Programme Area: Bioenergy

Project: Characterisation of Feedstocks

Title: D2 Methodology and Sampling Consistency Report; Database Design

Abstract:

The primary objective of this 2015/16/17 Project was to provide an understanding of UK produced biomass properties, how these vary and what causes this variability.
This deliverable comprises a set of briefing slides used to brief the ETI on the experimental hypotheses, approach, methodology (including with respect to health and safety) for the second phase (2016/17) of the Characterisation of Feedstocks project. The purpose of this deliverable was to enable the Project delivery team to demonstrate to the ETI that the planned approaches would enable the experimental hypotheses to be robustly tested and meet the ETI's needs.

Context:

The Characterisation of Feedstocks project provides an understanding of UK produced 2nd generation energy biomass properties, how these vary and what causes this variability. In this project, several types of UK-grown biomass, produced under varying conditions, were sampled. The biomass sampled included Miscanthus, Short Rotation Forestry (SRF) and Short Rotation Coppice (SRC) Willow. The samples were tested to an agreed schedule in an accredited laboratory. The results were analysed against the planting, growing, harvesting and storage conditions (i.e. the provenance) to understand what impacts different production and storage methods have on the biomass properties. The main outcome of this project is a better understanding of the key characteristics of UK biomass feedstocks (focusing on second generation) relevant in downstream energy conversion applications, and how these characteristics vary by provenance.

ETI Project Code: BI2010

Project Title: Characterisation of Biomass Feedstocks

Deliverable title: Methodology and sampling consistency report; database design

Reference: D2

Participant lead: Forest Research

Other participant: E.ON Technologies (Ratcliffe) Ltd.

Submission date: 10th June 2015

Version number: v2

Contents

Executive Summary

The purpose of this deliverable is to describe the rationale for both the experimental design and the decisions taken. Deliverable 2 also provides the technical details to enable the ETI to check the technical validity of the project, in particular that feedstocks and soils will be representatively sampled, sample integrity will be maintained, approved laboratory methods will be used, and that both the statistical analyses and database design will be fit for purpose.

Sample integrity is assured by using a small team of trained staff to complete field and laboratory sampling using agreed written protocols that address sampling consistency, condition and labelling. Bespoke protocols, which have been designed to take account of particular contract requirements, are signed off by both the Project Manager and Chief Technologist. The degree of replication varies according to the level of variation being considered and is described for variation due to site factors; variation among and also within sites; and variation within lab samples. Where relevant, EN standards informed our choice of the number of replicates. Varietal differences among clones of willow and poplar have been addressed to ensure that they do not confound the results and make it difficult to separate out the influence of soil type, climate and provenance. There is also variation within a given plant, yet with the exception of *Miscanthus*, it is not possible to take samples of the entire above-ground material, so we have considered which selection of material is most representative of commercially available feedstock. The Technology Centre of E.ON Technologies (Ratcliffe) Ltd. has a UKAS accredited facility and all analyses follow UKAS accredited methodologies.

These methodologies will be followed carefully and consistently to complete the field sample collection and subsequent chemical and physical analysis of freshly harvested and stored feedstocks. The chemical and physical properties of the feedstocks plus associated site characteristics and provenance information will be used to populate a database that will serve two purposes: statistical analysis to quantify feedstock variation and establish relationships between site factors/provenance and feedstock characteristics; and input data to the linked contract addressing the techno-economic assessment of biomass pre-treatment technologies. An intermediate database is scheduled for delivery via ETI to the linked contract in July and a completed database in November.

At the broadest level, Analysis of Variance (ANOVA) will be used to test for impacts of different production and storage methods. At the finer scale, multivariate statistical techniques will be used to identify, for example, relationships between the principal mineral contents of the soil and the chemical and physical properties of the feedstock. Environmental, management and operational variables will also be used in an attempt to identify differences in feedstock characteristics. The main statistical techniques used will be principal component analysis and redundancy analysis.

Database design has been kept simple (as Excel spreadsheets linked by a unique identifier for each field sample) in the interim until there is an opportunity to discuss it formally with the contractor who will use our datasets for modelling.

Introduction

The purpose of this deliverable is to enable the ETI, its reviewer(s) and its members to understand the rationale for both the experimental design selected and the decisions taken when the project team was faced with the reality of commercial operations and the time available. Deliverable 2 also provides the technical details to enable the ETI to check the technical validity of the project, in particular that feedstock and soils will be representatively sampled, that sample integrity will be maintained (between the sample site and lab in particular), that approved laboratory methods (where applicable) will be used, and that the database design will be fit for purpose.

For ease of reference the Deliverable Description and Acceptance Criteria in the Technology Contract are given in Appendix 1. The objectives of the deliverable are to clearly describe:

- how sampling consistency will be assured
- the design for the database
- the roles and responsibilities between all parties involved in sampling, data collection and lab analysis

D1 *Detailed schedule of work* provides a detailed project plan which should be read in tandem with this deliverable.

Rationale for the experimental approach

Key properties of energy crops

The choice of which crop attributes to assess is informed by the properties of importance to the utilisation and conversion of the biomass feedstocks considered within the project. The key factors are:

- Yield
- Inputs required
- Calorific value
- Moisture content
- Density
- Ash content
- Ash characteristics
- Other inorganic components

Properties such as yield and inputs of fertilizers affect the economics of the fuels as a crop, in financial terms or deployment.

Although the gross calorific value (CV) of dry, ash-free wood varies very little between species[1], the presence of other components such as oils or resins can tend to increase the overall CV. The most important factor in determining the realisable (net) CV in use is the moisture content, which tends to change naturally over time, or as a result of deliberate

[1] Wood Fuels Basic Information Pack (2000) ALTENER II

drying. Moisture content at harvest is of greatest relevance as this will determine the initial net CV and how much drying will be required. Density of the harvested crop will have an impact on transport and storage, though subsequent processing will determine bulk density.

The quantity and nature of the ash, and levels of other inorganic components will help determine the characteristics of the fuel during combustion. The ash content represents non-combustible material that does not contribute to energy generation, but must be periodically removed from the furnace itself (e.g. grate, fluidised bed or bottom ash hopper), ash that is carried through with the flue gases must also be removed via electrostatic precipitators or other filtration methods. Ash levels will impact on the "as received" calorific value of the fuel, although as previously discussed, moisture content is the most significant determinant of net CV (for the ash levels typical of clean biomass fuels). More significant is the specific combination of inorganic components in the ash as this will determine the ash softening and melting points, and thus potential for slagging and fouling, agglomeration of ash and potential for corrosion. Key species include alkali metals such as potassium, which can contribute to slagging and corrosion in association with chlorine. Many trace elements and other minerals can be volatilized forming fine particles and aerosols thus contributing to particulate matter (PM) emissions, as well as being toxic in many cases. Halogens such as chlorine and fluorine can also contribute to acid gas emissions and under certain conditions may act as pre-cursors for the formation of potentially carcinogenic dioxins and furans.

Properties of crop components

Many of the key crop attributes vary within the plant and frequently differ in woody plants cf. grasses, therefore the choice of which plant parts to sample depends on the species and the distribution of key attributes.

In woody plants, stem wood is made up of lignin, cellulose and hemi-cellulose, of which the proportion of lignin will make a small difference to the CV. Heartwood tends to be relatively dry, inert wood that provides the plant's mechanical strength, while sapwood consists of vascular tissue containing sap. In the case of xylem, this brings water and minerals, including nitrogen, from the roots to the leaves, while phloem carries the products of photosynthesis, including polysaccharides and proteins around the plant. Both xylem and phloem therefore contain higher levels of minerals, nitrogen and inorganic components than the largely inert heartwood. The sap content however varies at different times of the year. Bark provides a complex, protective layer around stems and branches and tends to have a particularly high mineral content and therefore levels of ash. Where present, leaves contain high levels of minerals, metabolites and polysaccharides, but relatively little structural material. They too, consequently, have high ash content.

Young stems - such as found in short rotation coppice (SRC) stands, which are harvested every 2- 5 years, and even short rotation forests (SRF), which are harvested every 15 years - tend to have less heartwood and relatively more sapwood than older, larger diameter stems. Although bark tends to be relatively thin, it potentially represents a higher proportion of the whole and chemical composition can be significantly different.

Biomass grasses also have a similar structure of a xylem and phloem system within the growing plant material. Biomass grasses typically consist of just a single year's growth, but can contain higher levels of minerals such as potassium. Depending on when and how the

grasses are harvested the levels of silica, potassium and chlorine can be higher than in typical woody materials. However some wood species like Eucalyptus and certain varieties of SRC poplar have had more than twice the levels of chlorine commonly experienced within certain biomass grasses, so a direct comparison between wood and biomass grasses is not always straight forward. The time of harvesting and the interval between cutting and removal from the site influences the levels of elements found within the chemical composition of the grass[2], as does the level of leaf material harvested with the crop as the leaves contain higher concentrations of silica and potassium.

Grasses generally produce more ash content than wood (due to the annual growth being largely condensed leaf material), and will typically have a lower ash melting temperature than most woods, however this does tend to be linked with the levels of potassium found within the sample material. Older stands of grasses, and ones that have been allowed to fully senesce prior to harvesting will typically see significant improvements in fuel compositional aspects, with calorific value (CV) levels equal to or better than most woods.

Site factors contributing to feedstock characteristics

The rationale for selecting each of the key site factors being investigated (soil type, soil composition, and climate zone) is outlined below.

Rate of tree growth and plantation density (number of trees per hectare) obviously influence yield but they also influence properties of significance to utilisation and conversion such as wood density. Local rainfall, temperatures and sunshine are likely to influence growth rate hence wood density. Larger stem and branch diameter tends to reduce the relative proportion of bark, and is consequently likely to reduce ash content, so factors that promote more rapid growth may be expected to influence ash content.

The second factor known to have an impact on growth is the soil characteristics. The soil fertility, including available nitrogen, phosphorus and potassium (NPK) and other mineral content, influences the availability of these to the growing plant[3]. Soil physical characteristics (including organic matter content and particle size distribution) influence the water retention and draining properties and the soil's ability to warm up quickly or slowly and retain or lose heat. All these factors may be expected to influence growth rate and biomass allocation within the plant, e.g. to stems, branches, roots and leaves; soil characteristics therefore influence ash content, and also wood density. Other aspects of soil which can affect growth can be soil compaction or stone content - if these are excessive the rooting of plants can be severely hindered causing restrictions on growth and possibly instability.

In addition, the presence of primary minerals and trace elements are expected to influence the extent to which these are found in the harvested biomass. Some minor elements have a vital role in forming ligands critical for plant metabolism, such as magnesium in chlorophyll. It is also thought that the presence of some heavy metals in the soil, either present as natural geological deposits, or as anthropogenic contaminants, can be taken up by plant

[2] Study of *Miscanthus x giganteus* ash composition – Variation with agronomy and assessment method (2012) Baxter, XC, Darvell, LI, Jones, JM, Barraclough, TJP, Shield, IF and Yates, NE

[3] The yield and quality response of the energy grass *Miscanthus x giganteus* to fertilizer applications of nitrogen, potassium and sulphur (2014) Shield, IF, Barraclough, TJP, Riche, AB and Yates, NE

roots giving rise to significant heavy metal levels in the harvested biomass. Certain biomass species are highly efficient at taking up metals from the soil and trapping them in the plant material - willow is exceptionally good at doing this.

Selection and breeding

Another factor that influences yield and plant form is plant variety. Both willow and poplar have undergone a period of development of new varieties to optimize desirable plant properties such as yield, and resistance to disease. Different varieties also show visible differences such as the number and diameter of stems when grown as coppice. The number and average diameter of stems may also be expected to influence the proportion of bark, and hence ash content, though it is not known whether there will be differences in the ash properties between different varieties grown on the same soil.

Individual varieties of both poplar and willow are propagated as clones some of which have been widely planted. Different clones show different yield characteristics under different conditions. The mix of clones selected by the grower is likely to vary from one site to another and will depend on a number of factors. Around 20 varieties are encountered on commercial sites, with about a dozen reasonably common. Most of the willow SRC sites sampled for this project used around 5 different clones, in varying proportions depending on expected yield. Many poplar clones were developed, with about 16 widely encountered; individual poplar SRF sites typically have a larger number of different clones than the willow SRC. Taking a random sample from an individual crop may therefore be expected to display, in addition to differences attributable to the site conditions, an influence from the specific mix of clones at that site. In practice, the impact of varietal differences could not be addressed in exactly the same way for willow SRC, poplar SRC and poplar SRF; in each case there was a balance to be stuck - obtaining a sample that represented what a commercial buyer would be given vs minimising the factors that would confound the statistical design of the project vs the reality of obtaining the sample in the field. The final approach is described in p 21.

In the case of biomass grasses such as *Miscanthus* there are numerous breeding programs to improve yields and fuel quality. Because biomass grasses are usually harvested after an annual cycle of growth, their rate of growth and minerals and heavy metal concentrations are likely to be affected more readily by the individual seasonal changes from one year to the next than woody crops. Because of latter's longer cycle, the overall effect of variable growing conditions, e.g. drought or low radiation, on the chemical composition can be smoothed out.

Time of harvesting and length of storage

For a particular site, plant properties will vary seasonally with senescence in autumn, dormancy over winter, release of dormancy in spring, followed by leaf and shoot extension during late spring and summer. Seasonal changes are especially marked in deciduous species. Consequently time of harvesting is expected to have a significant impact on biomass feedstock properties.

In most cases biomass will undergo a period of storage before use. Although usually necessitated by the realities of handling, processing and logistics, this also allows most forms of biomass to dry to some extent. Both harvest time and storage have been

demonstrated to influence a number of properties of *Miscanthus*[4]. Consequently the experimental approach is designed to investigate the effect of a period of storage on the properties of our energy crops.

Supplementary site and management factors to be analysed

A further set of site and management factors was identified on the basis of the client's brief and our understanding of possible influences on relevant crop characteristics. In order to unambiguously assess the influence of each of these parameters with reasonable statistical confidence we would need to collect a sufficient numbers of replicates, preferably with other parameters held constant. This would rapidly require not the 468 samples of the original proposal, but thousands, or tens of thousands. Owing to the limited number of growing sites available, it would be impossible to obtain anything like a complete set, as well as requiring a massive team of sample collectors to achieve it within the appropriate timing window, and being prohibitively expensive. A pragmatic alternative is to note these additional parameters at the time of sample collection, which will allow the relationship with feedstock characteristics to be analysed. Although there are slight variations depending on the feedstock, these parameters generally include:

- Slope percentage (%)
- Aspect of site
- Height above sea level
- Stoniness of the soil
- Fertilizer application and type of fertiliser
- Management practice
- Number of stems per ha
- State of dormancy

In addition, a number of visual observations of the quality of the crop were made, including height/length of crop and the diameter of steams. These can be used, together with the number of stems per ha, as an indication of yield.

Of the supplementary site factors, the angle of slope may be expected to influence the drainage properties of the site. Slope, aspect, exposure to sunshine and wind will impact on potential growth, as will the altitude of the site. The stoniness will influence drainage properties, availability of minerals and root growth.

Of the management factors, fertilizer application in combination with natural soil fertility will influence nutrient availability to the growing plant, and hence growth rates; if the input is in the form of sewage sludge it is possible that it may also contain elevated levels of heavy metals and other minerals not usually present.

Observations of the crop itself give an additional insight into the overall state of the crop and an indication of yield. The state of dormancy (fully dormant/bud expansion/bud burst/flushing/full leaf) of deciduous tree crops, gives a more relevant measure of the harvest time with respect to the onset of spring than the calendar date. It is expected that later stages of leaf development will be associated with increased vascular and metabolic activity

[4] The influence of harvest and storage on the properties of and fast pyrolysis products from *Miscanthus x giganteus* (2013) Greenhalf, CE, Nowakowski, DJ, Yates, NE, Shield, IF and Bridgwater, AV.

by the plant and therefore higher uptake of minerals from the soil and formation of complex metabolic products.

Fuel Analysis

The rationale for each of the fuel characteristics considered is given in Appendix 2.

Decisions taken in developing sample collection protocols

Introduction

In developing our experimental protocols we have needed to make a number of decisions in order to ensure maximum scientific rigour within the budgetary, time and practical constraints.

Timings

Delays to the start of the project have meant that the planned sampling during the fully dormant period for crops was not possible. As the season progressed, and in discussion with the ETI Strategy Manager, Programme Manager and Project Manager, it was decided that the project team should focus on securing one full set of mid-Spring samples for each of the resource types, rather than trying to sample two harvest times (one in mid-Spring when plants were being released from dormancy and one in very late Spring when the crop had resumed active growth) *provided* the second harvest time is not typical of commercial practice.

Soil types

The original plan was that the "agricultural type" crops, such as willow SRC, poplar SRC, and *Miscanthus*, would be sampled from sites classified as heavy, medium or light soil (i.e. clay, loamy or sandy), whereas the "forestry type" crops such as SRF, would be sampled from either mineral or organic (i.e. peaty) soil. Once the broadleaf SRF (poplar) had been chosen it was realized that there were no sites on peaty soil so it was decided that this too would be assessed according to the heavy/medium/light soil classification used for the SRC and *Miscanthus*.

Climate zones

When developing the bid document, six different climate zones were used as the basis of our approach to site selection because these had been identified as meaningful in previous trials of the interaction between site and yield of willow SRC. The selected climate zones were derived from the Forestry Commission GIS Ecological Site Classification for Great Britain.

The majority of crops to be investigated in this study are either broadleaves (willow or poplar) or a grass (*Miscanthus*). For all of these the best growing conditions, and the vast majority of commercial sites, are in England, with some in Wales and a few in Scotland. Almost all these sites fall within either our warm dry or warm moist climate zone classification. The exception is conifer SRF. Conifers are found throughout the UK, but the relative abundance is highest in Scotland and the northern counties of England. In these regions, the areas in which conifers do best are the warm moist and cool wet zones, and consequently these climate zones were selected for the conifer SRF sampling.

Varieties

For both willow and poplar there have been significant breeding programmes to improve yield, disease resistance or other properties. These different varieties are distributed as individual named clones. Some studies in willow have shown differences in physical properties between some different clones, especially those developed from more distantly related parent stock. It was therefore felt to be important that the issue of different clones was acknowledged when designing the sampling protocols for willow and poplar. Our objective (to obtain samples that are representative of the commercial product of both willow and poplar) was consistent but in practice the methodology used to achieve this differed; this is discussed below.

Conifer SRF

There are currently no conifer plantations in the UK specifically grown as SRF. This was known at the outset of the project and it was decided to make use of "SRF-like" trees – i.e. young trees of the age at which SRF might be harvested. This required decisions on a number of points:

- Conifer species
- Age of trees
- Climate zones
- Which parts of the tree to sample separately

In choosing species there were several issues to consider. Initially, species that may be widely available as a fuel in the immediate future, particularly as a result of disease, were considered. There is widespread felling of larch due to *Phytophthora ramorum* infection; burning the infected timber is an acceptable disposal method, however movements of infected larch wood require a movement licence and transport by a licensed operator, with regulations on handling procedures, to prevent spreading the infection further. The biosecurity regulations associated with this rendered larch impractical. Three pine species were considered, however this limited the range of sites. Finally Sitka spruce was selected because it is more likely to be planted as SRF, has a high growth rate, has no biosecurity restrictions and is available on a wide range of sites.

For the choice of age, stands of 14 ±2 years were selected to best represent likely commercial practice. This would also be expected to be of relevance to fuel obtained from early thinnings.

Conifers generally outperform broadleaf species in warm moist and cool wet areas, consequently these were chosen as the two climate zones to study.

In practice, it is likely that an SRF crop would be harvested and processed as an entirety, and consequently the fuel produced would contain a mix of these tree parts. Nevertheless it was decided, for several reasons, that there was value in sampling at a finer resolution than just a mix of all above-ground material. Firstly, for certain soil characteristics and ground conditions it is common practice to lay a mat of harvesting residue to minimize ground damage, and also to assist the return of organic matter and nutrients to the soil. Secondly conifer logs are routinely debarked in many sawmills as a preliminary processing step and although this is unlikely to take place when preparing biofuels unless necessary, it is

possible that demand for low ash fuel could make such a process economically feasible. Thirdly individual trees could be sold to two different markets, e.g. an energy market and more traditional markets for small round wood, in which case the split will be at the standard threshold of 7cm. Lastly, stemwood has different chemical characteristics from bark, from needles and from harvesting residues (which contain relatively high levels of bark, needles and young twigs). In any case it is always possible to recreate the characteristics of a mixed fuel from the constituent parts, provided the proportions of each are known, therefore the choice of sampling fractions from individual trees was considered. It was decided to measure the properties of three fractions: debarked stemwood, i.e. stems > 7 cm diameter; bark; and tops (stems ≤ 7 cm diameter plus their associated branches and needles).

Broadleaf SRF

There are almost no broadleaf SRF plantations in the UK. The Poplar Tree Company has many plantations of young poplar that are effectively being grown as SRF, however these are almost all within the warm dry climate zone and on medium to heavy soils so initially a number of alternative SRF analogues were considered to widen the range of growing conditions. Ash has many suitable characteristics but was rejected because of biosecurity restrictions related to ash dieback (*Chalara fraxinea*). Birch was discussed as a possible alternative but the available data on fuel characteristics suggest that it is less suitable than poplar. Sycamore was also considered but rejected. Poplar was deemed to be the most suitable for power generation owing to a more reliable chemical composition so finally, despite the limited geographical spread of poplar SRF available through the Poplar Tree Company, poplar was selected as the most appropriate broadleaf SRF.

It was found that none of the poplar SRF were on peaty soils so it was decided that the soil categorization should be as for the SRC and *Miscanthus* crops, i.e. heavy, medium or light. On further investigation it was found that it was also almost never planted on light soils.

As with the conifer SRF it was decided that the main trunk > 7 cm diameter and the tops and branches should be sampled and tested separately. Compared to Sitka spruce it is much harder to remove the relatively thin bark of poplar, and it is not common practice, so it was decided that this would not be tested separately from the stemwood.

There are many different poplar clones grown in commercial plantations, with perhaps 6-10 or more on a single site. As the SRF samples would be taken by felling a number of individual trees, it would not be feasible, or acceptable to the crop owners, to take samples from all clones present. Consequently it was decided to identify a subset of three different clones (Gaver, Ghoy and Gibecq) that were found on almost all the sites to be sampled, and take four individual trees of each of these. As the clones were usually planted in blocks taking from just one clone would have meant sampling only a relatively small proportion of the field. As the distribution of blocks of clones within each field was mapped out it was relatively straightforward to organize in advance where the samples should be taken from.

Willow SRC

In common with poplar, the issue arose of different varieties (clones) with potentially different properties. There are about a dozen clones commonly encountered in commercial sites, with the particular occurrence depending on the expected performance of the clones

available at the time of planting. Unlike poplar SRF, the different clones are not generally planted in large contiguous blocks, and the location of individual clones is not mapped.

Initially we considered identifying a single clone present at all sites as this would allow results from different sites to be directly compared without the possibility that clonal differences might mask the impact of site characteristics. However in discussion with the ETI Strategy and Programme Project Manager it was decided that, for the main investigation of causes of variability in feedstock characteristics, we should take samples that reflected the properties of the crop as a whole, as would be delivered to bioenergy markets. This does mean that if clones do indeed have significantly different properties, then mixed samples from sites with a different mix of clones might confuse the interpretation of soil and climate effects. In order to get a representative sample of the site as a whole it was decided to sample willow chips immediately after harvest by taking multiple small scoops from different positions within the pile of harvested chips for mixing and sub-sampling. This then also ensures that all parts of the plant are sampled, as would be delivered to the end user.

A supplementary study examined the variation within the field and here too varietal differences were considered. Firstly, in order to ensure good sample representation from across the site it was necessary to take the samples before harvest. Also, as it was considered that the within-field variation in properties could be relatively small, it was important that all other variables be minimized as far as possible. It was decided to take samples from just a single clone (Tora) and to focus on just one combination of climatic zone and soil type. Consequently the distribution of Tora within the three chosen sites was logged using GPS waymarks to allow the distribution of sample locations to be prepared in advance. Identification was undertaken in the company of an expert with the experience to be able to positively identify Tora before there was any leaf present. He then accompanied the team collecting the samples to ensure that the right clone was collected on each occasion.

Poplar SRC

There is now only one commercial poplar SRC site in existence, others having been grubbed up over the previous winter in response to commercial pressures. There are however a few poplar SRC trial sites and so, rather than abandon this resource type altogether, it was decided to sample the trials wherever possible and appropriate particularly ones with the same three clones (Gaver, Ghoy and Gibecq) used in the poplar SRF study.

As most of these trials will not be harvested this year, it is necessary to take samples from individual standing stems. As all the sites are well documented it should be possible to identify the three relevant clones.

Although the numbers of sites will restrict the statistical robustness of analysis of the results, it will be possible, at least in a superficial way, to compare the results with those from both willow SRC and poplar SRF.

Miscanthus

In the case of *Miscanthus,* only one variety (*Miscanthus x giganteus*) is routinely used commercially so there has not been the difficulty of addressing clonal differences.

Three sampling times of *Miscanthus* will be required from most sites, as opposed to the two from most other crops, i.e. one at the time of harvest, one at the time of baling (where this

was more than a few days after harvest) and one after the crop has been in storage for a month.

Stem samples representing the time of harvest are collected immediately after cutting. Despite the logistical challenge this approach presents, it does mean that at the same time as the freshly harvested samples are being collected, a rain gauge can be set up to measure rainfall over the period the crop lies in the swath before baling.

Stored samples

Obtaining stored samples of the different feedstocks presented different challenges and each required a different approach.

In the case of SRF, the usual approach would be to store the harvested material as roundwood to allow it to dry - this is more efficient than trying to dry significant quantities of chips owing to the difficulty of ensuring good airflow through a stack of chips. In the case of the conifer SRF, the logs for subsequent sampling could be embedded within piles of roundwood with the tops laid over the stack next to the forest road. For the poplar SRF however, it was advised that there was significant risk of theft so the test logs were brought back to a central Forest Research location for storage in a secure compound.

There was discussion as to the optimum length of time to leave the stored roundwood in order to give best chance of observing noticeable change in feedstock properties, while still achieving results within a timescale appropriate for the project: a storage duration of 3 months was selected. It was discussed whether the conifer SRF logs de-barked as a part of the initial sampling could be reused as an aid to accelerated drying and hence shortening storage times, but this was rejected for several reasons: de-barking is likely to enhance drying rates, it is not commercial practice and it might introduce additional differences other than simply accelerated drying. Consequently separate samples were taken to investigate the effect of storage which allowed the stems to be stored with bark intact. This was not an issue for the poplar SRF as this was not de-barked anyway. It was decided that for the stored SRF samples a storage time of 3 months would be used as a sensible compromise between achieving a realistic level of drying, even with bark on, and allowing the project to progress on a reasonable timescale.

Where willow SRC chips are stored in a large stack or storage facility it could be difficult to match the chips with the same field as the initial samples, and the soil samples. In most cases chips are tipped from the wagon after harvest onto the end of an existing stack. Coloured paint on the floor was then used to identify where the initial samples were taken, allowing the second samples to be taken a month later from the same population. In some cases however it may not be possible to identify the chips from a specific field unambiguously and a more generic sample will have to be taken. If this happens it will be noted as part of the sample record.

Having identified the appropriate zone of the stored chip stack for sampling, it is necessary to take samples not just from the superficial layer (as could be done for the freshly harvested chips) but to sample material from as deep within the stack as possible, as well as from more superficial layers. Having spoken to a number of organizations responsible for testing woodchip properties, it emerged that the most common way of achieving this was to dig into

the stack and take samples at different depths. Unfortunately that was not possible mainly because digging into a large stack would fall under construction health and safety legislation which would preclude FR staff.

Several devices to take samples from a range of depths within a pile of chips were trialled. The most effective device was a simple one consisting of a 10 cm diameter plastic waste tube with coarse teeth cut in one end and a hole to take a T bar at the other. This allows samples to be taken up to a depth of around 1.5 m which provides a realistic analysis of the stack, well beyond the superficial layers that are exposed to significant air penetration and drying effects. The thermal insulation properties of wood chips are such that even at depths of 1 m there is a significant temperature increase as a result of microbial action, suggesting sampling to 1.5 m effectively represents "deep" within the stack.

In the case of *Miscanthus* the crop is baled and various approaches were discussed in order to obviate the necessity to completely dismantle bales, which would not be acceptable to the majority of owners. It was therefore decided that since bales are generally stacked together a telehandler would be used, where available, to move the front bale and take samples from both the front face, which is exposed to drying and the back face which is effectively deep within the store. Owing to the density with which the *Miscanthus* is packed in the bale, it is thought that there is unlikely to be the gradation of conditions found within the first metre or so of a stack of wood chips.

Procedures to ensure sample integrity

In general, delivery of this aspect of the project is underpinned by using a small team of trained staff collecting field samples and also by effective liaison between the manager of these field staff and the manager of the analytical lab. Any procedures established specifically for this project are signed off by the Project Manager and Chief Technologist. The full protocols are described in Appendices 5 to 10.

Three aspects are considered – consistency, condition and labelling.

- Consistency

  All samples are collected following agreed written protocols specific to each resource type, level of variation being investigated (between sites or within-field), and the time since harvest. Data are recorded on hard copy forms to agreed formats. The data being recorded are shown in Appendix 3.

- Condition

  All samples are handled in a way that minimises the field-to-lab analysis time, prevents moisture loss and minimises change in physical or chemical properties, with particular emphasis on preventing sample contamination.

  While following these principles, variations are necessary to allow for the different resource types, the plant parts, level of variation and harvest times being

investigated (see Table 1). Different techniques are needed to obtain representative samples that meet the objectives of the project (see Box 1).

Box 1. Sampling techniques

Miscanthus is sampled for the assessment of fresh condition as either the standing crop, or immediately after cutting; the second sampling occasion – just before baling – is generally from the cut stems resting on top of the stubble but, if there is no phase of the crop lying out to dry off, then no second sample is taken; the third sampling occasion – one month after baling – is from the face of Heston bales. Owners were not willing for bales to be split open to sample the interior of bales therefore we will take samples from the face of three outer and three inner bales.

Willow SRC. Overall field condition, both fresh and after storage, is assessed by sampling from the piles of chips or billets. Within-field variation is sampled by cutting whole stems from representative stools from across the standing crop.

Poplar SRC. Overall field condition, is assessed by cutting whole stems from representative stools from across the standing crop.

Poplar SRF. Overall field condition is assessed at each site by felling one tree at each of 10 representative locations. The main trunk from the base to 7 cm top diameter is cut into 3 billets of equal length. Samples at the time of harvest are taken from the top and bottom billet with the middle billet set aside for sampling after the stipulated storage period. Because of the known risk of cut billets being stolen, these are not left on site for the storage period but transported to a common secure site for storage. The upper stem, i.e. with a diameter less than 7 cm, and associated branches of each tree is also sampled. In this case by taking 500 g from opposite ends of alternate trees, i.e. from the first tree the sample is started from the very tip moving down and from the next tree by starting from the 7 cm diameter cut end upwards to the tip.

Conifer SRF. As above for poplar SRF except that a) bark is also sampled, separately from the freshly felled trunks and b) cut billets are stored on their original site rather than a common site.

Soil, excluding any stones and plant material, is collected at each point by scraping off any top humus / litter layer and collecting 25 g of soil from both the 5 – 15 cm and 15 – 30 cm layers. All samples are mixed thoroughly, sub-sampled, sealed in bags and stored in a cool dry and dark place before dispatch to the analytical lab for next day (before noon) delivery.

- General

  During sample collection, all samples are handled so that no material is in contact with the ground and is not contaminated with soil; this is achieved by placing the samples directly in bags or buckets, or working on clean plastic sheeting as appropriate. Tools used to collect samples are chosen for ease of cleaning between samples. *S*amples at the point of collection in the field are sealed in robust polythene bags or buckets with tight clip lids. To avoid cross contamination and at the same time minimise the amount of sample discarded, shredders and chippers are run for a short time between samples until no more pieces are coming through thus minimising carry over. All dispatch is by courier for delivery by noon the following day.

  During lab procedures, all samples are handled in such a way as to prevent contamination.

- *Miscanthus*

  Fresh harvest: Each stalk is cut to fit into the sample bag, ensuring that the full length of each stalk is included. When the site sampling is complete, samples are sealed within another bag and dispatched to the Field Station at Thetford, the closest Forest Research site with facilities to shred *Miscanthus* stalks, for next day delivery. All samples are reduced to 2.5 cm lengths using the same shredder.

  i) Fresh harvest assessing overall field condition: shredded material from all sampling points within the field is bulked into a clean large container, mixed thoroughly and a 2 kg sample is sealed in a robust polythene bag and sent to the analytical lab for next day (before noon) delivery.

  ii) Fresh harvest assessing within-field variation: each sample once shredded is dispatched in a separate labelled bucket.

  Before baling. 48 hrs before the field is baled, samples are collected as above for the fresh harvest assessing overall field condition.

  After storage (as bales). 1 month after the crop was baled, samples are collected from the outside face of the outer and inner bales and treated as above for the fresh samples assessing overall field condition. Samples from the faces of the outer bale are representative of material on the very periphery of the stack of bales. Because the bales are densely packed during storage, samples from the faces of bales previously butting hard up against another will be representative of the material within the bale. Consequently samples obtained in this way will be similar to the interior of bale which could be obtained only by opening out a bale, something growers would not allow. A mix of material from the peripheral and inner faces was assumed representative of the totality of the stored bales.

- Willow SRC.

  Both fresh harvest and after storage (as chips or billets). Chips or billets removed from the piles of harvested willow SRC are moved in a clean bucket to a clean plastic sheet laid on the ground where all samples are

mixed and subsampled. The subsample is sealed in a robust polythene bag. Chips are sent to the analytical lab for next day (before noon) delivery. Billets, which require further reduction in size to be acceptable for the analytical lab, are sent for next day delivery to the research station for further processing. Once the billets are chipped chips are bulked in a large clean container, subsampled and dispatched to the analytical lab as for material that was harvested in the form of chips.

- Poplar SRC. The condition of the samples is maintained as described for billets of willow SRC

- Poplar SRF. Samples are removed with clean bow saws (rather than chain saws which are difficult to clean between cuts) and clean secateurs. Samples are bagged in clean robust polythene bags and dispatched for chipping. After chipping they are sealed into robust polythene bags and dispatched to the analytical lab by next day delivery.
  After storage. The middle trunk sections for later sampling are identified with bio-degradable tape and then stored by placing them on top of the two other trunk sections which are laid directly on the ground. The upper stem and branch sections for later sampling are placed on top of the stack.

- Conifer SRF. The condition of the samples is maintained as described for poplar SRF.

- Soil. Samples are taken from a freshly cut face of a soil pit, mixed on a clean plastic sheets, subsampled, sealed in bags and stored in a cool dry and dark place before dispatch to the analytical lab for next day (before noon) delivery.

- Labelling

  Two identical labels are used, one on the outside and one inside of the sample container. The sample identifier is unique and its format has been agreed between field and lab staff. This identifier will follow the sample through to the database.

Procedures to ensure representative sampling

The overarching approaches to achieve representative sampling are:

- To sample three sites for each combination of site climate and soil type.

  For most resource types when investigating variation among site factors this is two climates x three soil types (see Table 1) while only one climate x one soil type is considered when investigating within-field variation and leaf properties. Irrespective of the number of climate x soil type combination, the intention is to sample three sites.

- To sample sufficient locations at each site.

  When investigating variation among site factors 10 locations are sampled while 20 locations are sampled when investigating within-field variation. At each site the field shape and size are entered into GIS and the polygon overlain with a grid. Sampling locations are selected at random from across the grid to provide field staff with a unique set of co-ordinates (way marks) for each field and sampling time.

Table 1, Species	Climatic zone	Soils	Harvest	Fraction	Time of sample	Replicates	Plots
Miscanthus	warm moist warm dry	light medium heavy	February April	whole	at harvest in-field prior to baling 1 month uncovered	3 sites	108
SRC willow	warm moist warm dry	light medium heavy	January March	whole	at harvest 1 month outside	3 sites	72
SRC poplar	warm moist warm dry	light medium heavy	January March	whole	at harvest 1 month outside	3 sites	72
SRF conifer	cold wet warm moist	mineral organic	Oct/Nov Jan/Feb	trunk stems/branches	at harvest 3 months outside	3 sites	96
SRF conifer	cold wet warm moist	mineral organic	Oct/Nov Jan/Feb	bark	at harvest	3 sites	24
SRF broadleaved	warm dry warm moist	mineral organic	Oct/Nov Jan/Feb	trunk stems/branches	at harvest 3 months outside	3 sites	96
Within-field variation *Miscanthus* SRC Willow	Select one Select one	Select one Select one	March/May January	Whole Whole	at harvest at harvest	3 sites and 20 points 3 sites and 20 points	60 60
Leaf properties SRC Poplar SRC Willow	Select one Select one	Med/Heavy Light	September September	Leaves only Leaves only	before harvest before harvest	3 sites 3 sites	9 9
Pellets	n/a	n/a	n/a	whole	before and after pelleting		12
Total number of samples							**618**

- To minimise varietal (clonal) differences.

  There is some evidence that clonal differences within both willow and poplar can be substantial. Two approaches were considered to minimise the effect of varietal differences: ensure that the sample is representative of all clones planted at each sampling site or sample only one clone that is present at all sites. Following discussion with the ETI Strategy and Programme Manager there are three variants:

  - Assessing the overall condition of a field of willow SRC. Varieties are usually planted in very small poorly defined blocks and it is extremely difficult to correctly identify willow varieties, especially in the leaf-off condition. It is therefore not possible to sample a particular variety that is common to all site combinations. Instead samples are taken from the piles of harvested willow which are representative of the field as a whole provided there are sufficient sampling points. At each field 40 points from around the piles of harvested willow are sampled.

  - Assessing the overall condition of a field of poplar SRF or SRC. Varieties are usually in blocks of identifiable clones therefore the three clones (Gaver, Ghoy, and Gibecq), which are common to all our SRF sites, are sampled. In the case of poplar SRC, owing to the very small number of sites now available, not all these three clones are available at all sites, so samples were taken from as many of these three as were present.

  - Assessing within-field variation of willow SRC. A known expert, Dr Kevin Lindegaard [5] was subcontracted to identify blocks of one variety (Tora) found at each of the three sites chosen to assess within-field variation. The plots were marked and located with GPS. At each site representative sampling points were selected from these common varieties.

- To sample plant material that is representative of the harvested crop.

  - *Miscanthus* sampled in the field – whole stalks are sampled. Once baled material is cut from the face of bales that have been on both the outside and inside of the stack

  - Willow SRC for in-field variation and all poplar SRC – full stems are cut, alternating between the second largest diameter at breast height (dbh) and the second smallest dbh in order to best represent the crop

  - Poplar SRF and conifer SRF. The selected trees are separated into trunk vs upper stem and branches at 7 cm stem diameter on the assumption that if sections of individual trees are sold to two different markets, e.g. an energy market and more traditional markets for small round wood, the split will be at the standard threshold of 7cm.

[5] author of the 2013 publication 'Willow Varietal Identification Guide' which for the first time brings together all the information about the currently available biomass willow (*Salix spp*) varieties grown for short rotation coppice

The wood samples are taken as close to the mid-point of the trunk as possible on the assumption that these will be representative of the average of the thinner younger material toward the tip of the stems and the thicker older material towards the base. This is obtained in practice by sampling from the top of the bottom third and bottom of the top third (remembering that the middle third will be stored).
Representative samples of the upper stems and associated branches when freshly harvested are achieved by taking 500 g from opposite ends of alternate trees, i.e. from the first tree the sample is started from the very tip moving down and from the next tree by starting from the 7 cm diameter cut end upwards to the tip. Representative samples of the upper stems and associated branches after storage are obtained by sampling the remaining sections which have been left for the stipulated period. This procedure was developed for this contract to ensure that the samples of freshly harvested and stored upper stems and associated branches had similar diameters and ages, therefore any differences between fresh and stored material could be attributed to storage.

- To sample soil that is representative of the site,

  10 random sampling points per field are selected as described above. At each point, equal weights of soil (excluding stones) are taken from each of two depths (5-15 cm and 15-30 cm) down to the typical plough limit. These are amalgamated for all 10 points, mixed and subsampled.

- To analyse representative feedstock and soil material from the samples submitted to the analytical lab,

  all samples are milled and mixed prior to a portion being taken for analysis. For the major fuel parameters, analysis is undertaken in duplicate. The mean of the two replicates is used as the final data point.
  Surplus material is stored in conditions that minimise any sample deterioration (see next section for details) so that the analysis can be repeated if there are unusual results.

Use of EN standards for sample testing.

UKAS accredited methodologies are used for all lab testing (see Table 2 below). Preparation and moisture analysis of samples is undertaken following SOPs as covered in the laboratory's scope of accreditation under ISO17025, with the relevant international standards referenced in Table 2.

Further details of the early stages of sample preparation (covered by BS EN 14780, 14474-2, and 14474-3) are provided here. Samples are received at the processing laboratory in sealed, impermeable containers such as plastic bags or buckets, which prevent moisture loss to the atmosphere. As soon as possible after receipt, typically next day, they are pre-

dried at <80 ℃ for a minimum of 24 hours to remove the “Free” moisture; very wet samples may be dried for longer as required. This stabilises the material and allows milling to <4 mm without moisture loss.

Following drying, the material is milled to the required size fractions for further analysis, as per BS EN 14780, with the prepared materials stored in sealed plastic bottles or bags. As part of the fuel analysis, an “Inherent” moisture analysis is undertaken on a portion of the <4 mm material (this is combined with the “Free” moisture to give “Total” moisture of the as received fuel). This is done at 105 ℃ in accordance with BS EN 14474-2. While inherent moisture contents are usually <5 %, for some feedstocks it has been found that a significant proportion of the total moisture is inherent, requiring this second stage moisture determination at >100 ℃. As the bulk sample is not dried at 105 ℃, volatile matter loss is minimised. To account for any moisture losses when milling to <1 mm, a determination of the analysis sample moisture content is undertaken at 105 ℃ in accordance with BS EN 14474-3, with the “as analysed” results recalculated to either a dry basis or to an “as received” basis using the determined total moisture from the <4 mm fraction.

New SOPs and/or procedures

The procedures developed specifically for this contract are presented in full in the appendices; these have not yet been allocated SOP reference numbers. Flow charts illustrating the processes between field sampling and dispatch to the analytical lab are given in Appendix 11.

- *Miscanthus* sampling to assess its average condition over the field site (described as overall-field condition)
- *Miscanthus* sampling to assess within-field variation
- Willow SRC sampling of fresh chips or billets from stacks to assess its average condition over the harvested field (described as overall-field condition)
- Willow and poplar SRC sampling protocol to assess overall condition of stored material
- Poplar SRC and SRF sampling to assess overall field condition
- Conifer SRF sampling to assess overall field condition

Summary of existing FR and E.ON standard operating procedures (SOP)

Existing E.ON Technical Operating Instructions are listed and summarised in Table 2. Flow charts illustrating the sample preparation and analysis stages are provided in Appendix 12. Existing FR standard operating procedures are listed and summarised in Table 3.

Table 2: Technical Operating Instructions and Standard Operating Procedures for sample preparation and analysis by E.ON

Technical Operating Instruction Reference	Title	Scope	Relevant Standards
TOI_APA_FT_009	Procedures for Operation of the Sample Preparation Centre	This method details the sample preparation (to <4 mm and <1mm) as well as the 1st stage of Total Moisture determination	BS EN 14780
TOI_APA_FT_001	Determination of the Inherent (Equilibrium) Moisture Content in Fuel Samples	This method covers the analysis of moisture in <4 mm material. This is the 2nd stage of Total Moisture determination	BS EN 14474-2
TOI_APA_FT_002	Determination of Analysis Moisture in Fuel Samples	This method covers the analysis of moisture in <1 mm material. Used for calculating all parameters to a reference basis	BS EN 14774-3
TOI_APA_FT_003	Determination of Ash in Fuel Samples	This method covers the determination of total ash content in samples	BS EN 14775
TOI_APA_FT_004	Determination of Volatile Matter in Fuel Samples	This method covers the determination of volatile content in samples	BS EN 15148
TOI_APA_FT_005	Determination of Calorific Value in Fuel Samples by Bomb Combustion	This method covers the determination of Calorific Value in samples. It also covers additional sample preparation for Chlorine determination	BS EN 14918
TOI_APA_FT_006	Determination of Total Sulphur in Fuel Samples by Combustion and IR	This method covers the determination of Sulphur content in samples using Infra-Red	BS EN 15289
TOI_APA_FT_008	Determination of Chlorine in Fuel Samples by Bomb Combustion and ISE	This method covers the determination of Chlorine content in samples using an Ion Selective Electrode	BS EN 15289
TOI_APA_FT_011	Manual Sampling of Solid Fuels	This method covers the sampling of pellets	

TO00I_APA_FT_010	Determination of Carbon, Hydrogen and Nitrogen in Fuel Samples by Combustion and IR	This method covers the determination of Carbon, Hydrogen and Nitrogen in samples using Infra-Red	BS EN 15104
SOP_APA_FT_002	Quality Control Procedures in the Fuel Testing Labs	This document provides detail on general quality control in the Fuel Testing Labs. This document includes flow charts to detail the sample handling process	ASTM D3686
TOI_APA_OE_0.0114	Sample Preparation By Ashing Of Solid Fuels	These methods describe the sample preparation of the milled fuel samples received from the sample preparation centre by ashing of the fuel followed by extraction into aqueous solution of the elements contained by either microwave digestion or dissolution	Microwave digestion: BS EN 13656
TOI_APA_OE_0.0136	Microwave Assisted Dissolution Of Fuel Ash, Fused Alumina-Silicates And High Silica Materials For Element Composition		Dissolution: BS ISO 15238,
TOI_APA_OE_0.0062	Dissolution Of Solid Fuel, Fuel Ash And Related Materials For Trace Element Determination	Elements present in ash and fused silica materials can be associated with the surface of the material and can also be intrinsically bound within the matrix. The determination of element concentrations using ICP-OES, GFAAS or FIMS (CV-AAS) requires that the samples are in aqueous solution. Therefore, in order to determine total element concentration, the matrix itself must be broken down (digested) to release the elements into solution. The digestion of silica and aluminosilicate-based materials requires strong digestion conditions, for which HF/aqua regia is used. Following digestion, boric acid is added to complex the HF into a useable (safe) form. All elements are stabilised in the solution by the acid matrix. Analysis of the solution by ICP-OES, GFAAS or FIMS (CV-AAS) is then undertaken using matrix-matched standards.	BS EN ISO 11885 BS 6068-2.60 BS ISO 15238 ASTM D3683

TOI_APA_OE_0.0006	Trace Element Determination In Aqueous Solutions By Conventional Aspiration ICP-OES	Inductively Coupled Plasma Atomic Emission Spectroscopy is used for trace element determination in aqueous solutions. This document describes the operation of the Perkin Elmer Optima 7300 DV ICP spectrometer. Elements commonly analysed by this techniques are aluminium, boron, barium, beryllium, calcium, cadmium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, phosphorous, potassium, silicon, silver, sodium, strontium, sulphur, thallium, titanium, vanadium and zinc. This method is valid for the analysis of all aqueous samples, including acid extractions/digestions as prepared under TOI_APA_OE_0.0136 and TOI_APA_OE_0.0062. In each case a set of matrix matched standards are produced for calibration and Quality Control purposes. Determination of major ash-forming elements is UKAS accredited only for solutions prepared via	
TOI_APA_EO_0.0012	Atomic Absorption Spectroscopy: Perkin Elmer Aanalyst 600	This procedure describes the use of the AAnalyst 600 Atomic Absorption Spectrometer to determine a range of trace elements in aqueous solution. Elements commonly analysed upon this instrument include cadmium, silver, lead and aluminium. This method is valid for the analysis of aqueous samples including acid extractions/digestions as prepared under TOI_APA_OE_0.0136 and TOI_APA_OE_0.0062	BS EN ISO 15586 BS 6060-2.84 BS ISO 15238 ASTM D3683
TOI_APA_OE_0.0106	Trace Element Determination In Aqueous Solutions By Gaseous Hydride Generation ICP-OES	Inductively Coupled Plasma Atomic Emission Spectroscopy using gaseous hydride generation sample introduction is used for the analysis of arsenic, antimony and selenium in water and acid digests, including samples generated under TOI_APA_OE_0.0062 and TOI_APA_OE_0.0034. Prior to analysis in the ICP the samples must be chemically pre-treated by a method specific to the analyte to be measured. This procedure describes the pre-treatment of the aqueous solutions and the operation of the Perkin Elmer Optima 7300 DV ICP Spectrometer	BS EN ISO 11969 BS 6068-2.55 BS 6068-2.45 ISO 9965

SOP_APA_OE_001	Subcontracting Analysis	This document provides guidance on the suitability and procedures for sub-contracting analysis to external laboratories. Sub-contracted laboratories will hold UKAS accreditation to ISO 17025 for the analysis in question if possible, otherwise a minimum requirement of ISO 90001 accreditation will be applied.	
TOI_APA_OE_0.0034	Bomb Combustion Of Solid Or Liquid Fuels For The Determination Of Hg, F, Cl, Br And Se	This document describes the procedure to be used to prepare aqueous sample solutions for the determination of mercury, fluoride, chloride, bromide and selenium from solid or liquid fuels. The procedure involves combustion of the milled fuel sample received from the sample preparation centre in an oxygen combustion bomb. The bomb washings are collected and pre-treated (depending on the analyte to be determined) prior to analysis.	See individual element standards
TOI_APA_EO_0.0013	Inorganic Anions By Ion Chromatography	This procedure describes the use of Ion chromatography to determine inorganic anions (including chloride and bromide) in aqueous solutions, such as those prepared under TOI_APA_OE_0.0034.	BS EN ISO 10304-1
TOI_APA_OE_0.0014	Determination Of Fluoride By Ion Selective Electrode	This procedure describes the determination of fluoride in an aqueous solution using ion selective electrode. The procedure is valid for the analysis of aqueous solutions derived from potable, ground and waste waters as well as aqueous abstracts of soluble fluoride from solid samples and from bomb washings from the combustion of solid and liquid fuels, as prepared under TOI_APA_OE_0.0034	ASTM D3761

TOI_APA_OE_0.0083	Determination Of Mercury In Aqueous Solutions From Bomb Combustion Of Fuel And Pisces Solutions From Ash Digestion Using FIMS	This document describes the determination of mercury in aqueous solutions derived from the bomb combustion (TOI_APA_OE_0.0034) or acid dissolution (TOI_APA_OE_0.0062) of fuels using the Perkin Elmer Flow Injection Mercury System (FIMS) FIMS is a Cold Vapour Atomic Absorption techniques (CV-AAS) for the determination of mercury concentration. Mercury in the aqueous sample is reduced to the zero oxidation state by reaction with stannous chloride. The mercury is vaporised and carried by argon into the long path-length absorption tube. Multi-point calibration is required.	ASTM D3684

* Analysis of bromine by this method is not in E.ON Technologies (Ratcliffe) Ltd. UKAS scope, however the method is accredited for other anions

Table 3: Standard Operating Procedures registered by FR.

SOP number	Title	Scope
SOP0001 v.4	Writing Standard Operating Procedures (SOPs)	All procedures and methods used by Forest Research (FR) staff must be documented. Where a technique is to be repeated it must be described by writing a Standard Operating Procedure (SOP) which must conform to the standard layout.
SOP0024 v.3	Use of balances (analytical, top pan and micro)	Describes procedure for determining weight using analytical, top pan, and micro balances, includes checking accuracy prior to use using 'check' weights. Precision varies between type of device and model. Generally, devices suitable for measuring very small to medium weights, with a higher degree of precision. Heavier weights and those requiring lower precision suit a spring balance (SOP0088). Some balances are highly sensitive to electrostatic charges. Pesticide efficacy trials have additional requirements for archiving of calibration and maintenance records.
SOP0048 v.2	Marking and labelling field experiments	Describes how to label field experiments. Markers are used to aid navigation and are a useful backup to maps and layout plans. Must never be used to guide treatments or assessments. Applies to experiments only - are not a requirement for demonstration sites. Requirements and procedure for mapping is described separately (SOP0084).
SOP0066 v.3	The use of a cold store	Details procedure for use and maintenance of non-freezing cold stores, various types and size, located as external free standing units or within research buildings. A cold store is generally suitable for short to medium term storage, but maximum period before samples spoil or degrade is dictated by factors such as wetness, whether woody or fleshy, dried or fresh, plant or soil etc. Not suitable for long-term storage of plant material (requires sub-zero temperature), and does not cover use of a fridge (SOP0501), freezer (SOP0488), or humi-store. All samples must be inspected at least once per month, and be discarded if show signs of deterioration. Pesticide efficacy trials have additional requirements for archiving of calibration and maintenance records. This SOP only covers hydrocarbons installations and not ammonia, carbon dioxide or any other gas refrigerants as these have different safety implications for the user should the gas escape into store.

SOP0084 v.3	Mapping the location and layout of field experiments and monitoring sites	Describes the procedure for recording the location and layout of field experiments (including sites used for ongoing monitoring purposes), and also for supplying details to the respective database managers. This SOP should not be used for recording the location of survey plots.
SOP0088 v.3	Use of suspended spring balances to determine weight	Procedure for using a spring balance to determine the weight of an object or sample. May be used to determine weights from 10 g up to a limit of approximately 200 kg, depending upon the type and model. 'Spring' balances do not usually have an accuracy of greater than +/- 0.5%, so must not be used where a high degree of accuracy is required. Pesticide efficacy trials have additional requirements for archiving of calibration and maintenance records.
SOP0127 v.3	The protocol for naming electronic files containing assessment data	This SOP outlines the structure, system and purpose of the file naming convention for all electronic files containing assessment data (including digital photographs). It does not detail naming conventions to be used for correspondence, or paper and other hard copy records.
SOP0232	Determining tree height assessment points	Describes height assessment points (all tree sizes) commonly used in FR and conventions for measurement. Measurement of felled trees (i.e. length) is covered in SOP0415. The equipment used to assess height is covered by separate SOPs.
SOP0261	Terrain classification	Describes method for broad classification of terrain for any site. Easy to undertake and gives objective information on terrain for experiment and operational planning. 'Working surface' is assessed: condition (soil type and moisture regime); roughness (obstacles); and slope (gradient and form). Each factor in five classes: combination of which allows a simple numerical site descriptor. Examples used are typical instances, and not intended as precise definitions.

SOP0305	Collection of soil samples for soil moisture and bulk density analysis	Describes the method for collecting and forwarding soil samples for subsequent (laboratory) analysis of soil moisture and bulk density (SOP0169). The procedure is suitable for any soil type (i.e. mineral, organic or mixed profile), and in any moisture condition. Samples are taken from a standard soil pit (SOP0533). Samples require prompt forwarding to the laboratory to prevent them from drying out or absorbing atmospheric moisture, and further preparation and careful storage (SOP0149) prior to analysis. Although prepared for use with the BioSoil project, this procedure may also be adopted for other studies where appropriate.
SOP0322	Use of handheld GPS units	This SOP covers the use of handheld GPS units as an aid to navigation and mapping. Different manufacturers follow slightly different steps, but all GPS units can capture a point, or any linear feature. Conversely, pre-determined points can be plotted in a Geographical Information System (GIS) and the co-ordinates uploaded to the GPS unit. This allows an operator to navigate to these points in the field by following a directional bearing.
SOP0323	Bark sampling for attempted isolation of *Phytophthora ramorum*	Describes procedure for collecting bark samples for processing (SOP0271) to confirm / rule out *P. ramorum* infection. Procedure is time-limited and isolation of Phytophthoras from samples (SOP0271) must be done within 24 hours because pathogen is ephemeral and quickly replaced by other organisms once bark is dead.
SOP0522	Sample collection for dendrochronology studies using a manual increment corer, bow-saw, or chainsaw	Describes the collection of: i) increment core samples from living trees, >10 cm diameter at breast height, using manual increment corers / augers, and ii) disc sections from stem or branch wood cut using either a bow-saw or chainsaw. Includes instructions for selection of trees and sampling positions, and for the transport, drying and storage of samples. The technique has been written to enable subsequent dendrochronological analysis (not described here), but may be adopted for other procedures where similar samples are required. Note: Successful dendrochronological analysis depends upon a high degree of care being taken during all stages of this procedure.
SOP0533	Digging an exploratory soil pit	Describes the simple procedure for digging a pit in order to obtain information on a soil's type and other characteristics.

SOP0534	Soil / site mapping	Describes method for using field data from exploratory soil pits (SOP0533) in conjunction with aerial photography, pre-survey investigation of topography and lithology (SOP0536), and various other site factors (SOP0294) in order to compile a soil / site map.

Details of the database structure and software selection.

To maximise its accessibility within the present contract and any subsequent investigation, the final database is expected to be presented as Excel spreadsheets, with accompanying metadata describing the data and each data field. The critical point is that each sample has a unique identifier therefore the Excel format can be converted to a more structured relational database, e.g. Access, if agreed. Our own analysis of biomass characteristics will use Genstat (see D1 for further detail of the statistical approach).

Contracts for the linked modelling project, which uses the data generated by BI2010 Characterisation of Biomass Feedstocks, have not been concluded so we have not been able to liaise formally over the database structure and software selection. Again Excel is sufficiently flexible for our data, provided they are described appropriately, to be pulled into the format required by the linked modelling project.

Internal roles and responsibilities

Clear roles and responsibilities between all parties involved in sampling, data collection and laboratory analysis are essentially as proposed in the bid document. The roles and responsibilities are:

- The Project Manager is Dr Helen McKay (Forest Research; FR)
- The Chief Technologist is Steve Croxton (E.ON).
- Day-to-day management for sample analysis lies with Dr Will Quick (E.ON)
- Stewart Bradley (E.ON) is responsible for receipt of the samples at the analytical lab and is responsible for the day-to-day management of the sample preparation team and the fuel analysis laboratory.
- Duncan Credland (E.ON) is team leader of the Operational and Environmental Analysis lab undertaking elemental analysis of the samples and managing submission of the fuel samples to the sub-contractor for ash fusion analysis.
- Dr Susan Weatherstone (E.ON) is responsible for management of the data produced by the analysis laboratories and population of the database.
- Day-to-day management of site selection and collection of their associated data lies with Michael Wall (FR).
- Liz Richardson (FR) is responsible for developing sampling protocols, using GIS to locate the sampling points at each site, organising her field staff to complete the field sample collection and dispatch to the analytical lab or research centre for chipping and subsequent dispatch to the analytical lab.
- Andy Peace (FR) advises on the statistical aspects of sampling design and data analysis, including quality assurance of data.

Conclusions and next steps

The original design and approach to ensure adequate replication to account for various sources of variation, which was outlined in our bid documentation and formalised in the contract, has been followed. Draft protocols have been modified slightly when faced with the reality of the operational practices being followed by growers but our protocols remain robust in terms of ensuring consistent, representative and uncontaminated samples. Roles and responsibilities are well established and operating efficiently.

Database design has been kept simple (as Excel spreadsheets linked by a unique identifier for each field sample) in the interim until there is an opportunity to discuss it formally with the contractor who will use our datasets for modelling. Having agreed the database structure it will be populated with field and lab assessments at regular intervals following standard data QA procedures.

Stage Gate Review 1 takes place on 16th April to review the performance of the research Consortium and consider if the desired outcomes of the 'Characterisation of Biomass Feedstocks' contract can and will be met.

Appendices

Appendix 1. Description of D2 and Acceptance Criteria

Deliverable No:	**D2**		
Deliverable Name:	**Methodology and sampling consistency report; database design**		
Work Package(s)	**2,4,5**	**Deliverable lead**	**Forest Research (FR)**
Deliverable description / scope / content:	Provision of a document outlining how sampling consistency will be assured, including, but not limited to, how and when samples will be collected, how they are stored, how long before they are sent to the labs, how long before they are processed. Where an appropriate Forest Research Standard Operating Procedure (SOP) exists then the SOP number will be quoted and a short summary of the key points given. If deemed appropriate, a new SOP will be drawn up to set out the sampling protocol. Where no appropriate SOP is applicable, the procedure to be adopted will be described and a plan detailing when a formal SOP will be produced. Presentation of final database design and structure.		
Deliverable Purpose(s):	The purpose of this deliverable is to enable the ETI, its reviewer(s) and its members to check that samples will be representatively sampled, that sample integrity will be maintained (between sample site and lab in particular), that approved laboratory methods (where applicable) will be used, and that the database design will be fit for purpose.		
Deliverable Objective:	Deliver a document clearly describing how sampling consistency will be assured and present the design for the database. Clear roles and responsibilities between all parties involved in sampling, data collection and lab analysis are to be defined.		
Dependent on:	Project kick-off meeting.		
Prerequisite to:	Balance of Project		

Acceptance Criteria:	• Satisfies the Deliverable Description, Deliverable Purpose(s) and Deliverable Objective, as described above.
	• Formal Deliverable prepared in accordance with the Generic Acceptance Criteria.
	• Comprises (at least):
	  ○ A document which at least: details how sample integrity will be maintained from field to laboratory analysis; how representative sampling is ensured; shows that where applicable, EN standards are used for sample testing. Existing FR and E.ON standard operating procedures (SOP) will be summarised and referenced; new SOP's and/or procedures will be presented in full. Clear roles and responsibilities between all parties involved in sampling, data collection and laboratory analysis are to be defined.
	  ○ Details of the database structure and software selection for the database to be issued in Deliverable D3.		

Appendix 2. Rationale for each of the fuel characteristics analysed.

Chemical or component being analysed	Biomass analysis result area of interest	Soil analysis result	Provenance data required to link comparative area of concern which could infer a relationship between biomass composition and data collected	Expected reasoning behind analysis selections	Why the end user comparative area is of interest
Nitrogen (N)	N concentration in fuel	Organic matter (OM) content, Total N levels, soil type, sand/silt/clay percentage (%)	Type of fertiliser used, when applied, how much. Previous cropping. Soil temperature, annual rainfall due to leaching risk, slope, drainage, stone content. Delayed harvest collection, storage comparison	Different harvest timings, different soil N levels, differing amounts of fertiliser applied and type applied are all likely to impact on N levels within a biomass fuel. Slope, stone content, annual rainfall and soil type are important as this will potentially indicate the growing medium's ability to hold or lose nitrogen	Nitrogen in fuel levels will directly impact on NO_x emissions
Potassium (K)	K concentration expressed as K or K_2O in ash	K results, soil type, sand percentage (%)	Type of fertiliser used, when applied, how much, previous cropping, rainfall, slope, drainage – leachable in certain soils, stone content. Delayed harvest collection, early/late harvest, extent of senescence	Delayed harvest timing and different parts of the plant are expected to exhibit different levels of K. Quantity and type of fertiliser applied could impact on availability of K Different soils will have varying levels of potassium. Delayed baling following cutting is expected to see a reduction in potassium levels in certain biomass types due to natural leaching, especially after heavy rain	Formation of alkali salts in the furnace, resulting in deposition and corrosion within boiler system, together with slagging and ash agglomeration issues due to low melting temperatures. Potential for alkali salt aerosols to contribute to particulate emissions at furnace exit. Potassium is also a recognised poison for catalysts used in flue gas clean-up applications.

Chemical or component being analysed	Biomass analysis result area of interest	Soil analysis result	Provenance data required to link comparative area of concern which could infer a relationship between biomass composition and data collected	Expected reasoning behind analysis selections	Why the end user comparative area is of interest
Hydrogen (H)	H concentration in fuel	Not specifically analysed in soil	Not expecting any correlation between provenance data and levels of hydrogen seen within biomass sample	Analysed in biomass samples as part of C,H,N determination process.	Used in Net CV calculations
Chlorine (Cl)	Cl concentration in fuel	Cl levels in soil, soil type, location (is it coastal, flood plain). Differences between plant parts. Clay percentage (%) as it is expected sandier soils will leach more Cl	Fertiliser and agricultural-chemical pesticide application details (when, what, how much quantity). Drainage aspects, annual rain fall, slope/topography, age of biomass crop. Delayed harvest collection comparison	Harvest timing and plant part being analysed are expected to show significant variations at different times of the growing season. Annual rainfall (more rainfall could mean more leaching of Cl from certain biomass types), this also links to delayed collection after harvesting. It is thought the soil type (and its location) will impact on the level of Cl able to be naturally held within the soil. The amount and type of fertilisers (some bagged potassium products (e.g. MOP (Muriate of Potash)) contain very high levels of chlorine).	Chlorine, in partnership with other elements (alkalis and other metals) is a well-known initiator of furnace corrosion in biomass applications. Chlorine will contribute to acid gas emissions if released as HCl from the furnace, and may act as a precursor for formation of chlorinated organic pollutants, Chlorine may also contribute to fine aerosol particulate emissions if released as alkali chloride aerosol from the furnace. Low temperature corrosion (air heater/ductwork) will also be affected by HCl concentrations.

Chemical or component being analysed	Biomass analysis result area of interest	Soil analysis result	Provenance data required to link comparative area of concern which could infer a relationship between biomass composition and data collected	Expected reasoning behind analysis selections	Why the end user comparative area is of interest
Trace elements (Hg, Pb, Cd, As, Se, Sb, Ba, Be, Cr, Co, Cu, Mo, Ni, V, Zn)	Trace element concentrations in fuel	Aiming to link if levels in soil are seen to be elevated above average if biomass lab sample sees elevated levels. Soil classification, clay percentage (%)	Previous applications to soil of any waste materials, manures, composts, was site reclaimed land, overall location data	It is expected the largest contributor for trace element content within biomass material is from the soil and location. Also the history of applied organic fertiliser products (sewage sludge, composts and digestates) will be important in determining potential soil levels. Soil type and annual rainfall are also important in identifying if certain trace elements may leach from the soil	Certain elements (e.g. Zn, Pb) are linked with corrosion, particularly in combination with chlorine. Low melting point elements (e.g. Zn, Pb) may cause blockage of air nozzles and bed agglomeration in fluidised beds. All elements, particularly volatile species will contribute to gaseous emissions. Arsenic is also a recognised poison for catalysts used in flue gas clean-up applications. High concentrations of certain elements (e.g. Pb) could be an occupational health hazard in ash, and may affect ash categorisation as 'hazardous'/'non-hazardous' impacting on its re-use/recycling.
Carbon (C)	C concentration in fuel,	Soil OM percentage (%) levels, soil classification	Previous cropping history, soil temperature, application of fertilisers – what used, when, age of biomass crop, details of any manure/wastes applied	Carbon levels are not thought to be directly impacted by the level of soil carbon being available, but it will be interesting to see if Carbon levels increase on soils with a higher Carbon percentage (%).	Carbon is oxidised to CO_2

Chemical or component being analysed	Biomass analysis result area of interest	Soil analysis result	Provenance data required to link comparative area of concern which could infer a relationship between biomass composition and data collected	Expected reasoning behind analysis selections	Why the end user comparative area is of interest
Sulphur (S)	S concentration in fuel	Soil S levels, soil classification	Location, drainage, rainfall (as S leaches fairly readily), topography, stone content, fertiliser/manure/waste applied details. Delayed collection and storage comparative	Soil type, slope, stone content, annual rainfall and location will all be important in determining if the soil is likely to leach more sulphur. Also when the biomass is harvested, and the length of time after harvest before collecting is expected to see a reduction in certain biomass types due to natural leaching of sulphur from the biomass. Type of fertiliser used could also indicate if sulphur has been applied or is naturally low in the location. Location is likely to have an impact on sulphur levels as a site which is closer to an industrial area could expect to see higher levels of sulphur.	Levels of sulphur in fuel will have a direct correlation with sulphur dioxide emissions. Depending on boiler temperatures sulphur can be also be associated with corrosion. However, sulphur can preferentially react with alkali metals over chlorine and can act as protection against alkali chloride corrosion.
Sodium (Na)	Na concentration expressed as Na or Na_2O in ash.	Soil results, Soil type, soil classification	Location, fertiliser/manure/waste/compost application details. Stone content Rainfall	Sodium is likely to be closely linked to location and soil type. Sodium will be readily absorbed by the plant in preference to potassium as it is an easier chemical exchange for the plant – although often not a beneficial one. Certain soils can have elevated levels of sodium depending on their location, previous cropping history and previous fertiliser usage.	Similar to potassium, sodium is linked with corrosion, boiler bed agglomeration, and slagging, due to low melting temperatures. Also alkali salt aerosols contributing to particulate emissions.

Chemical or component being analysed	Biomass analysis result area of interest	Soil analysis result	Provenance data required to link comparative area of concern which could infer a relationship between biomass composition and data collected	Expected reasoning behind analysis selections	Why the end user comparative area is of interest
Aluminium	Al concentration expressed as Al or Al2O3 in the ash concentration	Soil results, soil type, soil classification	Location, fertiliser/manure/waste/compost application details.	Aluminium, like sodium and heavy metals, is linked to soil type and location, and to details of any waste materials applied to the soil/field.	Alumino-silicate in the ash may mitigate alkali-metal mediated corrosion/slagging/fouling
Phosphate	P concentration expressed as P or P_2O_5 in ash	Soil results, soil type, clay percentage (%), soil classification	Location, fertiliser/manure/waste/compost application details.	Phosphate levels will vary by soil classification, previous cropping history, amount and type of fertiliser applied. As phosphate is fairly immobile within a plant differences seen post harvesting are not expected to be significant. It is expected a difference would be seen between early and late harvest timings before the plant sequesters a percentage of the phosphate back to the roots or is lost in leaf/needle fall.	Phosphorous is a recognised poison for catalysts used in flue gas clean up applications. Phosphorous may also be implicated in corrosion.
Moisture	Percentage (%) Moisture in fuel	Soil percentage (%) of sand/clay/silt, soil classification	Location, timing of collection, temperature, topography, crop canopy, rainfall	It is thought certain soil types can influence overall plant moisture at time of harvesting. If soils and annual rainfall is high it is thought biomass moisture percentage (%) at point of harvesting could likely be elevated. Drainage of soil, slope, stone content, exposure (how windy) and soil type will all potentially impact biomass moisture content – likely to be less significant for SRF plantations.	High moisture content will reduce combustion plant efficiency. Potential impact on fuel handleability/ dustiness/ degradation in storage

Chemical or component being analysed	Biomass analysis result area of interest	Soil analysis result	Provenance data required to link comparative area of concern which could infer a relationship between biomass composition and data collected	Expected reasoning behind analysis selections	Why the end user comparative area is of interest
Ash	Percentage (%) of ash content and	Soil type – namely clay (silica) content, OM levels	Part of the plant tested, when sample collected, age of plant, planting density, what fertiliser/manure/compost applied	Which part of the plant being analysed is expected to show the most significant difference. Ash levels would expect to be seen increasing in younger plants, or in plantations where planting density is high. Published work has previously linked soil silica content (clay percentage (%)) to an increase in ash percentage of certain biomasses. Type of harvesting technique, and timing will also potentially impact on levels of ash percentage (%) in the sample	High ash content may cause increased slagging and fouling within the boiler, due to the increased mass flow of ash in the furnace. Ash handling systems need to be designed to deal with the expected ash quantities of ash produced. Fluidised bed medium may need more regular replacement.
Halides (bromine and fluorine)	Concentration in fuel	Soil type	Which part of the plant has been tested, linked to when the biomass sample was collected within the growth cycle of the plant. Understanding if any application of waste products or irrigation water have been applied, and specifically identifying any links with field location	Halides exist within soils and plant material naturally at very low levels. Identifying if elevated results can be linked to soil types, timing of harvest operation, site location, if site has a history of application of waste materials	Impact on gaseous emissions of acid gases HBr and HF. Bromine is implicated in degradation of fabric filters for dust collection, as well as aggressive corrosion of air heaters and ductwork.

Chemical or component being analysed	Biomass analysis result area of interest	Soil analysis result	Provenance data required to link comparative area of concern which could infer a relationship between biomass composition and data collected	Expected reasoning behind analysis selections	Why the end user comparative area is of interest
Ash composition (SiO_2, Al_2O_3, Fe_2O_3, TiO_2, CaO, MgO, NaO, K_2O, Mn_3O_4, P_2O_5, BaO, SO_3)	Concentration of each element and as the oxide in ash.	Some of the trace elements are analysed in the soil samples (either as heavy metals or other), but in the main these chemicals are not directly analysed in the soil	Directly linking compounds within the ash composition to provenance data is a continuation of review of the elements. The percentage level of ash and how the biomass plant was managed, where grown and how harvested/stored are not expected to show significant differences to the ash composition		Ash composition will have impacts on the rate of mill and furnace wear (abrasion and erosion). As noted above (K and Na), ash composition has a direct impact on the melting point/agglomeration of ash particles causing furnace slagging and bed agglomeration. Other ash components may mitigate these effects
Ash Fusion Temperature	Initial Deformation Temperature (IDT), Softening/Sphere Temperature (ST), Hemisphere Temperature (HT), Flow/ Fluid Temperature (FT)	Related to ash composition (see above)	Related to ash composition (see above)	Related to ash composition (see above)	Provides an indication of the likelihood of ash slagging and bed agglomeration
Volatile Matter	Percentage (%) volatile matter in fuel	Not sampled for within the soil analysis	It will be interesting to see if there is any correlation between VM and when the crop was harvested, which soil type or region grown.		Volatile matter will impact on flame stability, combustion burnout performance and NOx emissions. It is unlikely that the range seen for biomass will be significant.

Chemical or component being analysed	Biomass analysis result area of interest	Soil analysis result	Provenance data required to link comparative area of concern which could infer a relationship between biomass composition and data collected	Expected reasoning behind analysis selections	Why the end user comparative area is of interest
Bulk Density	Bulk density of dried fuel	Not applicable to soil sample	Provenance data expected to be of influence to BD is plantation thickness, age of plantation, soil type location/region, level of exposure, fertiliser regime, which part of the plant being analysed, species type	Bulk density is more closely linked with logistics/handling and storage cost aspects, rather than the utilization of the fuel within a boiler. Identifying if BD can be directly influenced by certain aspects of provenance data could assist with improving the BD in the future for certain fuel types. – Not all samples are being assessed for BD	Bulk density will affect transport and storage costs, as well as capacities of bulk handling systems.
Net and Gross Calorific Value	NCV and GCV of fuel	Not applicable to soil sample	Provenance data expected to partially link to this will be harvest timing, type of species, which part of plant being analysed, plantation age, and plantation density – storage length and type – linked to moisture content. To a lesser extent soil type and management practices	It is largely believed moisture content is the key driving factor for this calculation. Identifying if any of the other provenance data is attributable to affecting the NCV and GCV will be interesting – but is not expected to be the case	Calorific value has a direct impact on boiler and ancillary plant size, overall power plant capacity, efficiency and logistics.

Appendix 3. List of provenance data collected

Provenance data
Desk collection (before or after field sampling)
1. Site unique identifier
2. Species
3. Age
4. Site type
5. Grid reference
6. Fertiliser application (including sewage sludge) : dates, forms, application rates
7. Pesticide application: dates, forms, application rates
8. Cultivation: dates, depths
9. Drainage
10. Sale price of most recent crop
11. Varieties
12. Spatial distribution of varieties

Field collection
1. Sampling phase
2. Location of waypoint position
3. Photo of the crop at each waypoint*
4. Photo of the ground at each waypoint*
5. Visual assessment of stoniness*. This is a subjective assessment based on the experience of the sample collector as to the presence or otherwise of obvious significant stone content. This is supplemented by the photographic record.
6. Air temperature at 1.5 m height*
7. Soil temperature at 10 cm depth*
8. Visual assessment of recent weather conditions i.e. snow or frost present, recent heavy rain etc.
9. Site aspect for the site as a whole
10. Slope percentage (%) for the site as a whole
11. Rainfall between *Miscanthus* cutting and baling

*(except where sampled from chip piles)

Appendix 4. List and identifier of laboratory methods

Determination of the Inherent (Equilibrium) Moisture Content of Solid Fuel Samples	TOI_APA_FT_001
Determination of Moisture Content of General Analysis Sample of Solid Fuel	TOI_APA_FT_002
Determination of Ash Content of General Analysis Sample of Solid Fuel	TOI_APA_FT_003
Determination of Volatile Matter Content of General Analysis Sample of Solid Fuel	TOI_APA_FT_004
Determination of Calorific Value of Solid Fossil Fuels	TOI_APA_FT_005
Determination of the Sulphur Content of Fuel Samples Using the LECO SC-432 High Temperature Analyser	TOI_APA_FT_006
Determination of Chlorine in Solid Fuel using the SPECTRO TITAN Instrument	TOI_APA_FT_007
Determination of Chlorine in Solid Fuel by Bomb Combustion and an Ion Specific Electrode	TOI_APA_FT_008
Procedures for Operation of the Sample Preparation Centre	TOI_APA_FT_009
Determination of Carbon, Hydrogen and Nitrogen in Biomass and Oil using the LECO CHN Instrument	TOI_APA_FT_010
Determination of Ash composition SiO2, Al2O3, Fe2O3, TiO2, CaO, MgO, NaO, K2O, Mn3O4, P2O5 BaO SO3	TOI_APA_OE_0.006 TOI_APA_OE_0.062 TOI_APA_OE_0.138
Trace Elements Ba, Be, Cr, Co, Cu, Mo, Ni, V, Zn	TOI_APA_OE_0.006 TOI_APA_OE_0.062
Trace Elements As, Sb, Se	TOI_APA_OE_0.106 TOI_APA_OE_0.062
Trace Elements Cd, Pb	TOI_APA_OE_0.012 TOI_APA_OE_0.062
Trace Element Hg	TOI_APA_OE_0.083 TOI_APA_OE_0.034
Trace Element F	TOI_APA_OE_0.014 TOI_APA_OE_0.034
Trace Element Br	TOI_APA_OE_0.013* TOI_APA_OE_0.034

* This analysis (bromine analysis by ion chromatography) is not in ETG UKAS scope

Appendix 5. *Miscanthus* sampling protocols to assess its average condition over the field site (described as overall-field condition)

Miscanthus **protocol to assess overall-field condition**

Site criteria:

Species – *Miscanthus*
Age 1 year
Site area – >1 ha with a minimum width of 25 m

Office work:

Select 36 sites – 6 warm, moist / light
6 warm, moist / medium
6 warm, moist / heavy
6 warm, dry / light
6 warm, dry / medium
6 warm, dry / heavy

Three of the above sites will need to be identified for the additional in-field variation sampling.

Produce 36 polygon maps with grids.

$$\text{Grid distance} = \frac{\text{square root of A}}{10} \times 100$$

Produce 3 in-field variation polygon maps with grids.

$$\text{Grid distance} = \frac{\text{square root of A}}{20} \times 100$$

The 3 in-field variation maps will have separate sampling grids that do not pick up the original 10 sample points.

Produce waypoint shape file to download to GPS.

Phasing:

Phase 1, sampled within 24 hours after harvest (this includes the in-field variation sampling)
Phase 2, sampled 3 weeks in-field after harvest
Phase 3, sampled 1 month after bailing

Once notification of the harvest date is received from Terravesta, the site will be scheduled for sampling the day after harvest subject to liaison with the site owner or manager.

Field work: sites 049 – 084, Phase 1

Ensure there are sufficient robust sample bags for each site.
The sample bags should be labelled with the site number from the map issued, MISC and the phase
i.e. site 049 will have a bag marked as 049/Misc/1
If more than 1 bag is used for a sample type, write 1 of X, 1 of 2…. etc for however many are used.

A rain gauge made from a flat bottomed, straight sided water bottle with the top cut off, inverted and secured is to be dug into a place at each site on the phase 1 visit. It should be

in or close to the sample field and positioned so that it is in the open and not overhung by trees or interfering with farm operations.

Locate waypoint position; there will be 10 per site.

Take a photo of the crop and a photo of the ground at each waypoint, the photos will be labelled site number/waypoint/C for crop or G for ground and sent to the Field Station Manager, Fineshade when site is complete.

Visually assess if the site should be classed as stony or not and record on the site field form.

Collect 300 g of harvested material at each location point; this is likely to be in lengths. Ensure no material has been in contact with the ground and is not contaminated with soil. Cut each length up to fit into the sample bags, all material from each length will be included.

Take air temperature at 1.5 m height with a Vertex at each waypoint and record on site field form.

Complete soil sample:

50 g of soil excluding any stones and plant material will be collected at each way point:
Scrape off the top humus / litter layer, if any.
Dig a spade width hole to 30 cm from the adjacent ground level.
Collect 25 g of soil from 5 – 15 cm and 15 – 30 cm layers and bag together.
Push soil temperature probe into ground horizontally at 10 cm depth. When display has settled and no longer changes, record temperature on site field form.
Do not fill in soil pit at this stage.
On a clean tarpaulin or in a clean bucket mix all 10 samples thoroughly and bulk a representative sample of 300 g in to a sample bag (double bag if necessary).
Label bag as follows: Site Number i.e. 049/Soil

Soil samples should be sealed and stored in a cool dry and dark place until all team site samples are collected.

Proceed to next waypoint and repeat.

All *Miscanthus* samples from the same site can be put in the same bag.

Visually assess and record recent weather conditions i.e. snow or frost present, recent heavy rain etc.
Record the site aspect on the site field form
Record the percentage (%) slope for the site on the site record form

Miscanthus samples should be sealed when site is complete and dispatched to TSU, Thetford for next day delivery. The Field Station Manager, Fineshade should be informed of dispatch

Soil samples should be sent when all team sites are complete to arrive before 12 noon of the following day to: Sample reception (Stewart Bradley), E.ON Technologies (Ratcliffe) Ltd, Technology Centre, Ratcliffe-on-Soar, Nottingham, NG11 0EE. The Field Station Manager, Fineshade should be informed of dispatch.

48 hours before baling: Phase 2
Regular contact with the land owner is required to ensure sampling happens as close to baling as possible.

Ensure there are sufficient robust sample bags for each site.
The sample bags should be labelled with the site number from the map issued, MISC and the phase
i.e. site 049 will have a bag marked as 049/MISC/2
If more than 1 bag is used for a sample type, write 1 of X, 1of 2…. etc for however many are used.

Locate waypoint position; there will be 10 per site.

Collect 300 g of harvested material at each location point; this is likely to be in lengths. Ensure no material has been in contact with the ground and is not contaminated with soil. Cut each length up to fit into the sample bags, all material from each length will be included.

Take air temperature at 1.5 m height with a Vertex at each waypoint and record on site field form.

Locate soil pit.
Push soil temperature probe into ground horizontally at 10cm depth. When display has settled and no longer changes, record temperature on site field form.
Fill in soil pit.

Proceed to next waypoint and repeat.

All *Miscanthus* samples from the same site can be put in the same bag.

Visually assess and record recent weather conditions i.e. snow or frost present, recent heavy rain etc.

Locate rain gauge, remove and using a ruler measure in cm rainfall, record on the site field form.

Miscanthus samples should be sealed when site is complete and dispatched to TSU, Thetford for next day delivery. The Field Station Manager, Fineshade should be informed of dispatch.

Once notification of the baling date is received from Terravesta, the confirmed date will be recorded on the site field form and the site will be scheduled for sampling 1 month later.

1 month after baling: Phase 3
Ensure there are sufficient robust sample bags for each site.
The sample bags should be labelled with the site number from the map issued, MISC and the phase
i.e. site 049 will have a bag marked as 049/MISC/3
If more than 1 bag is used for a sample type, write 1 of X, 1of 2…. etc for however many are used.
The landowner will need to make a telehandler available: The bales will be Heston bales.

Record on the site field form if the bales are stored in a barn or outside, if necessary take a photo and label: Site Number/Misc/3
Three outer bales will be selected and 500 g of material will be picked from the outer edge of each bale, without breaking any strings. Ensure no material has been in contact with the ground and is not contaminated with soil.
An outer bale will be moved by telehandler giving access to 3 inner bales.

500 g of material will be picked from each of the 3 inner bales, if possible from areas that have not been exposed previously, without breaking any strings. Ensure no material has been in contact with the ground and is not contaminated with soil.

All *Miscanthus* samples from the same site can be put in the same bag.

Miscanthus samples should be sealed when site is complete and dispatched to TSU, Thetford for next day delivery. The Field Station Manager, Fineshade should be informed of dispatch.

The bale samples will not be able to be attributed to a specific field which would have been the original sample site.

Field work: sites 082 - 084, in-field variation sites. Phase 1
These 3 sites will have an additional 20 sample point.
At all 20 points another sample will be done as follows

Ensure there are sufficient robust sample bags for each site. At least 20 will be required
The sample bags should be labelled with the site number from the map issued, MISC, the waypoint number and IF
i.e. site 082 will have a bag marked as 082/MISC/ 1 to 20/IF
If more than 1 bag is used for a sample type, write 1 of X, 1of 2…. etc for however many are used.

Locate waypoint position; there will be 20 per site.
Collect 3 kg of harvested material at each location point; this is likely to be in lengths. Ensure no material has been in contact with the ground and is not contaminated with soil. Cut each length up to fit into the sample bags, all material from each length will be included.

Each waypoint is to be sampled independently so 1 bag per waypoint, the bag should be sealed when a waypoint is complete and dispatched to TSU, Thetford for next day delivery. The Field Station Manager, Fineshade should be informed of dispatch.

Processing protocol: TSU Thetford - week 0 and 3 and 1 month after bailing

A Qualcast SDS2810, 2800W quiet garden shredder will be used for this process.
TSU Thetford will receive:

1 x 3 kg sample bag from 36 sites, week 0, sites 049 - 084
20 x 3 kg sample bags from 3 sites, week 0, sites 082 - 084
1 x 3 kg sample bag from 36 sites, week 3, sites 049 - 084
1 x 3 kg sample bag from 36 sites, 1 month post bailing, sites 049 – 084

The bale samples will not be able to be attributed to a specific field which would have been the original sample site.

Each bag will be shredded separately:

The 3 kg week 0 bags, labelled Site Number/MISC/1 from each site will be shredded into 2.5 cm pieces.
The shredded material will be bulked into a suitable container and mixed thoroughly.
A 2 kg or full bucket sample will be sealed in buckets supplied by E.ON, a label will be placed in a sealed clear plastic bag on top of the chips inside the bucket and a label will be put on the lid and the side of the bucket. The labels will have the site number, the sample type and phase written on them i.e. 049/Misc/1. If more than 1 bucket is used for a sample type, write 1 of X, 1 of 2…etc. for however many are used on the label.

The 20 x 3 kg week 0 bags, labelled Site Number/MISC/Waypoint Number/IF from each waypoint of each site will be shredded into 2.5 cm pieces.
The shredded material will be bulked into a suitable container and mixed thoroughly.
A 2 kg or full bucket sample will be sealed in buckets supplied by E.ON, a label will be placed in a sealed clear plastic bag on top of the chips inside the bucket and a label will be put on the lid and the side of the bucket. The labels will have the site number, the sample type, waypoint number and IF written on them i.e. 084/MISC/1/IF. If more than 1 bucket is used for a sample type, write 1 of X, 1 of 2…etc. for however many are used on the label.

The 3 kg week 3 bags, labelled Site Number/MISC/2 from each site will be shredded 2.5 cm pieces.
The shredded material will be bulked into a suitable container and mixed thoroughly.
A 2 kg or full bucket sample will be sealed in buckets supplied by E.ON, a label will be placed in a sealed clear plastic bag on top of the chips inside the bucket and a label will be put on the lid and the side of the bucket. The labels will have the site number, the sample type and phase written on them i.e. 049/Misc/2. If more than 1 bucket is used for a sample type, write 1 of X, 1 of 2…etc. for however many are used on the label.

The 3 kg 1 month after baling bags, labelled Site Number/MISC/3 from each site will be shredded into 1” (2.5 cm) pieces.
The shredded material will be bulked into a suitable container and mixed thoroughly.
A 2 kg or full bucket sample will be sealed in buckets supplied by E.ON, a label will be placed in a sealed clear plastic bag on top of the chips inside the bucket and a label will be put on the lid and the side of the bucket. The labels will have the site number, the sample type and phase written on them i.e. 049/Misc/3. If more than 1 bucket is used for a sample type, write 1 of X, 1 of 2…etc. for however many are used on the label.

Between different samples and different sites the shredder will be cleaned so no cross contamination occurs.

Samples will be dispatched for next day, before noon, delivery on the day of chipping to:
Sample reception (Stewart Bradley),, E.ON Technologies (Ratcliffe) Ltd, Technology Centre,

Ratcliffe-on-Soar, Nottingham, NG11 0EE. The Field Station Manager, Fineshade should be informed of dispatch.

Appendix 6. *Miscanthus* sampling protocols to assess within-field variation

Miscanthus **protocol to assess within-field variation**

Site criteria:

Species – *Miscanthus*
Age 1 year
Site area – >1ha with a minimum width of 25m

Office work:

Select 3 sites – 3 warm, dry / light

Produce 3 in-field variation polygon maps with grids.

$$\text{Grid distance} = \frac{\text{square root of A}}{20} \times 100$$

The 3 in-field variation maps will have separate sampling grids that do not pick up the original 10 sample points.

Field work: sites 082 - 084, in-field variation sites. Phase 1

These 3 sites will have an additional 20 sample point.
At all 20 points another sample will be done as follows

Ensure there are sufficient robust sample bags for each site. At least 20 will be required
The sample bags should be labelled with the site number from the map issued, MISC, the waypoint number and IF
i.e. site 082 will have a bag marked as 082/MISC/ 1 to 20/IF
If more than 1 bag is used for a sample type, write 1 of X, 1of 2…. etc for however many are used.

Locate waypoint position; there will be 20 per site.
Collect 3 kg of harvested material at each location point; this is likely to be in lengths. Ensure no material has been in contact with the ground and is not contaminated with soil. Cut each length up to fit into the sample bags, all material from each length will be included.

Each waypoint is to be sampled independently so 1 bag per waypoint, the bag should be sealed when a waypoint is complete and dispatched to TSU, Thetford for next day delivery. The Field Station Manager, Fineshade should be informed of dispatch.

Processing protocol: TSU Thetford - week 0 and 3 and 1 month after bailing

A Qualcast SDS2810, 2800W quiet garden shredder will be used for this process.
TSU Thetford will receive:
20 x 3 kg sample bags from 3 sites, week 0, sites 082 - 084

Each bag will be shredded separately:

The 20 x 3 kg week 0 bags, labelled Site Number/MISC/Waypoint Number/IF from each waypoint of each site will be shredded into 1" (2.5 cm) pieces.
The shredded material will be bulked into a suitable container and mixed thoroughly.
A 2 kg or full bucket sample will be sealed in buckets supplied by E.ON, a label will be placed in a sealed clear plastic bag on top of the chips inside the bucket and a label will be put on the lid and the side of the bucket. The labels will have the site number, the sample type, waypoint number and IF written on them i.e. 084/MISC/1/IF. If more than 1 bucket is used for a sample type, write 1 of X, 1 of 2…etc. for however many are used on the label.

Between different samples and different sites the shredder will be cleaned so no cross contamination occurs.

Samples will be dispatched for next day, before noon, delivery on the day of chipping to: Sample reception (Stewart Bradley),, E.ON Technologies (Ratcliffe) Ltd, Technology Centre, Ratcliffe-on-Soar, Nottingham, NG11 0EE. The Field Station Manager, Fineshade should be informed of dispatch.

Appendix 7. Willow SRC sampling of fresh chips or billets to assess its average condition over the harvested field (described as overall-field condition)

Willow SRC sampling of fresh chip stack to assess overall-field condition

Site Criteria

Species - willow
Harvesting method - chip or billet
Timing - within 24 hours of harvest
Stack size - minimum of ca. 60 fresh tonnes (i.e. ca 130 - 200 m3).[6]

Office work

Select 36 sites:
6 warm, moist / light
6 warm, moist / medium
6 warm, moist / heavy
6 warm, dry / light
6 warm, dry / medium
6 warm, dry / heavy.

NB: Owing to a shortage of sites, some classes may have fewer than 6 sites

Field work:

For chip harvested sites take one E.ON 10 litre bucket with lid to each site. Take two buckets for billet harvested sites. If buckets are not available, then take sufficient tough polythene bags.

A tough plastic sheet (minimum size 2 m * 2 m), a large plastic bucket, a clean shovel, labels and a camera will also be required.

Use the same sampling procedure for chip stacks and billet stacks:

For sites harvested on two or more days, identify the stack (or part of any stack) which was harvested within one day i.e. harvested on the day of arrival or on the previous day. In the absence of local knowledge do this by eye.

Measure[7] the accessible circumference of the stack at ca 1 m height. Divide the circumference figure by 40 to give the spacing between samples to be taken.

If there is more than one fresh stack (or part of a stack) at the site, then divide the sum of the circumferences by 40 to give the spacing between samples to be taken. NB: omit any stack if there is any doubt that it is newly harvested.

Using a clean shovel, take 40 of ca 1 litre samples, evenly spaced around the accessible perimeter of the fresh stack/s. For fresh chip, harvested within the period of a day, this will be all way round the stack/s. Take the samples from ca 1 m above ground level, after first moving aside the outer 5 cm of surface chip at the sample point (which may have dried slightly)

[6] i.e. minimum of 1 ha of 20m^3 fresh / ha growth p.a. * 3 yrs

[7] Pacing will be sufficient

Spread the large plastic sheet on firm level ground. Moving around the stack/s as sampling progresses, place each shovel-sample within the large plastic bucket, tipping it out when necessary into a conical pile at the centre of the plastic sheet.

When the 40 samples have been taken, mix the conical pile of chips thoroughly, and spread it out over the sheet. Do not spread within 15 cm of the edge of the sheet (to avoid possibility of ground contamination of chips at the edge of the sheet).

For chips, fill one E.ON 10 litre bucket with ca 20 of ca 0.5 litre scoops distributed evenly from the surface of the mixed chips on the plastic sheet.

For chips, the bucket should be labelled on the lid and the side of the bucket with the date and site number from the map issued and SRC-W i.e. site 013 will labelled as 013/SRC. A similar label will be placed in a sealed clear plastic bag on top of the chips inside each bucket and the bucket lid replaced and sealed securely.

For billets, fill two E.ON 10 litre buckets with ca 10 of ca 1 litre scoops each, distributed evenly from the surface of the mixed chips on the plastic sheet.

For billets, the two buckets should be labelled on the lids and the sides of the buckets with the date and site number from the map issued and SRC-W i.e. site 013 will labelled as - 013/SRC.W, 1 of 2 and 013/SRC-W, 2 of 2. A similar label will be placed in a sealed clear plastic bag on top of the billets inside each bucket and the bucket lids replaced and sealed securely.

Despatch

Chips: The 1 bucket from each site will be despatched for next day, before noon, delivery on the day of chipping to: E.ON Technologies (Ratcliffe) Ltd, Technology Centre, Ratcliffe-on-Soar, Nottingham, NG11 0EE. The Field Station Manager, Fineshade should be informed of dispatch.

Billets: The 2 buckets from each site will be despatched for next day delivery to Research Worker (Colin Gordon) at NRS. The relevant Field Station Managers should be informed of dispatch.

Billet Processing protocol: Research Worker (Colin Gordon) at NRS

A suitable chipper or shredder will need to be hired for a short period if all sites can be done in the same week or fortnight.

Colin will receive:
2 x 10 litre buckets of billets from each billet harvested site (each bucket at least 2.5 kg).
The number of sites will depend on the number of sites harvested as billets.

Each site (of 2 buckets of billets) will be chipped separately to 2.5 cm chips. After chipping, the chips will be bulked into a suitable container and mixed thoroughly. A minimum of 3 kg sample will be sealed in one of the buckets (already labelled) in which the sample was received. One of the within-bucket labels, in its sealed clear plastic bag, will be placed on top of the chipped billets inside the bucket. The labels will have the site number and the sample type written on them i.e. 013/SRC – W.

The chipper will be cleaned between different samples so that no cross contamination occurs.

Samples will be dispatched for next day, before noon, delivery on the day of chipping to: E.ON Technologies (Ratcliffe) Ltd, Technology Centre, Ratcliffe-on-Soar, Nottingham, NG11 0EE. The Field Station Manager, Fineshade should be informed of dispatch.

MW 16/2/15v18215

Appendix 8. Willow SRC sampling protocols to assess within-field variation

Willow SRC within-field crop and soil sampling protocol

Site criteria

Species – willow
Age 3 years from cut-back
Site area - minimum 1 ha and 25 m wide

Office work

In-field Variation Study: three warm dry / heavy sites have been pre-selected for in-field variation sampling of the standing crop.

- For in-field variation biomass sampling, produce 3 polygon maps with grids.
  Grid distance = $\frac{\text{square root of A}}{20} \times 100$

- For soil sampling, for each site produce polygon maps with grids.
  Grid distance = $\frac{\text{square root of A}}{10} \times 100$

Infield Variation Study, Field work, Biomass sampling

These 3 sites will have 20 sample points

- Ensure there are sufficient robust sample bags for each site. At least 20 will be required.

- The sample bags should be labelled with the site number (from the map issued) / SRC-W / waypoint no / IF i.e. site 046 will have a bag marked as 046/SRC-W/1 to 20/IF

- If more than 1 bag is used for a sample type, write 1 of X, 1 of 2… etc. for however many are used.

- Locate waypoint position; there will be 20 per site.

- Take a photo of the crop and a photo of the ground at each waypoint, the photos will be labelled site number/waypoint/C for crop or G for ground and sent to the Field Station Manager, Fineshade when site is complete.

- Visually assess if the waypoint area should be classed as stony or not stony and record on the site assessment sheet.

- Take air temperature at 1.5 m height with a vertex at each waypoint and record on site assessment sheet.

- Visually assess and record recent weather conditions i.e. snow or frost present, recent heavy rain etc.

- Select the closest Tora willow stool to the waypoint. A minimum weight of 4 kg is required at each waypoint.

- Select the stem with the second largest diameter at breast height (dbh).

- Measure and record the stem dbh and the number of live stems on the stool on the site assessment sheet.

- Fell the stem, measure stem length to tip & mid diameter, and record on the site assessment sheet.

- Cut the entire stem into billets and place in a polythene bag/s. *Include any twigs or dead/dying leaves that may be attached and any side branches.*

- If the weight gathered from the stem is **equal to or more than 4 kg**, move onto the next waypoint.

- If the weight gathered from the first stem is **less than 4 kg**, choose the next largest dbh stem on the stool and repeat the exercise.
  NB: from 4 kg to a possible c 7 kg may be gathered from a waypoint this way.

- If the weight gathered from the first two stems **is still less than 4 kg**, choose the next largest dbh stem on the stool and repeat the exercise again, and so on until at least 4 kg is gathered from whole stems. *Do not take a part stems.*

- If there are insufficient stems on the stool, move to the next stool and repeat until at least 4 kg is gathered from whole stems. The largest dbh stem from the stool will remain in situ.

- Move to the next waypoint and repeat the exercise until all the waypoints have been completed.

- Record site details of aspect and percentage (%) slope on the site assessment sheet.

All sites, Field work, Soil sampling
At each of 10 waypoints per site, 50 g of soil excluding any stones and plant material will be collected at each way point.

To collect each soil sample:
- Scrape off the top humus / litter layer, if any.
- Dig a spade width hole to 30 cm from the adjacent ground level.
- Collect 25 g of soil from the 5 – 15 cm and 20 – 30 cm layers and bag together.
- Push soil temperature probe into ground horizontally at 10 cm depth. When the display has settled and no longer changes, record temperature on site assessment sheet.
- Fill soil pit in

Proceed to next waypoint and repeat.

On completing all 10 soil sample collections:
- On a clean tarpaulin or in a clean bucket mix all 10 samples thoroughly and bulk a representative sample of 300 g in to a sample bag (double bag if necessary).

- Label the bag as follows: Site No / Soil. i.e. 046/Soil

Soil samples should be sealed and stored in a cool dry and dark place until all team site samples are collected.

Biomass processing protocol: TSU Thetford

A Qualcast SDS2810, 2800 W quiet garden shredder will be used for this process.

TSU Thetford will receive:
Twenty of 4 kg to 7kg samples in bags from each of the three in-field variation sites i.e. 60 samples in total.
NB: It is possible that some samples will be in two or more bags labelled 1 of 2, 2 of 2 etc.

Each sample will be chipped twice into 2.5cm pieces separately. Between different samples the chipper will be cleaned so no cross contamination occurs.

After chipping, 3 kg of each sample will be sealed in a bag and labelled.
The labels will have the site number (from the map issued) / SRC-W / waypoint no / IF. Each sample will them be double bagged with the same label on the outer bag.

Samples will be dispatched for next day, before noon, delivery on the day of chipping to: Sample Reception (Stewart Bradley), E.ON Technologies (Ratcliffe) Ltd, Technology Centre, Ratcliffe-on-Soar, Nottingham, NG11 0EE. The Field Station Manager, Fineshade should be informed of dispatch *or* delivered straight to the lab.

Appendix 9. Poplar SRC sampling protocol to assess overall condition of fresh material (described as overall-field condition)

Poplar SRC standing crop and soil sampling protocol

Site criteria

Species – poplar, clones – Gaver, Ghoy and Gibecq.
Age 3 years from cut-back (ideally but +/- 1 year)
Site area – not limited, potentially minimum of 1 plot of 10 stools per clone

Office work

Sites have been sought from two climate zones (warm-moist, warm-dry) and three soil types (light, medium, heavy). Site availability is restricted to three sites covering warm-dry / heavy (1 of) and medium (2 of), with either one, two or three clones on the three sites.

- At least 1 waypoint location will be made for each clone (i.e. a minimum of 1, 2 or 3 waypoints depending on site) centrally within the area (plot) covered by the clone
- Where sites have more than 1 plot of a specific clone, waypoints will be increased to a maximum of 4 waypoints per clone distributed as evenly across the site as possible
- Where sites have only 1 plot of a specific clone there will be only one waypoint for that clone
- Ensure that there is a suitable map of the layout of clones within the crop available for use at the site, so that the correct clone can be identified for each sample location when waypoints are reached.

Field work

Ensure there are sufficient robust sample bags for each site.
The sample bags should be labelled with the site number from the map issued, the material it contains and the sample phase 1,. i.e. site 031 will have 1 bag at phase 1. 031/SRC-P/1.
If more than 1 bag is used write 1 of X, 1 of 2……etc for however many bags are used.

Sampling biomass at waypoints

For each clone:

- Locate 1st waypoint position
- Take a photograph of the ground and the coppice crop at each waypoint, targeting as far as possible the stools of the target clone and including both stool and crowns within shot. The photos will be labelled site number / waypoint / G for ground or C for crop and sent to the Field Station Manager, Fineshade when site is complete.
- Visually assess if each waypoint area should be classed as stony or not and record on the site field form.
- Take air temperature at 1.5m height with a vertex at each waypoint and record on the site field form.
- Original planting spacing: At each waypoint take the spacing by measuring at least 5 planting positions (or as many as possible if less), from the middle of the planting position to the middle of the planting position in and across rows.
  If 5 planting positions are used the result should be divided by 4 to give the correct distance.

- Please note if twin row planting is present.

- Determine the total weight ('**W**') of material to be collected at the waypoint as follows:

For **3 clone site**:
1,200 g divided by the number of waypoints for that waypoint's clone at the site i.e. 1 waypoint for clone = 1,200 g, 2 waypoints for clone = 600 g, 3 waypoints for clone = 400 g, 4 waypoints for clone = 300 g

For **2 clone** site:
1,800 g divided by the number of waypoints for that waypoint's clone at the site i.e. 1 waypoint for clone = 1,800 g, 2 waypoints for clone = 900 g, 3 waypoints for clone = 600 g, 4 waypoints for clone = 450 g

For **1 clone** site:
3,600 g divided by the number of waypoints for that waypoint's clone at the site i.e. 1 waypoint for clone = 3,600 g, 2 waypoints for clone = 1,800 g, 3 waypoints for clone = 1,200 g, 4 waypoints for clone = 900 g

- Select the **closest stool** of the target clone to the waypoint.

- Select the stem with the second largest diameter at breast height (dbh).

- Measure and record the stem dbh and the number of live stems on the stool on the site assessment sheet.

- Fell the stem, measure stem length to tip & mid diameter, and record on the site assessment sheet.

- Record dormancy of stool stems – dormant / bud burst / flushing / full leaf.

- Cut the entire stem into lengths and place in the polythene bag/s. Include any twigs or dead/dying leaves that may be attached and any side branches.

- Select the **second closest stool** of the target clone to the waypoint.

- Select the stem with the second smallest dbh.

- Measure and record the stem dbh and the number of live stems on the stool on the site assessment sheet.

- Fell/cut the stem, measure stem length to tip & mid diameter, and record on the site assessment sheet.

- Record dormancy of stool stems – dormant / bud burst / flushing / full leaf.

- Cut the entire stem into lengths and place in the polythene bag/s. Include any twigs or dead/dying leaves that may be attached and any side branches.

- If the weight gathered from the two stems is **less than 'W' g**, go to the **third closest stool** and repeat the exercise and so on. Do not take a part stems. Multiples of 2 stems are to be collected from each waypoint.

- Move to the next waypoint and repeat the exercise until all the waypoints have been completed.

- Visually assess and record recent weather conditions i.e. snow or frost present, recent heavy rain etc.

- Record aspect, percentage (%) slope and drainage (Poor / Moderate / Good) for the whole site on the site field form.

- Samples from the waypoints can bebagged together and should achieve at least 3.6 kg per site.

Poplar samples should be dispatched to TSU Thetford for next day delivery. TSU Thetford and the Field Station Manager, Fineshade should be informed of dispatch.

All sites, Field work, Soil sampling

- Determine the total weight ('**S**') of soil to be collected at the waypoint as follows:

600 g divided by the number of waypoints i.e. 1 waypoint = 600 g, 2 waypoints = 300 g, 3 waypoints = 200 g, 4 waypoints = 150 g, 5 waypoints = 120 g, 6 waypoints 100 g and so on (with a minimum of 50 g)

At each waypoint, '**S**' g of soil excluding any stones and plant material will be collected.

To collect each soil sample:
- Scrape off the top humus and litter layer, if any.

- Dig a spade width hole to 30 cm from the adjacent ground level.

- Collect **half x 'S'** g of soil from the 5 – 15 cm and **half x 'S'** g from the 20 – 30 cm layers and bag together.

- Push soil temperature probe into ground horizontally at 10cm depth. When the display has settled and no longer changes, record temperature on site assessment sheet.

- Fill soil pit in

Proceed to next waypoint and repeat.

On completing all soil sample collections:
- On a clean tarpaulin or in a clean bucket mix all samples thoroughly and bulk a representative sample of 500 g in to a sample bag (double bag if necessary).

- Label the bag as follows: Site Number / Soil. i.e. 031/Soil

Soil samples should be sent to arrive before 12 noon of the following day to: Sample Reception (Stewart Bradley), E.ON Technologies (Ratcliffe) Ltd, Technology Centre, Ratcliffe-on-Soar, Nottingham, NG11 0EE. The Field Station Manager, Fineshade should be informed of dispatch.

Biomass processing protocol: TSU Thetford

A Qualcast SDS2810, 2800 W quiet garden shredder will be used for this process.

TSU Thetford will receive:
At least 1 x 3.6 kg plus sample bag from each site.
NB: It is possible that some samples will be in two or more bags labelled 1 of 2, 2 of 2 etc.

The sample bag(s) from each site will be chipped into 2.5 cm pieces.
The chipped material will be bulked into a suitable container and mixed thoroughly.
A 3 kg sample will be sealed in a bag and labelled.
The labels will have the site number, the sample type and phase written on them i.e. 031/SRC-P/1.
Each sample will them be double bagged with the same label on the outer bag.

Between different samples the chipper will be cleaned so no cross contamination occurs.

Samples will be dispatched for next day, before noon, delivery on the day of chipping to: Sample Reception (Stewart Bradley), E.ON Technologies (Ratcliffe) Ltd, Technology Centre, Ratcliffe-on-Soar, Nottingham, NG11 0EE. The Field Station Manager, Fineshade should be informed of dispatch *or* delivered straight to the lab.

Appendix 10. Willow and poplar SRC sampling protocol to assess overall condition of stored material

Willow and poplar SRC sampling of stored chip stack to assess overall condition

Site Criteria

Species - willow (or poplar)
Harvesting method - chip
Timing - four weeks after harvest
Stack size - minimum of c. 60 fresh tonnes (i.e. ca 130 - 200 m^3).[8]

Office work

Select 36 sites: 3 warm, moist / light
3 warm, moist / medium
3 warm, moist / heavy
3 warm, dry / light
3 warm, dry / medium
3 warm, dry / heavy.

NB: Owing to a shortage of sites, some classes may have fewer than 6 sites

Field work:

Take sufficient 10 litre sample 'buckets' with lids.

Take the chip-stack sampling probe-pipe, 2 m long, plus handle.
A tough plastic sheet (minimum size 2 m * 2 m), a large plastic bucket and a clean shovel for mixing chip samples, labels and a camera will also be required.

Identify the target stack/s (or part of any stack) which was harvested at the same time i.e. harvested over a 1-5 day period and subsequently stored undisturbed as a stack/s at the same location in the same way. In the absence of local knowledge do this by eye.

Take photos of the stack from all accessible directions (2 to 4). Label with site Number/ Chip stack / photo Number.

Record the air temperature at the stack/s site. Visually assess and record recent weather conditions i.e. snow or frost present, recent heavy rain etc.

Record the height, width and length of each stack to be sampled (the 'target' stack/s), noting also for each:

- Whether the cross sectional profile is generally convex or an inverted ,V' shape
- Whether the stack to be sampled is *free standing* or directly *abuts* another that is <u>not</u> to be sampled (owing e.g. to an age difference).

Measure[9] the accessible circumference of the target stack/s at ca 1 m height. Divide the circumference figure by 10 to give the spacing between samples to be taken.

[8] i.e. minimum of 1 ha of 20m^3 fresh / ha growth p.a. * 3 yrs

[9] Pacing will be sufficient

If there is more than one target stack (or part of a stack) at the site, then divide the sum of the circumferences by 10 to give the spacing between samples to be taken. NB: omit any stack if there is doubt over its harvesting period or treatment, to ensure that samples are taken from stacks that are as similar as possible.

Using a chip-stack sampling probe-pipe, take 10 cores, from holes bored evenly spaced around the accessible perimeter of the fresh stack/s. Take each core in the following way:

a. Spread the large plastic sheet on firm, level ground convenient to accept the chip samples from all 10 sampling holes.
b. Locate the part of the chip-stack to be sampled.
c. Hold the handle end of the probe-pipe at chest height, angled slightly down wards, with the cutting end resting on the surface of the chip stack, aimed at the notional centre of the stack, at least 0.6 m above the notional ground level.
d. Lean on, and rotate, the probe-pipe so that it bores a hole into the stack under manual pressure. If necessary, withdraw the probe-pipe, collect the sample so-far in the bucket and then continue down the same hole until reaching the centre of the chip stack or the full extent of the probe-pipe, whichever occurs first.
e. Collect all the chips extracted from the hole and mix thoroughly in the large plastic bucket.
f. Tip the bucket contents in a conical pile in the centre of the large plastic sheet and mix again with the shovel. Do not spread within 6" of the edge of the sheet (to avoid possibility of ground contamination of chips at the edge of the sheet).
g. Move to the next sampling location and repeat (b) to (f) until core samples have been collected from all 10 locations.

Collect the final sample from the bulked and mixed sample chip pile as follows:

h. Ensure that the pile of sampled chips (on the plastic sheet) from the 10 locations is thoroughly mixed and free of any contamination from adjacent soil etc.
i. Take a part shovel-load of chips from each of at least 12 locations distributed evenly across and through the sample pile, aiming to collect at least 3 kg (approx. 12 – 15 litres) of chips. NB: the volume to weight conversion factor may vary, so use discretion to when loading the shovel.
j. Collect <u>at least</u> 3 kg of chips within a 10 litre sample bucket.
k. If the sample bucket contains less than a 3 kg sample, then part-fill a second sample bucket to achieve a total of at least 3 kg of sample. If a second sample bucket *is* used, fill it to at least ½ its capacity, even if this will be more than a total 3 kg sample. NB: this is to avoid shipping samples in near-empty containers.

Securely seal the sample bucket/s and label externally with the site number from the map issued and SRC-W i.e. site 017 will labelled as 017/SRC-W. Before sealing, place a duplicate label inside the bucket in a plastic bag.

Despatch
The sample buckets from each site will be despatched for next day, before noon, delivery on the day of chipping to: Sample reception (Stewart Bradley),, E.ON Technologies (Ratcliffe) Ltd, Technology Centre, Ratcliffe-on-Soar, Nottingham, NG11 0EE. The Field Station Manager, Fineshade should be informed of dispatch.

V1.1 MW/GH 2415

Appendix 11. Poplar SRF sampling protocol to assess overall-field condition

Poplar SRF protocol to assess overall-field condition

Site criteria:

Species – Poplar, clones - Gaver, Ghoy and Gibecq.
Site area - >1 ha with a minimum width of 25 m

Office work:

Select 18 sites – 3 warm, moist / light
3 warm, moist / medium
3 warm, moist / heavy
3 warm, dry / light
3 warm, dry / medium
3 warm, dry / heavy

Three waypoint selections will be made for each clone, distributed across the site.

Phasing for each site:

9 fixed sample points;
Phase 1, trees 1 – 9, felled and sampled straight away
Phase 2, trees 1 - 9, Phase 1 tree sampled 3 months later at secure compound
Phase 3, trees 10 – 18, felled when phase 2 sampled and sampled straight away
Phase 4, trees 10 – 18, Phase 3 tree sampled 3 months later at secure compound.

Plot 1 will contain trees 1 and 10
Plot 2 will contain trees 2 and 11
And so on.

Field work:

Ensure there are sufficient robust sample bags for each site a minimum of 2 will be required but more may be needed per site.
The sample bags should be labelled with the site number from the map issued, the material it will contain (Stem or Top for all phases) and the sample phase 1.
i.e. site 085 will have 2 bags at phase 1, 085/Stem/1, 085/Top/1
If more than 1 bag is used for a sample type, write 1 of X, 1 of 2…. etc for however many are used.

Phase 1:

Locate waypoint position; there will be 9 per site.

Take a photo of the crop and a photo of the ground at each waypoint, the photos will be labelled site number/waypoint/C for crop or G for ground and sent to the Field Station Manager, Fineshade when site is complete.

Visually assess if the waypoint area should be classed as stony or not and record on the site field form.

Put a 0.01 ha plot in and count number of live stems ≥7 cm present.

Select the closest single stemmed poplar tree to waypoint with a diameter at breast height (dbh) ≥7 cm.
Measure and record dbh on the site assessment sheet.

At each waypoint fell one tree (i.e. tree numbers 1 – 9), measure tree length to tip and record on the site assessment sheet.
Cut top off at 7 cm top diameter, measure stem length from butt to 7 cm top diameter and record on the site assessment sheet.
Cut a single billet length from the middle of the stem measuring 1.2 m length. This is the phase 2 sample and should be labelled with bio tape with site number and tree number then put to one side for transportation back to Thetford.
Two types of samples will be taken from this tree as follows:

i. A disc of 300 g will be cut with a bow saw from the top end of the bottom billet and the bottom end of the top billet; these will be bagged in the site sample bag for stem only.
ii. The top of the tree will be sampled; for every odd numbered tree, sampling will start from the 7 cm cut off point and for every even numbered tree, sampling will start from the top of the tree including the leader. Each twig and branch will be removed in turn, to no more than half the length of the top, 500 g of material is the aim of the collection, this can be left in lengths and will be bagged in the site sample bag for top only. If 500 g is not achieved stop at the half way point of the length of the top and collect no more at the way point.

Cut the top off where sampling has finished, the remaining top is for phase 2 and should be labelled with bio tape with site number and tree number then put to one side for transportation back to Thetford.

Record dormancy of tree – dormant / bud burst / flushing / full leaf

Take air temperature at 1.5 m height with a Vertex at each way point and record on site field form.

Visually assess and record recent weather conditions i.e. snow or frost present, recent heavy rain etc.

For phase 2:
The 1.2 m sample billet from phase 1 will have biodegradable tape tied round it and be labelled with Site Number and Tree Number to identify it in 3 months' time.
The remaining top from phase 1 will have biodegradable tape tied round it and be labelled with Site Number and Tree Number to identify it in 3 months' time.

The billets and tops will be transported to a secure compound at High Ash near Mundford. The labelled billets from phase 1 will be left in situ for 3 months, stacked on top of pre-prepared bearers. The remaining labelled tops from phase 1 will be placed over the stack.

Complete soil sample:

50 g of soil excluding any stones and plant material will be collected at each way point:
Scrape off the top humus / litter layer, if any.
Dig a spade width hole to 30 cm from the adjacent ground level.
Collect 25 g of soil from 5 – 15 cm and 15 – 30 cm layers and bag together.
Push soil temperature probe into ground horizontally at 10 cm depth. When the display has settled and no longer changes, record temperature on site field form.
Do not fill in soil pit at this stage.
On a clean tarpaulin or in a clean bucket mix all 9 samples thoroughly and bulk a representative sample of 300 g in to a sample bag (double bag if necessary).
Label bag as follows: Site Number i.e. 085/Soil

Soil samples should be sealed and stored in a cool dry and dark place until all team site samples are collected.

Proceed to next waypoint and repeat.

Record aspect, percentage (%) slope and drainage (Poor / Moderate / Good) for the whole site on the site field form.

All stem samples from phase 1 of the same site can be put in the same bag.
All top samples from phase 1 of the same site can be put in the same bag.

Stem samples should be sealed when site is complete and dispatched to Research Worker (Colin Gordon) at NRS for next day delivery. The relevant Field Station Managers and Research Worker should be informed of dispatch.

Top samples should be sealed when site is complete and dispatched to TSU Thetford for next day delivery. The Field Station Manager, Fineshade should be informed of dispatch.

Soil samples should be sent when all team sites are complete to arrive before 12 noon of the following day to: Sample reception (Stewart Bradley),, E.ON Technologies (Ratcliffe) Ltd, Technology Centre, Ratcliffe-on-Soar, Nottingham, NG11 0EE. The Field Station Manager, Fineshade should be informed of dispatch.

3 months after Phase 1 sampling: Phase 2 - TUS Fineshade team:
Ensure there are sufficient robust sample bags for each site a minimum of 2 will be required but more may be needed per site.
The sample bags should be labelled with the site number from the map issued, the material it will contain (Stem or Top) and the sample phase 2.
i.e. site 085 will have 2 bags, 085/Stem/2, 085/Top/2.
If more than 1 bag is used for a sample type, write 1 of X, 1 of 2…. etc for however many are used.

Locate the previously stacked billets and tops 1 – 9 for each site: 2 types of samples will be taken from these trees as follows:

i. A disc of 5 cm will be cut with a bow saw from each end of the billet and discarded. A disc of 500 g will be cut with a bow saw from each end of the billet and a 1 kg disc will be cut with a bow saw from the centre of the billet; these will be bagged in a sample bag for stem only.
ii. The top of the tree will be sampled; for every even numbered tree, sampling will start from the cut-off point and for every odd numbered tree, sampling will start from the top of the tree including the leader. Each twig and branch will be removed in turn until 500 g of material is collected, this can be left in lengths and will be bagged in the site sample bag for top only. If 500 g is not achieved stop and collect no more.

Visually assess and record recent weather conditions i.e. snow or frost present, recent heavy rain etc.

All stem samples from phase 2 of the same site can be put in the same bag.
All top samples from phase 2 of the same site can be put in the same bag.

Stem samples should be sealed when site is complete and dispatched to Research Worker (Colin Gordon) at NRS for next day delivery. The relevant Field Station Managers and Research Worker should be informed of dispatch.

Top samples should be sealed when site is complete and dispatched to TSU Thetford for next day delivery. The Field Station Manager, Fineshade should be informed of dispatch.

3 months after phase 1 sampling: Phase 3

Ensure there are sufficient robust sample bags for each site a minimum of 2 will be required but more may be needed per site.
The sample bags should be labelled with the site number from the map issued, the material it will contain (Stem or Top) and the sample phase 3.
i.e. site 085 will have 2 bags, 085/Stem/3, 085/Top/3. If more than 1 bag is used for a sample type, write 1 of X, 1 of 2…. etc for however many are used.

Locate waypoint position:

Select the next closest single stemmed poplar tree to waypoint with a dbh ≥7 cm.
Measure and record dbh on the site assessment sheet.
At each waypoint fell one tree (i.e. tree numbers 10 – 18), measure tree length to tip and record on the site assessment sheet.
Cut top off at 7 cm top diameter, measure stem length from butt to 7 cm top diameter and record on the site assessment sheet.
Cut a single billet length from the middle of the stem measuring 1.2 m length; this is the phase 4 sample and should be labelled with bio tape with site number and tree number then put to one side for transportation back to Thetford.
Two types of samples will be taken from this tree as follows:

i. A disc of 300 g will be cut with a bow saw from the top end of the bottom billet and the bottom end of the top billet; these will be bagged in the site sample bag for stem only.
ii. The top of the tree will be sampled; for every odd numbered tree, sampling will start from the 7 cm cut off point and for every even numbered tree, sampling will start from the top of the tree including the leader. Each twig and branch will be removed in turn, to no more than half the length of the top, 500 g of material is the aim of the collection, this can be left in lengths and will be bagged in the site sample bag for top only. If 500 g is not achieved stop at the half way point of the length of the top and collect no more at the way point.

Cut the top off where sampling has finished, the remaining top is for phase 4 and should be labelled with bio tape with site number and tree number then put to one side for transportation back to Thetford.

Record dormancy of tree – dormant / bud burst / flushing / full leaf

Take air temperature at 1.5 m height with a Vertex at each way point and record on site field form.

Visually assess and record recent weather conditions i.e. snow or frost present, recent heavy rain etc.

Locate soil pit.
Push soil temperature probe into ground horizontally at 10 cm depth. When display has settled and no longer changes, record temperature on site field form.
Fill in soil pit.
For phase 4:
The 1.2 m sample billet from phase 3 will have biodegradable tape tied round it and be labelled with site Number and tree Number to identify it in 3 months' time.

The remaining top from phase 3 will have biodegradable tape tied round it and be labelled with site Number and tree Number to identify it in 3 months' time.

The billets and tops will be transported to a secure compound at High Ash near Mundford. The labelled billets from phase 3 will be left in situ for 3 months, stacked on top of pre-prepared bearers. The remaining labelled tops from phase 3 will be placed over the stack.

Proceed to next waypoint and repeat.

All stem samples from phase 3 of the same site can be put in the same bag
All top samples from phase 3 of the same site can be put in the same bag

Stem samples should be sealed when site is complete and dispatched to Research Worker (Colin Gordon) at NRS for next day delivery. The relevant Field Station Managers and Research Worker should be informed of dispatch.

Top samples should be sealed when site is complete and dispatched to TSU Thetford for next day delivery. The Field Station Manager, Fineshade should be informed of dispatch.

6 months after phase 1 sampling: Phase 4
Ensure there are sufficient robust sample bags for each site a minimum of 2 will be required but more may be needed per site.
The sample bags should be labelled with the site number from the map issued, the material it will contain (Stem or Top) and the sample phase 4.
i.e. site 085 will have 2 bags, 085/Stem/4, 085/Top/4.
If more than 1 bag is used for a sample type, write 1 of X, 1 of 2…. etc for however many are used.

Locate the previously stacked billets and tops 10 – 18 for each site: 2 types of samples will be taken from these trees as follows:

i. A disc of 5 cm will be cut with a bow saw from each end of the billet and discarded. A disc of 500 g will be cut with a bow saw from each end of the billet and a 1 kg disc will be cut with a bow saw from the centre of the billet; these will be bagged in a sample bag for stem only.
ii. The top of the tree will be sampled; for every even numbered tree, sampling will start from the cut-off point and for every odd numbered tree, sampling will start from the top of the tree including the leader. Each twig and branch will be removed in turn until 500 g of material is collected, this can be left in lengths and will be bagged in the site sample bag for top only. If 500 g is not achieved stop and collect no more.

Visually assess and record recent weather conditions i.e. snow or frost present, recent heavy rain etc.

All stem samples from phase 4 of the same site can be put in the same bag.
All top samples from phase 4 of the same site can be put in the same bag.

Stem samples should be sealed when site is complete and dispatched to Research Worker (Colin Gordon) at NRS for next day delivery. The relevant Field Station Managers and Research Worker should be informed of dispatch.

Top samples should be sealed when site is complete and dispatched to TSU Thetford for next day delivery. The Field Station Manager, Fineshade should be informed of dispatch.

Processing protocol: months 0, 3 and 6

A suitable chipper will need to be hired for a short period if all sites can be done in the same week or fortnight.

Colin will receive

For phase 1, 1 sample each of stem from up to 18 sites, month 0.
For phase 2, 1 sample each of stem from up to 18 sites, month 3
For phase 3, 1 sample each of stem from up to 18 sites, month 3.
For phase 4, 1 sample each of stem from up to 18 sites, month 6.

Each phase, site and sample will be dealt with individually.

The Phases 1 and 3 stem samples (each 18 x 300g) plus the Phases 2 and 4 stem samples (each 9 x 2 kg) labelled Site Number/Stem/(*Phase)* from each site will be chipped into 1" (2.5 cm) chip size.
The chips will be bulked into a suitable container and mixed thoroughly.
A 4 kg sample will be sealed in buckets supplied by E.ON. A label will be placed in a sealed clear plastic bag on top of the chips inside the bucket and a label will be put on the lid and the side of the bucket. The labels will have the site number, the sample type and phase clearly written on them i.e. 085/Stem/(*Phase)*. If more than 1 bucket is used for a sample type, write 1 of X, 1of 2…. etc for however many are used on the label.

Between different samples and different sites the chipper will be cleaned so no cross contamination occurs.

Samples will be dispatched for next day, before noon, delivery on the day of chipping to: Sample reception (Stewart Bradley),, E.ON Technologies (Ratcliffe) Ltd, Technology Centre, Ratcliffe-on-Soar, Nottingham, NG11 0EE. The Field Station Manager, Fineshade should be informed of dispatch.

TSU Thetford will receive

For phase 1, 1 sample each of top from up to 18 sites, month 0.
For phase 2, 1 sample each of top from up to 18 sites, month 3
For phase 3, 1 sample each of top from up to 18 sites, month 3.
For phase 4, 1 sample each of top from up to 18 sites, month 6.

The 4.5 kg top samples labelled Site Number/Top/(*Phase)* from each site will be chipped into 1" (2.5cm) chip size.
The chips will be bulked into a suitable container and mixed thoroughly.
A 3 kg sample will be sealed in buckets supplied by E.ON. A label will be placed in a sealed clear plastic bag on top of the chips inside the bucket and a label will be put on the lid and the side of the bucket. The labels will have the site number, the sample type and phase clearly written on them i.e. 001/Top/(*Phase)*. If more than 1 bucket is used for a sample type, write 1 of X, 1of 2…. etc for however many are used on the label.

Samples will be dispatched for next day, before noon, delivery on the day of chipping to: Sample reception (Stewart Bradley),, E.ON Technologies (Ratcliffe) Ltd, Technology Centre, Ratcliffe-on-Soar, Nottingham, NG11 0EE. The Field Station Manager, Fineshade should be informed of dispatch.

Appendix 12. Conifer SRF sampling protocol to assess overall field condition

SRF conifer protocol to assess overall field condition

Site criteria:

Species – Sitka spruce (SS)
Age 12 - 16 years
Site area - >1 ha with a minimum width of 25 m

Office work:

Select 12 sites – 3 cold, wet / mineral
3 cold, wet / organic
3 warm, moist / mineral
3 warm, moist / organic

Produce polygon map with grids.

$$\text{Grid distance} = \frac{\text{square root of A}}{10} \times 100$$

Produce waypoint shape file to download to GPS.

Phasing for each site:

10 fixed sample points;
Phase 1, trees 1 – 10, felled and sampled straight away
Phase 2, trees 1 - 10, Phase 1 tree sampled 3 months later (No bark)
Phase 3, trees 11 – 20, felled when phase 2 sampled and sampled straight away
Phase 4, trees 11 – 20, Phase 3 tree sampled 3 months later (No bark)

Plot 1 will contain trees 1 and 11
Plot 2 will contain trees 2 and 12
And so on.

Field work:

Ensure there are sufficient robust sample bags for each site a minimum of 3 will be required but more may be needed per site.
The sample bags should be labelled with the site number from the map issued, the material it will contain (Bark for phases 1 and 3 only, Stem or Top for all phases) and the sample phase 1.
i.e. site 001 will have 3 bags at phase 1, 001/Bark/1, 001/Stem/1, 001/Top/1
If more than 1 bag is used for a sample type, write 1 of X, 1of 2…. etc for however many are used.

For phase 1:
Locate waypoint position; there will be 10 per site.

Take a photo of the crop and a photo of the ground at each waypoint, the photos will be labelled site number/waypoint/C for crop or G for ground and sent to the Field Station Manager, Fineshade when site is complete.

Visually assess if the waypoint area should be classed as stony or not and record on the site field form.

Select the closest single stemmed Sitka spruce tree to waypoint with a dbh ≥7 cm.
Measure and record dbh on the site assessment sheet.
At each waypoint fell one tree (i.e. tree numbers 1 – 10), measure tree length to tip and record on the site assessment sheet.
Cut top off at 7 cm top diameter, measure stem length from butt to 7 cm top diameter and record on the site assessment sheet.
Cut the stem into 3 billets of equal length.
Three types of samples will be taken from this tree as follows:

i. The top end of the bottom billet and the bottom end of the top billet will be peeled and 150 g of bark from each will be bagged in the site sample bag for bark only.
ii. A disc of 500 g will be cut with a bow saw from the top end of the bottom billet and the bottom end of the top billet and will be bark free; these will be bagged in the site sample bag for stem only.
iii. The top of the tree will be sampled; for every odd numbered tree, sampling will start from the 7 cm cut off point and for every even numbered tree, sampling will start from the top of the tree including the leader. Each whorl will be removed in turn, to no more than half the length of the top, 500 g of material is the aim of the collection, this can be cut up with secateurs or bill hook and will be bagged in the site sample bag for top only. If 500 g is not achieved stop at the half way point of the length of the top and collect no more at the way point.

Take air temperature at 1.5 m height with a Vertex at each way point and record on site field form.

Visually assess and record recent weather conditions i.e. snow or frost present, recent heavy rain etc.

For phase 2:
The middle billet from phase 1 will have biodegradable tape tied round it to identify it in 3 months' time.
The 3 billets from phase 1 will be left in situ for 3 months, stacked with the 2 end billets side by side length ways on the floor and the middle billet lain on top of them.
The remaining top of the tree from phase 1 will be placed over the stack.

Complete soil sample:

50 g of soil excluding any stones and plant material will be collected at each way point:
Scrape off the top humus / litter layer, if any.
Dig a spade width hole to 30 cm from the adjacent ground level.
Collect 25 g of soil from 5 – 15 cm and 15 – 30 cm layers and bag together.
Push soil temperature probe into ground horizontally at 10 cm depth. When the display has settled and no longer changes, record temperature on site field form.
Do not fill in soil pit at this stage.
On a clean tarpaulin or in a clean bucket mix all 10 samples thoroughly and bulk a representative sample of 300 g in to a sample bag (double bag if necessary).
Label bag as follows: Site Number i.e. 001/Soil

Soil samples should be sealed and stored in a cool dry and dark place until all team site samples are collected.

Proceed to next waypoint and repeat.

Record both aspect and percentage (%) slope for the whole site on the site field form.

All bark samples from phase 1 of the same site can be put in the same bag.

All stem samples from phase 1 of the same site can be put in the same bag.
All top samples from phase 1 of the same site can be put in the same bag.

Tree samples should be sealed when site is complete and dispatched to Research Worker (Colin Gordon) at NRS for next day delivery. The relevant Field Station Managers and Research Worker should be informed of dispatch

Soil samples should be sent when all team sites are complete to arrive before 12 noon of the following day to: Sample reception (Stewart Bradley),, E.ON Technologies (Ratcliffe) Ltd, Technology Centre, Ratcliffe-on-Soar, Nottingham, NG11 0EE. The Field Station Manager, Fineshade should be informed of dispatch.

3 months after phase 1 sampling: (Phase 2, 3 and 4)
Ensure there are sufficient robust sample bags for each site a minimum of 5 will be required but more may be needed per site.
The sample bags should be labelled with the site number from the map issued, the material it will contain (Bark for phase 3 only, Stem or Top for phases 1, 3 and 4) and the sample phase 2 or 3.
i.e. site 001 will have 5 bags, 001/Stem/2, 001/Top/2, 001/Bark/3,001/Stem/3, 001/Top/3
If more than 1 bag is used for a sample type, write 1 of X, 1 of 2…. etc for however many are used.

Locate waypoint position:

For phase 2:
Locate waypoint position
Locate the previously felled and stacked tree numbers 1 - 10: 2 types of samples will be taken from these trees as follows:

i. A disc of 500 g will be cut with a bow saw from each end of the middle billet and will be bark free; these will be bagged in a sample bag for stem only.
ii. The top of the tree will be sampled; for every even numbered tree, sampling will start from the cut-off point and for every odd numbered tree, sampling will start from the top of the tree including the leader. Each whorl will be removed in turn until 500 g of material is collected, this can be cut up with secateurs or bill hook and will be bagged in a sample bag for top only. If 500 g is not achieved stop and collect no more at the way point.

Take air temperature at 1.5 m height with a Vertex at each waypoint and record on site field form.

Locate soil pit.
Push soil temperature probe into ground horizontally at 10cm depth. When display has settled and no longer changes, record temperature on site field form.
Do not fill in soil pit at this stage.

Visually assess and record recent weather conditions i.e. snow or frost present, recent heavy rain etc.

For phase 3:
Select the next closest single stemmed Sitka spruce tree to waypoint with a dbh ≥7 cm.
Measure and record dbh on the site assessment sheet.
At each waypoint fell one tree (i.e. tree numbers 11 – 20), measure tree length to tip and record on the site assessment sheet.

Cut top off at 7 cm top diameter, measure stem length from butt to 7 cm top diameter and record on the site assessment sheet.
Cut the stem into 3 billets of equal length.
Three types of samples will be taken from this tree as follows:

i. The top end of the bottom billet and the bottom end of the top billet will be peeled and 150 g of bark from each will be bagged in the site sample bag for bark only.
ii. A disc of 500 g will be cut with a bow saw from the top end of the bottom billet and the bottom end of the top billet and will be bark free; these will be bagged in the site sample bag for stem only.
iii. The top of the tree will be sampled; for every odd numbered tree, sampling will start from the 7 cm cut off point and for every even numbered tree, sampling will start from the top of the tree including the leader. Each whorl will be removed in turn, to no more than half the length of the top, 500 g of material is the aim of the collection, this can be cut up with secateurs or bill hook and will be bagged in the site sample bag for top only. If 500 g is not achieved stop at the half way point of the length of the top and collect no more at the way point.

For phase 4:
The middle billet from phase 3 will have biodegradable tape tied round it to identify it in 3 months' time.
The 3 billets from phase 3 will be left in situ for 3 months, stacked with the 2 end billets side by side length ways on the floor and the middle billet lain on top of them.
The remaining top of the tree from phase 3 will be placed over the stack.

Proceed to next waypoint and repeat.

All bark samples from phase 3 of the same site can be put in the same bag
All stem samples from phase 2 of the same site can be put in the same bag
All stem samples from phase 3 of the same site can be put in the same bag
All top samples from phase 2 of the same site can be put in the same bag
All top samples from phase 3 of the same site can be put in the same bag

Tree samples should be sealed when site is complete and dispatched to Research Worker (Colin Gordon) at NRS for next day delivery. The relevant Field Station Managers and Research Worker should be informed of dispatch.

6 months after phase 1 sampling: (Phase 4)
Ensure there are sufficient robust sample bags for each site a minimum of 2 will be required but more may be needed per site.
The sample bags should be labelled with the site number from the map issued, the material it will contain (Stem or Top) and the sample phase 4.
i.e. site 001 will have 2 bags, 001/Stem/4, 001/Top/4
If more than 1 bag is used for a sample type, write 1 of X, 1of 2…. etc for however many are used.

Locate waypoint position:

For phase 4:
Locate the previously felled and stacked tree numbers 11 - 20: 2 types of samples will be taken from these trees as follows:

i. A disc of 500 g will be cut with a bow saw from each end of the middle billet and will be bark free; these will be bagged in a sample bag for stem only.
ii. The top of the tree will be sampled; for every even numbered tree, sampling will start from the cut-off point and for every odd numbered tree, sampling will start

from the top of the tree including the leader. Each whorl will be removed in turn until 500 g of material is collected, this can be cut up with secateurs or bill hook and will be bagged in a sample bag for top only. If 500 g is not achieved collect no more at the way point.

Take air temperature at 1.5 m height with a Vertex at each waypoint and record on site field form.

Locate soil pit.
Push soil temperature probe into ground horizontally at 10 cm depth. When display has settled and no longer changes, record temperature on site field form.
Fill in soil pit.

Visually assess and record recent weather conditions i.e. snow or frost present, recent heavy rain etc.

All stem samples from phase 4 of the same site can be put in the same bag
All top samples from phase 4 of the same site can be put in the same bag

Tree samples should be sealed when site is complete and dispatched to Research Worker (Colin Gordon) at NRS for next day delivery. The relevant Field Station Managers and Research Worker should be informed of dispatch.

Processing protocol: Research Worker (Colin Gordon) - months 0, 3 and 6

A suitable chipper is to be hired for the appropriate period.

Colin will receive:

For phase 1, 1 sample each of stem, top and bark from 12 sites, month 0.
For phase 2, 1 sample each of stem and top from 12 sites, month 3
For phase 3, 1 sample each of stem, top and bark from 12 sites, month 3.
For phase 4, 1 sample each of stem and top from 12 sites, month 6.

Each phase, site and sample type will be dealt with individually.

The 20 x 500 g stem samples labelled Site Number/Stem/(*Phase)* from each site will be chipped into 1" (2.5 cm) chip size.
The chips will be bulked into a suitable container and mixed thoroughly.
A 5 kg or 2 full bucket samples will be sealed in buckets supplied by E.ON, a label will be placed in a sealed clear plastic bag on top of the chips inside the bucket and a label will be put on the lid and the side of the bucket. The labels will have the site number and the sample type and phase clearly written on them i.e. 001/Stem/(*Phase)*. If more than 1 bucket is used for a sample type, write 1 of X, 1of 2…. etc for however many are used on the label.

The 5 kg top samples labelled Site Number/Top/(*Phase)* from each site will be chipped into 1" (2.5cm) chip size.
The chips will be bulked into a suitable container and mixed thoroughly.
A 3 kg sample will be sealed in buckets supplied by E.ON, a label will be placed in a sealed clear plastic bag on top of the chips inside the bucket and a label will be put on the lid and the side of the bucket. The labels will have the site number, the sample type and phase clearly written on them i.e. 001/Top/(*Phase)*. If more than 1 bucket is used for a sample type, write 1 of X, 1of 2…. etc for however many are used on the label.

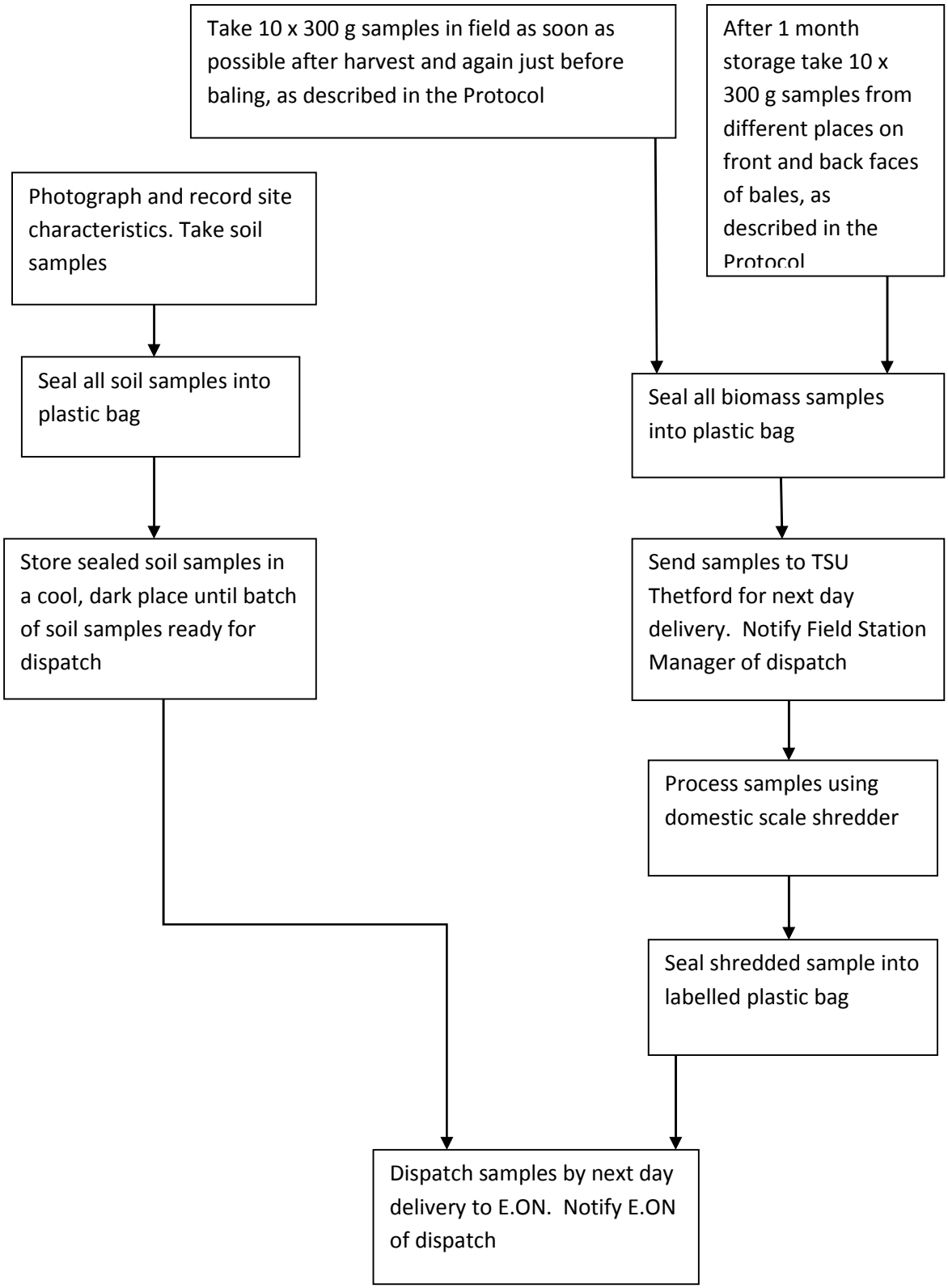
The 3 kg bark samples labelled Site Number/Bark/(*Phase)* from each site will be chipped into 1" (2.5cm) chip size.
The chips will be bulked into a suitable container and mixed thoroughly.
A 2 kg sample will be sealed in buckets supplied by E.ON, a label will be placed in a sealed clear plastic bag on top of the chips inside the bucket and a label will be put on the lid and the side of the bucket. The labels will have the site number, the sample type and phase clearly written on them i.e. 001/Bark/(*Phase)*. If more than 1 bucket is used for a sample type, write 1 of X, 1of 2…. etc for however many are used on the label.

Between different samples and different sites the chipper will be cleaned so no cross contamination occurs.

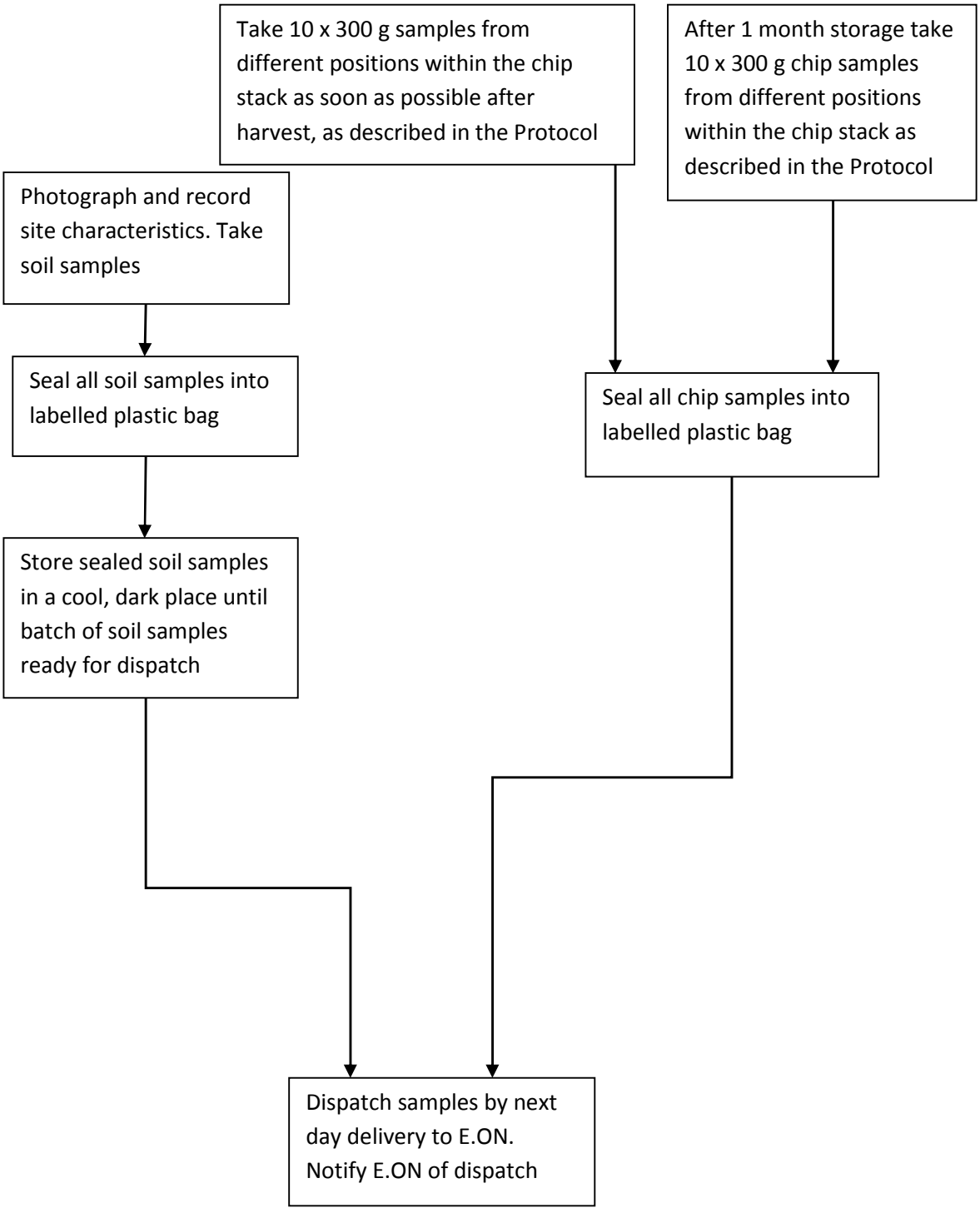
Samples will be dispatched for next day, before noon, delivery on the day of chipping to: Sample reception (Stewart Bradley), E.ON Technologies (Ratcliffe) Ltd, Technology Centre, Ratcliffe-on-Soar, Nottingham, NG11 0EE. The Field Station Manager, Fineshade should be informed of dispatch.

Appendix 13. Flow charts for field collection and dispatch of samples

Sample collection process flow chart: *Miscanthus*

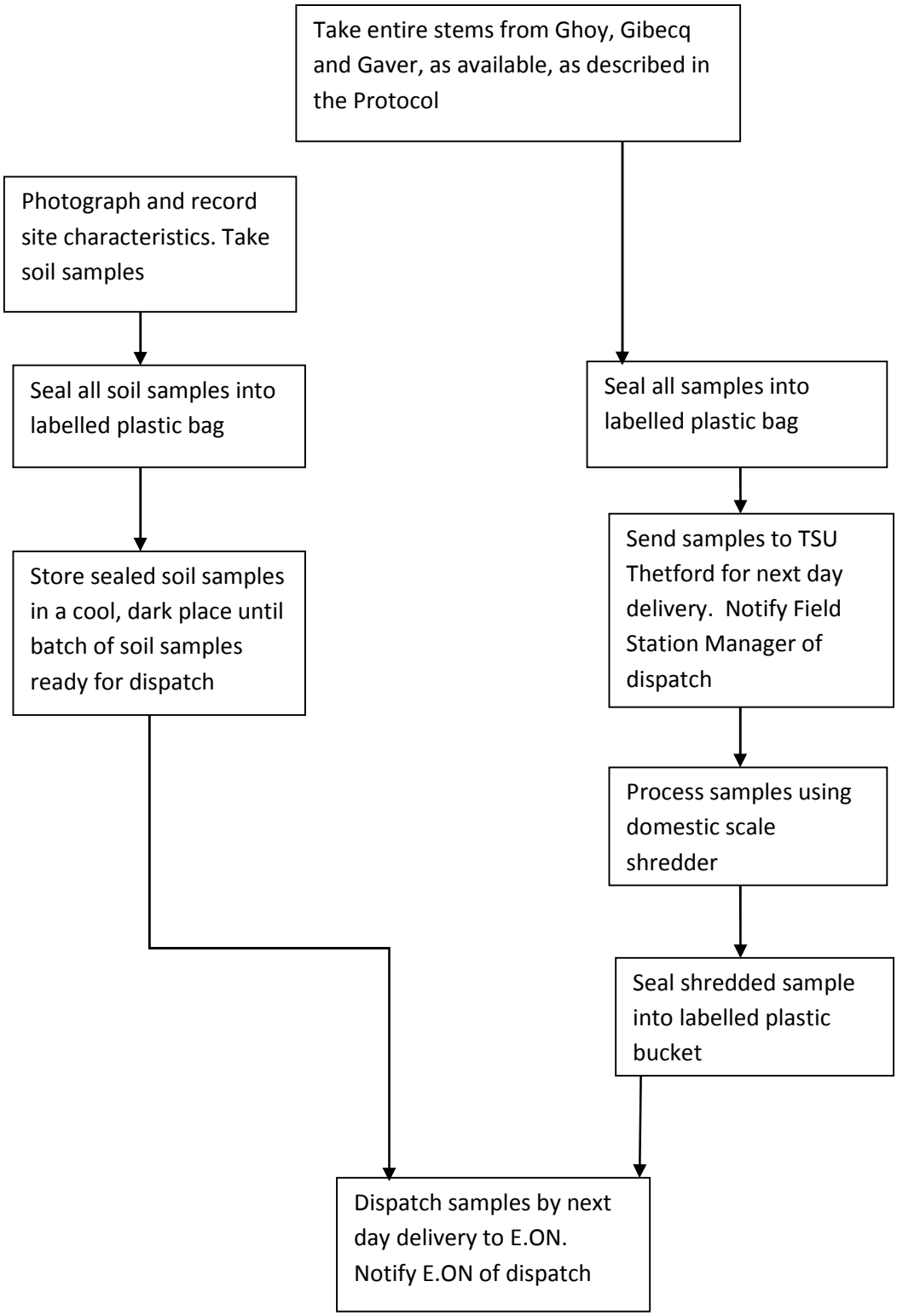


Sample collection process flow chart: willow SRC

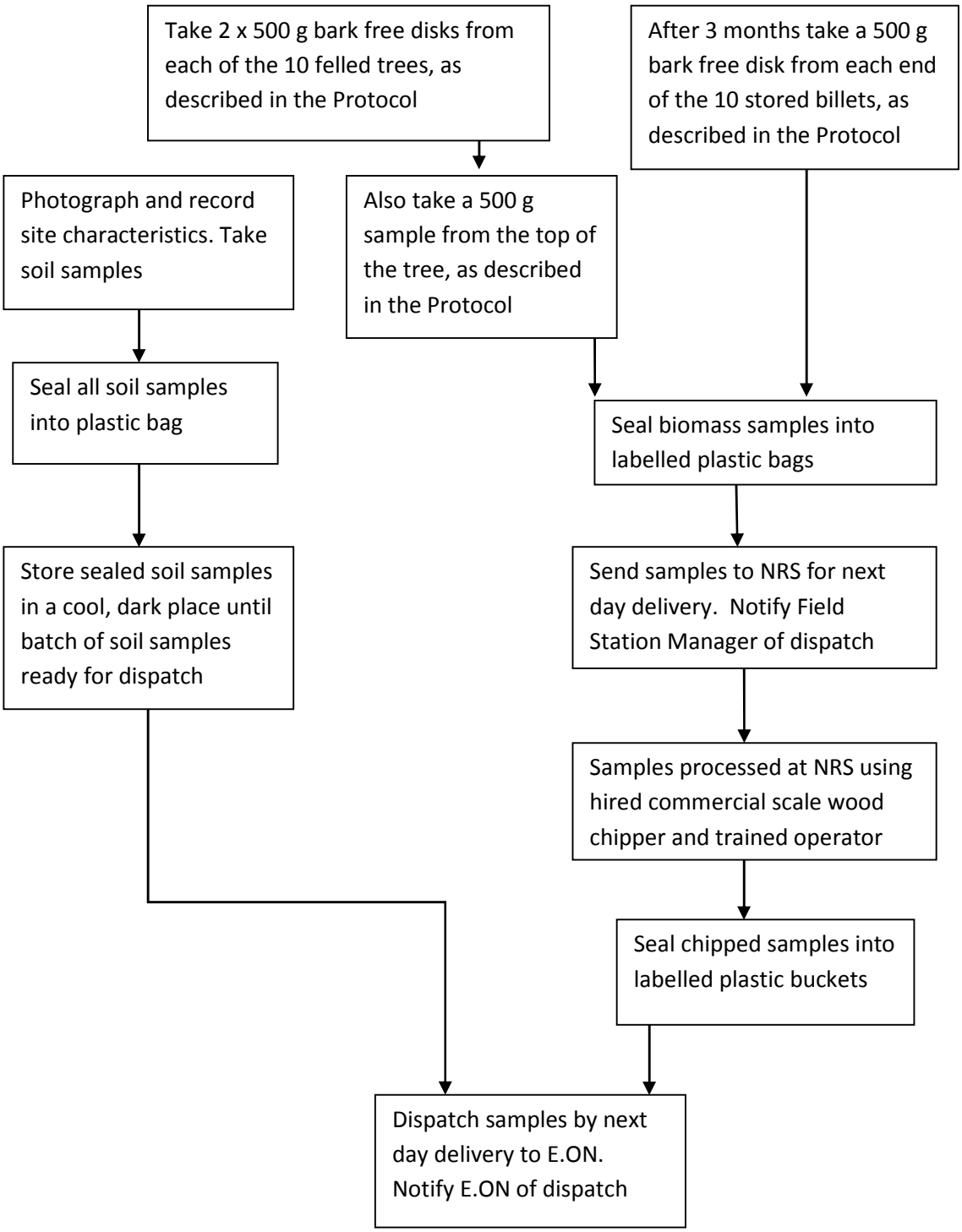


Sample collection process flow chart: willow SRC (within-field variation)

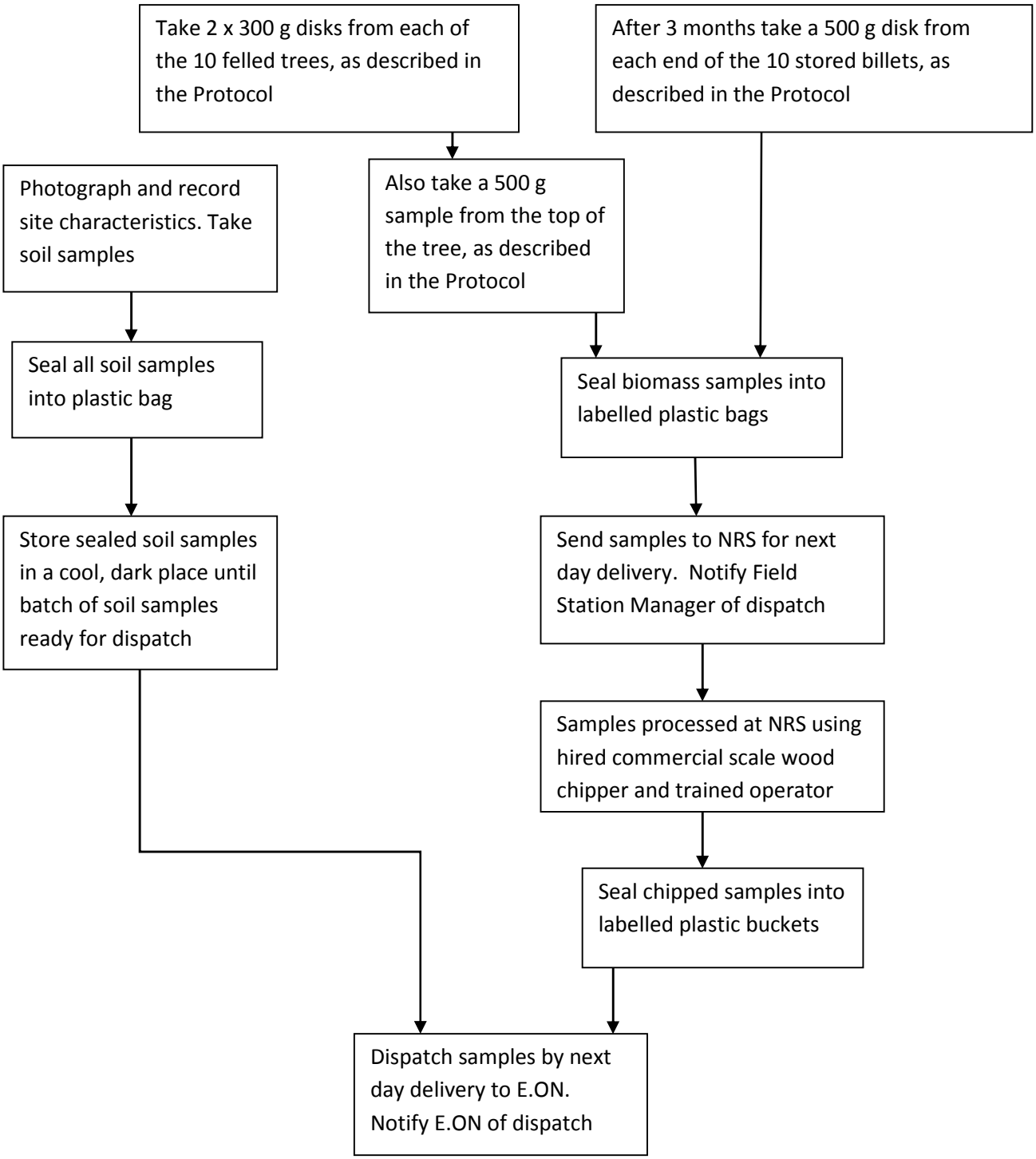
Sample collection process flow chart: poplar SRC



Sample collection process flow chart: conifer SRF



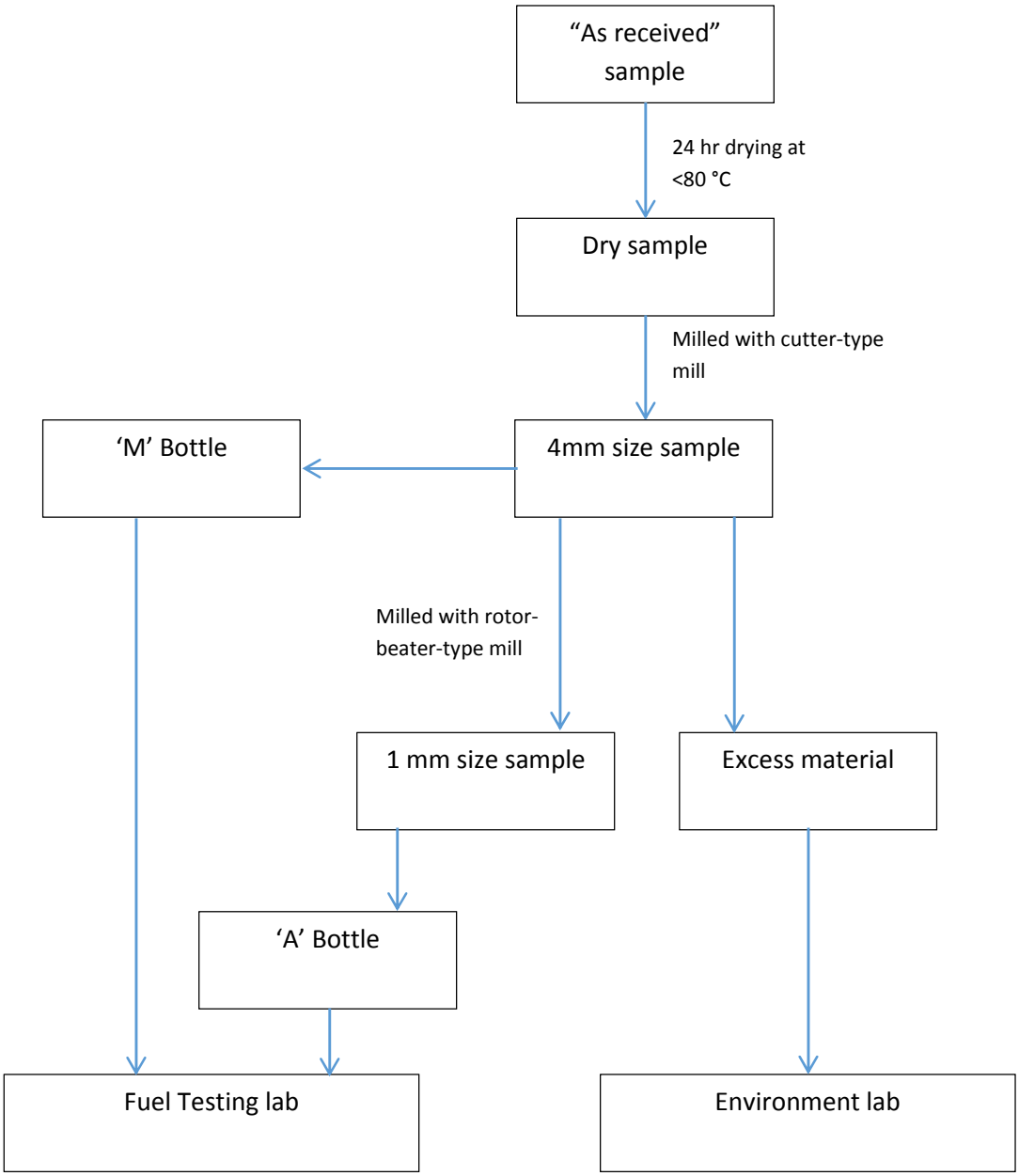
Sample collection process flow chart: poplar SRF



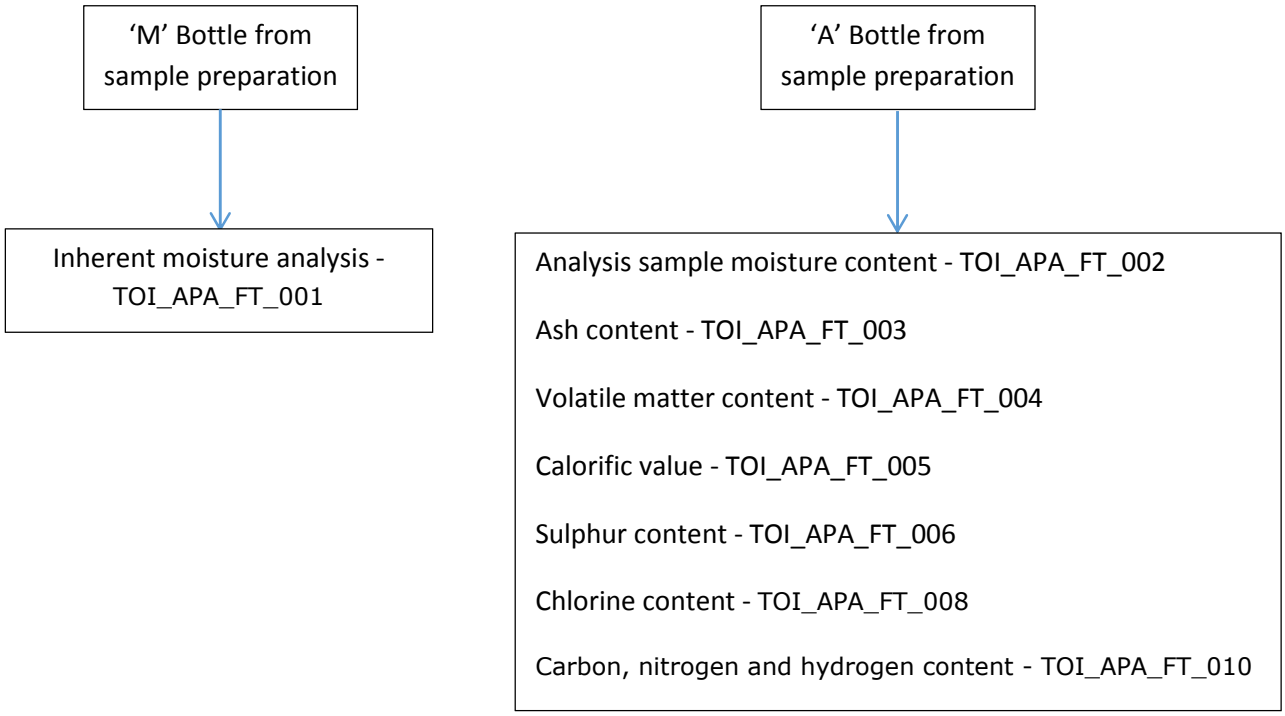
Appendix 14. Process flow charts for sample preparation and analysis at ETG

Process Flow chart: sample preparation

TOI-APA_FT_009



Process Flow chart: Fuel testing laboratory



Process Flow Chart: Environment laboratory