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Stratified Sampling to Define Levels of Petrographic Variation in Coal Beds: Examples from Indonesia and New Zealand

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Abstract - Stratified sampling of coal beds for petrographic analysis using block samples is a viable alternative to standard methods of channel sampling and particulate pellet mounts. Although petrographic analysis of particulate pellets is employed widely, it is both time consuming and does not allow variation within sampling units to be assessed - an important measure in any study whether it be for paleoenvironmental reconstruction or in obtaining estimates of industrial attributes. Also, samples taken as intact blocks provide additional information, such as texture and botanical affinity that cannot be gained using particulate pellets. Analysis of variance tests (ANOVA) on stratified samples from an Eocene coal bed in Kalimantan, Indonesia showed that the largest amount of microscopic variation occurs between coal types indicating they are petrographically dissimilar. The ANOVA tests also indicated that only a maximum of 125 point counts are needed to make precise estimates of block composition. Confidence limits of estimates from stratified sampling were calculated to be comparable to what would have been obtained using standard crushed particulate pellets. This means less work is needed for the same accuracy of estimates using a stratified sampling method over that of crushed particulate pellets for coal beds. Macroscopic point counting of vitrain bands can accurately account for those particles >1 mm within a coal interval. This point counting method is conducted using something as simple as string on a coal face with marked intervals greater than the largest particle expected to be encountered (although new technologies are being developed to capture this type of information digitally). Comparative analyses of particulate pellets and blocks on the same interval show less than 6% variation between the two sample types when blocks are recalculated to include macroscopic counts of vitrain. Therefore even in coarse-grained coals, stratified sampling can be used effectively and representatively.

Keywords: coal type, analysis of variance, stratified sampling, fine- and coarse-grained coals

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Introduction

It is the Holy Grail of any descriptive system to minimise time spent characterising something whilst maximizing the defining information. Descriptions of rocks are no different. For example, the bulk of what makes a sandstone, a sandstone and not a mudstone can be seen with the eye without further work. That is not to say that when certain questions need to be answered, additional analyses and/or more detailed descriptions are needed. With coal beds, however, it is generally the case that they receive little to no macroscopic characterisation. Rather, samples are usually extensively analysed, but only in the laboratory, after any distinguishable visible features have been destroyed through crushing and grinding.

There are several extant macroscopic classifications for coal (ASTM, 1982; Cameron, 1978; Davis, 1978; George, 1982; Schopf, 1960; Stopes, 1919, 1935). But most of these are qualitative in nature and only occasionally used, if at all. There are a few classification systems which allow quantitative assessments that can statistically test visible texture (*e.g.* Esterle *et al.*, 2002; Esterle *et al.*, 1992; Ferm *et al.*, 2000; Moore *et al.*, 2000; Moore *et al.*, 1993; Yu *et al.*, 1997), but again, like the other classification systems, these are under utilised.

One of the reasons that macroscopic characterisation of coal is usually not performed is because it is believed that it yields information of little value, in either applied or academic circumstances. However, this is simply not true; many of the fundamental properties of coal result from its organic composition (*e.g.* Anggara *et al.*, 2014; Beamish and Crosdale, 1998; Crosdale, 1995; Esterle *et al.*, 1994; Hower and Wild, 1994; Lamberson and Bustin, 1993; Mares and Moore, 2008; Moore, 2012; Moore *et al.*, 2002) which is mostly estimated through petrographic analysis. Current methods for obtaining estimates of the petrographic composition of coal are both simple and elegant. Various standards exist for this procedure but most are similar in design (ICCP, 1994, 1998, 2001; ISO, 2009). In general, these procedures consist of three broad steps: 1) collection of samples, 2) sample preparation in the laboratory and 3) microscopic quantification of proportions of components.

In the first step, a coal bed is sampled by either coring or careful channelling. The latter process is usually performed at a mine face or outcrop and consists of digging a column of coal approximately 10 cm deep by 10 cm wide with the length corresponding to the interval being sampled. This method allows proportional representation of all parts of a sampling interval. A sampling interval can consist of either a whole bed profile or a single interval.

In the second step, samples are crushed to less than 1 mm and a split of approximately 50 to 100 g is taken for petrographic analysis. Because of the small size of the ground particles the composition of a sample interval will be proportionally represented. Next, the split for petrographic analysis is embedded in epoxy-resin and placed within a ~2.5 cm diameter mould. Once hardened, the pellet is polished at one end with a series of progressively finer polishing compounds.

Estimates of proportions of microscopic components are made on the polished surface of the particulate pellet. Point count traverses are made with a spacing that should be greater than the largest particle. Because the crushed coal particles are randomly distributed about the polished surface of the pellet, confidence limits for the petrographic components follow the binomial sampling distribution. As predicted by the binomial distribution theory, 500 points are generally needed to estimate components with a proportion of 50% (the hardest to accurately estimate; Snedecor and Cochran, 1989) with confidence limits of ± 5%.

A disadvantage of the particulate pellet method is that some of the petrographic components in the original sample may be larger than the 1 mm size to which the coal is crushed. Although these particles will appear in proportion to their occurrence in the original material, they will be dislocated fragments amongst the other particles and binding medium. Therefore spatial relationships between particles are lost precluding any textural analyses.

In a study of an Eocene coal bed in southeast Kalimantan, Indonesia the standard method of point count analysis failed to discriminate between macroscopically recognisable coal types (Moore and Ferm, 1992). In addition, the standard analysis could not account for the cause in variability of some quality parameters such as grindability (Moore, 1990). As a consequence, an alternative method of sampling and measurement was required. This method utilised uncrushed blocks, which retain the texture of the coal that was the original basis of sampling. Using the block sampling method also allowed for the microscopic identification of the larger macroscopic components i.e. vitrain bands, which almost always consists of plant organs or tissues. Subsequent observations led to a botanically based petrographic classification which has been detailed in Esterle *et al*. (1992), Moore (1990), Moore and Ferm (1992) and Shearer and Moore (1994a, b; 1999).

An incumbent problem of using block samples is in accurately representing the macroscopic variation of a ply within a single block. For fine-grained or thinly (≤1 mm) banded coals this is not a problem as blocks of sufficient size can be taken which represent the macroscopic variation of an interval (Moore, 1990). However, coarse-grained or thickly (>1 mm) banded coals pose the problem of requiring block samples that would be of an impractical size. Therefore it was decided to macroscopically point count the larger (>1 mm) bright bands in the coal face adjacent to where block samples were collected. Because few bright bands were wider than 2 cm, macroscopic point counts were made with a 2 cm spacing. For each mark on the line the presence/absence of a bright band greater than 1 mm in width was recorded and the thickness of the bands also noted (Moore *et al*., 1993). Only one count was made on each bright band, even if the band was greater than 2 cm thick. A size of 1 mm was chosen as the lower limit for macroscopic point counting for two reasons. First, the size of bands less than 1 mm thick are difficult to estimate visually and second, the proportion of bands of this thickness are adequately represented on collectable block samples.

The major advantage with block sampling is in assessing sample representivity and in identifying the sources (or levels) of variation in the proportion of microscopic constituents. A sampling design was constructed which could assess the level of maximum variation *e.g.* between or within macroscopic coal types and whether blocks were representative of intervals from which they were sampled. This design utilised a stratified sampling method that allows both testing for differences between macroscopic coal types and also determines confidence limits for estimates of petrographic components. Therefore, the objectives of this paper are:

- to describe this sampling design and the subsequent treatment of data,
- to determine if the macroscopically recognisable coal types are consistently petrographically dissimilar,
- to quantify confidence limits for the estimates of the microscopic components,
- to compare these confidence limits with those obtained from standard petrographic analyses, and finally,
- to determine how representative blocks are compared to standard particulate pellets for the studied coals.

Location and Sampling

Coal samples used in testing stratified sampling methods for estimation of petrographic composition were collected from two locations in Indonesia (Figure 1). Coal samples used in testing the representivity of block sampling in coarse-grained coals were collected from two locations within a single mine from New Zealand (Figure 2). The coal from Indonesia comes from Eocene age deposits that outcrop in several south-west trending linear belts in southeast Kalimantan, Indonesia (Friederich *et al*., 1995, 1999). In these areas, coal bed thickness range from less than 0.5 to 13 m. The coal is subbituminous A to high volatile bituminous ($Ro_{(max)}$ approximately 0.55) in rank (Friederich *et al*., 1999; Hutton *et al*., 1994). Some of the inorganic layers in the Eocene coal beds have been attributed to volcanic ash fall (Ruppert and Moore, 1993).

The New Zealand sample location is within the Cretaceous age, Morley Coal Measures of the Ohai coalfield and is subbituminous to bituminous in rank (Butland and Moore, 2008; Shearer, 1992; Sykes, 1988). This locality represents deposits that accumulated from mostly Podocarp vegetation in a tectonic pull-apart basin (Shearer, 1995; Shearer and Moore, 1994b; Warnes, 1990). More on the geology and coal-forming characteristics can be found in Lyon and Giggenback (1994), Shearer (1995), Sherwood *et al*. (1992), Sykes (1985) and Warnes (1990).

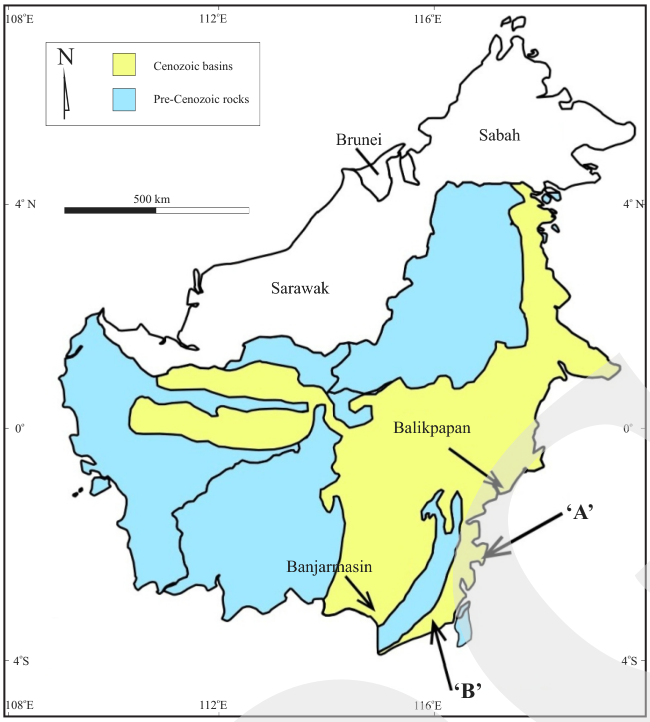

Figure 1. Index map showing location of sample sites 'A' and 'B' in Indonesia. Kalimantan provinces are differentiated based on distribution of Cenozoic sediments.

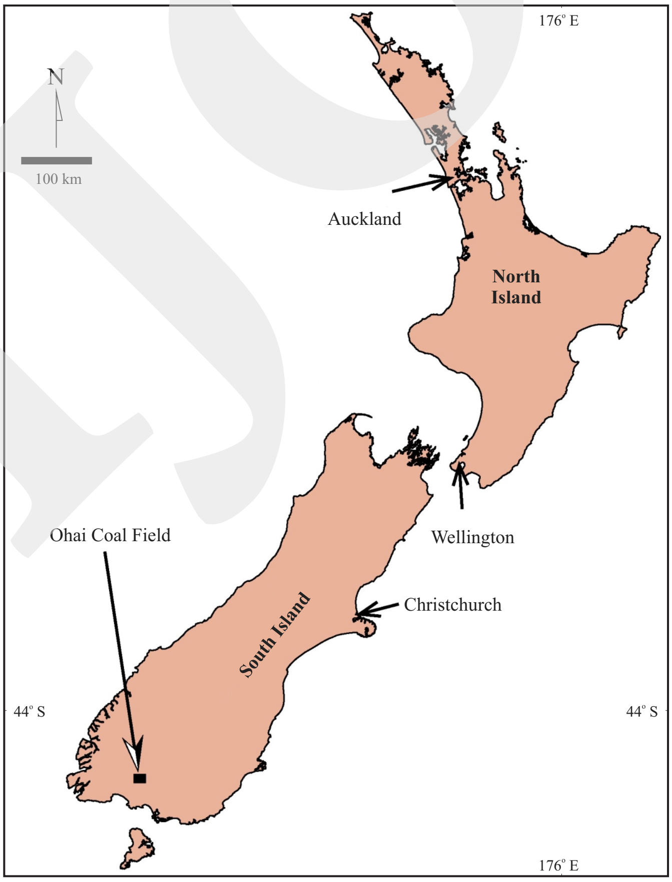

Figure 2. Index map showing location of sample site in the Ohai Basin, South Island, New Zealand.

Sampling Locations

At the Indonesian sites, coal was collected from two locations where the entire bed was available for sampling. In site 'A', coal was collected from a fresh mine face whereas continuous core was sampled for site 'B' (Figure 3). At the area surrounding site 'A' coal bed thickness varies from 3 to 6 m. Site 'A' consist entirely of bright structure-less coal with the exception of several thin (<5 cm thick) inorganic partings (Ruppert and Moore, 1993). Careful examination, however, showed that some parts of the bed included thin (1 to 2 mm) bands of glassy, homogeneous "vitrain" set in a bright structure-less matrix sometimes referred to as "attrital" layers (Schopf, 1960). Therefore two coal types were identified, a bright coal without banding ("bright non-banded") and a bright coal containing vitrain bands ("bright banded") (Figure 4; Moore *et al.*, 1992). The banded – non-banded coal types, in addition to the inorganic layers, led to a field description of the bed as being made up of layers of banded and non-banded bright coal ranging from 0.1 to 0.5 m in thickness.

Since the bright glassy bands were mostly less than or equal to 1 mm thick, small blocks of coal approximately 3 x 3 x 3 cm were considered to be of sufficient size that many bands would be included in the sample. Two such blocks were collected at random from two randomly selected layers of both banded and non-banded coal types. This procedure permitted comparison between coal types and allowed evaluation of variation at the layer and block sampling levels. As a re-

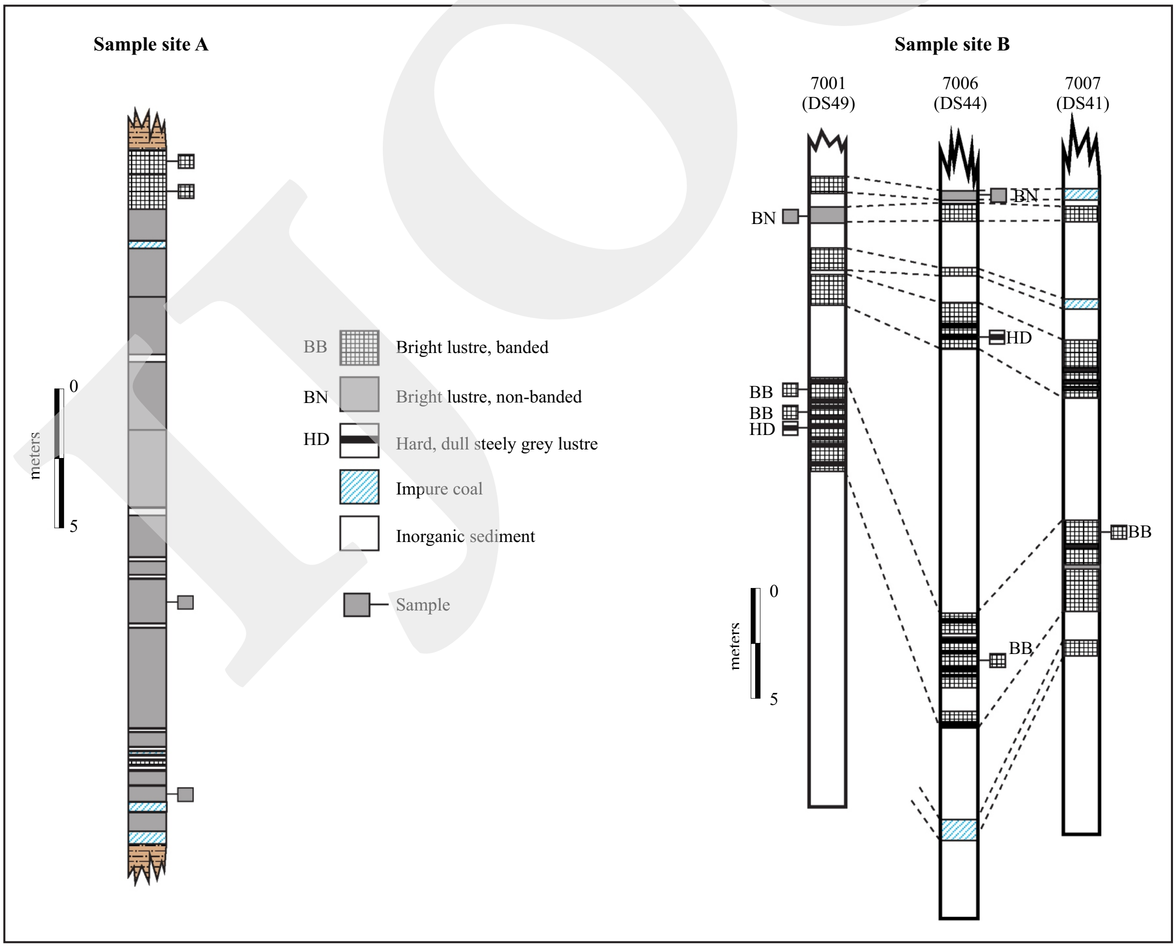

Figure 3. Coal type variation within the Eocene age coal at sites 'A' and 'B' in Kalimantan, Indonesia. Note that samples from site 'A' were taken from a fresh mine face, whilst samples from site 'B' were collected from fresh core. Distance between the three cores are approximately 400 m. Sampling procedures are described in the text.

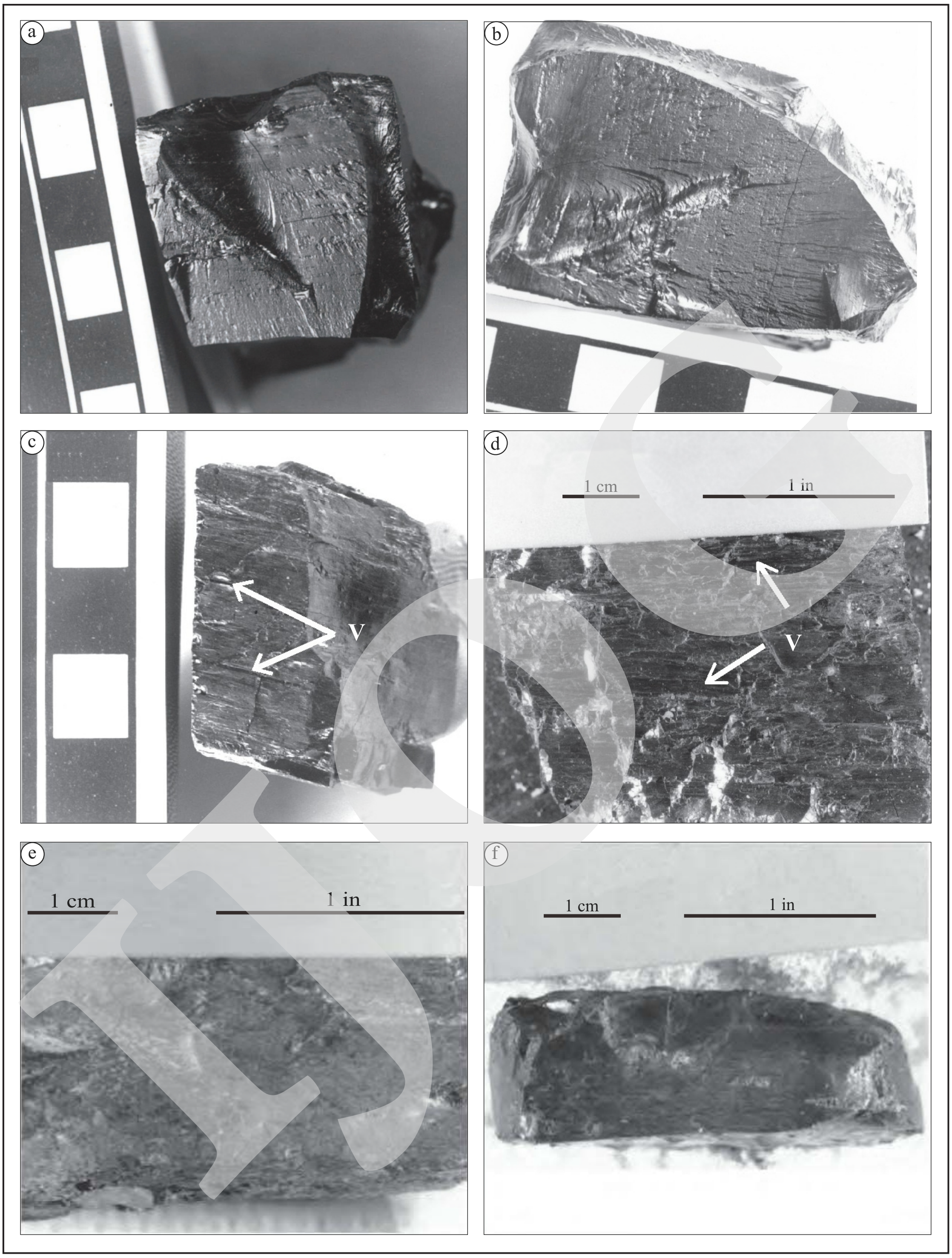

Figure 4. Macroscopic coal types of the Eocene coal in southeast Kalimantan, Indonesia (see also Moore *et al*, 1992). (a) and (b) bright, non-banded, scale in centimetres; (c) and (d) bright, banded, 'v' indicated vitrain bands, scale in centimetres; (e) and (f) dull, steely grey.

sult, 8 blocks were collected from site 'A', half of which represented banded coal and the other half non-banded. It should be noted that this sampling procedure does not rely on proportional representation except at the block level in which the block should be of sufficient size to include a large number of recognizable components. This is a form of stratified sampling that relies on the recognition of homogeneity within strata in order to reduce sample variance and increase efficiency of sampling.

At the second sampling location in Indonesia the same procedures were followed except that an additional coal type was recognised. This was a very hard, splintery, dull coal with a steel grey lustre that occurred in several layers but was generally <5 cm thick (Figure 4). Therefore the bed was described as alternating layers of banded bright, non-banded bright and hard, dull steel grey coal. Similar to site 'A', two blocks from two layers of each coal type were collected. An exception was made for the steel grey coal type because of its thinness (<5 cm) and therefore only one block each from two layers could be sampled.

A coal bed within the Morley Coal Measures from the Ohai coalfield in New Zealand was chosen to test if a coarse-grained coal can be accurately sampled and its petrographic composition estimated. Numerous vitrain bands, often clearly displaying growth rings were observed through out the coal (Shearer, 1992; Shearer and Moore, 1994b).

Two locations were sampled from the Morley coal both from the former Wairaki underground coal mine. At location #1, ten plies were channel sampled and at location #2, nine plies were sampled. Each ply had both a channel sample taken which was later crushed and prepared for particulate pellet analysis as well as two blocks of ~3x5 cm as part of the stratified sampling. Because of the mining method of leaving a metre of top and bottom coal, a complete section could not be sampled. The middle part of the bed was composed exclusively of a bright, well-banded coal type (Figure 5). Macroscopic point counts of two sections in the underground mine showed

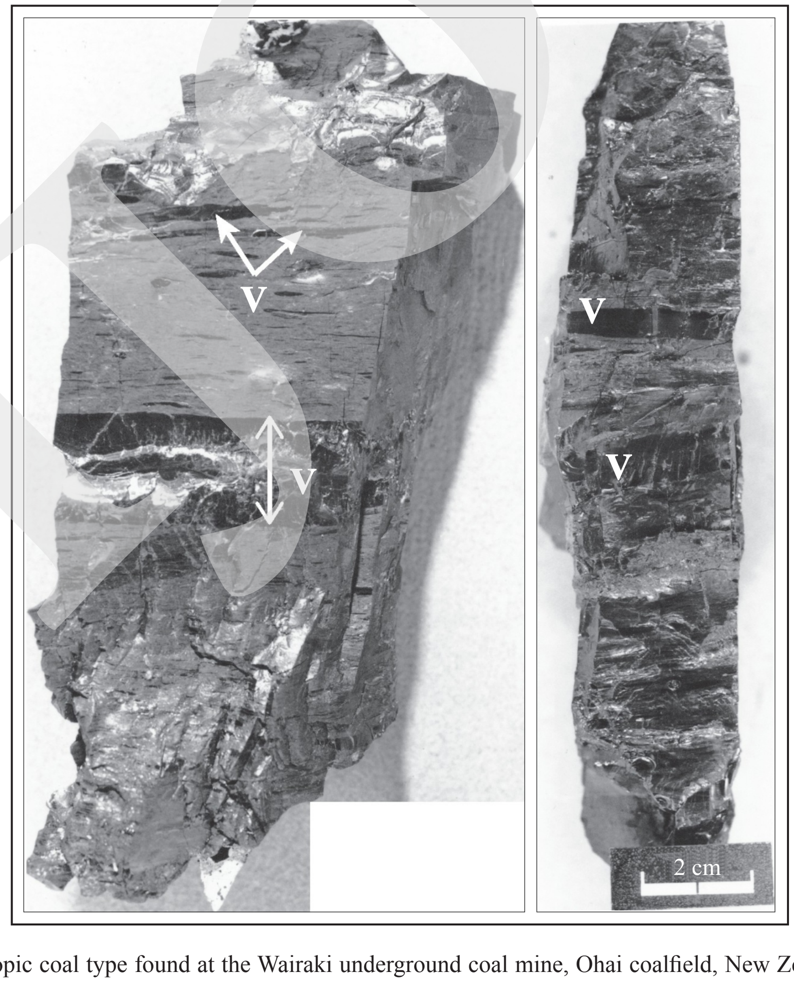

Figure 5. Macroscopic coal type found at the Wairaki underground coal mine, Ohai coalfield, New Zealand, 'v' indicates vitrain bands.

that the coal type was composed of approximately 25% vitrain bands. Macroscopic point counting followed procedures outlined in Moore *et al*. (1993) and Shearer and Moore (1994b). It should be noted that other means of characterisation of macroscopic banding are also available, most notably described in Yu *et al*., (1997).

Laboratory Procedures

In the laboratory all blocks were mounted intact in thermoplastic resin and surfaces were polished at right angles to bedding. After etching the surface with an acidified solution of potassium permanganate in order to enhance plant tissue structures (Moore and Swanson, 1993; Pontollilo and Stanton, 1994; Stanton and Moore, 1991), estimates of petrographic components were generated by point count traverses. The point count traverses were at right angles to the bedding and spaced at 2 mm which was larger than all but a few of the largest particles. A total of 500 points were counted on each block to make estimates comparable to particulate pellet analysis. In the blocks from site 'A' two traverses of 250 points were made on each block in order to evaluate variation within a block. Because of the very high degree of uniformity obtained from the results at site 'A' the procedures for site 'B' were modified so that four traverses of 125 points were counted on each block. This allowed comparison with site 'A' data to determine whether relatively stable estimates of petrographic components can be obtained with fewer numbers of points.

Identification of components at each point was made first with X 40 or X 125 magnification in order that plant organ/tissue type could be ascertained for the larger particles. Magnification was then increased to X 400 so that the type of tissue of the plant part could also be determined. Matrix components, because of their small size (generally less than 100 μm), were characterized at X 400 magnification. Analyses were first conducted in white reflected light and then in blue-light illumination in order to best recognize components such as spores/pollen, resins and cuticles. A total of 18 kinds of components were identified in the course of the study and their description is given in Moore (1990) and Moore and Ferm (1992). Because some of the frequencies of components are small, the organic materials have been grouped into three major classes. The only restriction for subdivision of classes was that the mean occurrence of a component had to be ≥ 5%. The three classes are:

Plant Organs and Tissues

Most are recognizable woody material displaying differing degrees of decay and could clearly be related to the bright vitrain bands in the banded coal type. These components range in size from 0.031 to 2 mm (Moore and Ferm, 1992). Based on morphology and frequency of occurrence two subclasses of plant organs/tissues can be identified:

- Moderately to well-preserved woody tissue;
- Poorly preserved plant organs/tissues which include some recognizable stems and/or roots with secondary growth, leaves and plant tissues that were so poorly preserved that categorization was difficult.

Particulate Matrix

It consists of a great variety of fragments that range in size from 0.125 to 0.0005 mm (0.5 to 125 μm) (Moore and Ferm, 1992). Based on frequency of occurrence, morphology, reflectance in white-reflected light and character in blue-light illumination two subclasses of particulate matrix are recognized:

- Fragments of cell walls and cell fillings,
- Other particulate matrix which include cell fillings without adjoining cell walls, algae, resins, spore/pollen grains and fungal remains.

Amorphous Matrix

The matrix consists of material with diffuse or indistinguishable grain boundaries. Based on blue-light fluorescent characteristics two types of amorphous matrix can be recognized:

- Amorphous humic gels, which in white light, are grey in colour and fill in spaces between other particles (under blue-light illumination this material does not fluoresce);
- Amorphous bitumen, which does fluoresce in blue-light and possesses a dark brown to black vitreous reflectance in white light.

During point counting, both standard maceral components and plant part/matrix data were captured using a system described in Moore and Orrell (1991) and Moore *et al*. (1989), although those data are not presented in this paper.

Vitrain layers, when examined microscopically always revealed they were composed exclusively of plant tissues, in various levels of decomposition (Figure 6). The differing levels of decay, of course, have been arrested and preserved as a result of the coalification process. The macroscopic matrix layers are microscopically composed of both particulate and amorphous matrix as well as some thin, non-visible to the naked eye plant organs and tissues (Figure 7). Oxidized material was not recognised in either macroscopic (fusain) or petrographic (inertininte) examination with the exception of microscopically identifiable fungal remains that have a high reflectance as a result of its original organic structure rather than from oxidation through combustion or microbial alteration (Moore *et al*. 1996). Petrographic characterisation of the coarse-grained coal from New Zealand used procedures consistent with the ICCP (1994, 1998, 2001). Although the primary microscopic classification systems are different between the sampling sites in New Zealand and Indonesia (i.e. plant part/matrix petrographic system for Indonesia and ICCP system for New Zealand), this is immaterial in this study since the locations are testing different, though certainly allied, characterisation questions.

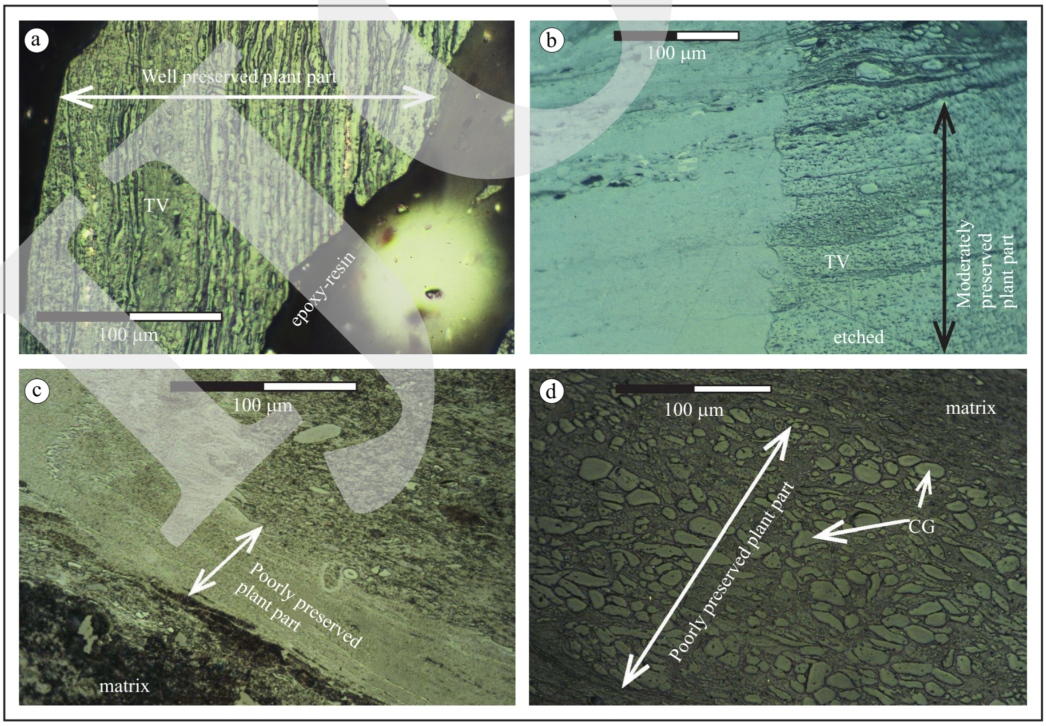

Figure 6. Photomicrographs of plant tissues in various states of decomposition; all samples were etched (see text). (a) Well preserved plant part showing cell tissue. In a standard maceral classification, this would be telovitrinite (TV) or possibly even telinite (x600). (b) Moderately preserved plant part, both unetched (left hand side of photomicrograph) and etched (right hand side). In standard maceral terminology this would be termed telovitrinite (‘TV’, x400). (c) Poorly preserved plant part with virtually no recognizable cell tissue (x600). (d) Poorly preserved plant part, with virtually no recognizable cell tissue but abundant cell fillings (corpogelinite, ‘CG’; x500).

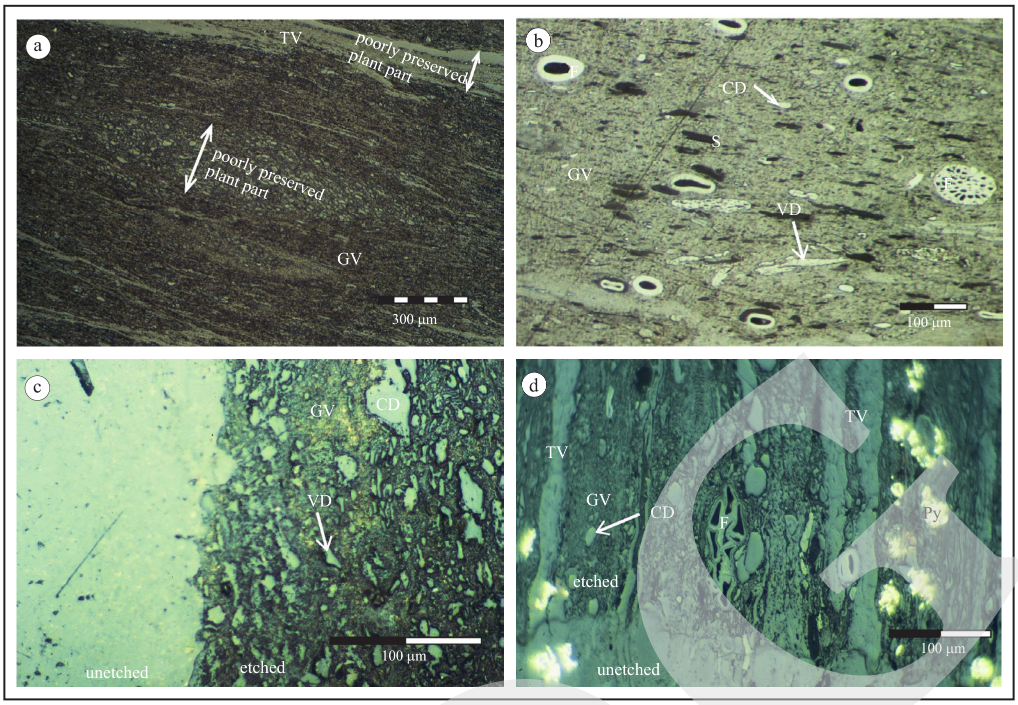

Figure 7. Photomicrographs of matrix layers illustrating typical microscopic organic composition; all samples were etched (see text). (a) Mostly matrix material (dark groundmass) which is a combination of amorphous humic gels ('GV' = gelovitrinite) and bitumen and particulate matrix consisting of fragments of cell walls, spores/pollen, resin and other material. Some poorly preserved plant part material can also be noted which would be termed telovitrinite ('TV') in a conventional maceral analysis. Note that the poorly preserved plant part in the centre of the photomicrograph is the same as the one shown in Figure 6 d. (b) Matrix of particulate and amorphous material. 'F' = funginite, 'CD' = collodetrinite, 'VD' = vitrodetrinite, 'S' = sporinite. (c) Matrix material illustrating particulate and amorphous material. The dark, non granular material between the organic material is the amorphous humic gels. (d) Predominately matrix material with some plant parts visible. 'Py' = pyritic material.

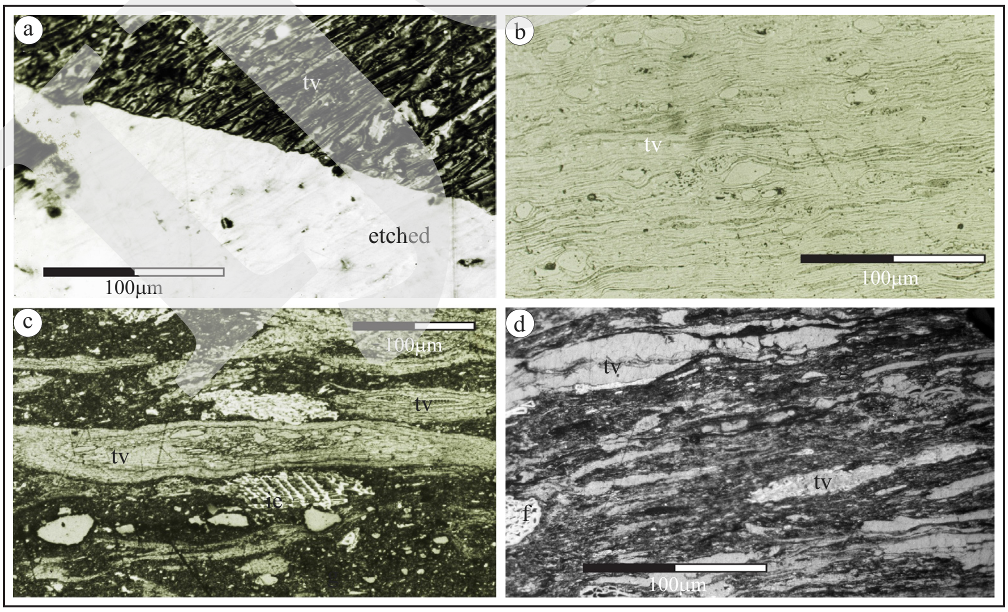

Figure 8. Photomicrographs of petrographic components of the coarse-grained coal, Morley Coal Measures; all samples were etched (see text). Note the over all prevalence of plant part material in the coal from the Morley Coal Measures over that from the Eocene coal in Indonesia. (a) Plant cell walls illustrating the usefulness of etching for identification. 'tv'= telovitrinite. (b) Plant cell wall tissue. (c) and (d) A combination of matrix and plant part material. The dark groundmass would mostly be amorphous humic gels. 'ie'=inertinite, 'f' = funginite, 'g'=gelovitrinite.

Microscopy of the New Zealand coals also showed that vitrain layers are exclusively composed of plant cell wall tissue (Figure 8; and also see Shearer (1992) and Shearer and Moore (1994a, b)). The microscopic composition of the matrix is fundamentally different from the Indonesian sample sites. The Cretaceous age Morley coal has an abundance of inertinite as well as a higher proportion of preserved plant material that is also larger in size than does the Eocene age coal examined in Indonesia.

Statistical Design

The basic form of data analysis used in this study is a hierarchical analysis of variance (ANOVA) with the following components:

Coal types
- Layers within coal types
  - Blocks within layers
    - Traverses within blocks

A schematic of the hierarchical sampling design is shown in Figure 9. This form of analysis allows comparison of the proportion of laboratory determined petrographic components with the coal types recognized macroscopically in the field. A hierarchical ANOVA test also calculates the total variance of a component from all samples and identifies the proportion of variance that is inherent in the different sampling levels. For example a component may vary in concentration from 20 to 60% over the whole data set. However, in one coal type the component may have a mean of 25% ±5% and in the other coal type a mean of 55% ±5%. Therefore, 75% (or [55-25]/[60-20]) of the total variation in the proportion of the component occurs between coal types. Or, in other words, just by being able to distinguish visibly recognisable coal types will capture 75% of the microscopic variation.

Hierarchical analysis also permits calculation of confidence limits for estimation of petrographic components for each coal type. This is useful because some coal types may have inherently more variation for some components than for others. In addition, the comparison of variance at each level of the design may indicate alternative sampling schemes that will increase the efficiency of sampling. Finally, confidence limits generated from the hierarchical analysis can be compared directly to confidence limits obtained through standard methods.

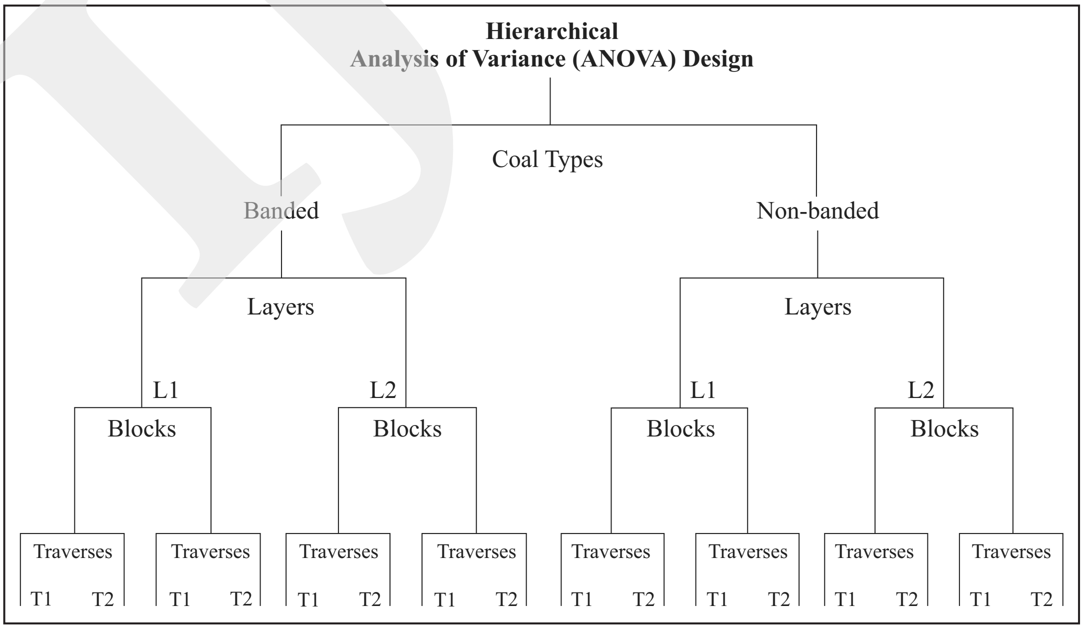

Figure 9. Sampling scheme used for hierarchical analysis of variance (ANOVA).

In assessing the results of the hierarchical ANOVAs two empirical rules, based on the mean and coefficient of variation (cv), should be followed. Firstly, the variability of components with means between 0 and 5% are considered difficult to assess or interpret because this variability approaches experimental error. As a result, these components are considered to be insignificant and are not used in interpretations. The second rule uses the coefficient of variation that describes the amount of variation of a component within a population. Since it is derived from dividing the standard deviation by the mean (cv = σ/μ) the cv allows for comparisons between populations. The cv is usually expressed as a percentage and cvs less than 25% are not generally thought of as indicating significant variation. That is, a component with a cv less than 25% has little variation within or across sampling levels. Therefore, the variance of these components can not be used in identifying significant sample variation.

Results

The variation of petrographic components as related to coal type can be noted in Figure 10. Although there is overlap, as would be expected, bright, banded coal have the most plant parts whereas bright non-banded, in general, have less and the hard, dull steely grey coal type has the least.

ANOVA Tests

The complete point count data for the two Indonesian study sites are given in Moore (1990). ANOVA tables for all components at both sites can be found in the Appendix of this paper. A summary of the partitioning of variances at the different sampling levels (i.e. coal types, layers, blocks and traverses), the means between coal types and the coefficient of variation for organic components are given in Table 1 for site 'A' and Table 2 for site 'B'.

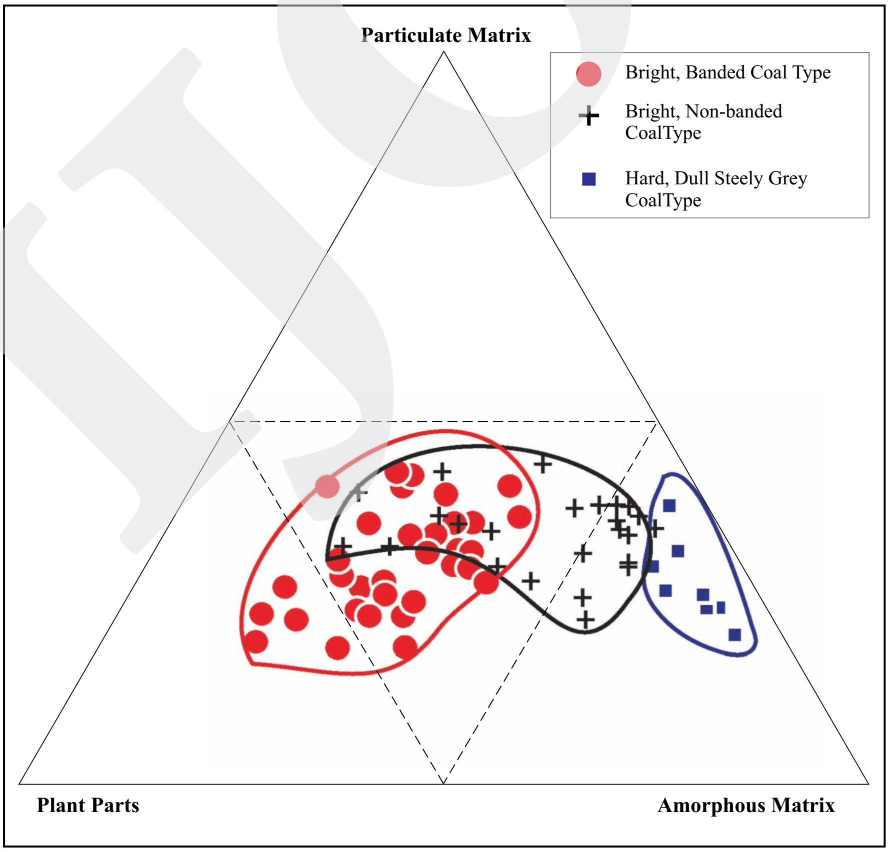

Figure 10. Ternary diagram showing petrographic variations between coal types. Each data point represents the result of a single point count traverse (see text for explanation).

Table 1. Analysis of Variance (ANOVA) of Sampling Levels for Indonesian Sample Site "A". The coal types being compared are bright non-banded and bright banded; s = standard deviation, cv = coefficient of variation

		% VARIATION				MEAN VALUES OF COAL TYPES				
Organic Component	**Maceral equivalent(s)†**	**Type**	**Layer**	**Block**	**Tra-verse**	**Banded**	**Non-banded**	**TOTAL MEAN**	**s**	**%cv**
Total Plant Parts	**Telovitrinite**	**77**	**20**	**1**	**2**	**43.6**	**13.5**	**28.5**	**17.6**	**62**
Moderate + Well -preserved woody tissue	*telinite + collotelinite*	88	9	1	2	37.3	1.9	19.6	19.6	100
Poorly preserved plant tissue		48	9	18	25	4.1	8.8	6.5	3.6	55
Total Particulate Matrix	**Detrovitrinite/Inertodetrinite**	**59**	**20**	**0**	**21**	**28.8**	**37.1**	**33.0**	**5.6**	**17***
Cell-wall and filling fragments	*vitrodetrinite/collodetrinite, sporinite, cutinite, resinite, funginite*	0	81	5	14	17.7	16.5	17.0	4.9	29
Other particulate matrix		70	15	0	15	8.8	15.3	13.9	2.8	37
Total Amorphous Matrix		**73**	**20**	**5**	**2**	**27.7**	**49.4**	**38.6**	**13.2**	**34**
Amorphous humic gels	*gelinite*	58	33	6	30	24.0	43.1	33.5	13.5	37
Amorphous liptinic gels	*bituminite*	0	88	0	12	3.7	6.4	5.0	3.2	64

* cv less than 25% (non-significant variation); † as defined by ICCP (1998 and 2001), bold names are maceral subgroups, italics are macerals

For sampling site 'A', the majority of variation in petrographic composition occurs in the coal type and layer level (Table 1). F-tests (see Appendix) further show that these variations are generally only significant at the coal type and layer levels whereas variations in petrographic composition between blocks within layers and traverses within blocks are insignificant (with 95% confidence) (Moore, 1990).

The major compositional differences between the coal types for site 'A' lie within the total proportion of plant organs and tissues; the banded coal types contain significantly more plant remains (mean = 44%) than does the non-banded coal type (mean = 14%). The banded coal contains less particulate matrix (mean = 29%) than does the non-banded type (mean = 37%). The greatest difference in the particulate matrix between the coal types occurs within the 'other' particulate matrix category. Examination of the detailed count data (Moore, 1990) show that the major contribution to this difference is from the higher proportion in the non-banded coal of resin and unidentifiable fragments of fluorescing material. Finally, the coal types differ in the amount of amorphous matrix. The non-banded coal type contains more amorphous humic (49%) material than occurs in the banded type (28%).

Hierarchical ANOVA of petrographic components at site 'B' are summarized in Table 2. The results are similar to analyses for site 'A' except for differences arising from the three coal types occurring at site 'B' and the use of four traverses (125 counts each) instead of the two 250 count traverses used at site 'A'. As in the case of site 'A' the majority of variation in petrographic composition is attributable to differences between coal types. However, variations between layers of the same coal type and between blocks within layers are higher than at sampling site 'A'. The amount of variation between traverses within blocks is still relatively small even with the reduced number of points per traverse.

The abundance of plant organs and tissues differ greatly between the coal types at site 'B'. Banded coal has the highest concentration of plant organs and tissues (mean = 38%) whereas as the hard, dull steely grey coal has the lowest (mean = 7%). Unlike sampling site 'A', differences between particulate matrix properties are small for all three coal types (note small cv in Table 2). The greatest difference between coal types is in the proportion of amorphous matrix which averages 30% for banded, 40% for non-banded and 65% for the dull steely grey coal type.

Calculation of Confidence Limits

While it is satisfying to know that coal types recognized in the field are reflected in the proportion of microscopically determined organic components, it is also necessary to use this information in generating confidence limits which can be compared with similar results

Table 2. Analysis of Variance (ANOVA) of Sampling Levels for Indonesian Sample Site "B". The coal types being compared are bright non-banded and braight banded; s = standard deviation, cv = coefficient of variation

		% VARIATION				MEAN VALUES OF COAL TYPES					
Organic Component	**Maceral equivalent(s)†**	**Type**	**Layer**	**Block**	**Tra-verse**	**Banded**	**Non-banded**	**Dull, steel-grey**	**TOTAL MEAN**	**s**	**%cv**
Total Plant Parts	**Telovitrinite**	**59**	**16**	**19**	**6**	**38.0**	**26.5**	**7.3**	**29.8**	**14.4**	**48**
Moderate + Well -preserved woody tissue	*telinite + collotelinite*	28	48	17	7	25.9	15.3	1.7	18.9	14.2	75
Poorly preserved plant tissue		0	50	36	14	11.2	9.1	5.7	8.0	7.3	90
Total Particulate Matrix	**Detrovitrinite/Inertodetrinite**	**0**	**27**	**62**	**21**	**32.3**	**33.0**	**28.0**	**33.0**	**6.9**	**21***
Cell-wall and filling fragments	*vitrodetrinite/collodetrinite,*	34	39	15	12	21.8	17.0	7.2	18.1	8.1	45
Other particulate matrix	*sporinite, cutinite, resinite, funginite*	51	0	39	10	7.9	13.6	16.0	13.9	5.6	40
Total Amorphous Matrix		**68**	**23**	**4**	**7**	**29.7**	**40.3**	**64.7**	**38.3**	**14.9**	**39**
Amorphous humic gels	*gelinite*	0	75	12	15	25.8	32.0	30.3	28.4	8.5	30
Amorphous liptinic gels	*bituminite*	65	32	1	2	3.9	8.3	34.4	9.9	13.1	132

from conventional petrographic methods. The general formulae for calculation of confidence limits of stratified samples, such as those in the Eocene coals are given below (see also Snedecor and Cochran, 1989).

To estimate the proportion of a component (organic or inorganic) within a complete coal bed section using a stratified sampling method the following formula is used (Snedecor and Cochran, 1989):

$$P' = P_1W_1 + P_2W_2 + \dots P_nW_n \quad \text{.....................(1)}$$

where:

P' = estimated proportion of the component in the whole bed,

$P_{1 \dots n}$ = estimated proportion of the component in coal types 1...n (note that each P is a different coal type),

$W_{1 \dots n}$ = proportion of each individual coal type making up the bed in a core or mine site (note that each W is associated uniquely with one coal type and is the proportion that the coal type makes up of the total coal section being estimated).

For calculation of confidence limits for P', the standard error (Se^2) for P is required. For any Se^2_P (Snedecor and Cochran, 1989):

$$Se^2_P = W^2 (\sigma^2_t + T\sigma^2_b + TB\sigma^2_l)/TBL \quad \text{..........(2)}$$

where:

σ^2_t = sample variance for traverses,

σ^2_b = sample variance for blocks,

σ^2_l = sample variance for layers,

W = the relative proportion of the coal type Awithin the whole bed,

T = number of traverses within a block,

B = number of blocks within a layer,

L = number of layers within a coal type.

The standard error for P' is:

$$Se^2_{P'} = W^2_1Se^2_{P1} + W^2_2Se^2_{P2} + \dots W^2_nSe^2_{Pn} \quad \text{...(3)}$$

The confidence limits for P' are then:

$$P' \pm Se^2_{p'}t \quad \text{..(4)}$$

where:

t = value of a students "t"- test for the required confidence level which is generally at a 95% confidence and therefore t = 1.96.

The confidence limits from the stratified sampling scheme for both sampling sites 'A' and 'B' are given in Table 3. For comparison, confidence limits for the estimates were also calculated using the binomial distribution; that is, confidence limits were also calculated as if the estimates were derived from particulate pellet analysis. Such confidence limits are calculated according to following formula (Snedecor and Cochran, 1989):

$$P \pm \sqrt{\frac{t[P(1-P)]}{n}} \quad \text{..(5)}$$

Table 3. Confidence Limits at 95% Significance of Microscopically Identified Organic Components as Calculated from Stratified Formulae and from Binomial Distribution. n = total number of points counted. All numbers in percent

Organic Component	Site 'B'[1]			Site 'B'[2]		
	Stratified C.L.[3]	Mean[5]	Binomial C.L.[4]	Stratified C.L.[3]	Mean[5]	Binomial C.L.[4]
Total Plant Parts	4.3 ±	18.3	± 1.2	7.3 ±	37.1	± 1.1
Moderate + Well -preserved woody tissue	2.8 ±	17.3	± 1.2	12.1 ±	25.2	± 1.0
Poorly preserved plant tissue	2.6 ±	4.0	± 0.6	5.8 ±	11.0	± 0.7
Total Particular Matrix	4.5 ±	35.8	± 1.5	5.7 ±	32.2	± 1.1
Cell-wall fragments	9.0 ±	16.7	± 1.2	5.3 ±	21.4	± 1.0
Other particulate matrix	4.5 ±	14.3	± 1.1	1.6 ±	8.2	± 0.6
Total Amorphous Matrix	4.6 ±	45.9	± 1.6	5.7 ±	30.4	± 1.1
Amorphous humic gels	4.8 ±	40.0	± 1.5	5.7 ±	26.0	± 1.0
Amorphous bituminite	5.8 ±	6.0	± 0.7	1.9 ±	4.4	± 0.5

C.L. = confidence limits; 1. n = 4000; 2. n = 7000; 3. calculated using formula (4); 4. calculated using formula (5); 5. calculated using formula (1)

The confidence limits for the binomial distribution are smaller than those that are obtained using stratified sampling (Table 3). Careful examination of formula (5) shows that confidence limits for the binomial distribution are highly dependent on the total number of points counted (n). Therefore it is not surprising that the confidence limits are small as the number of total point counts is large at both sampling sites (n = 4000 for site 'A' and n = 7000 for site 'B'). Still, the confidence limits obtained using the stratified sampling method would be an acceptable level of precision (generally ± 6%) in many if not most circumstances.

Comparison of Block *vs*. Pellet Point Counts

There was no significant variation of bright bands between plies in the point counts of the Morley coal bed. Both sampling sites had very similar vitrain band proportions, with site 1 having 23.6 $\pm$ 0.8% and site 2 having 24.6 $\pm$ 0.8% vitrain bands (n = 1244). In addition, the size measurements of the vitrain bands were also very similar for both sampling sites (Shearer, 1992; Shearer and Moore, 1994b). The mean and standard deviation of the widths of bright bands (calculated from the method of moments equations; Lewis (1984)) are -1.73 ø (3.35 mm) and 1.47 ø, respectively (n = 256) (Figure 11).

Results of the maceral analyses on particle pellet and block samples are similar except for the proportions of individual vitrinite macerals (Table 4). Counts of structured vitrinite were lower in the block samples than in particle pellets. This discrepancy is a function of the point counting technique in which the proportion of vitrain bands, composed mainly of structured vitrinite (96% telinite and corpocollinite and 4% suberinite; Shearer, 1992), was estimated at the macroscopic level but could not be counted representatively on blocks. Very few vitrinite bands greater than 1 mm in width occur in blocks, firstly because these bands are brittle and therefore blocks tend to break along vitrain bands and thus they may only occasionally be represented on blocks. Secondly, because of the size of vitrain bands (1-40 mm in width), the proportion of bands cannot be accurately represented within 3 x 5 cm blocks. Therefore vitrain bands are under-represented on blocks, and, as a consequence, so is structured vitrinite. However, when structured vitrinite counts from blocks are re-proportioned to account for the macroscopically determined proportions of vitrain bands, individual maceral counts for pellets and blocks are within ±6% of each other (Table 4; Figure 12). This same relationship has been shown to occur in other New Zealand coals (Newman *et al*., 1997).

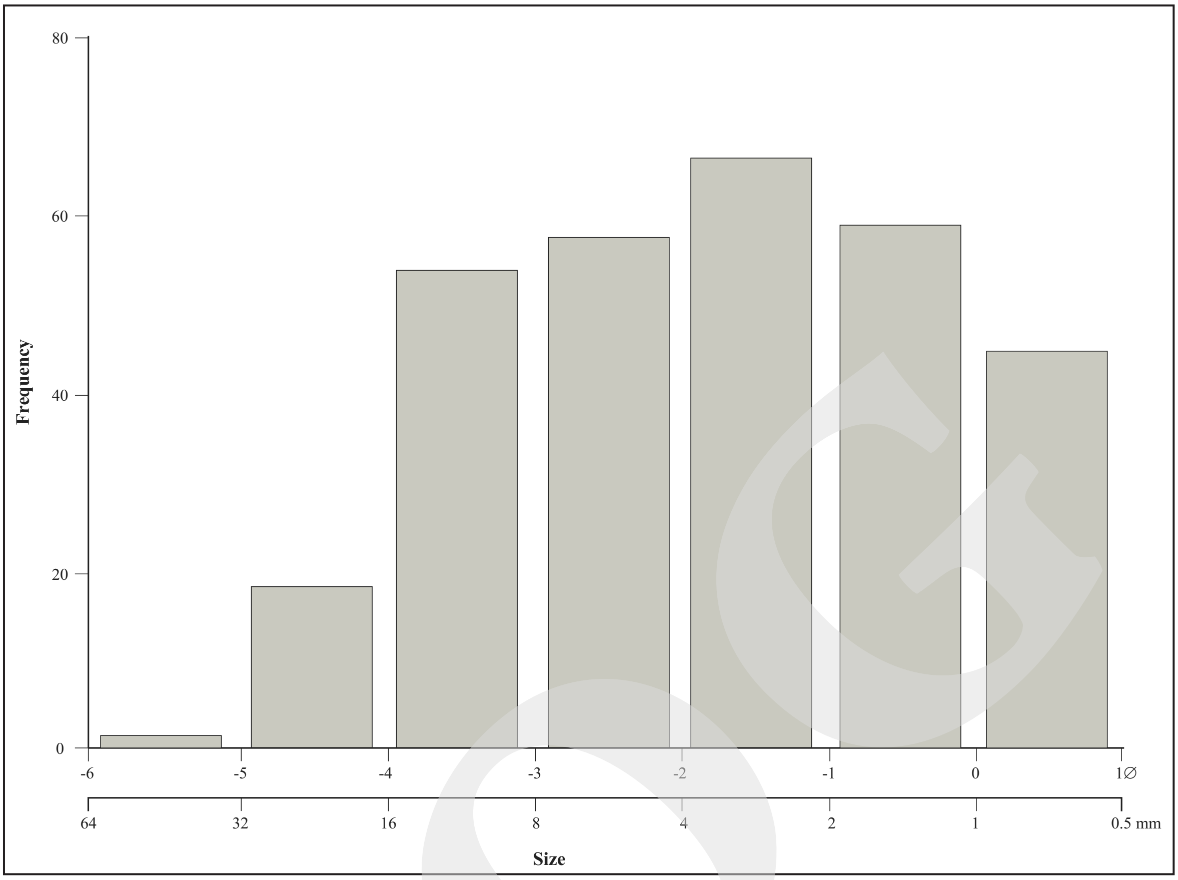

Figure 11. Size distribution for vitrain from coal in the Wairaki underground coal mine, Ohai coalfield, New Zealand.

Table 4. Comparison of Means and Standard Deviations (in parentheses) for Maceral Counts on Particulate Pellets and Block Samples from Wairaki Mine, Ohai Coalfield, New Zealand. Seam Section 1 contained 25% vitrian bands, while Seam Section 2 contained 24%. See Shearer (1992) for detail of petrographic data

		Wairaki Mine Seam Section 1				Wairaki Mine Seam Section 2			
Maceral Group	Maceral	Particulate Pellet[1] *n=10*	Block[2] *n=10*	Block Recalculated[3]	% Difference Pellet - Block	Particulate Pellet[1] *n=9*	Block[2] *n=9*	Block Recalculated[3]	% Difference Pellet - Block
Vitrinite	Telovitrinite	44.2 (14.1)	22.6 (7.2)	40.7	3.5	32.0 (6.1)	18.1 (5.7)	38.2	-6.2
	Gelovitrinite - vitrodetrinite	40.8 (11.1)	60.5 (7.2)	46.2	-5.4	44.4 (4.8)	60.4 (5.9)	45.5	-1.1
Liptinite	Cutinite	0.6 (0.4)	1.0 (1.2)	0.7	-0.1	0.5 (0.6)	0.5 (0.5)	0.4	0.1
	Resinite	0.7 (0.5)	0.6 (0.7)	0.5	0.2	0.8 (0.7)	0.7 (0.9)	0.5	0.3
	Sporinite	0.4 (0.3)	1.8 (1.3)	1.4	-1.0	1 (0.6)	2.5 (1.5)	1.9	-0.9
	Suberinite	3.1 (1.8)	2.6 (2.1)	2.0	1.1	4.5 (1.9)	3.2 (1.5)	2.4	2.1
	Liptodetrinite	3.7 (2.1)	4.4 (1.6)	3.4	0.3	5.8 (2.2)	4.5 (1.8)	3.4	2.4
Inertinite	Fusinite	0.7 (0.6)	0.2 (0.3)	0.2	0.5	0.5 (0.2)	0.3 (0.3)	0.2	0.3
	Semifusinite	2.4 (1.6)	2.7 (1.7)	2.1	0.3	4.8 (3.2)	4.1 (2.2)	3.1	1.7
	Degradosemifusinite*	0.4 (0.5)	0.3 (0.3)	0.2	0.2	0.4 (0.5)	0.3 (0.4)	0.2	0.2
	Funginite	0.3 (0.3)	0.6 (0.6)	0.5	-0.2	0.4 (0.2)	0.5 (0.4)	0.4	0.0
	Inertodetrinite	3.1 (2.2)	3.3 (2.4)	2.5	0.6	5.2 (4.0)	4.9 (3.6)	3.7	1.5
	Total Vitrinite	85 (5.7)	82.7 (5.5)	86.8	-1.8	76.4 (9.0)	78.5 (7.2)	83.8	-7.4
	Total Liptinite	8.5 (4.0)	10.3 (3.8)	7.9	0.6	12.6 (4.1)	11.6 (3.4)	8.9	3.7
	Total Inertinite	6.5 (3.5)	7.0 (4.0)	5.3	1.2	11.1 (6.9)	9.9 (5.1)	7.5	3.6

[1]Each particulate pellet had 500 point counts using both white and blue light.
[2]Each block sample had 250 point counts using both white and blue light.
[3]Each block was recalculated so that the the % vitrain bands determined in the macroscopic point count for each mine section was added to the telovitrinite totals.
*Defined in Shearer (1992).

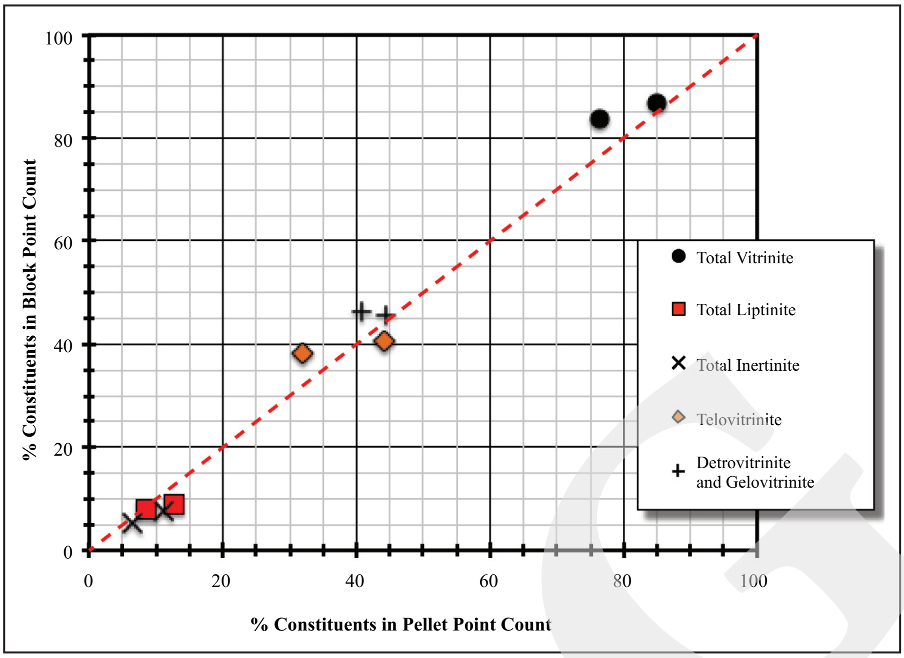

Figure 12. Comparison of average organic composition (from 19 plies) between particulate pellet and block samples.

Discussion

The data presented in Tables 1 and 2 and the Appendix for the Indonesian sites, allows future sampling procedures to be designed. The low proportion of variation seen between traverses within blocks at both sampling sites indicates that the number of points can be reduced from 500 to 125 per block. Although the variation between blocks at site 'A' is low, at site 'B' they are somewhat higher. This may result from the preponderance of the banded coal type at site 'B'. The larger components comprising banded coal types may be more difficult to estimate on the size of blocks collected. Therefore, in a new sampling scheme it may be advantageous to increase either the size or the number of blocks within layers of banded coal.

Still the largest amount of variation within either sampling site occurs between coal types and between layers within coal types. The large amount of variation seen between coal types is not unexpected because these types are visually distinct. However, variation seen between layers indicates that there are some differences between layers of the same coal type. There are two alternatives for further modification of the sampling plan to address this variation. In the first alternative layers can be further subdivided into subclasses of coal types. This is probably not a viable option because further subdivision may generate a classification that is too complex for field use. The other alternative is to increase the number of layers sampled within coal types. Examination of formula (2) shows that total variance of a coal type will be reduced as the number layers increases because layers (L) are in the denominator.

Therefore, in a modified scheme for stratified sampling, the number of traverses per block is decreased to one (with 125 counts) and the number of blocks counted per layer remains at two or possibly increasing to 3. The reduction of work at levels where variation is low i.e. the traverse level, allows more emphasis to be placed on layers where variation is highest. Therefore, in the case of the Eocene age, Indonesian coal the number of layers that can be examined for the same amount of work has quadrupled. That is, whereas in the initial survey 4000 points were needed to assess 4 layers of coal (or 2000 point counts per coal type), 16 layers (or 8 per coal type) can now be characterized using the same number of point counts in the modified sampling design. In effect this also allows the operator to estimate the composition of more of the bed either within a core or at a mine site in a statistically valid and representative manner.

As shown in Table 3, the confidence limits obtained for estimates from the stratified sampling method are slightly higher than when determined from the binomial distribution. The confidence limits produced from the standard method (i.e. particulate pellets) only show the reproducibility of the technique to obtain the same estimates and therefore gives no information about the variability of the interval being sampled. With stratified sampling using block samples, the amount of variability within an interval can be classed as high (in the case of poor recognition of homogeneity) or low (in the case of a true assessment of homogeneity) and thus provide useful information about the variability of a interval being sampled.

The confidence limits of the estimates obtained through stratified sampling will also be reduced from modification of the sampling design in a manner that has already been outlined above. For example, in sampling site ‘A’ increasing the number of layers per coal type to 3 will decrease the confidence limits from ± 4.3% to ± 3.7% (for the purposes of this example means and variances can be assumed to remain relatively unchanged from the original sampling plan). For the same amount of work (1500 point counts) the binomial distribution would yield confidence limits of ± 2.0%. The real advantage of the stratified sampling method is in allowing a large number of samples to be characterized with much less effort than the conventional method of point counting. In the modified stratified sampling plan, examination of 10 layers per coal type will reduce the confidence limits to ± 2.0% with only 2500 total points being counted. In contrast, using conventional methods on particulate pellets would require 20,000 points to be counted to characterize the two coal types.

An additional problem with using particulate pellets is that this procedure does not necessarily follow the binomial distribution theorem (Bustin, 1991). In some cases as little as 200 to 300 points may be sufficient to estimate the proportion of a component (within ± 5%) whereas in other cases as many as 800 points are needed for the same level of precision. The recommendation by Bustin (1991) is that counts on samples should be made in replicates to best assess the variation among petrographic components.

Finally, two problems with stratified sampling may potentially exist and need to be addressed. Firstly, means of compositional data are affected by closure (totals = 100%). As has been pointed out by Aitchison (1989) and Woronow and Love (1990) changes in a mean value from one sample to the next can be the result of either an actual increase (or decrease) in the concentration of that component or from a change (increase or decrease) in the concentration of another component. Therefore, interpreting changes in estimates is difficult and Woronow and Love (1990) recommend transformation of compositional data into “f-values”. These quantify the change in a component compared to a fixed variable thereby excluding the effects of closure. This procedure can be applied to the data presented here and may provide better estimates of the change in components for use in interpretations such as reconstructing paleoenvironments. Transformation of data does not affect the manner in which the stratified sampling experiment has been conducted. However in studies such as this one concerned only with large relative changes in compositional data between suites, calculation of f-values are not necessary.

The second problem that may arise is block sampling of “coarse-grained” coals. The Eocene coal used in this study is relatively “fine-grained” (macroscopic components mostly <1 mm thick) and therefore a relatively homogeneous and representative block can be obtained with dimensions of approximately 3 x 3 x 3 cm. However, coarse-grained coal beds, such as the ones studied in the Ohai coalfield in New

Zealand may have larger (2 to 50 cm thick) macroscopic components than can not be practically sampled. As shown with the Morely Coal Measures data, however, if macroscopic point counts are conducted and recalculated into the microscopic analysis very good estimates of microscopic composition are obtained.

Summary

Stratified sampling using block samples is a viable alternative to standard methods of channel sampling and particulate pellet mounts. Although the particulate pellet analysis is employed widely it is both time consuming and does not allow variation within sampling units to be assessed - an important measure in any study whether it be for paleoenvironmental reconstruction or in obtaining estimates of industrial attributes. Also blocks provide samples in which additional information can be gained such as particle size and texture.

In the case of the Indonesian Eocene coal ANOVA tests show that:

1. The largest amount of microscopic variation occurs between coal types showing they are petrographically dissimilar.
2. Within coal types variation is most important in the between layer level of sampling.
3. A maximum number of 125 point counts are needed to make precise estimates of block composition.

These data allow the stratified sampling plan to be modified to improve efficiency. The reduction of total point counts per block permits more layers to be examined and since layers are where the highest levels of variation occur within coal types, total variance will be reduced.

Confidence limits of estimates for petrographic composition generated with stratified sampling reflect the degree of homogeneity (or inhomogeneity) of sampling units. These confidence limits can often be reduced once the level of maximum variation is determined and the sampling design has been redirected to address that variance. In particulate pellet analysis confidence limits of estimates are predicted by the binomial distribution. However, as shown by Bustin (1991) the binomial distribution does not always work for particulate pellet analysis. In addition, the confidence limits derived from the binomial distribution (or the method of replication suggested by Bustin (1991)) gives no information about the variability of a sampling unit, only on the reproducibility of the technique to obtain the same estimates.

Combined with macroscopic point counting, coals that have large particles (such as vitrain bands) can still be accurately assessed using block petrographic counts. Comparative analyses of particulate pellets and blocks from a coal bed within the Morley Coal Measures in the Ohai coalfield of New Zealand show less than 6% variation between the same sample interval when blocks are recalculated to include macroscopic counts of vitrain bands as plant parts (‘telovitrinite’). Therefore even in ‘coarse’ grained coals, stratified sampling can be used effectively and representatively.

Acknowledgements

This paper is ‘late’ in more than one sense of the word (it is with sadness that I point out the string of ‘lates’ below, all of whom were not ‘late’ when the original research was conducted). A statistical treatment of stratified sampling was a significant part of my thesis, which I regretfully was unable to publish – until now. Recent discussions with colleagues, however, have reiterated that this work still has relevance and provides a platform for additional studies. I’d like to acknowledge the invaluable assistance of P.T. Arutmin Indonesia and BHP Minerals International Inc. (now BHP Billiton) for providing the original financial and field support. Mike Friederich, the late Ted Milligan, the late Al Johnnet, Sony Pangestu, Lim

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