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sabre bulletin

CL:AIRE SABRE bulletins describe specific, practical aspects of research from the LINK Bioremediation Project SABRE, which aimed to develop and demonstrate the effectiveness of *in situ* enhanced anaerobic bioremediation for the treatment of chlorinated solvent DNAPL source areas. This bulletin describes the development of a two-stage site characterisation methodology to guide and inform an assessment of DNAPL distribution at the SABRE site.

Site Investigation Techniques for DNAPL Source and Plume Zone Characterisation

1. INTRODUCTION

Establishing the location of the Source Area BioREmediation (SABRE) research cell was a primary objective of the site characterisation programme. This bulletin describes the development of a two-stage site characterisation methodology that combined qualitative and quantitative data to guide and inform an assessment of dense non-aqueous phase liquid (DNAPL) distribution at the site.

DNAPL site characterisation has traditionally involved multiple phases of site investigation, characterised by rigid sampling and analysis programmes, expensive mobilisations and long decision-making timeframes (Crumbling, 2001a) , resulting in site investigations that are costly and long in duration. Here we follow the principles of an innovative framework, termed Triad (Crumbling, 2001a, 2001b; Crumbling et al., 2001, Crumbling et al. 2003), which describes a systematic approach for the characterisation and remediation of contaminated sites. The Triad approach to site characterisation focuses on three main components: a) systematic planning which is implemented with a preliminary conceptual site model from existing data. The desired outcomes are planned and decision uncertainties are evaluated; b) dynamic work strategies that focus on the need for flexibility as site characterisation progresses so that new information can guide the investigation in real-time and c) real-time measurement technologies that are critical in making dynamic work strategies possible.

Key to this approach is the selection of suitable measurement technologies, of which there are two main categories (Crumbling et al., 2003). The first category provides qualitative, dense spatial data, often with detection limits over a preset value. These methods are generally of lower cost, produce real-time data and are primarily used to identify site areas that require further investigation. Examples of such "decision-quality" methods are laser induced fluorescence (Kram et al., 2001), membrane interface probing (McAndrews et al., 2003) and cone penetrometer testing (Robertson, 1990), all of which produce data in continuous vertical profiles. Because these methods are rapid, many profiles can be generated and hence the subsurface data density is greatly improved. These qualitative results are used to guide the sampling strategy for the application of the second category of technologies that generate quantitative, precise data that have low detection limits and are analyte-specific. These methods tend to be high cost with long turnaround times that preclude on-site decision making, hence applying them to quantify rather than produce a conceptual model facilitates a key cost saving. Examples include instrumental laboratory analyses such as soil solvent extractions (Parker et al., 2004) and water analyses (USEPA, 1996). Where these two categories of measurement technologies are used in tandem, a more complete and accurate dataset is achieved without additional site mobilisations.

The aim of the site characterisation programme at the SABRE site was to delineate the DNAPL source zone rapidly and identify a location for the *in situ* research cell. The site characterisation objectives were to; a) test whether semi-quantitative measurement techniques could reliably determine geological interfaces, contaminant mass distribution and inform the initial site conceptual model; and b) quantitatively determine DNAPL source zone distribution, guided by the qualitative site conceptual model.

2. FIELD SITE DESCRIPTION

The field site area (Figure 1) is approximately 2400 m^2 and historically was occupied by a mono-chloro acetic acid (MCAA) production plant, which was active between 1963 and 1990. The MCCA process required the storage of trichloroethene (TCE), acetate and acetic anhydride in 22-44 m^3 tanks, which supplied washing facilities that occupied a 20 m^2 area. Three sumps were present below ground and were known to contain TCE throughout the operation of the plant. Contamination from spillages, accidental releases and drain/sump leakages may have occurred during the 25-27 year operational period. A previous site investigation (2002[1]) observed high concentrations (2,500 mg/kg) of TCE in the subsurface and concentrations in the groundwater approaching TCE aqueous phase solubility (1100 mg/l). Cis-1,2-dichloroethene (cDCE), vinyl chloride (VC) and ethene were present at the site,

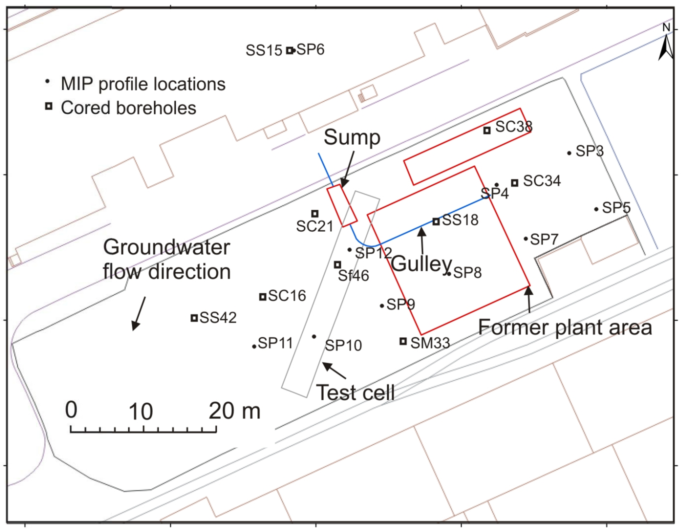

Figure 1. Site location map showing both membrane interface probe (MIP) and core positions.

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[1] Unpublished and confidential data were provided by the site owner

suggesting microbial reductive dechlorination activity, which was assumed to be enhanced by the historic release of acetate from the production plant. The detailed spatial distribution of the contaminants, and associated breakdown products, was not resolved in this early site characterisation phase.

Preliminary investigation of the geology and hydrogeology at the site prior to this study had been undertaken sporadically. The geology is indicative of a fluvial system comprising 1 m of made ground, underlain by 2-3 m of an upper, clay-rich alluvium and lower, sandy alluvium. The river terrace gravel aquifer, which is the focus for the *in situ* bioremediation studies, is located between approximately 3 m and 7 m below ground level (bgl). The fluvial sequence is underlain by the Mercia Mudstone bedrock, which approaches approximately 50 m thickness (Lelliott et al., 2009). Water levels on the site were recorded at approximately 1-2 m bgl and a south-southwest hydraulic gradient of 0.0018 was observed. Groundwater flow velocities of between 0.04 and 0.08 m/d were calculated, assuming a porosity of 25% and hydraulic conductivity values derived from slug tests at seven aquifer depths ranging from 0.14 to 26 m/d.

3. DNAPL SITE INVESTIGATION METHODS

3.1 Overview

The site characterisation plan was guided by a multidisciplinary, international consortium of researchers and environmental engineers who were concurrently working on engineering design and optimisation, treatability studies and process modelling. To achieve a dynamic work strategy during the practical implementation of the plan, the consortium was involved with decisions on a day to day basis, addressing uncertainties and receiving updates on site activities in real-time. The 3-week long dynamic work strategy was established using the methods stated in Table 1. Qualitative field data collection methodologies were chosen to provide 'decision-quality' volatile organic compound (VOC) screening and geological data over the whole site, which sequentially informed the conceptual site model. This site characterisation toolbox enabled the source zone to be located rapidly. Delineation of both vertical and horizontal contaminant distribution enhanced understanding of the spatial heterogeneity at the site and guided the next phase of site characterisation. Quantitative methodologies (core sampling and VOC sub-sampling) were employed to validate qualitative methods (membrane interface probe (MIP) and Sudan IV) and provide low detection limit data to estimate DNAPL mass.

Table 1. Overview of experimental approach.

Method	Data type	Data collected	References
ERT survey	Qualitative	Spatial data on geological interface topography and underground structures	(Chambers et al., 2002)
MIP profiling	Qualitative	Distribution of contamination	(McAndrews et al., 2003)
EC profiling	Qualitative	Geological interface depths	(McAndrews et al., 2003)
Sudan IV tests	Qualitative	DNAPL distribution	(Cohen and Mercer, 1993)
Core sampling	Quantitative	VOC, f_{oc}, geological interface data	(USEPA, 2002)

Notes:
ERT - Electrical resistivity tomography
MIP - Membrane interface probe
EC - Electrical conductivity

3.2 Geophysical Survey

Electrical resistivity tomography (ERT) provides a means of non-invasively imaging the subsurface. The rapid, high-resolution volumetric coverage that can be achieved using this technique allows preliminary assessment of ground conditions at the site-scale, which is valuable for both borehole placement decisions and delineation of heterogeneity. Resistivity data were collected using an AGI SuperSting R8 IP system, and multi-electrode surface arrays. Stainless steel electrodes were inserted to a depth of approximately 150 mm; bentonite slurry was applied to each electrode to improve the electrical contact with the ground. A dipole-dipole array configuration (a = 1.5, 3.0, 4.5, and 6.0 m and n = 1 to 8), which included a full set of reciprocal measurements for data quality assessment, was employed for data acquisition. The survey comprised measurement sets collected using orthogonal survey lines to facilitate the detection of narrow elongated features, such as foundations, which can be invisible to ERT if only one line orientation is used (Chambers et al., 2002). The maximum effective depth of investigation of the survey was approximately 12 m bgl. Limited processing of the measured data was carried out in the field using a 2D inversion code (Loke and Barker, 1996a) in order to assess data quality and verify that datasets were complete. Final data processing and model generation was carried out using a 3D smoothness constrained nonlinear least-squares algorithm (Loke and Barker, 1996b), in which the forward problem was solved using a finite difference method.

3.3 MIP/EC Survey

The combined MIP and EC instrument provides real-time results which are near-continuous with depth and enable a qualitative indication of the location of geological formations and an indication of the vertical heterogeneity of VOC contaminants. The process is rapid, does not require laboratory analyses and provides decision-quality data. The truck-mounted instrument was used to generate vertical profiles at 12 locations. The details of the profiling technique are described in full by McAndrews et al. (2003). Briefly, the EC probe measures conductivity variation within the deposit, from which the lithological properties can be inferred. The MIP probe generates a qualitative vertical profile of contaminant mass by analysing VOC vapour which is generated by a heated membrane at the bottom of the probe and transported in nitrogen carrier gas to a GC-PID (gas chromatograph- photo ionisation detector), FID (flame ionisation detector) and a DELCD (dry electrolytic conductivity detector). The response is measured over a range of 0 to 5 volts. An adhoc site-specific correlation of voltage versus concentration of MIP gas flow (data not shown) suggested that a voltage range over 4 volts was indicative of gaseous TCE concentrations >100 ppm, the magnitude of which suggests the presence of NAPL. The MIP response depends on porosity, water content, probe temperature and sorption and hence a rigorous calibration is difficult. The detection limit is higher than traditional analytical methods, however when characterising a DNAPL source zone, decisions are made on high concentration data, rather than on lower concentrations characteristic of the plume zone.

3.4 Core Recovery and Sampling

3.4.1 Drilling

A sonic drilling technique was selected to provide depth-specific, minimally disturbed, reliable core recovery in the unconsolidated lithology. Core recovered in this way enabled accurate geological interfaces (Lelliott et al., 2009) and sample positions to be reported. Furthermore, the cores were enclosed in 100 mm liners, which enabled sub-sampling with negligible VOC losses.

3.4.2 VOC Sampling

Samples were recovered for VOC analysis to provide definitive analyte-specific concentrations. Sub-sampling was undertaken immediately after the core was recovered and liner split open. Samples were prepared in an on-site laboratory according to USEPA Method 8265 (USEPA, 2002). Two samples of 0.5 and 5 g of aquifer material were taken at each location to ensure that analysis would be within the dynamic range of the laboratory instruments. The aquifer samples were added to headspace vials with 3 g of sodium sulphate, internal standards and 10 ml of deionised water. The samples were refrigerated at 4°C prior to transfer to the laboratory. Blanks were taken at a frequency of 1 in 20 and duplicates at 1 per borehole. Samples were cooled and transported to a commercial laboratory where they were analysed by GC-FID. Detection limits were 0.1 mg/kg for TCE, cDCE and VC.

3.4.3 Sudan IV testing

Sudan IV tests provide an instant identification of whether NAPL is present in a sample. This low cost, simple technique provided a check on laboratory analyses and MIP probe results and enabled on-site decisions with regard to source zone extent. Samples (~10 g) were shaken with ~0.01 g of Sudan IV dye and ~5 ml water following the methodology of Cohen and Mercer (1993). Where positive, the sample will be evenly dyed red, because individual globules of dyed NAPL will be distributed throughout the sample following sample agitation.

3.4.4 DNAPL distribution

Vertical profiles of VOCs were interpreted from the combined qualitative and quantitative measurement technologies. To determine the DNAPL source zone distribution, a method was required that discriminated between the dissolved phase and DNAPL. Once the proportion (expressed as a percentage) of the vertical profile that contained DNAPL could be determined, a spatial plot of DNAPL distribution across the site could be generated. This DNAPL percentage occupancy was calculated for MIP probe results by assigning vertical DNAPL zones to cores where PID values were between 4 and 5 volts. For the quantitative VOC core data, DNAPL presence was determined by both results from Sudan IV tests and also from soil VOC results that indicated that DNAPL was present. The soil concentration that was indicative of DNAPL presence, was calculated using partitioning calculations (Feenstra et al., 1991) as shown in Equation 1,

$$C_{sw} = \frac{(C_s B_d)}{(K_d B_d + \phi_w + H * \phi_a)} \qquad \text{Equation 1}$$

where C_{sw} is soil water concentration, B_d is bulk density, C_s is total soil concentration, K_d is partitioning coefficient, $ø_w$ is water-filled porosity, $ø_a$ is air-filled porosity, H is Henry's constant. A "DNAPL threshold" value of 622 mg/kg was calculated for TCE (Parameters: $ø_w = 0.25$, $C_{sw} = 1100$ mg/l, $B_d = 1600$ kg/m^3 and $K_d = 0.57$ ml/g. Saturated conditions were assumed and hence $H^*ø_a$ is equal to zero).

4. RESULTS AND DISCUSSION

4.1 Geology

Geological data were provided successively by three methodologies including an ERT survey, MIP profiles and core data. The ERT survey

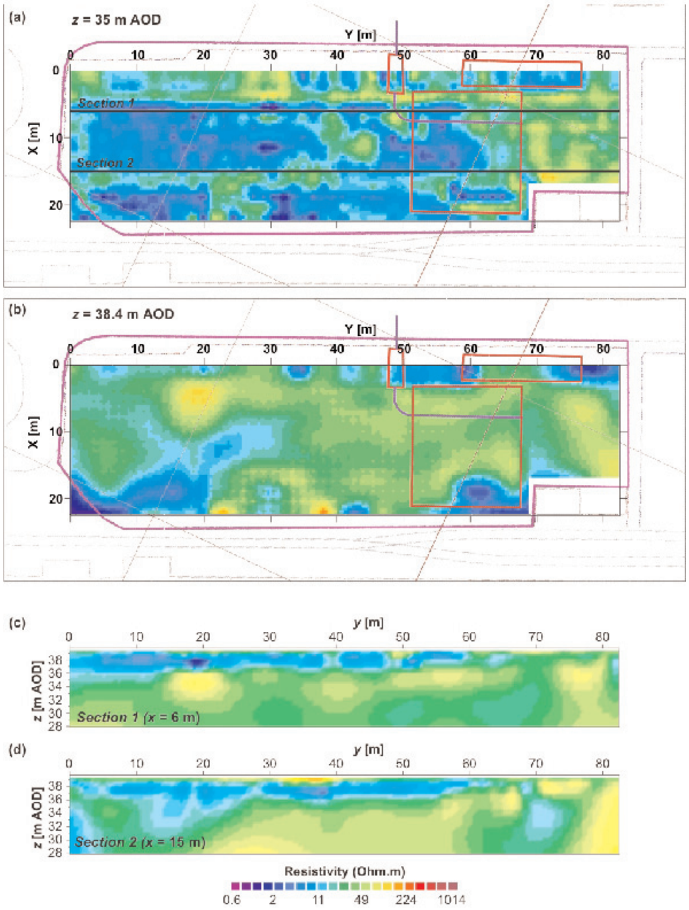

Figure 2. Resistivity images showing a) plan view at 35 m aod, b) plan view at 38.4 m aod and depth 2-D profiles at Section 1 (c) and Section 2 (d), the locations of which are shown on image a).

(Figure 2) was originally designed to determine interface topology at each geological interface to a depth of 6 m. However, high clay content of the various lithologies at the SABRE site decreased the differential resistivity variation between interfaces and hence compromised the use of this technique for the lower river terrace gravel-mudstone interface. The green-blue resistivity variation in Figure 2c and d at around 36 m above Ordnance Datum (m AOD) is likely to represent the topography of the alluvium-river terrace gravel interface. The interface is mildly undulating in the western part of the site, but the resistivity interface disappears in the easternmost portion of the site. It is hypothesised that the composition of the alluvium is more similar to the river terrace gravels in the eastern zone, hence the resistivity variation is lowered, concealing the river terrace gravel-alluvium interface. This change is visible in Figure 2a in which higher resistivity is coloured green and represents lower clay content, whilst lower resistivity is coloured blue and depicts the higher clay content of the alluvium layers. The ERT survey was additionally instrumental in locating the positions of subsurface foundations and concrete sumps (Figure 2a); an observation that was highly influential in positioning boreholes.

Qualitative geological interface data were provided by the EC sensor on the MIP probe. High EC values represent deposits exhibiting greater clay content (i.e. the upper and lower alluvium and mudstone) and hence the alluvium-river terrace gravel and river terrace gravel-mudstone interfaces could be differentiated. The interpreted geological interfaces are presented in Figure 3. The 6 m depth profiles of all the cores are relatively consistent, displaying relatively low spatial variation over the 30 m × 80 m site.

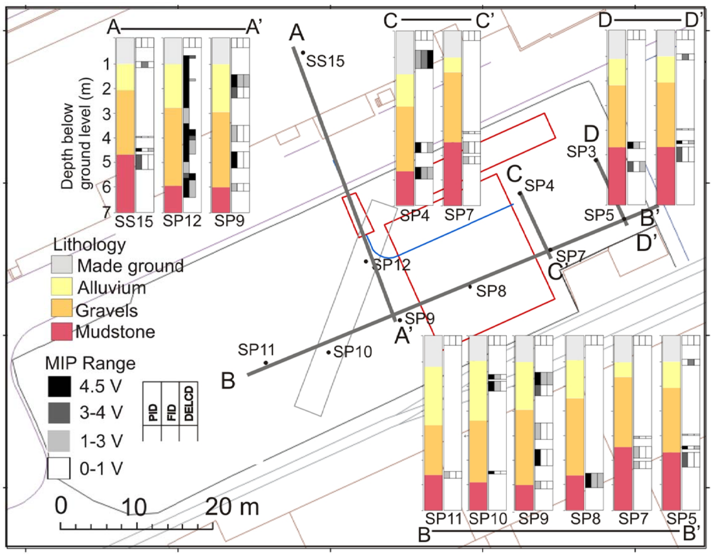

Figure 3. Geological interfaces and contaminant distribution inferred from EC and MIP measurements.

4.2 Site-wide Contaminant Distribution

Thirteen MIP profiles were drilled in an even distribution across the site. At each location, continuous vertical data for high concentration VOC presence was recorded to a depth of 7 m (Figure 3). The highest VOC response was recorded at SP12, located adjacent to a sump drain. At this location, high concentration PID responses were evident throughout the made ground, alluvium, river terrace gravels and also within the underlying mudstone. This MIP data indicated that DNAPL was present throughout the profile depth and provided the primary evidence for the location of the source zone. Positive MIP responses were observed in the alluvium at numerous locations (SP5, SP9 and SP10) and in the river terrace gravels at all locations suggesting possible further contaminant sources or lateral DNAPL transport. High concentration zones were evident immediately above the mudstone in positions other than SP12, including SP3, SP4, SP8 and SS15. It is possible that these high concentration responses reflect the lateral movement of DNAPL on the mudstone surface. Whilst these results cannot be used to infer the presence or absence of DNAPL, they do provide an outline of the likely source zone. The scale of vertical heterogeneity observed by MIP profiling is a function of the instrument carryover and probe penetration speed, however variation on the order of 0.1 to 0.5 m was apparent.

Eight cores were drilled across the site as shown in Figure 1 to quantify contaminant mass. The cores were positioned to avoid the possible test cell location and to decrease uncertainty between high concentration MIP profiles. Cores were sampled for VOCs at 0.5 m intervals; a sample density that was expected to coincide with the major contaminated zones.

Six of the seven cores located within the site area, recorded TCE concentrations over 622 mg/kg (SC16, SS18, SC21, SM33, SC34 and SC38), indicating DNAPL presence (Figure 4). Confirmation of DNAPL was provided by the positive dye test results recorded for samples over 622 mg/kg. Within these six cores, DNAPL was distributed sporadically throughout the alluvial and river terrace gravel deposits, with the exception of SS18 which recorded almost continuous DNAPL presence throughout the river terrace gravels. DNAPL was present at most locations as residual saturation rather than pooled DNAPL. Values of non-wetting phase saturation within the gravels of SS18 ranged from 0.44% to 3.35%, and near the gravel-mudstone interface, DNAPL saturation values in SS18, SC21, SC34 and SM33 were 3.09%, 1.77%, 0.71% and 12.16% respectively. These results suggest that DNAPL migrated downwards from the near-surface, through the aquifer leaving residual DNAPL trapped both within the lower permeability alluvium and higher permeability gravels. At the base of the gravels, downward migration was partially or fully prevented by the underlying capillary barrier (low permeability mudstone) and at limited sampling locations higher DNAPL saturations indicative of pooled DNAPL were observed. One core was located upgradient of the site (SS15) and was observed to be relatively clean. TCE daughter products, cDCE and VC, were present in all cores on the experimental site (Figure 4) suggesting that reductive dechlorination was occurring prior to the project. This is unsurprising given that acetate was released to the subsurface with the TCE and hence would have acted as an electron donor.

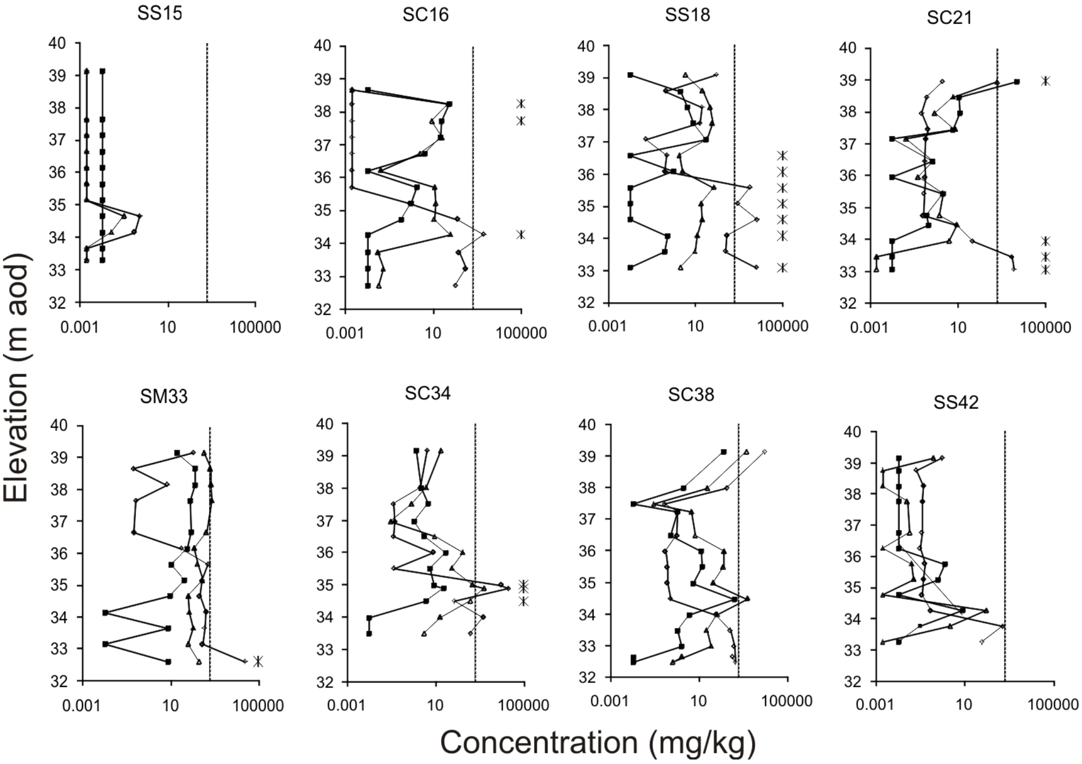

Figure 4. VOC core sample data including TCE (diamonds), cDCE (squares) and VC (triangles). ∗ denotes positive Sudan IV test.

A combination of MIP results and VOC core analysis results (Figure 5) indicate that the source zone has two hotspots, one coincident with the drain to the sump and one within the perimeter of the former plant area. DNAPL is also present at the base of the gravel sequence to the southeast of the western hotspot and in an area to the east of the main source zone. The position of the SABRE experimental cell was guided by the resulting DNAPL mass distribution, and was placed parallel to the direction of groundwater flow with the upgradient portion of the cell coincident with the DNAPL source zone and the downgradient portion of the cell located within the dissolved-plume (Figure 5).

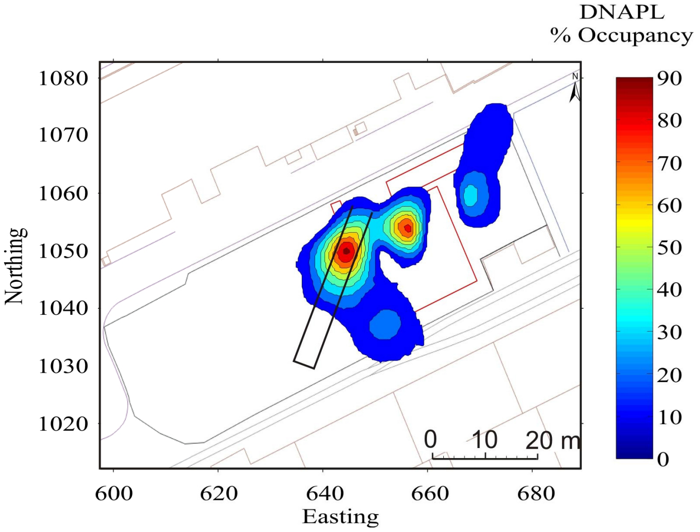

Figure 5. Plot of DNAPL percentage occupancy within the shallow aquifer, defining extent of DNAPL source zone. Outline of chosen cell position in black.

4.3 Comparison of Measurement Techniques

Following the reconnaissance phase investigation, a study of the source zone comprised high resolution sampling of core material. These core samples (core SF46) provided an opportunity to compare quantitative data from VOC sub-sampling with qualitative data from a MIP profile (SP12). Figure 6 shows a composite plot that presents the geological interface depths from core logs and EC profiles and a contaminant profile determined from MIP profiling, Sudan IV testing and VOC sampling.

The geological interface depths determined from the MIP and core profile indicate reasonable, but not exact correspondence, which was partly due to poor recovery within the upper 3 m. It is suggested that the recovery and hence accuracy of geological interfaces were compromised over this depth because of coarse-grade gravels and pebbles within the made ground that became lodged within the core liner. In comparison, the geological interfaces determined using the EC sensor on the MIP probe, were assumed to be largely unaffected by drilling, but are subject to an interpretation of the conductivity. Whilst neither technique is absolute, EC provides a rapid method to map the geological interfaces and is particularly powerful when validated by core data.

The contaminant distribution determined via the two methodologies show overall agreement. The MIP profile (PID) and quantitative VOC analyses show positive (4-5 volts) or high concentrations (above 622 mg/kg) between 2 m and 6 m bgl, indicative of DNAPL presence. This is confirmed by positive Sudan IV test results in the same depth region. Slightly lower concentrations were identified at 3.3 m and 5.3 m bgl in the MIP profile, which were coincident with a decrease in

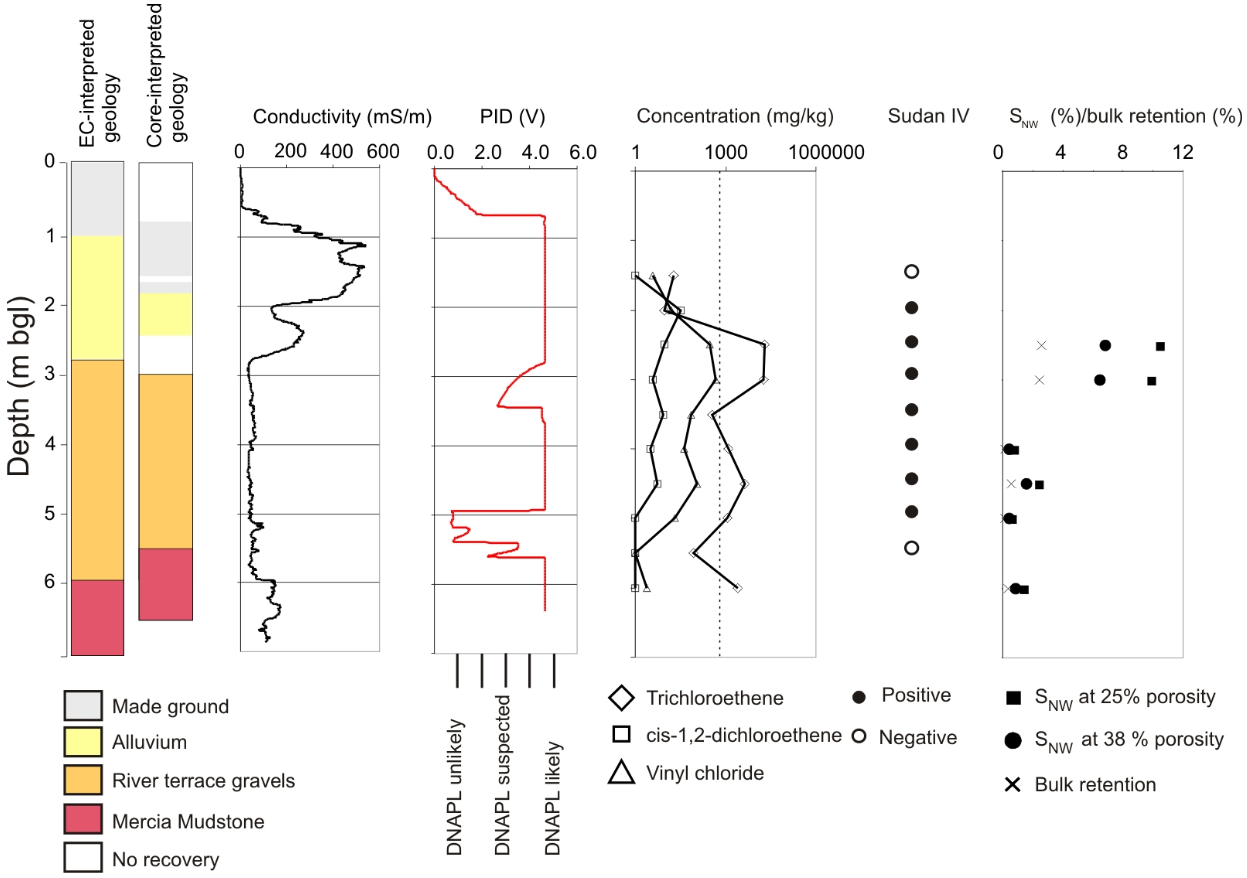

Figure 6. Comparison of qualitative and quantitative techniques for adjacent locations SP12 (EC-inferred geology and PID-inferred contaminant distribution) and SF46 (VOC analysis data, Sudan IV data and non-wetting phase saturation results.

the quantitative VOC analysis results. There was less agreement where residual saturation values indicated a grossly contaminated zone at around 2.5 m bgl (Residual saturation; 10.4%, using a porosity of 0.25); this zone could not be resolved by the MIP profile because the response was off-scale for much of the profile and hence insensitive to the increase in VOC mass. These results validate the use of MIP technology to differentiate between areas that are clean and those that are contaminated with dissolved and DNAPL-phase liquids.

5. CONCLUSIONS

The DNAPL source zone at the SABRE site was characterised using a two-stage site investigation strategy, based on the Triad methodology. The suite of on-site, real-time DNAPL characterisation tools were complimentary to the laboratory VOC results and provided robust decision-making data that informed and guided the site investigation. The combined use of quantitative and qualitative data provided the optimum balance of data density versus data quality, thereby reducing uncertainty in spatial heterogeneity. The decision to locate the *in situ* research cell was informed by both DNAPL distribution and hydraulic data.

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