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**Letter to the Editor: A critique of the present use of some geochemical techniques in geoforensic analysis**

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## Letter to the Editor: A critique of the present use of some geochemical techniques in geoforensic analysis

### 1. Introduction

A number of papers have been published recently concerning the geoforensic analysis of soils and sediments which generally utilise experimental verification and geological rationale [1–5]. This rapidly expanding field relies particularly upon the automated techniques of chemical analyses of soils and sediments which were developed to help solve geological problems. We contend that this development is at variance with the recently articulated geoforensic philosophy which is fundamental for accurate conclusions to be drawn in real-case forensic investigations [6–8]. We therefore raise four main issues; firstly the problems of sample preparation which will involve the homogenisation of a sample prior to analysis; secondly, the use of a series of analytical techniques and procedures which may be dependent upon one another and therefore do not provide independent corroborative results; thirdly, the use of inappropriate comparator samples to justify the discriminatory abilities of a particular analytical technique; and fourthly (and perhaps most importantly) the desire to ‘match’ or positively associate samples rather than adopt a fundamental forensic protocol of excluding samples from having associations with each other.

We include below examples of these four main concerns and comment also upon specific points raised in each of the following papers:

### 2. Specific comments on paper 1 [2]

Pye et al. [2] present an argument for the use of Inductively Coupled Plasma spectrometry (ICP) as a powerful tool for analysing soil/sediment samples for forensic purposes. They show that the ICP has the ability to produce a large quantity of reproducible, accurate and precise elemental data. As such they have addressed the issue of reproducibility mentioned [6] as a comment to their earlier paper [1] for elemental analysis of soil samples and they go some way toward covering the very pertinent points made by Jarvis et al. [9].

There are however, a number of comments and queries that we would like to present pertaining to the paper published in this journal by Pye et al. [2]. These comments concern firstly the practicability of the ICP technique for geoforensic investigation and secondly, and more specifically, relate to issues regarding the experimental design as outlined by Pye et al. [2].

We contend that the use of ICP analysis on forensic soil/sediment samples to derive results that can be interpreted to provide an indication of provenance poses a significant problem that has not been addressed by Pye et al. [2] due to the requirement of homogenisation of samples prior to analysis. As mentioned above, it is not possible to identify pre-, syn- or post-event mixing and thus the derived conclusions cannot be tested.

Pye et al. [2] demonstrate that ‘... in this case the two soils can clearly be distinguished, differences between the two soils

being far larger than the variability within either soil’ (p. 190). Whilst in this case there is a good discrimination between these two specific sites, it is not clear whether such a discrimination will always be apparent between samples in a real-case forensic situation where samples are taken from two or more distinct areas that are more geologically similar to each other. In their experimental case, it is apparent that the two sites are very contrasting in terms of their geology and landuse; the soils at Simon’s Wood are ‘... essentially natural and developed primarily on unconsolidated quartz-rich, gravely sands’ whilst the soils at Arborfield Bridge are ‘... a mixture of locally derived soil from the surrounding area, decayed leaf matter, and materials derived from fly-tipping onto an asphalt and concrete surface’ ([3] p. 60). In the light of these two contrasting sample sites we wonder whether it is possible to assert that forensic distinction can usually be made between different sites on the basis of this analytical technique. Soil samples taken from distinct sites where the soil has formed on a similar geology may not be so easily distinguished in this way. This issue has been raised before [6] although not taken into account by the authors here. It is important that such an issue is addressed before the technique can be recommended to the geoforensic practitioner.

Pye et al. [2] go some way in beginning to address the issue of distinguishing between far more similar soil samples than those discussed in their paper when they state that ‘... when similar soils are being considered ... several independent techniques should always be taken into account’ (p. 190). In support of their statement Pye et al. [2] cite their 2004 paper [1] which outlines four analytical techniques that can be applied to such samples (colour, particle size, carbon/nitrogen ratios and elemental analysis by ICP). However, the techniques that were used by Croft and Pye [1] cannot be considered to be truly independent of each other.

Further, Pye et al. [2] contend that the ICP has become the ‘... technique of choice for many scientists, especially those concerned with forensic applications, who need to measure simultaneously the abundance of a large suite of elements in small samples’ (p. 179). This statement is contrary to the wholly pertinent findings of Jarvis et al. [9] in the conference proceedings edited by Pye and Croft [10] who recommend that ‘... the technique is used only when sufficient sample is available to allow an assessment of uncertainty’ (p. 177) and who invoke great caution in the use of ICP in forensic enquiry unless other independent techniques are used in conjunction. It has been demonstrated [9] that when undertaking geochemical analysis on samples in a geological context, it is more informative to measure the concentrations of fewer elements that have a lower coefficient of variation than a larger number of elements which exhibit a greater degree of variation. For example, the precision and accuracy of oxides and trace element analysis is normally greater using XRF analysis even if the quantity of sample required for such analysis is greater than for ICP.

We are therefore concerned by the recommendation of Pye et al. [2] that where sample size precludes triplicate analysis of a sample ‘... there may be greater benefit in analysing the sample using three different techniques rather than three times using the same technique’ (p. 191). This point is also raised by

Jarvis et al. [9] in Pye and Croft [10]. Whilst we agree that the use of a number of truly independent techniques is necessary for any reliable and accurate interpretations to be made from the geoforensic analysis, we are concerned that Pye et al. [2] seem to suggest that some ICP analysis is better than none at all in such circumstances of insufficient sample availability. Perhaps in such circumstances it would be better not to use elemental chemical analyses at all (given the inherent problems that such techniques pose, as outlined above). If geochemical analyses were required, perhaps XRF analysis (which has been recognised as having greater accuracy [9]) or XRD analysis, that is able to provide mineralogical data from small samples [11], or indeed QemSCAN analysis [12] would be more appropriate.

### 2.1. Experimental design

Pye et al. [2] determine five different types of precision (type 1: short-term instrumental measurement precision (seconds– minutes); type 2: medium-term instrumental measurement precision (minutes–hours); type 3: variability due to sample solution preparation; type 4: variability due to sub-sampling from the parent sample; type 5: long-term instrumental measurement precision (weeks–months)), but they only use samples taken from specific geographical locations to establish types 1,3 and 4 precision. However, there is also selected use of Certified Reference Materials (CRM) to determine types 2 and 5 precision. May we venture to ask why such selectivity is employed by using two different types of samples to determine different forms of precision? Surely it would be more rigorous and beneficial to establish each type of precision on both different types of samples (those taken from specific geographical locations and the CRM samples) in order to provide a complete picture of the state of the instrumental precision (types 1, 2 and 5) and also the sampling preparation variability (types 3 and 4).

### 2.2. Summary

ICP analyses of small samples of soil/sediment is in our opinion, and that of others mentioned here, most certainly not ‘...the technique of choice for many scientists, especially those concerned with forensic applications, who need to measure simultaneously a large suite of elements in small samples’ ([2] p. 179). Our conclusions concur with the work of Jarvis et al. [9] and Rawlins and Cave [13] who raise specific concerns as to the use of ICP analyses on forensic soil/sediment samples. Rawlins and Cave [13] assert that not only are small samples associated with greater sampling error than larger samples (as found in geological investigations), but also that in the analysis of rock powders, a sample mass of 1g is necessary to provide an accurate representation of major element concentrations, whilst considerably more material is required to represent trace element contents adequately [14]. Indeed, ‘...sufficient soil material may not be available in many forensic cases for a representative analysis to be undertaken, which reduces our confidence in comparing the geochemistry of forensic samples [and] other local (control) samples’ ([13] p. 206). We concur.

### 3. Specific comments on paper 2 [3]

Pye et al. [3] employ four analytical techniques to provide an indication as to the inter- and intra- site variability of the soil at two locations by analysing nine soil/sediment samples taken from a 1 m grid at each site. Indeed, by assessing the results of these four different techniques they seek to provide an indication as to the potential of each form of analysis to discriminate between the samples derived from each of the two sites. These are worthy pursuits and of great interest to geoforensic scientists, however, there are a number of comments and questions that we feel are important to raise.

#### 3.1. Elemental composition

In order to establish the spatial variability between samples taken from each site, Pye et al. [3] employed elemental analyses by ICP-AES and ICP-MS. They also sought to assess whether it was possible to distinguish between samples taken from the two different sites. They contend in their Fig. 3 [3] (p. 66) that the ICP analyses (namely the results from SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Nd and U) successfully discriminate the samples taken from the two sites (Simon’s Wood and Arborfield Bridge). In a previous article by Pye et al. [2] they also provide the results from ICP analyses for samples taken from these same specific sites and demonstrate that on the basis of the elemental composition, the two sites can be distinguished ([2] p. 189). However, on closer inspection of the results presented in both papers for samples from the same site [2,3], it is alarming to discover that one of the elements which Pye and his various co-workers [2,3] contend to be very useful in distinguishing between the two sites may not be so

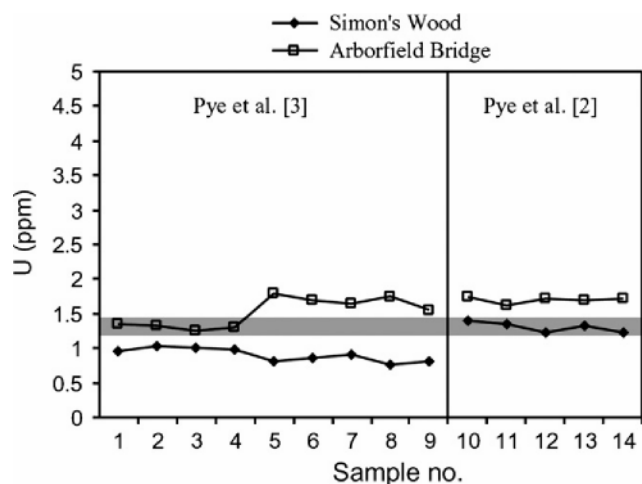


Fig. 1. Combined graph of Uranium concentration for soil/sediment samples compiled from Simon's Wood and Arborfield Bridge taken from Pye et al. [2] and [3]. The grey area shows that measurements from samples taken from the two sites do not discriminate between the two locations.

useful after all. Our Fig. 1 presents the ICP analyses recorded in

both papers (see Appendix A) for the Uranium content in the samples tested from both Simon's Wood and Arborfield Bridge.

Clearly, from reference to Fig. 1, Uranium proves not to be such a successful elemental component with which to distinguish between samples from these two sites. Samples 1–4 derived from Arborfield Bridge [3] have a Uranium content of approximately 1.4 ppm whilst the samples from Simon's Wood have a Uranium content of approximately 1.0 ppm. However, in the previous paper [2], the samples from the Arborfield Bridge (10–14) contain a higher level of Uranium (approximately 1.7 ppm), whilst the samples from Simon's Wood are found to have a very similar level to the Arborfield Bridge samples 1–4 of approximately 1.4 ppm. The grey area portrayed in our Fig. 1 demonstrates the similarity between samples 1–4 from Arborfield Bridge and samples 10–14 from Simon's Wood. Thus, the Uranium content does not appear to discriminate between all of the soil samples derived from each of these two distinct sites.

Furthermore, a difference of 1 ppm (between 1.0 and 2.0 ppm) between two samples is a minimal difference. Thus, it should not be possible to contend that Uranium is a viable element with which to distinguish between all of the samples taken from these two sites. Equally, little is known of any detailed Uranium concentration databases of a similar scale, or indeed even at a national scale, with which these findings can be compared. Without a suitable database for reference it is not possible to establish whether any such variance between readings taken from samples at the same site is relevant or not.

To contend that it was the combination of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , Nd and U that discriminated soil/sediment samples from the two sites, would beg the question as to the geological relevance of such an association of these compounds and elements. With the discovery in a court of law that Uranium could no longer be included in this discriminatory suite, it is likely that the evidence would be dismissed. Of course, such undetected false-positive and false-negative interpretations may be occurring not

only with two different studies from the same site, but also from different studies from different sites, thus diluting the relevance of the findings for the sake of the lack of the necessary detailed database.

One may also wish to ask why the samples from Simon's Wood show such a difference in the two papers of Pye et al. [2,3]. Is this due to a distinct lithological boundary that has not been identified by any other technique that they employed, or could it simply be a machine calibration error? Whatever the cause, this finding casts significant doubt on the use of these data in a forensic context.

### 3.2. The experimental design

The discrimination of sample sites undertaken in both of Pye and his co-workers' papers [2,3] should be quite easy to effect. The geology of the two sites is very distinct, with the soils at Simon's Wood being situated on Eocene formations (Camberley Sand Formation) and the Arborfield Bridge site being situated on Quaternary alluvium. Furthermore, the soils from the Simon's Wood site (an area of mixed woodland) are deemed to be naturally derived from the parent rock whilst the soils from the Arborfield Bridge site, which lies between two rivers, comprise both materials derived from the local site but also from 'fly-tipping onto an asphalt and concrete surface' ([3] p. 60). These sites therefore, present very different characteristics and it is indeed no surprise that the soils should be distinguished by different geological analytical techniques. Furthermore, if the aim of this paper [3] is to present analytical techniques that are useful in discriminating between samples in a forensic context, it would be of more use to test the different techniques on samples derived from two distinct areas of more similar geology and landuse. This is a similar point to which we made comment [6] in response to a previous paper by Croft and Pye [1]. Unfortunately in this present case [2,3], although it would be quite possible to distinguish between the samples from these two sites from the results derived from many other 'low-tech' chemical techniques (and indeed from a whole host of physical based techniques), ICP analyses in this particular instance provided similar Uranium results for the two sites (Simon's Wood and Arborfield Bridge) taken from wholly unrelated forensic cases.

We re-iterate that there is a fundamental problem when using any analytical techniques in forensic geoscience which require the homogenisation of a sample prior to analysis [7,8]. Any samples analysed in a forensic case are likely to contain materials derived from a number of different sources (pre-, syn- or post-forensic event). Thus it is quite possible that these materials will have derived from different sources and be made up of different physical and chemical constituents which are then amalgamated during homogenisation. There is then a considerable possibility of achieving false-positive or false-negative interpretations or conclusions from such data. It is not that the analysis and derived data are not correct or precise, it is simply that the sample (due to anthropogenic mixing) is not appropriate for such forms of analyses. Further constraints to the use of ICP in forensic analysis have been regularly voiced



[9,13,15] and this issue must be addressed by Pye and his co-workers, especially when they are attempting to use ICP data in a forensic context.

### 3.3. The philosophical approach

A fundamental tenet of forensic geoscience is that it is not possible to 'match' or positively associate soil/sediment samples, or to assert that two samples have derived from the same location [6–8]. This issue was also raised in response [6] to Croft and Pye's previous paper [1]. However, in this recent paper [3], Pye et al. state that '... there are three possible conclusions that can be drawn from such comparisons: (1) the questioned sample definitively did not come from the location of interest (i.e. is excluded); (2) the questioned sample could have come from the location of interest; (3) the questioned sample almost certainly did come from the location of interest' ([3] pp. 59–60). In other words, option (1) excludes the samples from having derived from the same source and option (2) can be re-phrased to say that the samples cannot be excluded from having derived from the same location (but they state '... the samples could have come from the location of interest' and this wording makes their statement fundamentally and philosophically wrong). Their 3rd option however, states that it is almost certain that the samples were derived from the same location. Once again, this is fundamentally and philosophically wrong. Indeed evoking this 3rd option in a courtroom will have very great influence upon a jury. Furthermore, the fact that the four methods of analysis are not all independent (elemental composition, colour, particle size distribution, carbon and nitrogen isotope ratios can all be dependent variables), the resultant conclusions made by a forensic scientist in a courtroom may well be devastatingly wrong. For truly meaningful results from the analysis of soil/sediment samples we follow the accepted contention adopted by Walls [16] that not only should the aim of the forensic geoscientist be to exclude samples from having derived from the same source (rather than include or 'match' them [17,18 (p. 2), 19 (p. 49), 20 (p. 72), 21 (p. 220)]), but it is also imperative that the forms of analysis employed are totally independent from each other.

### 4. Specific comments on paper 3 [4]

We are concerned with the comments made by Pye and Croft in their paper [4] regarding the expense and time-consuming nature of SEM analyses. Whilst automation, undertaken to provide rapid and accurate results (much in the same way as DNA analysis is perceived to do), has been considered by some to be the 'holy grail' of geoforensic analysis, we advocate great caution in this approach. Good science takes time and invariably whatever form of analysis is undertaken on soil/sediment samples, the presence of a skilled scientist to interpret the data appropriately and accurately is of great importance. Broeders [22] summarises this issue succinctly, although not specifically for soil/sediment analysis, when he says that the various forms of forensic science '... do not use the same scientific paradigm – and – consequently do not report their conclusions in the same format' ([22] p. 153). Adopting

Broeders' argument [22], soil/sediment analyses can be considered to be probabilistic evidence as opposed to categorical or quantitative. As such, the approach of the geoforensic scientist must be sensitive to the type of evidence with which they are dealing. We suggest that the use of probabilistic evidence (rather than categorical or quantitative) is contrary to the desire of Pye and Croft to '... screen samples more quickly on the basis of bulk elemental composition' ([4] p. 53). Whilst such an approach may be feasible when dealing with other forms of evidence (such as DNA), it is not appropriate for soil/sediments.

We are concerned that Pye and Croft [4] appear to [1–3] be advocating that their '... preferred method of soil comparison would be based on a combination of methods which examine quantitative elemental composition in combination with colour, particle size distribution, mineralogy and detailed study of individual particle types present' ([4] p. 62). Whilst no geoforensic scientist would dispute the necessity for a number of different techniques to be employed on soil/sediment samples, we are very concerned that they do not appear to appreciate the necessity for those techniques to be independent from each other. It simply is not possible to confer value on corroborative results derived from different analytical techniques when there is a possibility that those techniques may be dependent upon one another. Mineralogy may well have an influence on, or mutual relationship to, the soil/sediment colour, the particle size distribution and the elemental composition of a soil/sediment sample [7]. This matter must be addressed by Pye and Croft before they can make recommendations to the geoforensic community as to the most appropriate suite of analytical techniques for soil/sediment analysis in a forensic context. Their preferred method of soil method comparison is based on false premise.

Pye and Croft conclude that '... the comparison of elemental peak height ratios determined by EDXRA can be a useful tool for rapid screening of soil samples' ([4] p. 52). However, on inspection of their Figs. 3 and 4, only Fig. 3c (a bivariate plot of Si/Ca vs. Si/Al) provides a good discrimination between the soil/sediment samples from the three different sites. Figs. 3a, b and 4 do not provide a compelling discrimination between more than two of the groups. This appears to raise more questions as to the usefulness of this technique. If only one specific bivariate plot (taken from a wealth of data) is able to identify any discrimination between the samples, Pye and Croft [4] need to address whether this particular discrimination still has evidential value.

### 5. Specific comments on paper 4 [5]

Pye et al. [5] contend that it is important to make 'like with like' forensic comparisons between soil/sediment samples such that '... it is preferable to compare the questioned sample with an approximately equivalent size fraction obtained from the control samples' (p. 12). Such a statement concurs with the widely held view in the published literature [8]. However, whilst it is important to establish the most equivalent size

fraction for elemental analyses in the geological sciences, in reality, in the forensic arena, comparative analysis is constrained by the nature and size of the samples available. Further, complications of both physical and philosophical nature also come into play. These points are not properly considered by Pye et al. [5] as mentioned above.

Pye et al. [5] outline the important factors that must be taken into account during forensic investigations. In particular they mention that ‘... during transfer of sediment/soil material onto clothing, footwear or other items of forensic interest, particle size fractionation may take place’ ([5] p. 1). This has been demonstrated experimentally in the literature with regard to the particle size analysis of soil/sediment samples taken from footwear [23]. Indeed, elemental analysis undertaken on soil/sediment samples recovered from clothing has also been shown to exhibit demonstrable differences to the source soil/sediment even when comparing samples of the same grain size fraction [24]. Further, these comparator trace particulates can persist upon clothing for very many hours even when the clothing has been worn and subsequently washed [25].

These findings further compound the problem that Pye et al. highlight in their paper [5] regarding particle size fractionation during transfer. The longevity of trace particulates means that a further consideration must be taken into account when analysing samples recovered from clothing; specifically, if particulates persist for long time periods, articles of clothing will have trace particulates present which derive from transfers that occurred before the forensic event. When soil/sediment is transferred onto articles of clothing during forensic-event transfer, it is likely that the previously transferred trace materials that have persisted on the clothing will be incorporated into the soil/sediment derived from the forensic event in question. Thus, a mixture of trace materials is created on the clothing. This must be taken into account when analysing and interpreting the results from such samples. It will only be possible to identify whether mixing has taken place, and thereby avoid reaching a false-negative or false-positive conclusion, if a form of analysis is employed which does not require the homogenisation of the sample prior to analysis [7]. The problem with particle size distribution analysis and elemental chemical analyses is that sample homogenisation is prerequisite.

Whilst Pye et al. [5] appear to appreciate the problem of mixing to some extent when they say ‘...there is a danger of concluding incorrectly that two samples are unrelated unless comparisons are based on standardised particle size fractions’ ([5] p. 3) there is a further issue that they must address. A fundamental philosophical problem belies this comment which infers that the aim of the analysis is to ‘relate’ samples to each other. Indeed Pye et al. [5] also comment that their results in this paper regarding elemental data provide ‘...an adequate basis on which to identify associations and differences between samples’ ([5] p. 1). We contend that the aim of all geoforensic analysis must be to attempt to ‘exclude’ samples from having derived from a similar provenance as that of the comparator sample. A fuller discussion of this can be found above and elsewhere [6,7].

## 6. Conclusions

Whilst we suggest here that there are demonstrable problems with relying solely upon chemical analytical techniques, the introduction of truly independent corroborative analytical techniques would be necessary to validate any interpretative conclusions made of such analyses. We would contend that it would be necessary to validate interpretations and conclusions by physical geoforensic techniques which involve visual identification. Geological studies have their part to play [8,26], but it is not possible to simply transpose geological techniques to the forensic arena without due regard to the different aims of forensic geoscience. Geoforensic science seeks to exclude samples from having derived from a similar provenance; techniques used to provide corroborative evidence must be independent (and ideally not only using chemical analysis techniques which require prior homogenisation of the sample which itself may be unsuitable for soil/sediment samples that may have experienced pre-, syn- or post-forensic event mixing).

It is of great concern to us that the philosophical approach of forensic geoscientists is appropriate as there is too much at stake in the courtroom [27].

## Appendix A.

	SiO <sub>2</sub> (%)	Al <sub>2</sub> O <sub>3</sub> (%)	Nd (ppm)	U (ppm)
Simon's Wood				
Pye et al. [3]				
A1	86.98	1.8	7.1	0.95
A2	80	2	7.82	1.02
A3	70	1.9	7.2	1
A4	78	2	7.1	0.98
A5	82	1.9	3.77	0.8
A6	86	1.73	6	0.85
A7	81	1.9	7	0.9
A8	70	2	5	0.75
A9	66.97	2.22	6.5	0.8
Mean	78.07	1.93	6.06	0.90
Max	86.98	2.22	7.82	1.02
Min	66.97	1.73	3.77	0.75
CV (%)	8.9	7.7	18.3	10.5
Pye et al. [2]				
Mean	86.33	2.04	8.30	1.40
CV (%)	0.3	0.5	0.6	1.2
Mean	85.12	2.06	10.58	1.35
CV (%)	0.4	0.4	0.2	1.4
Mean	86.15	2.03	9.56	1.23
CV (%)	0.1	0.3	0.7	0.3
Mean	85.87	2.04	9.48	1.33
CV (%)	0.70	0.90	10.40	5.80
Mean	90.19	2.01	9.44	1.22
CV (%)	2.40	2.30	18.50	6.50
Arborfield Bridge				
Pye et al. [3]				
B1	48	5.2	16	1.35
B2	48	5.98	15.5	1.32
B3	51	5.3	14.46	1.24
B4	45.08	5.8	15	1.3
B5	55.08	5.8	20	1.78
B6	50	5.4	20	1.7
B7	51	5.3	20	1.65
B8	51	5.12	20.33	1.75

## Appendix A (Continued)

B9	52	5.15	20	1.55
Mean	50.64	5.52	17.86	1.52
Max	55.08	5.98	20.33	1.78
Min	45.08	5.12	14.46	1.24
CV (%)	5.90	5.80	13.90	14.10
Pye et al. [2]				
Mean	51.75	5.75	21.28	1.73
CV (%)	0.6	0.7	0.8	1.2
Mean	49.72	5.88	19.35	1.61
CV (%)	0.5	0.4	1.6	1.3
Mean	51.27	5.81	20.09	1.71
CV (%)	0.7	0.7	0.6	0.7
Mean	50.91	5.81	20.24	1.68
CV (%)	1.90	1.10	4.30	3.40
Mean	45.69	5.63	20.29	1.72
CV (%)	1.00	0.50	0.40	1.20

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