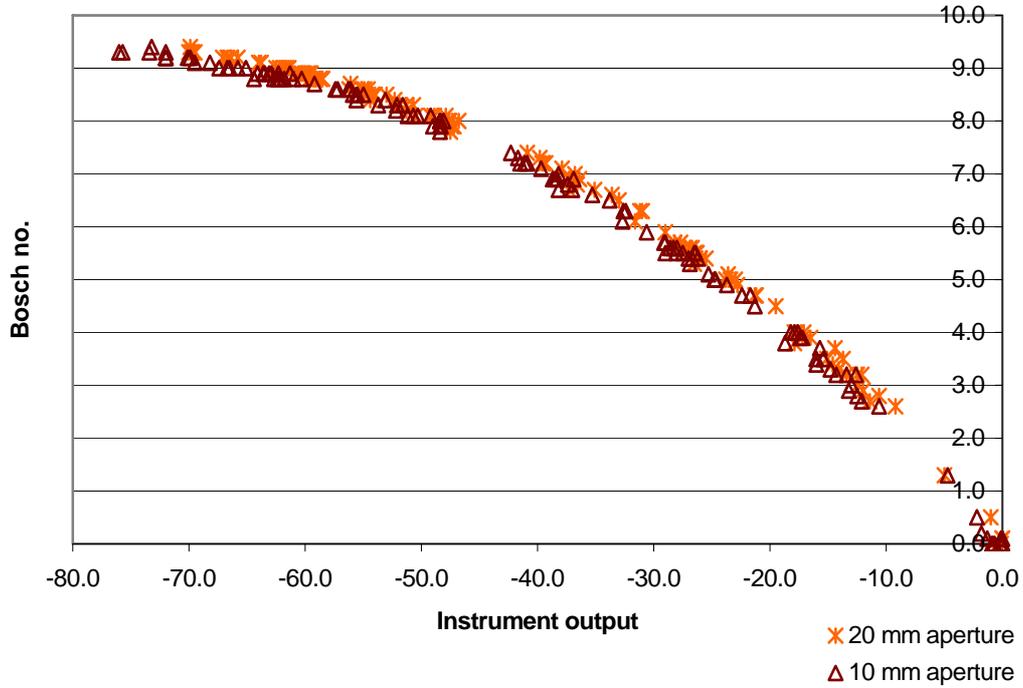


Figure 3 Chart of Bosch no. against DR-Lange for all filters with 2 aperture size

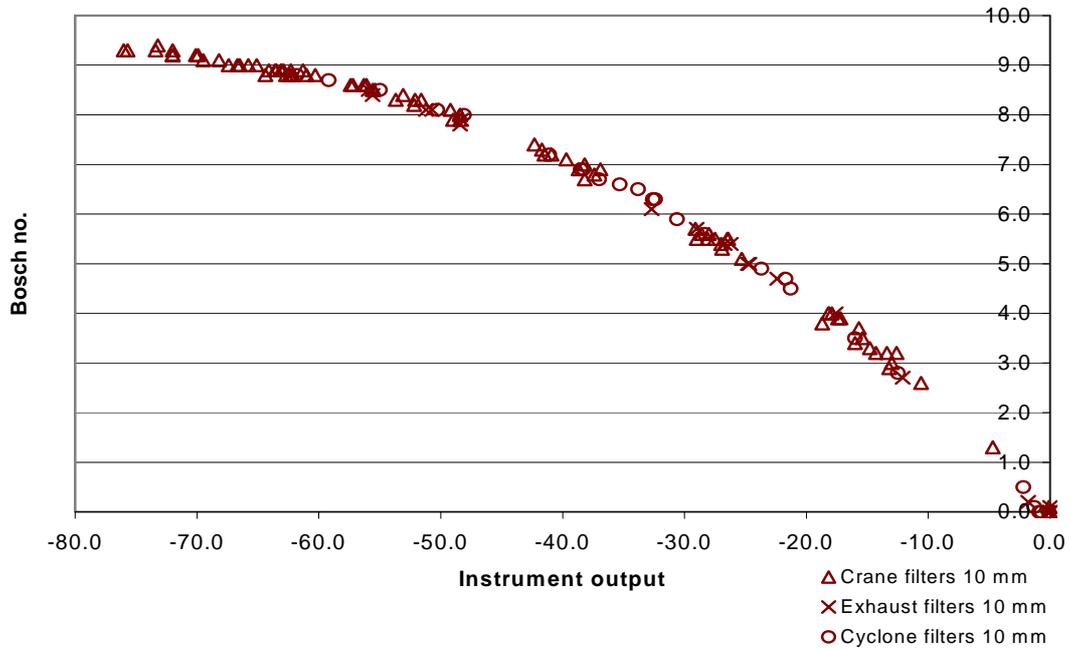


Figure 4 Chart of Bosch no. against DR-Lange for different filter sources using a 10 mm aperture

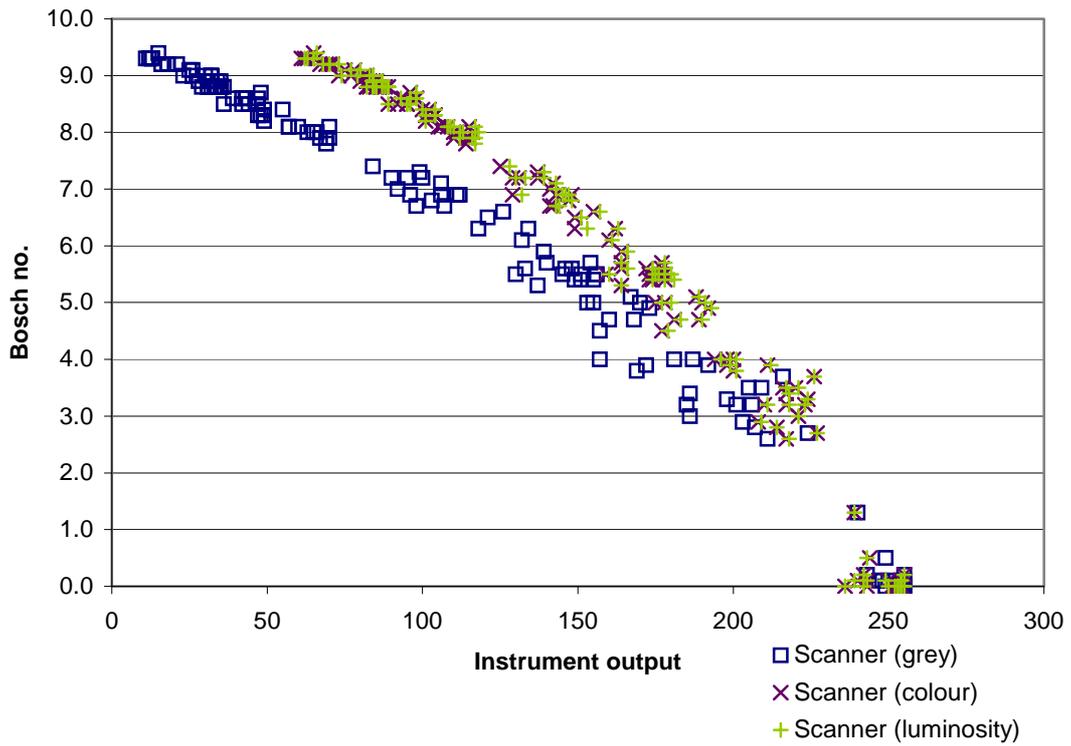


Figure 5 Chart of Bosch no. against scanner for all filters

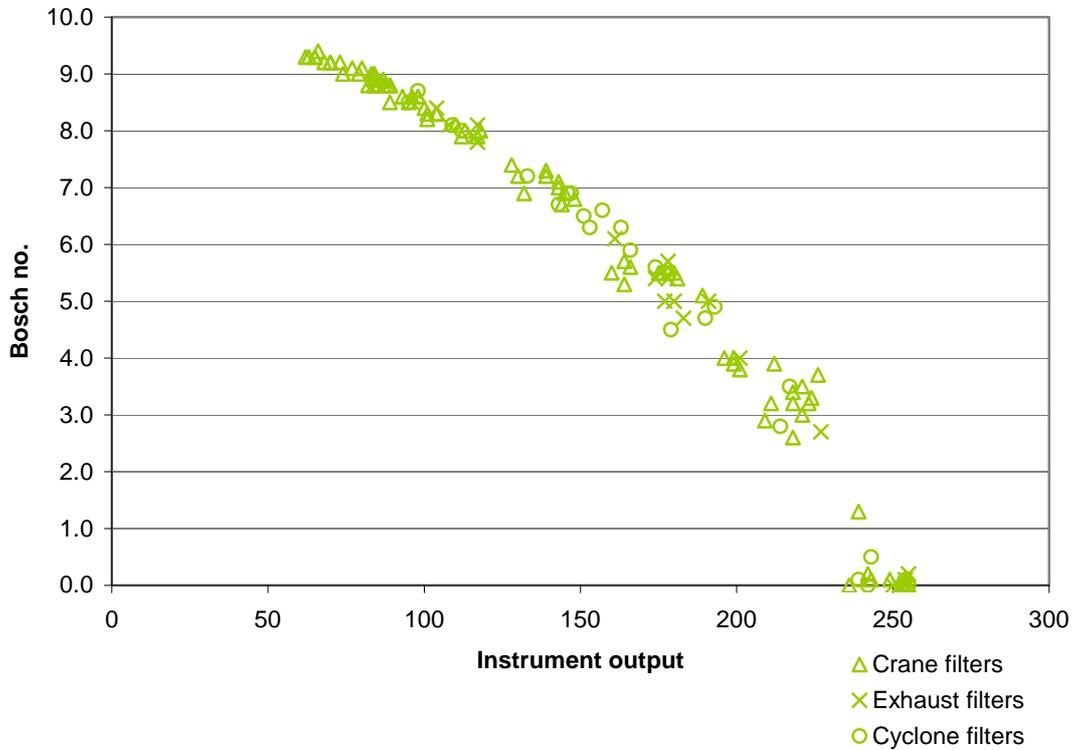


Figure 6 Chart of Bosch no. against scanner (luminosity) for different filter sources

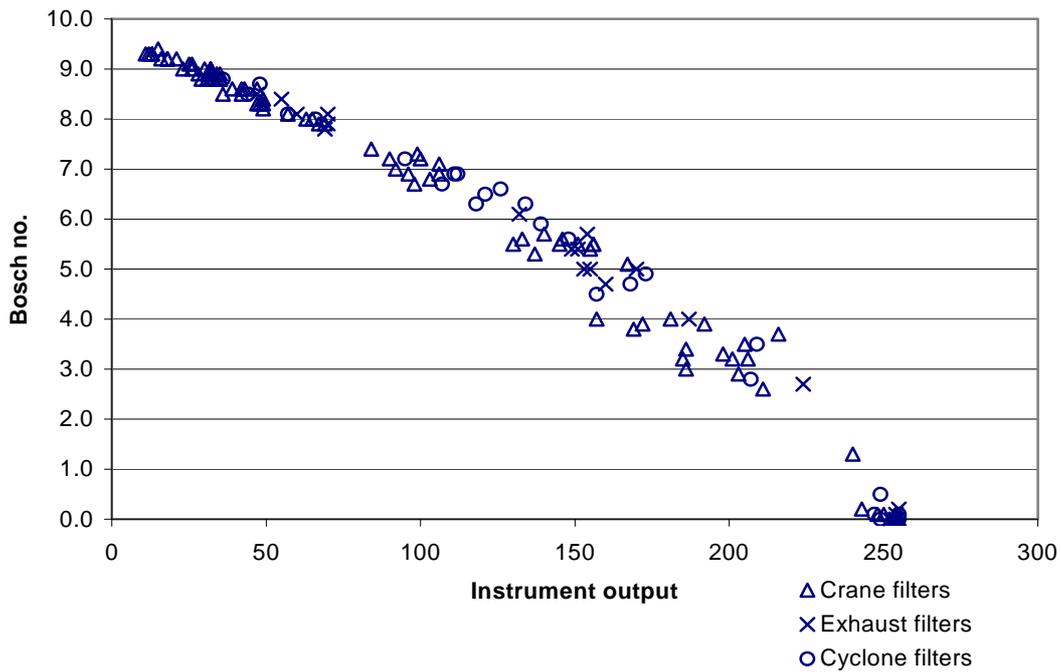


Figure 7 Chart of Bosch no. against scanner (greyscale) for different filter sources

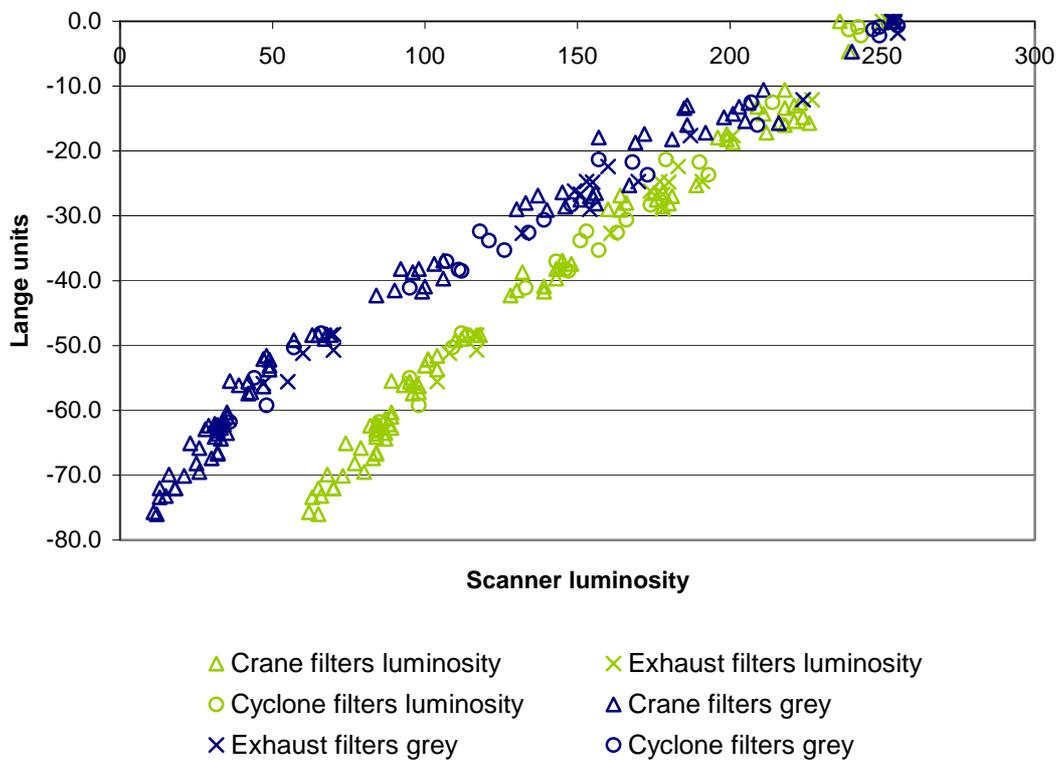


Figure 8 Chart of DR-Lange against scanner for all filters

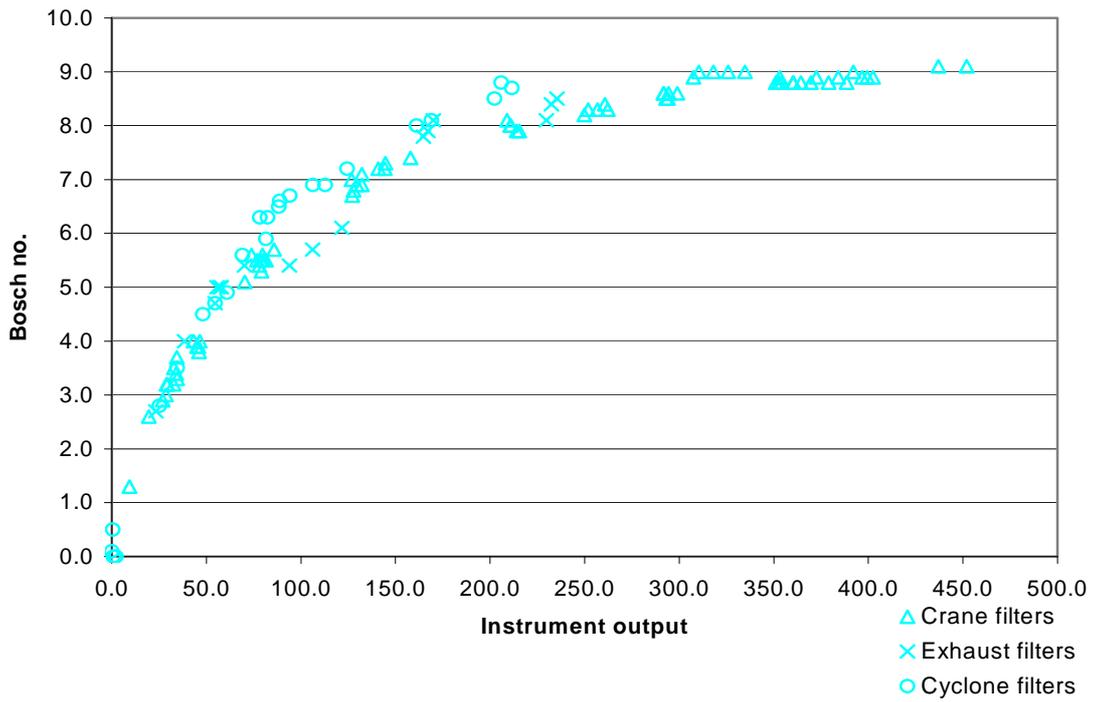


Figure 9 Chart of Bosch no. against Magee transmissometer for different filter sources

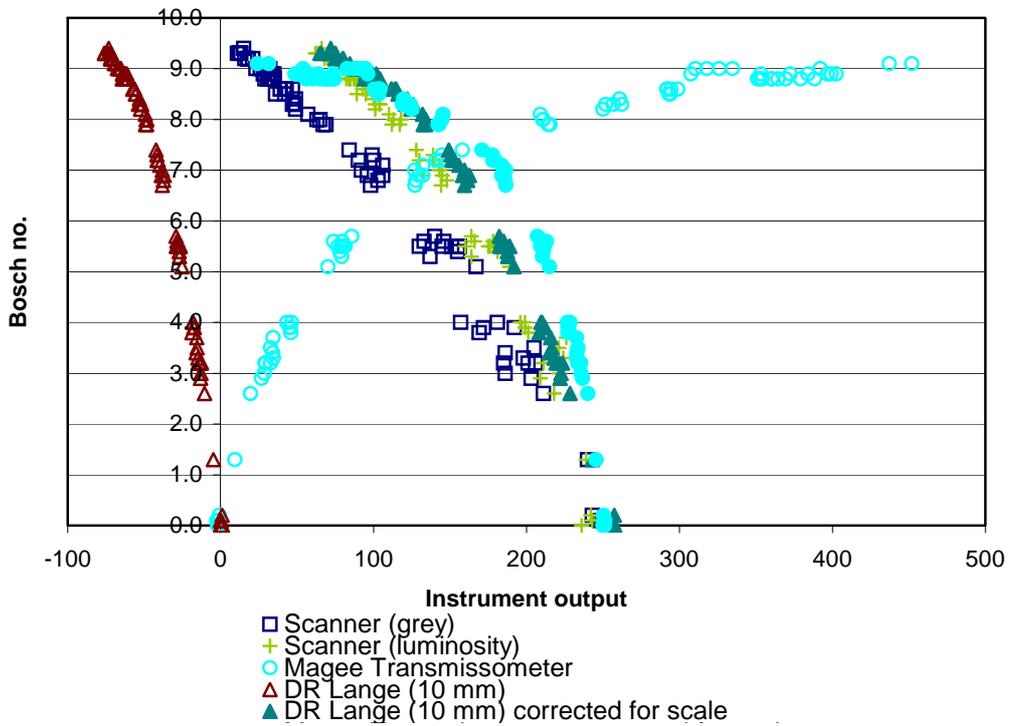


Figure 10 Chart of Bosch no. against all other light data for crane filter samples

Comparison of the results for Bosch no. and each of the other techniques (Figure 10) shows that each bears a strong relationship with it and could act as a suitable replacement. Repeatability is an issue for the scanner methods but this is less pronounced for the colour and luminosity results than for the grey scale scans (Figure 5). The greater spread of results seen on the chart will to some degree also be a consequence of the larger scale of the scanner method. Only data from the luminosity histogram is displayed in the other charts as the results from it and the colour histogram are so similar (Figure 5).

The aperture size of the DR-Lange instrument and therefore the amount of sample seen by the instrument has little effect on the result (Figure 3). This demonstrates a level of uniformity in the deposit on the filter and supports the assumption that light techniques which never view the entire sample can be reliably compared with the combustion technique which analyses the whole sample.

The chart of the results from the DR-Lange instrument against the scanner results (Figure 8) shows a near linear relationship between the two demonstrating that the two are equivalent. In fact, once the scale differences are corrected for (by multiplying by 2.5 and adding 255), the DR-Lange results overlay the scanner data quite neatly (Figure 10) with a tighter spread of results. The two methods are therefore highly comparable with a slight improvement in repeatability for the DR-Lange instrument.

The scale difference between the Magee transmissometer and the scanner can also be corrected for (by multiplying by 2 and subtracting from 500) and this has also been plotted (Figure 10) to allow for easier comparison between the different techniques and it can then be seen that there is a much greater rate of change of signal (sensitivity) for high Bosch no.s with the Magee transmissometer than with the other techniques.

Comparing Bosch no. with each of the other techniques it is clear that there is no significant difference in the relationship with Bosch no. when the sample source is varied for the scanner or DR-Lange techniques (Figures 4, 6 and 7). Assuming this to be the case for all sources, the following equations, from which the Bosch no. for a sample filter can be calculated, have been derived for the best fit curves of the above charts:

For the scanner (colour or luminosity):	$\text{Bosch no.} = -0.0002x^2 + 0.0099x + 9.3199$
For the DR-Lange DR-Lange II :	$\text{Bosch no.} = -0.0016x^2 - 0.2384x + 0.0964$

In each case x = the output from the instrument.

The equivalent equation for the greyscale scanner has not been calculated as there is too much variation in the results for such an equation to be meaningful.

Some variation between different sources can be observed at higher loadings when Bosch no is compared with results from the Magee transmissometer (Figure 9) and there are clear differences when compared with the EC content determined by combustion (Figure 11).

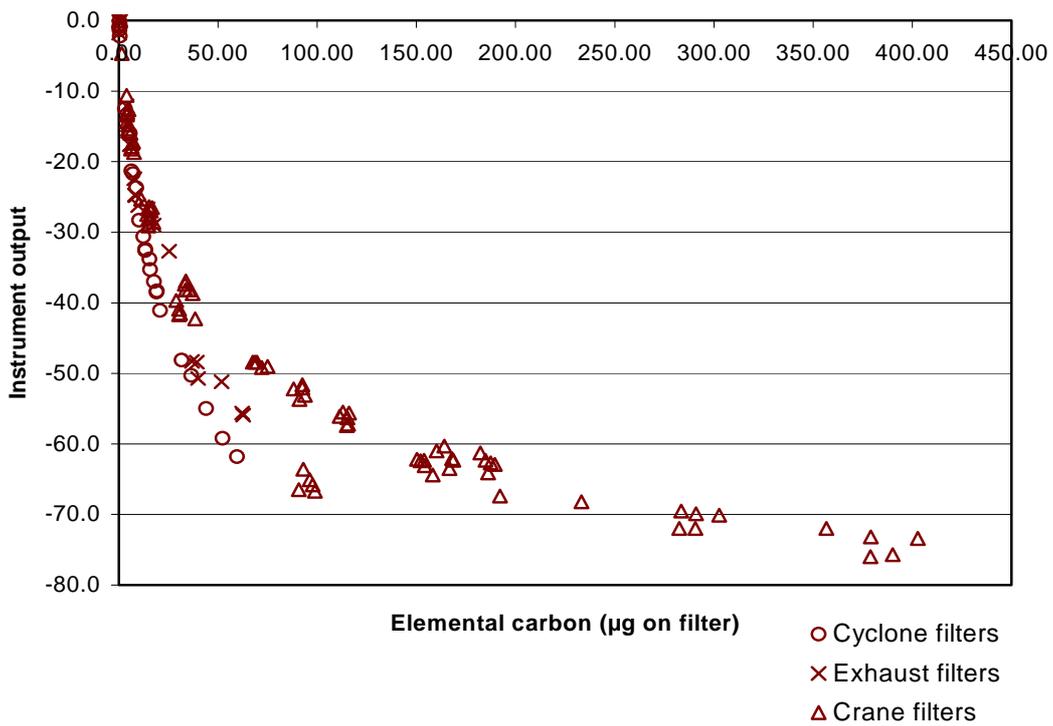


Figure 13 Chart of DR-Lange data against elemental carbon for different filter sources

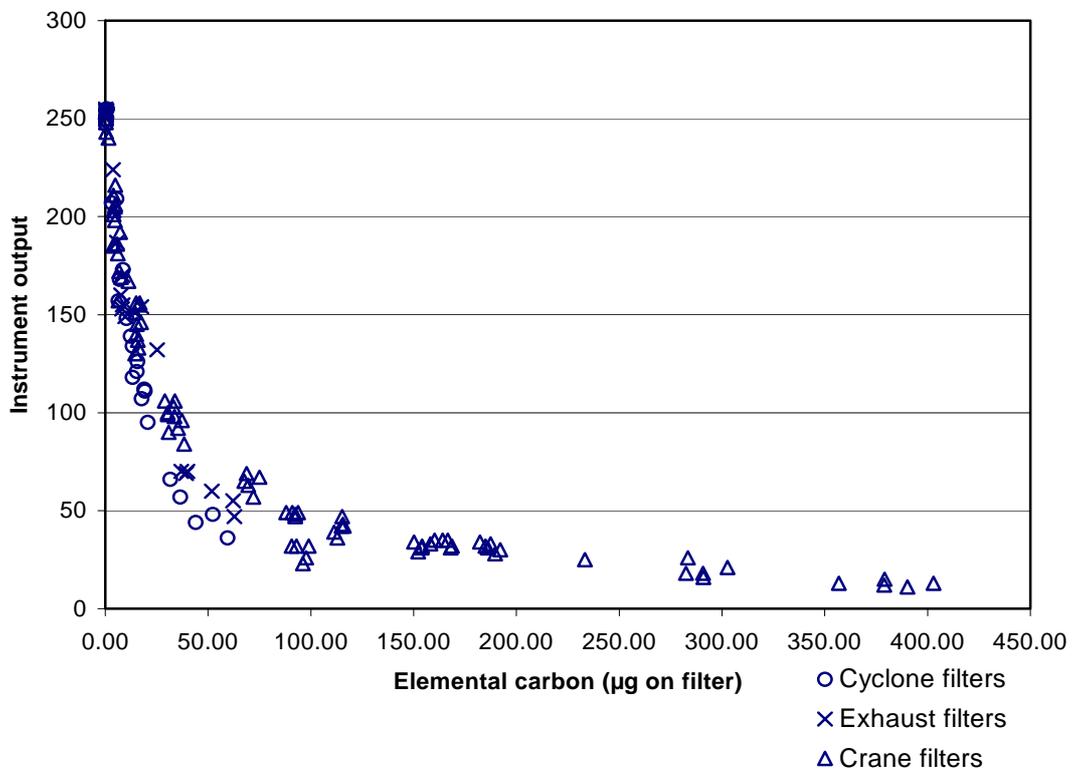


Figure 14 Chart of scanner (greyscale) data against elemental carbon for different filter sources

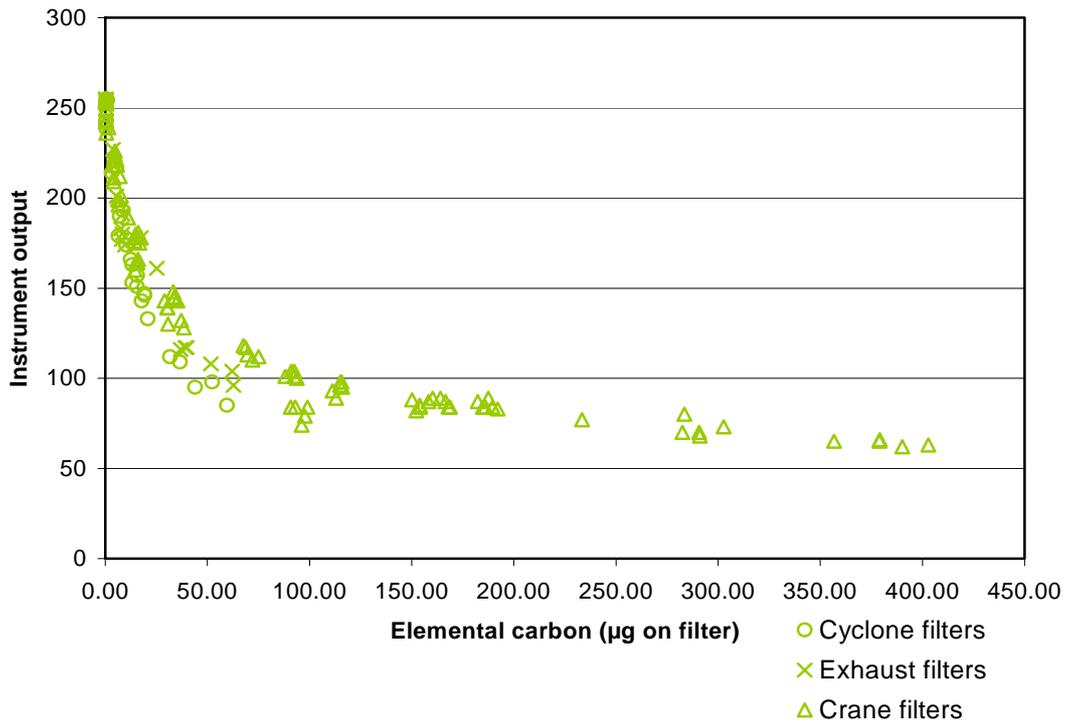


Figure 15 Chart of scanner (luminosity) data against elemental carbon for different filter sources

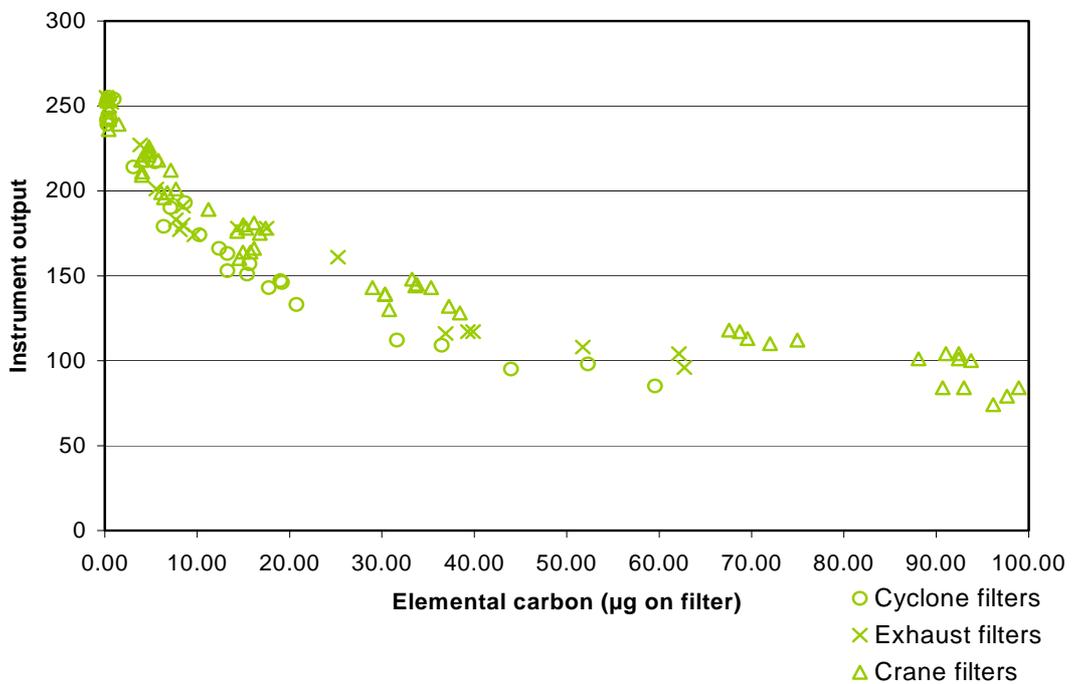


Figure 16 Segment of chart of scanner (luminosity) data against elemental carbon for different filter sources

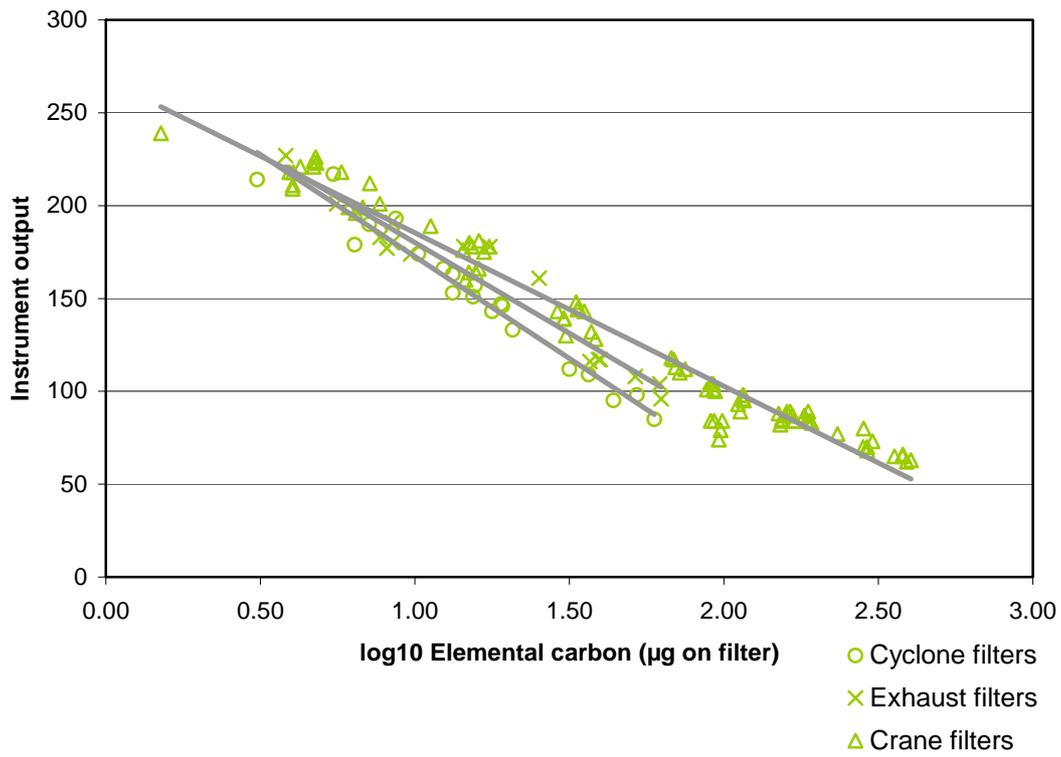


Figure 17 Chart of scanner (luminosity) data against log10 elemental carbon for different filter sources

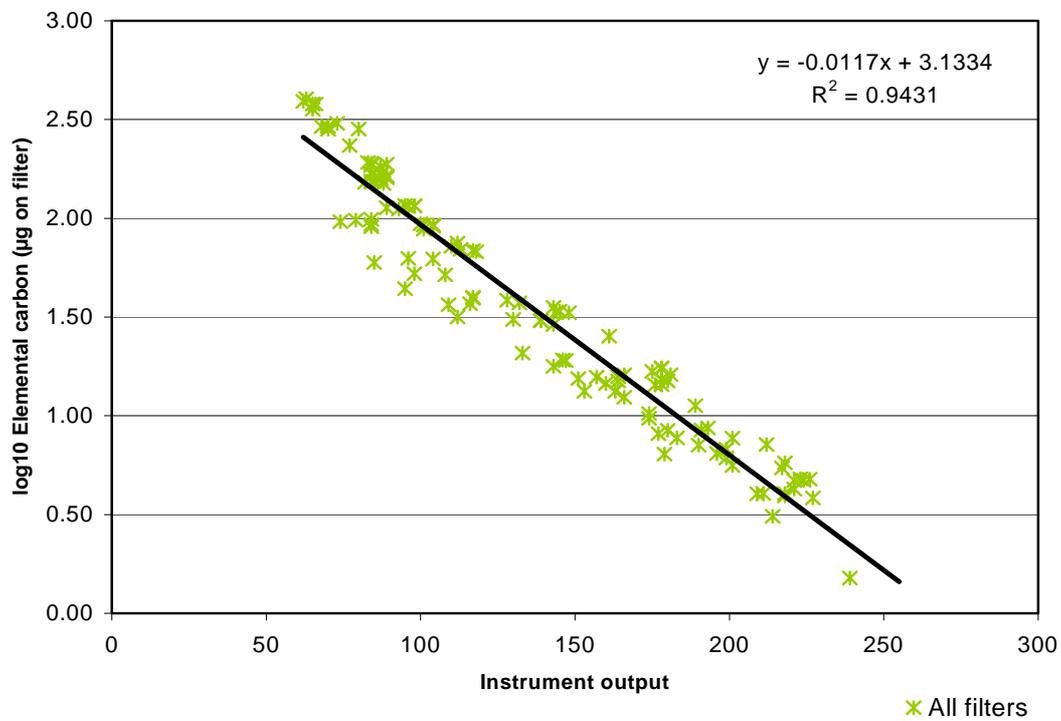


Figure 18 Chart of log 10 elemental carbon against scanner (luminosity) data

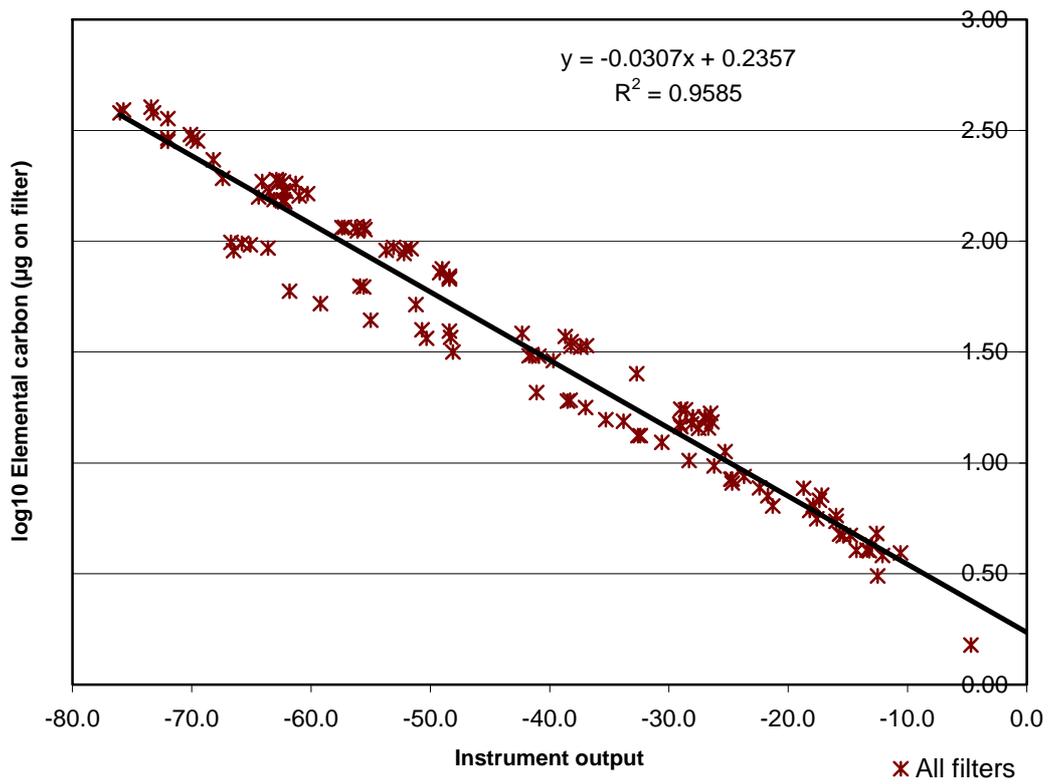


Figure 19 Chart of log 10 elemental carbon against DR-Lange data

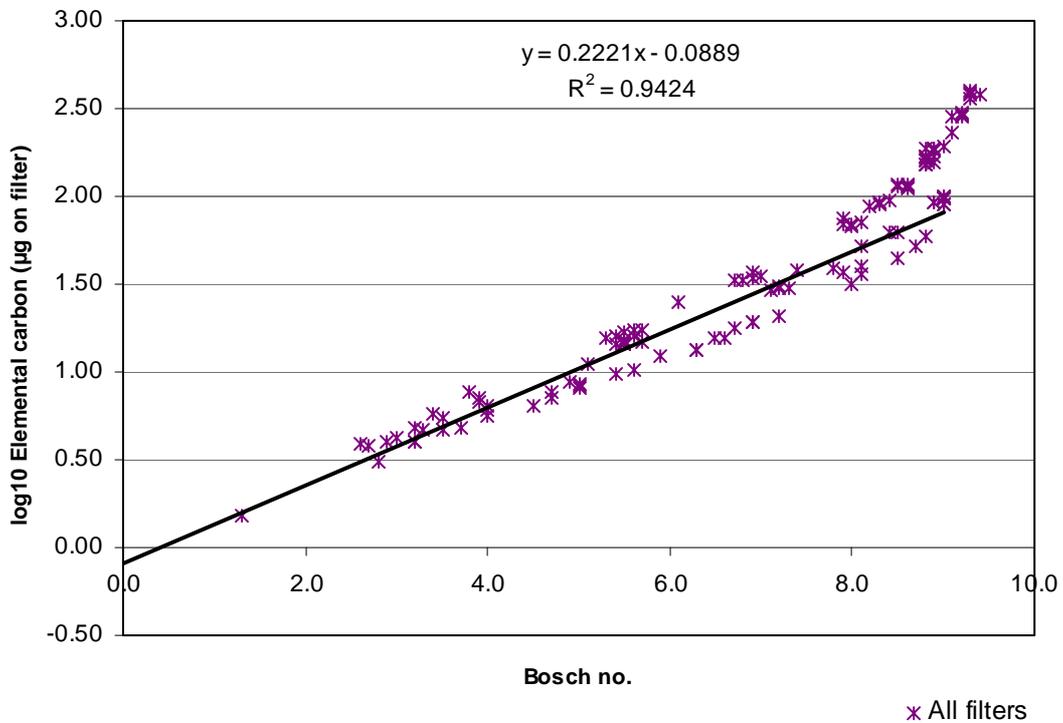


Figure 20 Chart of log 10 elemental carbon against Bosch no.

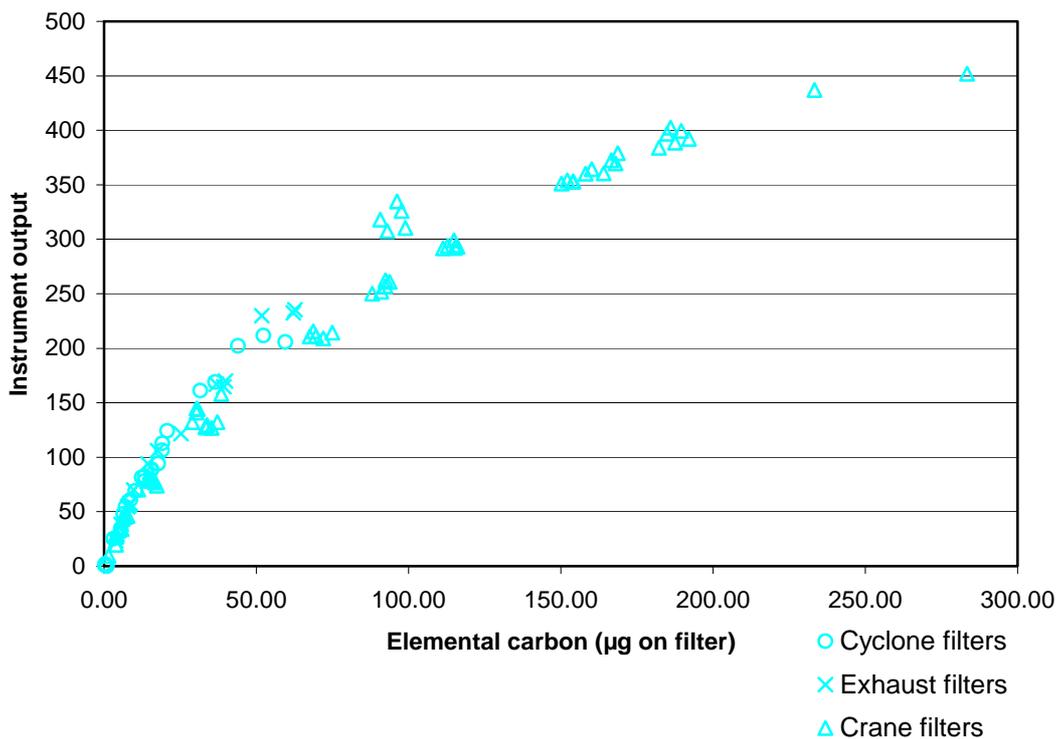


Figure 21 Chart of Magee transmissometer against elemental carbon for different filter sources

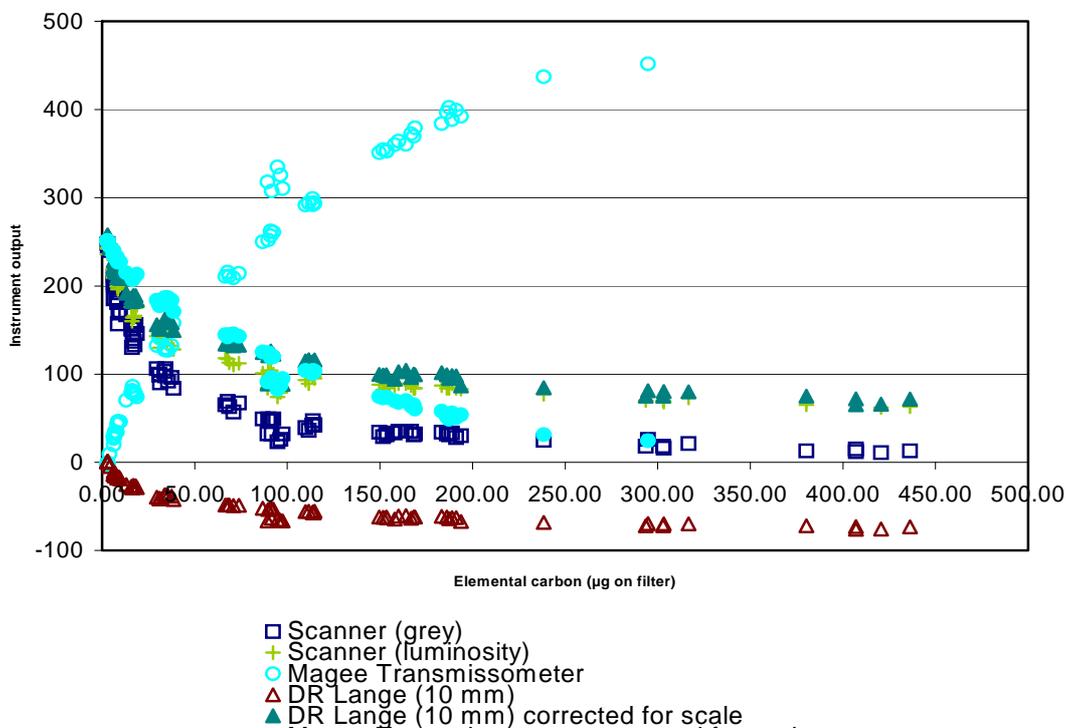


Figure 22 Chart of all light data against elemental carbon for crane filter samples

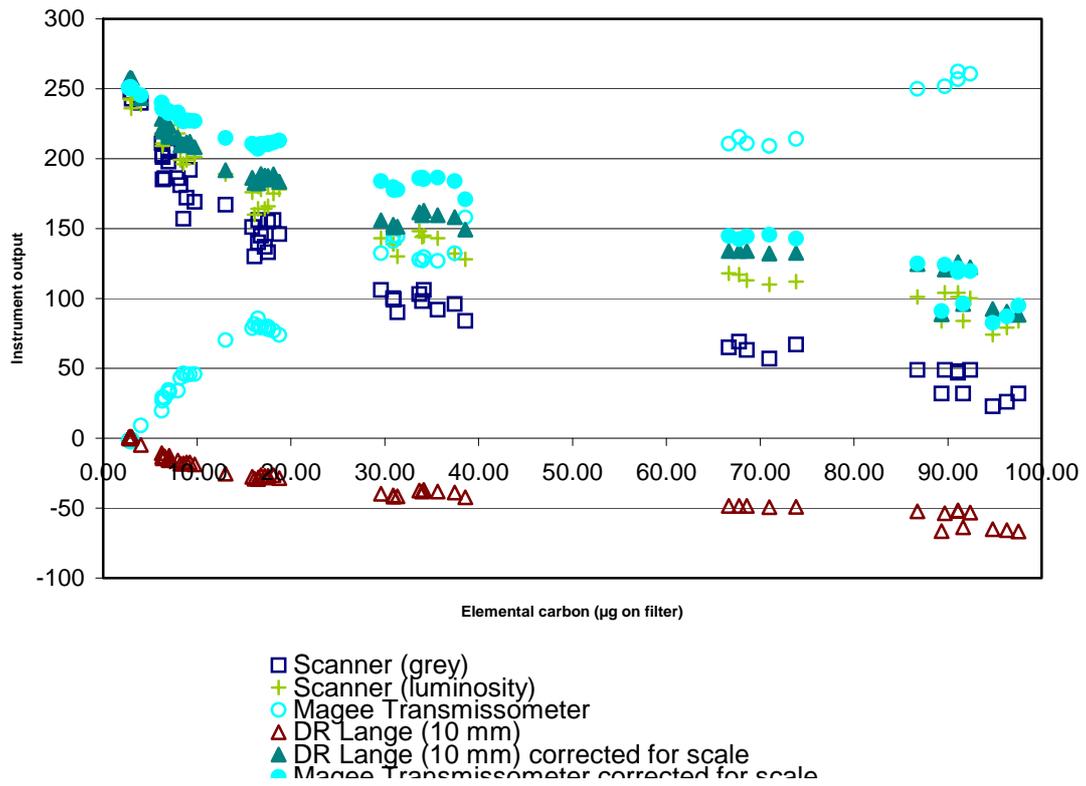


Figure 23 Segment of Chart of All light data against elemental carbon for crane filter samples

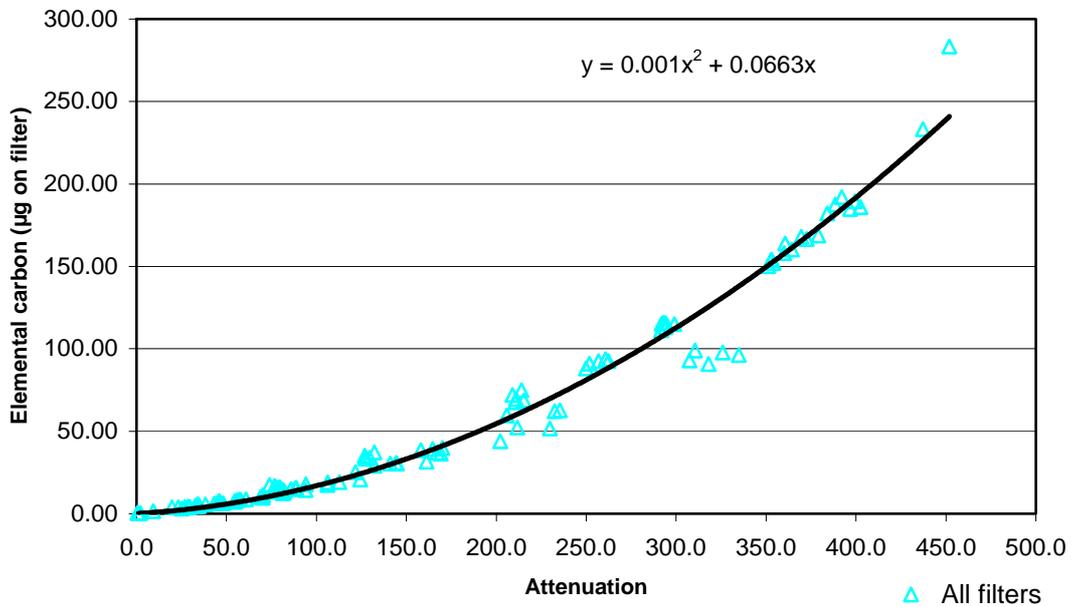


Figure 24 Chart of elemental carbon against Magee transmissometer

The above charts (Figures 12, 13, 14, 15, 21 and 22) demonstrate that the results from each light technique have a strong relationship with the loading of EC, as determined by combustion analysis with the Analytik Jena instrument. It is also clear that this relationship is dependent on the source of the filter analysed. From the decay of the relationship in each of these charts it can be seen that, apart from the Magee transmissometer, each instrument is no longer useful for determining EC above a certain loading.

Each of the light techniques (except the Magee transmissometer) has a logarithmic relationship with EC. Therefore, plotting the logarithm of EC against the instrument data gives the charts in Figures 17, 18, 19 and 20 and a linear relationship can be observed for the scanner luminosity, Bosch no. and the DR-Lange instrument.

Determining a best fit line for these charts allows the equation of this line to be used to calculate EC from a measurement from any of the light instruments. In addition, the point at which the chart departs from linearity allows an estimation of the maximum EC loading which the method can reliably quantify (Table 1). No value has been given for the DR-Lange instrument as there is no discernable point at which linearity is lost but the chart of output against elemental carbon (Figure 13) indicates that above approximately 150 µg EC the analytical variability begins to become large compared to the change due to increased loading. This represents a practical limit for the instrument.

Table 1 Formulae for calculating EC for each technique

Instrument	Best fit equation	Formula for EC	Estimated end of linearity (log ₁₀ EC)	Estimated end of linearity (EC)
	x = instrument output			
Scanner (luminosity)	$\log_{10}EC = -0.0117x + 3.1334$	$EC = 10^{(-0.117x + 3.1334)}$	2.0	100
DR-Lange	$\log_{10}EC = -0.0307x + 0.2357$	$EC = 10^{(-0.0307x + 0.2357)}$	Not determined	150
Bosch	$\log_{10}EC = 0.2221x - 0.0889$	$EC = 10^{(0.2221x - 0.0889)}$	1.8	63

Similar relationships have been derived in the past for EC and Bosch no. (D. Dabill, 2005). A chart showing the luminosity and EC data for filters from the different sources (Figures 15 and 16) shows that the relationship is dependent on the sample source and, in fact, this is what was found previously for Bosch no.

The number reported by the transmissometer 'Attenuation (ATN)' is the natural logarithm of the relative intensities of the sample filter and the blank, multiplied by 100 for arithmetic convenience.

$$ATN = 100 \times \ln(I_0/I)$$

Therefore the chart of attenuation against EC (Figure 21) is not logarithmic like the others. However, it ought to be linear like the other logarithmic charts. Although it is not, there is still a strong relationship with EC and this extends right up until the point where no transmitted light is detected. Therefore, the instrument can no longer give a result above approximately 300 µg EC on the filter.

Plotting a chart of EC against attenuation (Figure 24) gives a quadratic curve and the relationship below

$$EC = 0.001x^2 + 0.0663x$$

where x = attenuation. This equation is valid up to approximately 240 µg EC.

4.3 CONCLUSIONS

4.3.1 Replacement of the Bosch meter

The data comparing Bosch no. with the output from the scanner show that Bosch no. can be calculated from the scanner or DR-Lange result. It is therefore practicable for a site that has a calibration for EC from Bosch no. to move to either technique as a replacement and calculate EC by calculating Bosch no. from scanned luminosity or DR-Lange output first. However, this extra step adds a degree of uncertainty that could affect the accuracy of results. Since the analytical technique is non destructive there is the possibility of this being an interim procedure whilst a set of filters is gradually built up that can then be analysed by combustion to prepare a direct scanned luminosity or DR-Lange to EC calibration.

The charts in Figures 3, 5 and 9 show that the DR-Lange instrument has the best correlation with the Bosch no. and is therefore the best direct replacement.

4.3.2 Analytical range

The range of each technique has been determined as in Table 2 below

Table 2 Upper limit of the analytical range for each technique

Instrument	Maximum filter loading (µg EC)
Scanner	100
DR-Lange Micro colour II	150
Magee OT21 Transmissometer	230
Bosch meter	63

Whether these maxima represent a problem for the determination of EC on filters from real workplace environments is dependent on what concentrations of EC exist in those environments combined with the sampling protocols used.

As an example, taking the $100 \mu\text{g}/\text{m}^3$ value used in Germany, a typical 4 hour, half-shift sampling period at 2.2 litres per minute using the cyclone sampler will produce a sampled air volume of $2.2 \times 240 = 528$ litres = 0.528 m^3 and a filter loading of $100 \times 0.528 = 53 \mu\text{g}$. This is within the range of all the techniques and, as such, the analytical range would not normally be the primary criterion when selecting a technique.

4.3.3 Costs

Both the DR-Lange Micro colour II and the Magee OT21 transmissometer cost in the region of £8,000, whereas the scanner technique uses only standard office hardware and a commonly available software program.

4.3.4 Effectiveness at measuring EC

All the techniques investigated provide practical options for the measurement of DEEE as EC. The DR-Lange and Magee instruments have a higher limit at which they can accurately measure EC than the scanner technique. The scanner technique is also less precise and repeatable than these two.

If precision and range are important factors then the DR-Lange instruments and the Magee Transmissometer should be considered. For general monitoring, the scanner technique is acceptable. All techniques require calibration against EC for each site and sampling method but if a site has a Bosch no. to EC calibration the scanner and DR-Lange methods can be converted to Bosch no. and then to EC. This should be considered a temporary arrangement until a set of filters has been created as the extra step necessarily increases the uncertainty of the EC result.

5. REFERENCES

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Non destructive techniques for analysis of Diesel engine exhaust emissions (DEEE) on filters

Diesel engine exhaust emissions represent a hazard to worker health. The amount of elemental carbon (EC) is the routine measure of DEEE. Historically, an instrument, the Bosch meter, has been used in underground mines in the UK. The meter measures 'blackness' of a filter paper as a proxy for EC and HSL showed that there was a relationship between blackness and EC.

The Bosch meter is no longer available and this report details the investigations into a replacement system. The instruments tested were those that were commercially available at the time the research was undertaken: a Magee Scientific OT21 transmissometer, a DR-Lange Micro colour II and a system where a filter is scanned using a normal office scanner and then measured using a tool in the Adobe Photoshop software program.

Each has been assessed against the Bosch meter and the amount of EC as determined by two-stage combustion on an Analytik Jena elemental analyser.

The DR-Lange Micro colour II was found to be the closest like for like replacement for the Bosch meter.

When compared with EC, the Magee Scientific OT21 transmissometer was found to be the most effective instrument, having the best precision and range of analysis. The DR-Lange Micro Colour II and scanner/photoshop were found to be acceptable for normal use and each was an improvement on the Bosch meter.

The scanner/photoshop method is considerably less expensive to purchase

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