

## SECTION 4: INVESTIGATION OF POTENTIAL SOURCES OF VARIATION

### 4.1 INTRODUCTION

The design of FIRI is such that for each laboratory, we have some basic, though limited, information on the laboratory procedures, including the method of pretreatment applied to the samples, the modern standard, and the background material used. These can be considered as *factors* in the experiment and through statistical analysis, we can investigate whether they offer a statistically significant explanation of the observed variation. The different levels of the factors are described in Table 4.1. In addition, the laboratory type is also considered as a further factor (with 3 levels of LSC, GPC, and AMS).

Therefore, this section considers each sample, in turn, and explores the proportion of variation, which can be explained by each of the factors. For these analyses, extreme values (outliers) have been omitted (as identified in Section 3).

The structure of the section for each sample includes the summary statistics with the number of omitted values from the analysis, a boxplot showing the distribution of the results for the different levels of the factor of interest, and the output from a formal *analysis of variance* (a formal test of the hypothesis that the mean age/activity is the same for each level of the factor). This output takes the form of a table, where the key statistic is the *p-value*. Conventionally, at a 5% significance level, if the *p-value* is less than 0.05, then we reject that the mean age/activity is the same for all levels of the factor and conclude there are statistically significant differences. In such a case, a follow-up analysis can be used to identify the magnitude of any differences.

Table 4.1 Classifications used for modern standard and background material

a) Modern standard	
Original description	Analysis classification/level
ANU sucrose	ASuc
Benzene	Benz(ene)
NIST OxI	NBS1
NIST OxII	NBS2
GIN/HD-95,C-3	Other
NIST I/II	NBS12
b) Background	
Original description	Analysis classification
Anthracite	Anth(racite)
Benzene	Benz(ene)
Calcite	Calc(ite)
Charcoal	Charc(oal)
Bituminous coal	Coal
Graphite	Graphite
Doublespar/IAEA C1	Marble
IAEA C4/wood/limestone	Other

### 4.2 LABORATORY TYPE AS A SOURCE OF VARIATION

In this section, the analysis is focused on whether there are statistically significant differences in the mean activity/age among the different laboratory types.

4.2.1 Sample C: Turbidite

Table 4.2 Summary statistics of age for Sample C

Type	N	Nr of omitted values	Mean	Median	StDev
AMS	34	0	18,175	18,175	135
GPC	17	1	18,152	18,200	288
LSC	33	10	18,110	18,120	244
AMS	34	0	18,175	18,175	135

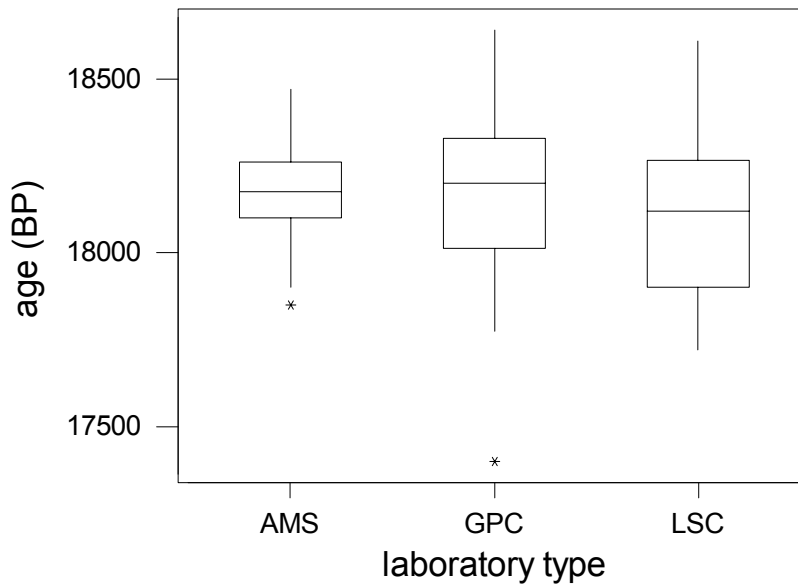


Figure 4.1 Distribution of age by laboratory type for Sample C

Table 4.3 Analysis of variance for Sample C

Source	DF	SS	MS	F	P
type	2	70472	35236	0.75	<b>0.478</b>
Error	81	3830128	47286		
Total	83	3900600			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
AMS	34	18175	135	(-----*-----)
GPC	17	18152	288	(-----*-----)
LSC	33	18110	244	(-----*-----)

Pooled StDev = 217

18060      18120      18180      18240

4.2.1.1 Conclusion

Since the p-value is >0.05 in Table 4.3, there is no evidence of statistically significant differences among laboratory types. The results from each laboratory type broadly overlap.

4.2.2 Sample E: Humic Acid

Table 4.4 Summary statistics of age for Sample E

Type	N	Number omitted	Mean	Median	StDev
AMS	64	1	11,804	11,800	117
GPC	23	4	11,743	11,722	173
LSC	38	14	11,757	11,736	177

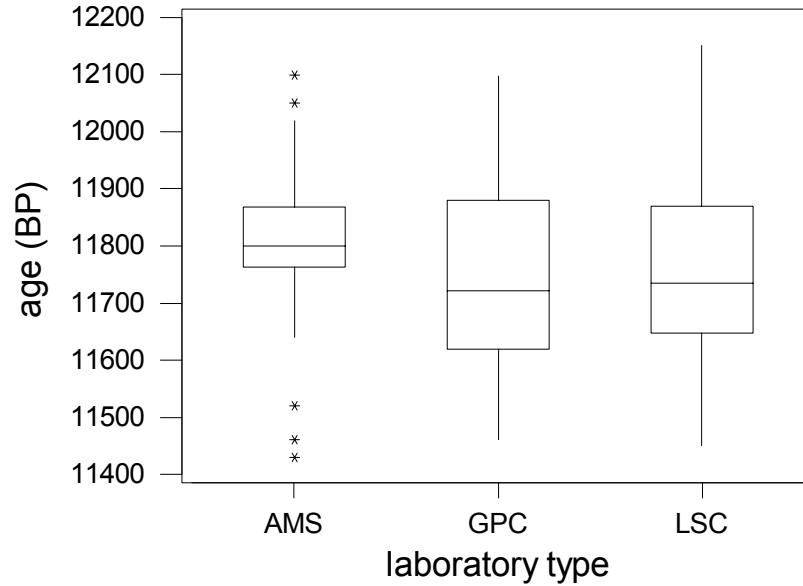


Figure 4.2 Distribution of age by laboratory type for Sample E

Table 4.5 Analysis of variance for Sample E

Source	DF	SS	MS	F	P
type	2	86788	43394	1.97	<b>0.143</b>
Error	122	2680561	21972		
Total	124	2767349			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
AMS	64	11804	117	(-----*-----)
GPC	23	11743	173	(-----*-----)
LSC	38	11757	177	(-----*-----)

4.2.2.1 Conclusion

Since the p-value is >0.05 in Table 4.5, there is no evidence of statistically significant differences among laboratory types. The results from each laboratory type broadly overlap.

**4.2.3 Sample D: Belfast Wood**

Table 4.6 Summary statistics of age for Sample D

Type	N	Number omitted	Mean	Median	StDev
AMS	41	0	4530.3	4520.0	52.0
GPC	19	1	4506.7	4509.0	56.5
LSC	38	12	4521.7	4537.5	106.7

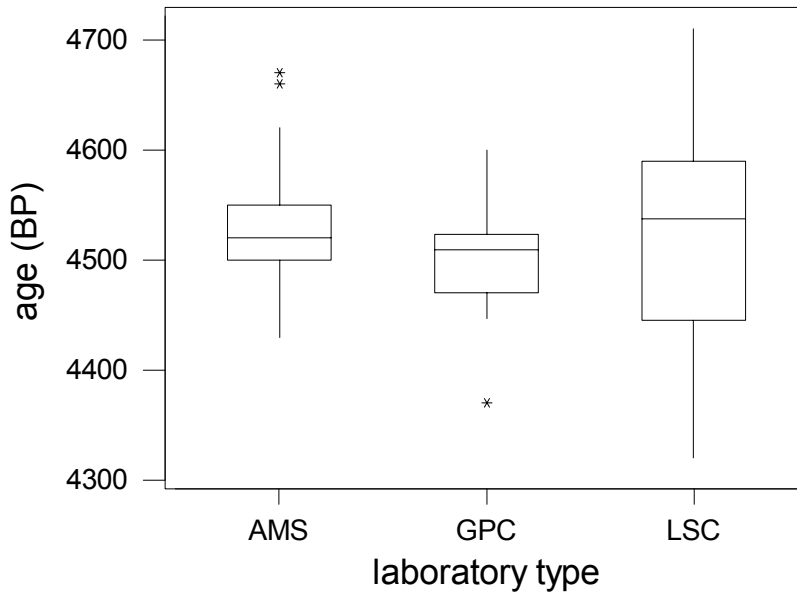


Figure 4.3 Distribution of age by laboratory type for Sample D

Table 4.7 Analysis of variance of Sample D

Source	DF	SS	MS	F	P
type	2	7231	3616	0.59	<b>0.559</b>
Error	95	586712	6176		
Total	97	593943			

Level	N	Mean	StDev
AMS	41	4530.3	52.0
GPC	19	4506.7	56.5
LSC	38	4521.7	106.7

Individual 95% CIs For Mean Based on Pooled StDev				
Level	N	Mean	StDev	CI
AMS	41	4530.3	52.0	(-----*-----)
GPC	19	4506.7	56.5	(-----*-----)
LSC	38	4521.7	106.7	(-----*-----)

Pooled StDev =	78.6	4475	4500	4525	4550
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**4.2.3.1 Conclusion**

Since the p-value is  $>0.05$  in Table 4.7, there is no evidence of statistically significant differences among laboratory types. The results from each laboratory type broadly overlap.



**4.2.5 Sample G: Barley Mash (pMC)**

Table 4.10 Summary statistics of activity (pMC) for Sample G

Type	N	Number omitted	Mean	Median	StDev
AMS	34	0	110.33	110.35	0.68
GPC	18	1	110.85	111.00	1.06
LSC	32	15	110.53	110.60	1.10

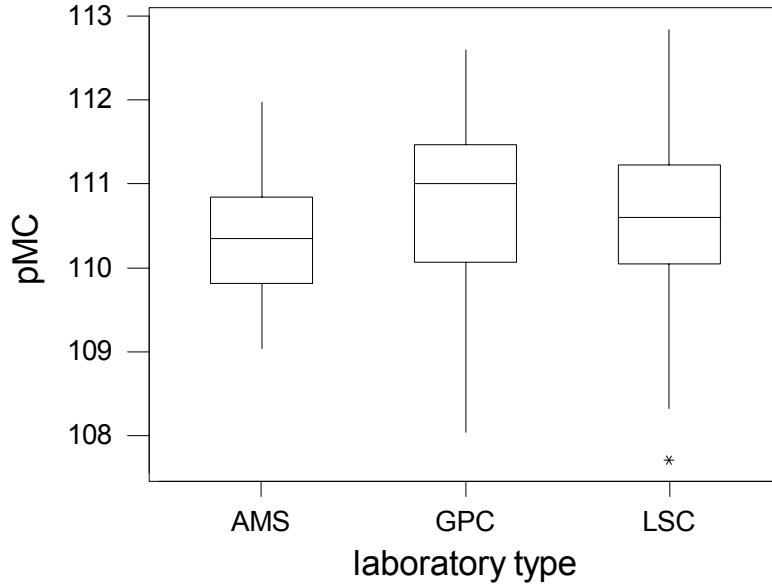


Figure 4.5 Distribution of activity by laboratory type for Sample G

Table 4.11 Analysis of variance for Sample G

Source	DF	SS	MS	F	P
type	2	3.154	1.577	1.76	<b>0.178</b>
Error	81	72.530	0.895		
Total	83	75.684			

Level	N	Mean	StDev
AMS	34	110.335	0.685
GPC	18	110.852	1.064
LSC	32	110.526	1.105

Individual 95% CIs For Mean Based on Pooled StDev			
AMS	110.25	110.60	110.95
GPC	110.25	110.60	110.95
LSC	110.25	110.60	110.95

Pooled StDev = 0.946

**4.2.5.1 Conclusion**

Since the p-value is >0.05 in Table 4.11, there is no evidence of statistically significant differences among laboratory types. The results from each laboratory type broadly overlap.

4.2.6 Sample J: Barley Mash

Table 4.12 Summary statistics of activity (pMC) for Sample J

Type	N	Number omitted	Mean	Median	StDev
AMS	35	0	110.58	110.56	0.61
GPC	18	1	110.60	110.70	0.83
LSC	34	12	110.63	110.84	1.20

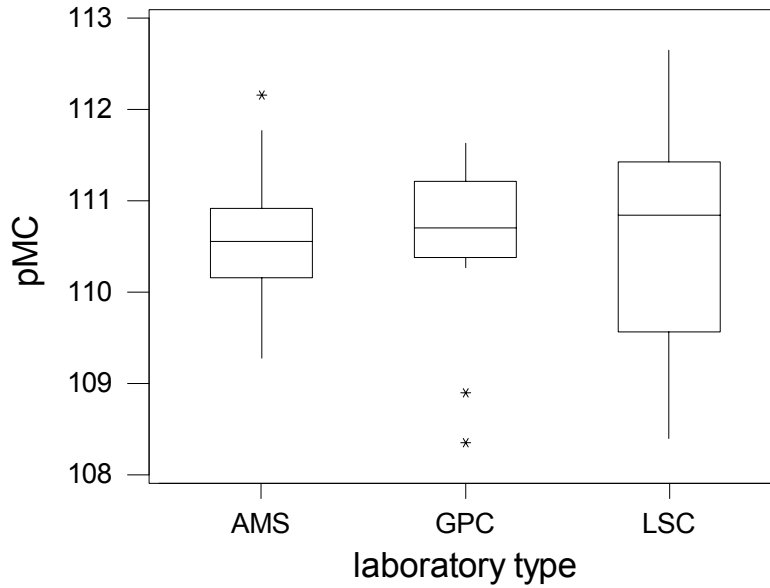


Figure 4.6 Distribution of activity by laboratory type for Sample J

Table 4.13 Analysis of variance for Sample J

Source	DF	SS	MS	F	P
type	2	0.047	0.024	0.03	<b>0.973</b>
Error	84	71.700	0.854		
Total	86	71.748			

Level	N	Mean	StDev
AMS	35	110.583	0.606
GPC	18	110.600	0.834
LSC	34	110.635	1.198

Individual 95% CIs For Mean Based on Pooled StDev				
Level	N	Mean	StDev	CI
AMS	35	110.583	0.606	(-----*-----)
GPC	18	110.600	0.834	(-----*-----)
LSC	34	110.635	1.198	(-----*-----)

Pooled StDev =	0.924	110.25	110.50	110.75	111.00
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4.2.6.1 Conclusion

Since the p-value is >0.05 in Table 4.13, there is no evidence of statistically significant differences among laboratory types. The results from each laboratory type broadly overlap.

**4.2.7 Sample H: Hohenheim Wood**

Table 4.14 Summary statistics of age for Sample H

Type	N	Number omitted	Mean	Median	StDev
AMS	36	0	2228.7	2230.0	48.2
GPC	19	1	2221.7	2200.0	95.4
LSC	33	13	2233.7	2230.0	98.1

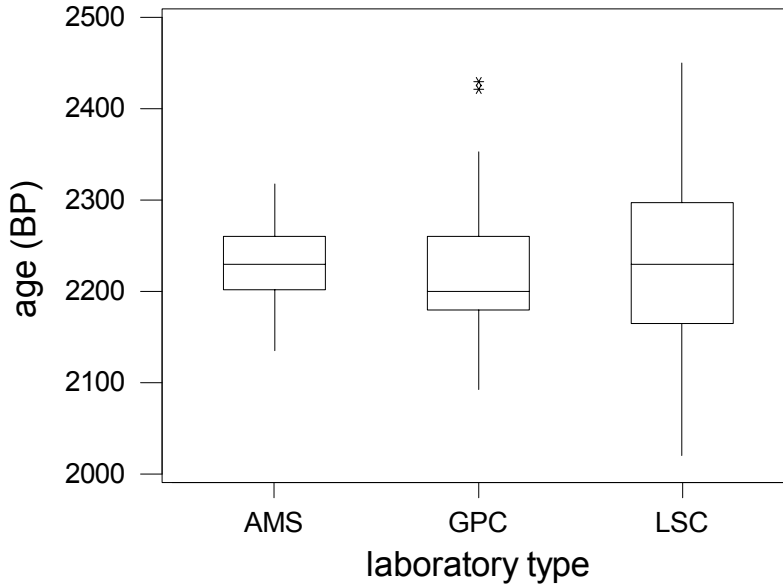


Figure 4.7 Distribution of age by laboratory type for Sample H

Table 4.15 Analysis of variance for Sample H

Source	DF	SS	MS	F	P
type	2	1742	871	0.13	<b>0.875</b>
Error	85	553268	6509		
Total	87	555011			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
AMS	36	2228.7	48.2	(-----*-----)
GPC	19	2221.7	95.4	(-----*-----)
LSC	33	2233.7	98.1	(-----*-----)

Pooled StDev = 80.7

2200      2225      2250

**4.2.7.1 Conclusion**

Since the p-value is >0.05 in Table 4.15, there is no evidence of statistically significant differences among laboratory types. The results from each laboratory type broadly overlap.



4.2.8 Sample I: Belfast Cellulose

Table 4.16 Summary statistics of age for Sample I

Type	N	Number omitted	Mean	Median	StDev
AMS	35	0	4499.1	4490.0	74.1
GPC	16	3	4438.0	4450.0	68.6
LSC	35	11	4508.4	4520.0	128.1

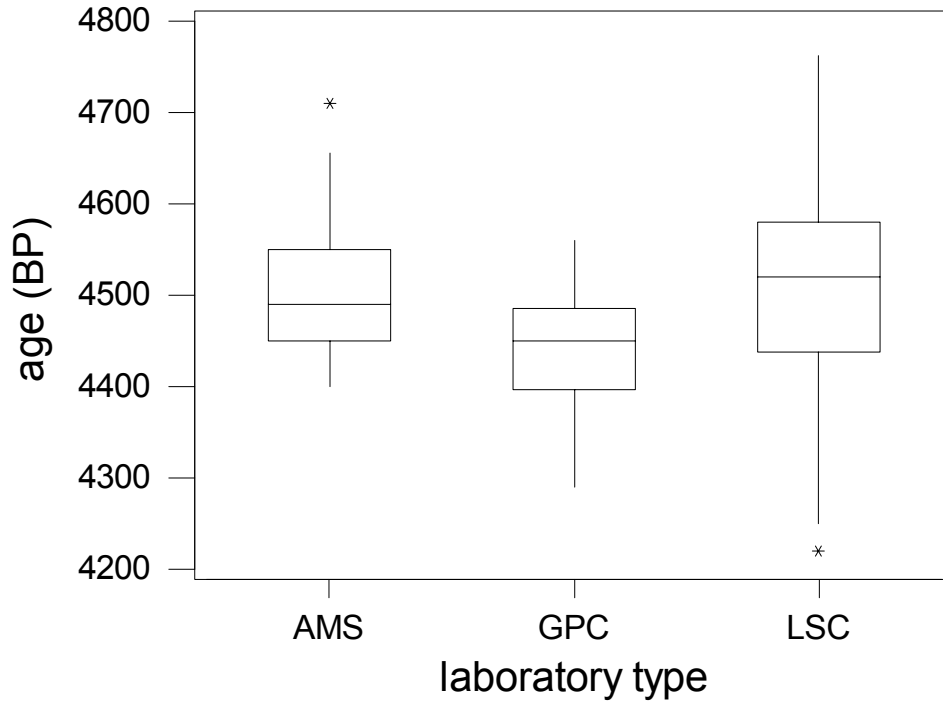


Figure 4.8 Distribution of age by laboratory type for Sample I

Table 4.17 Analysis of variance for Sample I

Source	DF	SS	MS	F	P
type	2	57840	28920	2.94	<b>0.058</b>
Error	83	815080	9820		
Total	85	872921			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
AMS	35	4499.1	74.1	(-----*-----)
GPC	16	4438.0	68.6	(-----*-----)
LSC	35	4508.4	128.1	(-----*-----)

Pooled StDev =	99.1	4400	4450	4500	4550
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#### 4.2.8.1 Conclusion

A statistically significant difference (at 10%) is found among the laboratory types. GPC laboratories quote an average age that is lower than either AMS or LSC laboratories.

#### 4.2.9 Conclusions

In the case of Samples F and I, a statistically significant difference among the laboratory types was found at a 10% level. In general for all other samples, there is no evidence of a difference, on average, among laboratory types. We can conclude that laboratories are comparable on the average age/activity. However, where we have seen lack of comparability is in the number of outliers, with LSC laboratories (as can be seen from the preceding tables) having by far the largest number of measurements omitted. The other striking feature from the tables is the comparison of the standard deviations, which provide a measure of the scatter or variation in the population. In all cases, the AMS laboratory results have the smallest variation, in some cases by as much as a factor of 2.

### 4.3 MODERN STANDARD AND BACKGROUND MATERIAL AS SOURCES OF VARIATION

Two other factors of potential use in explaining the observed variation are the background and modern standard materials used. This section, thus, explores these 2 factors, with the reporting format identical to that used in Section 4.2. The classification of materials (and the analysis code used) is given below

Table 4.18 Coding for a) modern standard material and b) background material

#### a) Coding for modern standard material

Original description	Analysis classification/level
ANU sucrose	ASuc
Benzene	Benz(ene)
NIST OxI	NBS1
NIST OxII	NBS2
GIN/HD-95,C-3	Other
NIST 1/II	NBS12

#### b) Coding for background material

Original description	Analysis classification
Anthracite	Anth(racite)
Benzene	Benz(ene)
Calcite	Calc(ite)
Charcoal	Charc(oal)
Bituminous coal	Coal
Graphite	Graphite
Doublespar/IAEA C1	Marble
IAEA C4/wood/limestone	Other

4.3.1 Sample C: Marine Turbidite

Table 4.19a Descriptive statistics for age by modern standard used

Standard	N	Number omitted	Mean	Median	StDev
ASuc	8	1	18,294	18,225	217
Benz	3	2	17,918	17,820	211
NBS1	25	5	18,143	18,180	215
NBS12	9	0	18,123	18,100	188
NBS2	27	2	18,149	18,120	231
Other	5	0	18,107	18,138	248
Unknown	7	1	18,115	18,140	135

Table 4.19b Descriptive statistics for age by background material used

Background	N	Number omitted	Mean	Median	StDev
Anth	12	0	18,118	18,191	304
Benz	14	3	18,070	18,120	205
Calc	3	0	18,347	18,230	229
Coal	4	0	18,095	18,144	139
Graph	2	1	18,240	18,240	189
Marble	22	3	18,162	18,145	207
Other	14	3	18,157	18,227	216
Unknown	13	1	18,163	18,160	178

4.3.1.1 Graphical Analysis

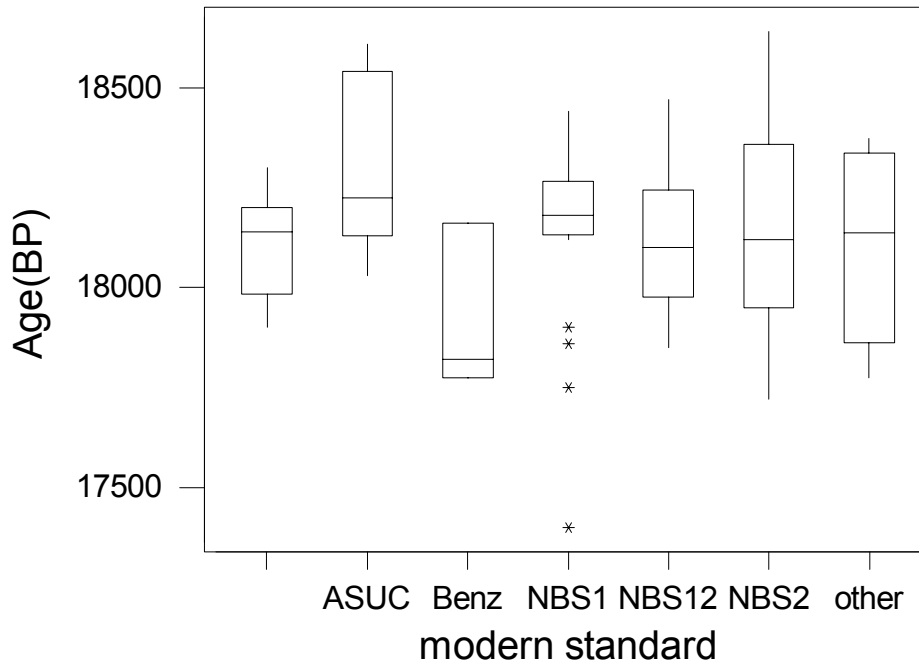


Figure 4.9a Distribution of age (yr BP) by modern standard material

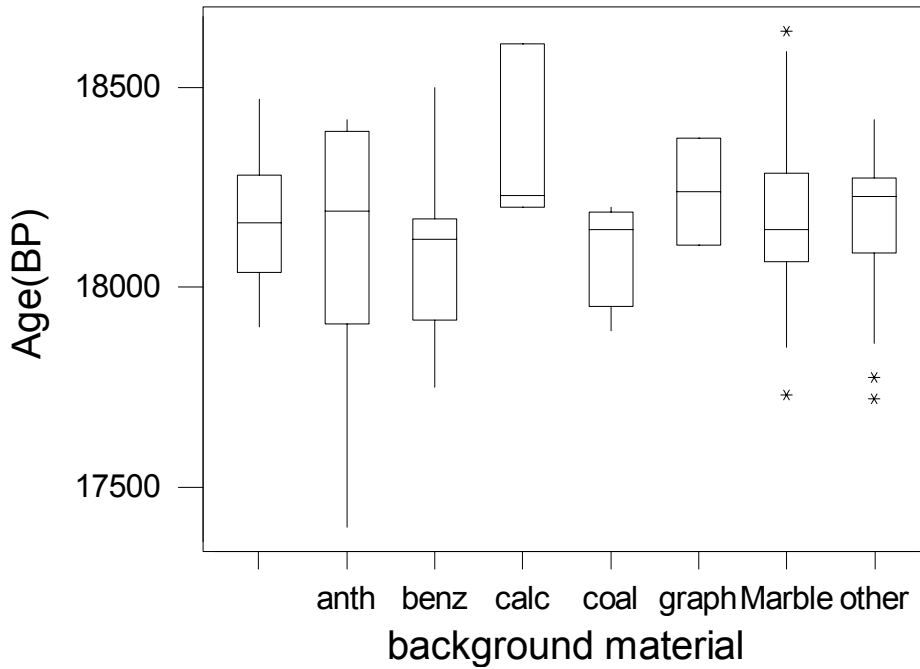


Figure 4.9b Distribution of age by background material

4.3.1.2 Formal Analysis

The formal analysis is carried out using a one-way analysis of variance (ANOVA); the hypothesis of interest is that the mean <sup>14</sup>C age is the same, irrespective of the modern standard or background material. The results are again summarized by the p-value.

Table 4.20a Analysis of variance of age by modern standard

Source	DF	SS	MS	F	P
Age	5	342707	68541	1.41	<b>0.230</b>
Error	71	3442176	48481		
Total	76	3784883			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
ASUC	8	18294	217	(-----*-----)
Benz	3	17918	211	(-----*-----)
NBS1	25	18143	215	(---*---)
NBS12	9	18123	188	(-----*-----)
NBS2	27	18149	231	(--*--)
other	5	18107	248	(-----*-----)

Pooled StDev =	220	17750	18000	18250	18500
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Table 4.20b Analysis of variance of age by background material

Source	DF	SS	MS	F	P
Age	6	244377	40729	0.80	<b>0.576</b>
Error	64	3271008	51109		
Total	70	3515384			

Individual 95% CIs For Mean Based on Pooled StDev			
Level	N	Mean	StDev
anth	12	18118	304
benz	14	18070	205
calc	3	18347	229
coal	4	18095	139
graph	2	18240	189
Marble	22	18162	207
other	14	18157	216

Pooled StDev =	226	18000	18200	18400
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4.3.1.3 Conclusion

For Sample C, there is no evidence that either the modern standard or background material used is a statistically significant factor in explaining the variation observed (p-value >0.05 in both cases).

4.3.2 Sample D: Belfast Wood

Table 4.21a Descriptive statistics age by modern standard material

Standard	N	Number omitted	Mean	Median	StDev
ASUC	6	1	4558.5	4565.0	95.4
Benz	3	3	4416.7	4420.0	90.0
NBS1	28	3	4505.4	4510.5	52.9
NBS12	7	0	4567.7	4550.0	51.4
NBS2	37	3	4537.8	4540.0	82.6
Other	7	1	4485.4	4482.0	35.3
Unknown	10	2	4516.8	4517.0	108.1

Table 4.21b Descriptive statistics age by background material

Background	N	Number omitted	Mean	Median	StDev
Anth	19	1	4530.3	4511.0	74.2
Benz	14	4	4515.1	4502.5	124.6
Calc	2	0	4530.0	4530.0	28.3
Charc	2	0	4525.0	4525.0	134.4
Coal	11	0	4487.9	4510.0	55.9
Graph	9	0	4513.7	4500.0	41.9
Marble	6	2	4505.5	4527.5	102.9
Other	18	3	4521.7	4519.0	56.6
Unknown	17	3	4551.8	4540.0	74.6

4.3.2.1 Graphical Analysis

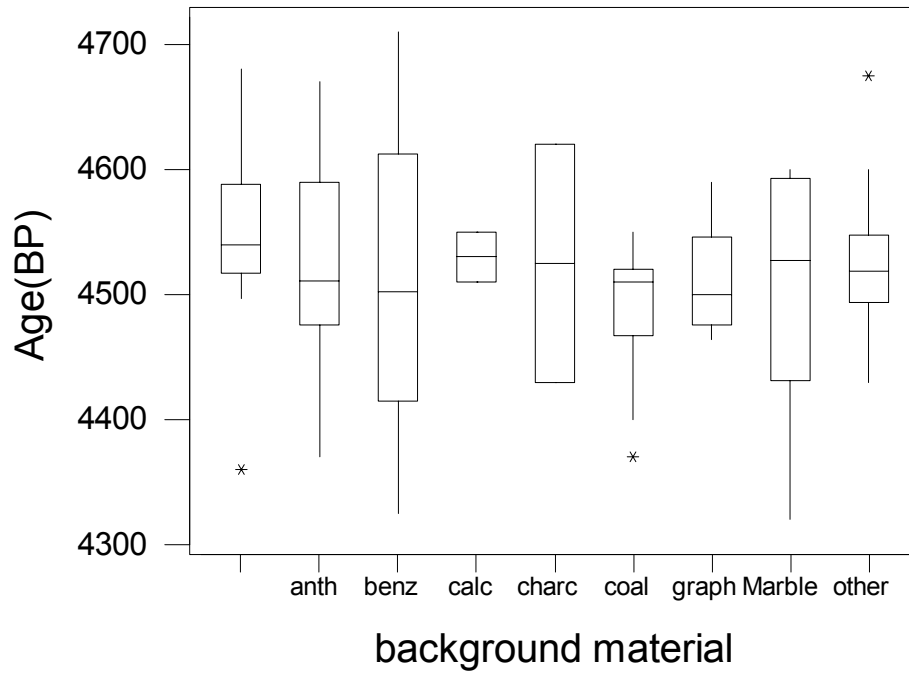


Figure 4.10a Distribution of age by background material

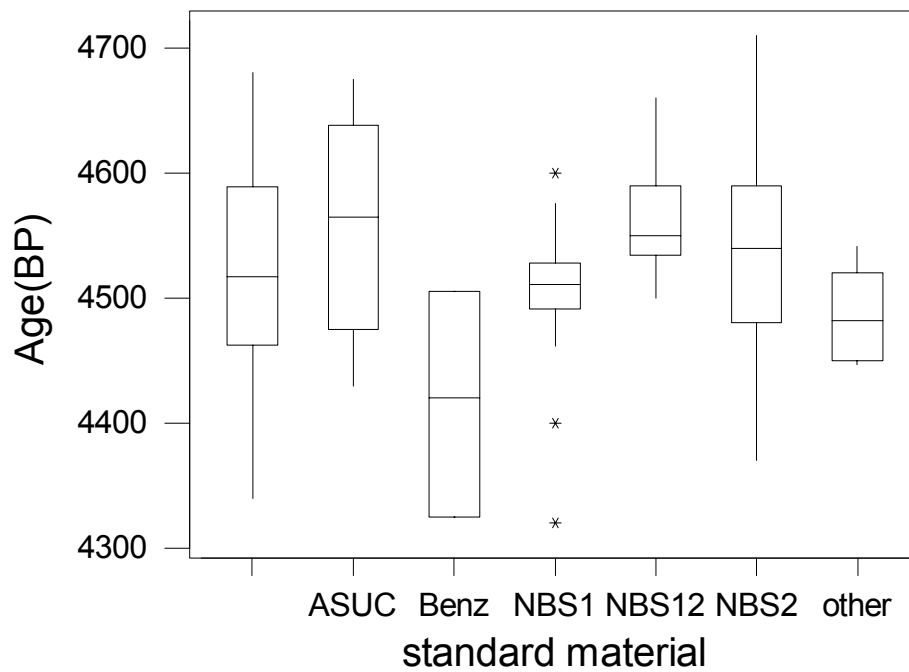


Figure 4.10b Distribution of age by modern standard material

4.3.2.2 Formal Analysis

Table 4.22a Analysis of variance of age by modern standard material

Source	DF	SS	MS	F	P
age	5	82173	16435	3.32	<b>0.009</b>
Error	82	406320	4955		
Total	87	488493			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
ASUC	6	4558.5	95.4	(-----*-----)
Benz	3	4416.7	90.0	(-----*-----)
NBS1	28	4505.4	52.9	(--*--)
NBS12	7	4567.7	51.4	(-----*-----)
NBS2	37	4537.8	82.6	(--*--)
other	7	4485.4	35.3	(-----*-----)

Pooled StDev =	70.4			4400	4480	4560
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Table 4.22b Analysis of variance of age by background material

Source	DF	SS	MS	F	P
age	7	14421	2060	0.32	<b>0.944</b>
Error	73	472634	6474		
Total	80	487055			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
anth	19	4530.3	74.2	(----*----)
benz	14	4515.1	124.6	(-----*-----)
calc	2	4530.0	28.3	(-----*-----)
charc	2	4525.0	134.4	(-----*-----)
coal	11	4487.9	55.9	(-----*-----)
graph	9	4513.7	41.9	(-----*-----)
Marble	6	4505.5	102.9	(-----*-----)
other	18	4521.7	56.6	(----*----)

Pooled StDev =	80.5			4480	4550	4620
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4.3.2.3 Conclusions

For Sample D, the modern standard is found to be statistically significant in explaining the observed variation. There are statistically significant differences among the mean ages for results based on the different modern standards. The modern standard material as a factor accounts for approximately 17% of the total variation observed. Laboratories using benzene as a modern standard material quote, on average, lower ages for this sample. The background material was not found statistically significant.

**4.3.3 Sample F: Belfast Wood**

Table 4.23a Descriptive statistics: age by background material

Background	N	Number omitted	Mean	Median	StDev
Anth	16	3	4507.9	4502.0	83.0
Benz	14	4	4507.1	4495.0	82.4
Calc	2	0	4472.5	4472.5	46.0
Charc	2	0	4525.0	4525.0	91.9
Coal	8	1	4461.8	4477.0	71.4
Graph	10	1	4554.9	4555.0	73.8
Marble	6	2	4551.8	4550.0	98.9
Other	18	2	4519.1	4511.5	66.6
Unknown	14	3	4488.9	4491.5	69.4

Table 4.23b Descriptive statistics: age by modern standard

Standard	N	Number omitted	Mean	Median	StDev
Asuc	5	3	4546.0	4550.0	63.5
Benz	5	1	4470.2	4459.0	116.0
NBS1	28	3	4502.4	4497.0	48.0
NBS12	9	0	4566.3	4550.0	63.8
NBS2	29	6	4518.4	4513.0	97.4
Other	8	0	4503.3	4487.0	42.1
Unknown	6	3	4442.8	4460.0	59.4

4.3.3.1 Graphical Analysis

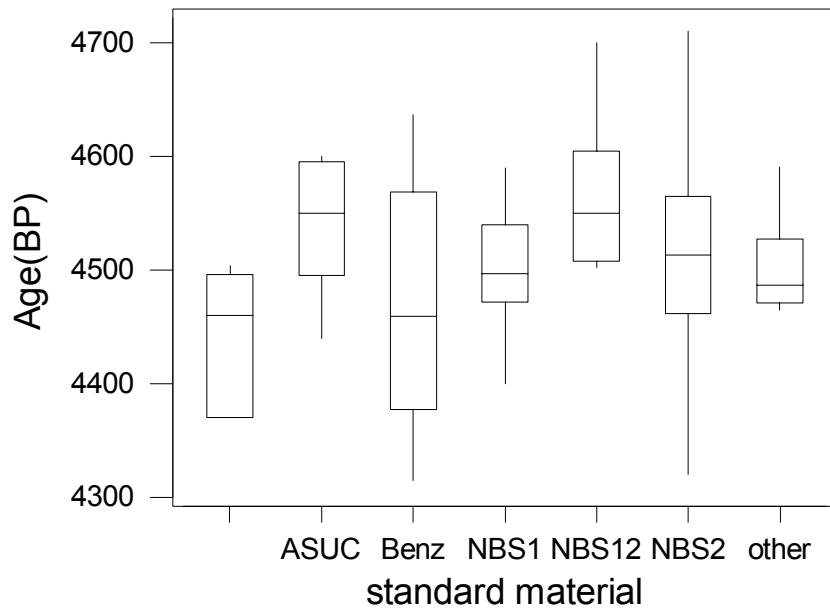


Figure 4.11a Distribution of age by modern standard material



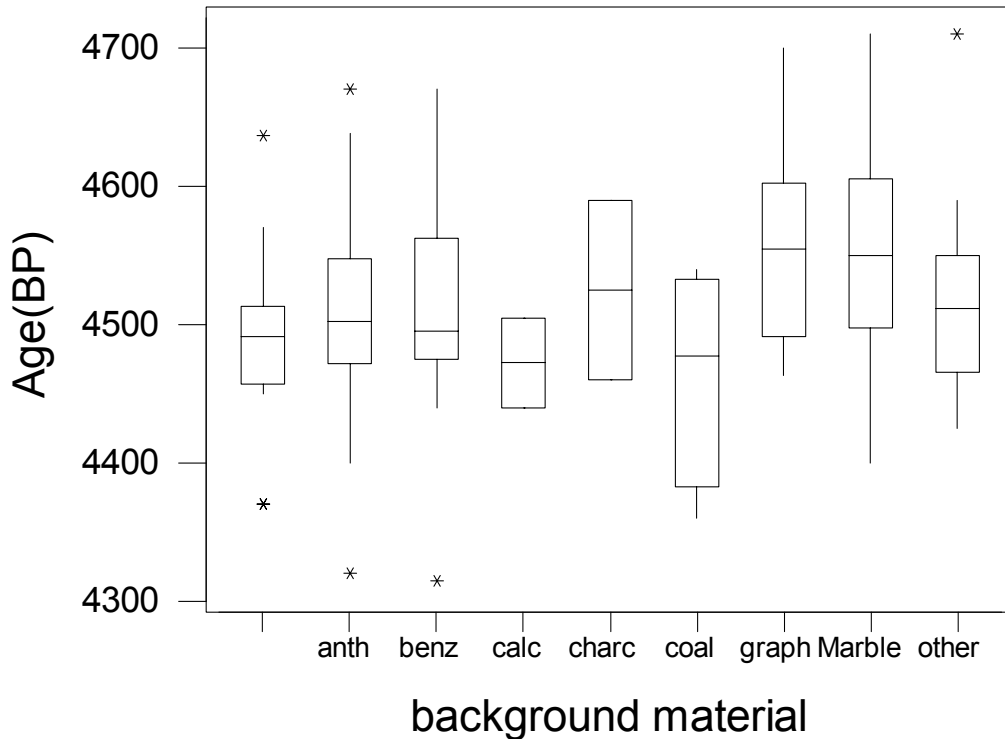


Figure 4.11b Distribution of age by background material

4.3.3.2 Formal Analysis

Table 4.24a Analysis of variance of age by background

Source	DF	SS	MS	F	P
age	7	52484	7498	1.24	<b>0.294</b>
Error	68	411176	6047		
Total	75	463660			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
anth	16	4507.9	83.0	(---*---)
benz	14	4507.1	82.4	(---*---)
calc	2	4472.5	46.0	(-----*-----)
charc	2	4525.0	91.9	(-----*-----)
coal	8	4461.8	71.4	(-----*-----)
graph	10	4554.9	73.8	(-----*-----)
Marble	6	4551.8	98.9	(-----*-----)
other	18	4519.1	66.6	(---*---)

Pooled StDev = 77.8

Table 4.24b Analysis of variance of age by modern standard

Source	DF	SS	MS	F	P
Age	5	44382	8876	1.56	<b>0.180</b>
Error	78	442573	5674		
Total	83	486955			

Individual 95% CIs For Mean Based on Pooled StDev					
Level	N	Mean	StDev	-----+-----+-----+-----	
ASUC	5	4546.0	63.5	(-----*-----)	
Benz	5	4470.2	116.0	(-----*-----)	
NBS1	28	4502.4	48.0	(---*---)	
NBS12	9	4566.3	63.8	(-----*-----)	
NBS2	29	4518.4	97.4	(---*---)	
other	8	4503.3	42.1	(-----*-----)	
-----+-----+-----+-----					
Pooled StDev =		75.3		4440	4500 4560

#### 4.3.3.3 Conclusions

Neither modern standard nor background materials proved to be statistically significant in explaining the observed variation.

#### 4.3.4 Sample E: Humic Acid

Table 4.25a Descriptive statistics: age by modern standard

Standard	N	Number omitted	Mean	Median	StDev
ASUC	8	2	11,712	11,715	130
Benz	4	2	11,681	11,682	58
NBS1	49	7	11,779	11,800	144
NBS12	5	0	11,781	11,770	87
NBS2	37	6	11,813	11,809	167
Other	14	0	11,785	11,771	175
Unknown	8	2	11,710	11,721	91

Table 4.25b Descriptive statistics: age by background material

Background	N	Number omitted	Mean	Median	StDev
Anth	20	3	11,847	11,855	139
Benz	18	4	11,761	11,700	208
Calc	2	0	11,715	11,715	78
Charc	13	0	11,832	11,800	90
Coal	18	0	11,804	11,805	141
Graph	15	0	11,734	11,772	158
Marble	9	2	11,754	11,760	176
Other	17	5	11,729	11,731	126
Unknown	13	5	11,748	11,760	103

4.3.4.1 Graphical Analysis

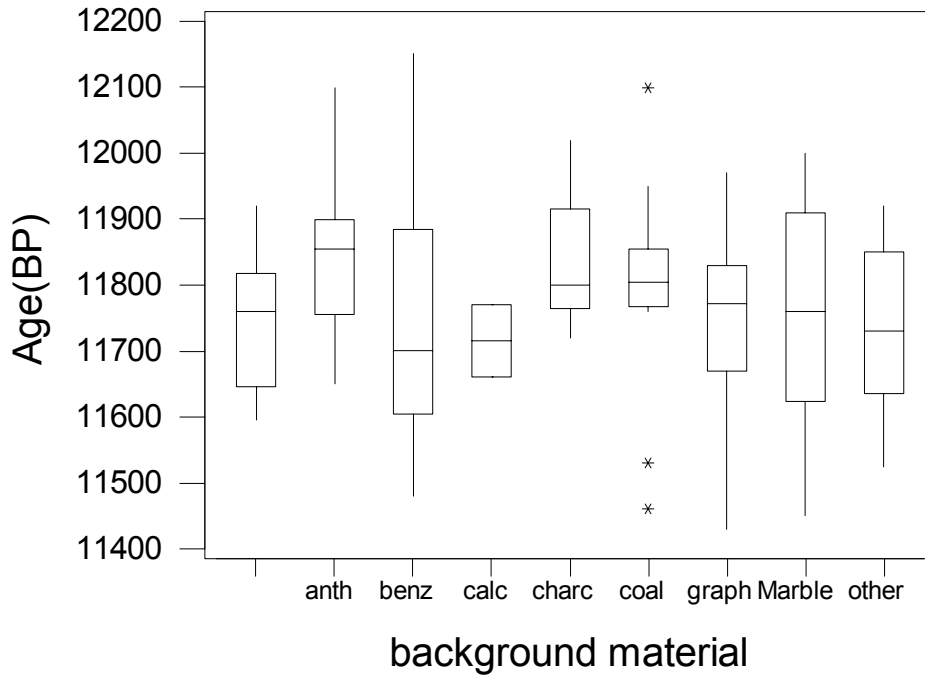


Figure 4.12a Distribution of age by background material

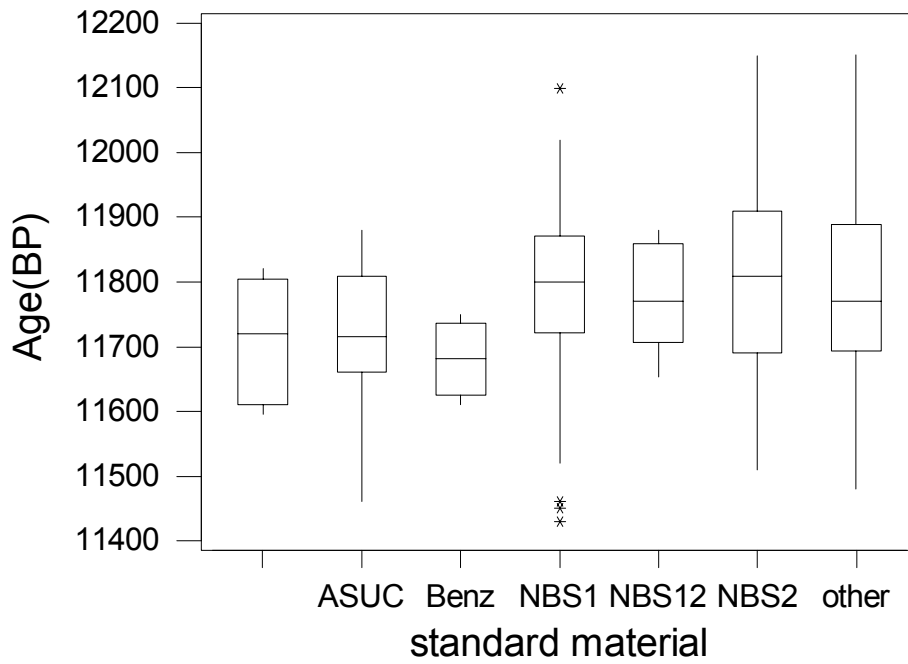


Figure 4.12b Distribution of age by modern standard material

4.3.4.2 Formal Analysis

Table 4.26a Analysis of variance of age by modern standard

Source	DF	SS	MS	F	P
Age	5	117339	23468	1.02	<b>0.409</b>
Error	111	2552299	22994		
Total	116	2669638			

Individual 95% CIs For Mean Based on Pooled StDev					
Level	N	Mean	StDev	-----+-----+-----+-----	
ASUC	8	11712	130	(-----*-----)	
Benz	4	11681	58	(-----*-----)	
NBS1	49	11779	144	(---*---)	
NBS12	5	11781	87	(-----*-----)	
NBS2	37	11813	167	(---*---)	
other	14	11785	175	(-----*-----)	
-----+-----+-----+-----					
Pooled StDev =		152		11640	11760 11880

Table 4.26b Analysis of variance of age by background material

Source	DF	SS	MS	F	P
background	7	234848	33550	1.46	<b>0.190</b>
Error	104	2392794	23008		
Total	111	2627641			

Individual 95% CIs For Mean Based on Pooled StDev					
Level	N	Mean	StDev	--+-----+-----+-----+-----	
anth	20	11847	139	(---*---)	
benz	18	11761	208	(-----*-----)	
calc	2	11715	78	(-----*-----)	
charc	13	11832	90	(-----*-----)	
coal	18	11804	141	(-----*-----)	
graph	15	11734	158	(-----*-----)	
Marble	9	11754	176	(-----*-----)	
other	17	11729	126	(-----*-----)	
--+-----+-----+-----+-----					
Pooled StDev =		152		11520	11640 11760 11880

4.3.4.3 Conclusions

For Sample E, neither modern standard nor background materials are statistically significant in explaining the observed variation.

4.3.5 Sample G: Barley Mash

Table 4.27a Descriptive statistics: activity (pMC) by background material

Background	N	Number omitted	Mean	Median	StDev
Anth	15	3	110.30	110.86	1.48
Benz	16	3	110.46	110.85	1.28
Calc	2	0	110.27	110.27	0.01
Charc	2	0	110.90	110.90	0.70
Coal	7	0	110.14	110.13	1.70
Graph	10	0	110.33	110.49	0.64
Marble	6	2	110.83	110.75	0.99
Other	16	3	110.35	110.28	0.81
Unknown	12	3	110.71	110.80	0.66

Table 4.27b Descriptive statistics: activity (pMC) by modern standard

Standard	N	Number omitted	Mean	Median	StDev
ASUC	6	1	109.39	109.75	1.32
Benz	5	1	111.10	111.03	1.10
NBS1	26	3	110.46	110.22	0.92
NBS12	7	0	110.36	110.26	0.62
NBS2	29	6	110.39	110.53	1.36
Other	7	1	110.82	110.84	0.40
Unknown	6	2	110.68	110.75	0.19

4.3.5.1 Graphical Analysis

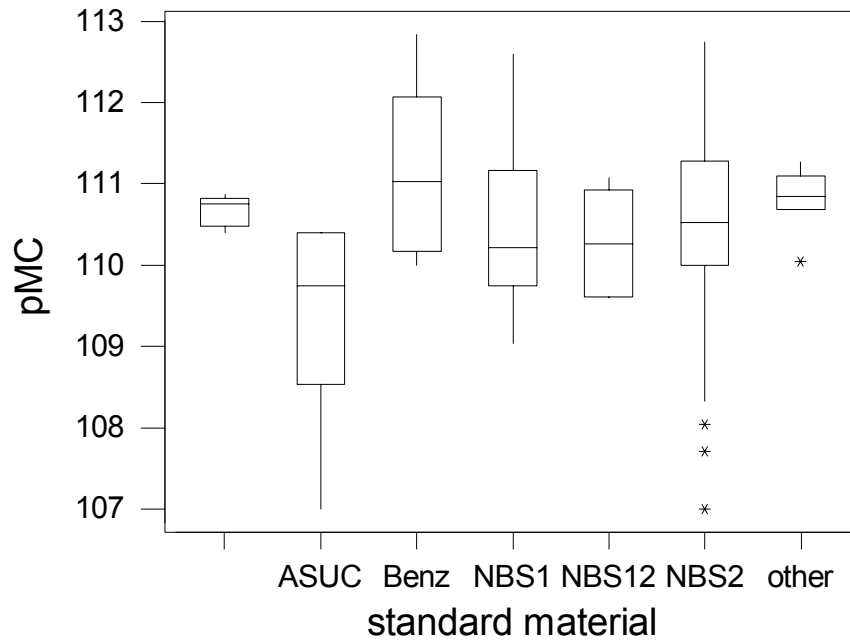


Figure 4.13a Distribution of activity by modern standard

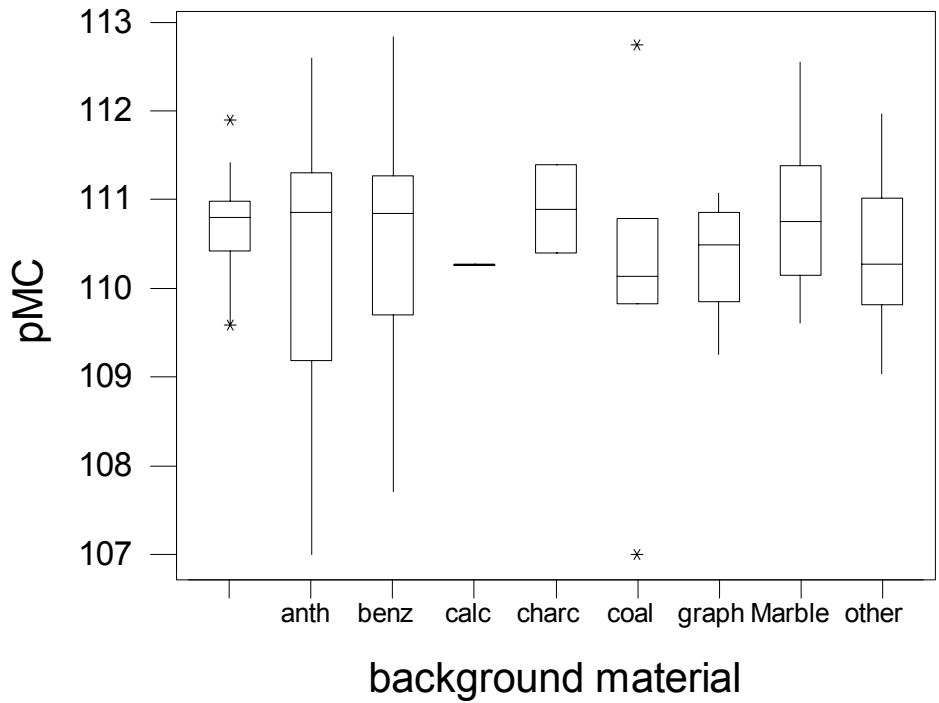


Figure 4.13b Distribution of activity by background material

4.3.5.2 Formal Analysis

Table 4.28a Analysis of variance of activity by background material

Source	DF	SS	MS	F	P
background	7	2.42	0.35	0.25	<b>0.971</b>
Error	66	91.67	1.39		
Total	73	94.09			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
anth	15	110.30	1.48	(-----*-----)
benz	16	110.46	1.28	(-----*-----)
calc	2	110.27	0.01	(-----*-----)
charc	2	110.90	0.70	(-----*-----)
coal	7	110.14	1.70	(-----*-----)
graph	10	110.33	0.64	(-----*-----)
Marble	6	110.83	0.99	(-----*-----)
other	16	110.35	0.81	(-----*-----)

Pooled StDev =	1.18	109.2	110.4	111.6	112.8
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Table 4.28b Analysis of variance of activity by modern standard

Source	DF	SS	MS	F	P
standard	5	9.86	1.97	1.63	<b>0.162</b>
Error	74	89.45	1.21		
Total	79	99.31			

Individual 95% CIs For Mean Based on Pooled StDev			
Level	N	Mean	StDev
ASUC	6	109.39	1.32
Benz	5	111.10	1.10
NBS1	26	110.46	0.92
NBS12	7	110.36	0.62
NBS2	29	110.39	1.36
other	7	110.82	0.40

Pooled StDev =	1.10	109.0	110.0	111.0	112.0
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4.3.5.3 Conclusions

Neither modern standard nor background materials are statistically significant in explaining the variation in activity.

4.3.6 Sample J: Barley Mash

Table 4.29a Descriptive statistics: activity (pMC) by modern standard

Standard	N	Number omitted	Mean	Median	StDev
ASUC	4	1	109.89	109.83	1.19
Benz	5	1	110.63	110.22	1.30
NBS1	27	2	110.52	110.50	0.77
NBS12	8	0	110.77	110.56	0.78
NBS2	28	7	110.64	110.71	0.98
Other	8	0	111.13	111.01	0.59
Unknown	7	2	110.40	110.90	1.12

Table 4.29b Descriptive statistics: activity (pMC) by background material

Background	N	Number omitted	Mean	Median	StDev
Anth	17	1	110.45	110.70	1.15
Benz	13	5	111.23	111.03	0.80
Calc	2	0	110.00	110.00	0.24
Charc	2	0	111.39	111.39	0.01
Coal	7	0	110.51	110.92	0.81
Graph	10	1	110.85	110.61	0.64
Marble	7	2	110.68	110.70	0.75
Other	16	2	110.29	110.23	0.67
Unknown	13	2	110.38	110.70	1.11

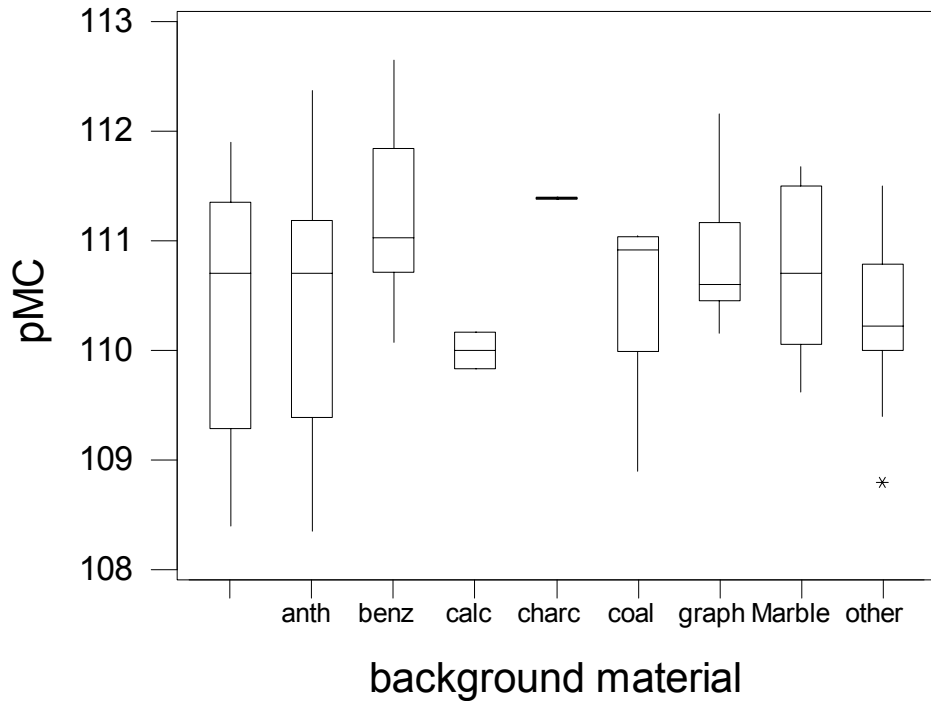


Figure 4.14a Distribution of activity by background material

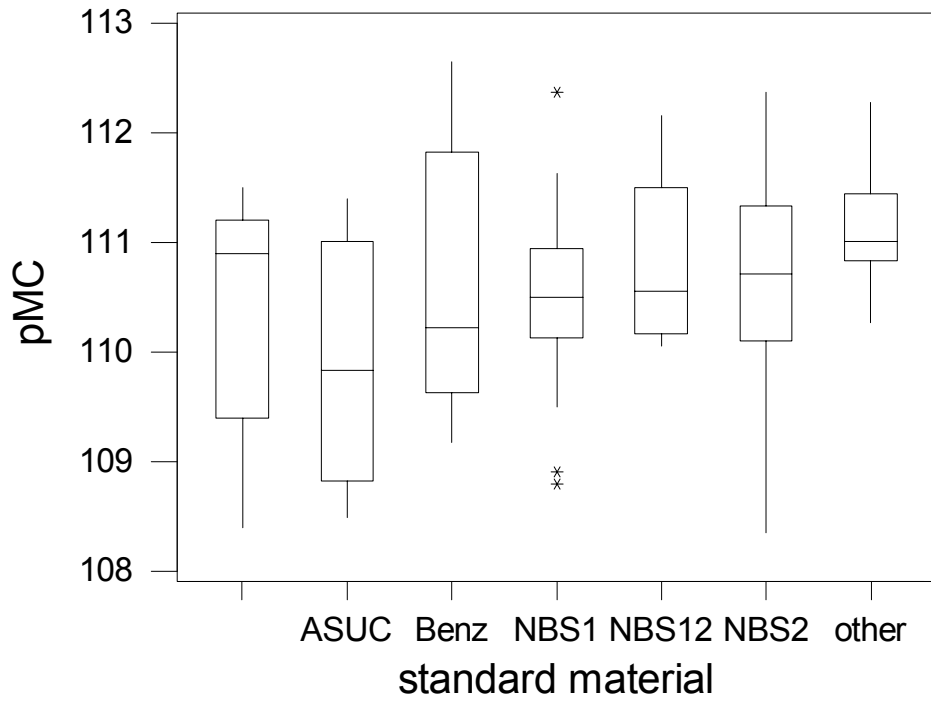


Figure 4.14b Distribution of activity by modern standard



4.3.6.2 Formal Analysis

Table 4.30a Analysis of variance of activity by modern standard

Source	DF	SS	MS	F	P
Standard	5	4.711	0.942	1.18	<b>0.329</b>
Error	74	59.228	0.800		
Total	79	63.939			
Individual 95% CIs For Mean Based on Pooled StDev					
Level	N	Mean	StDev	-----+-----+-----+-----	
ASUC	4	109.888	1.190	(-----*-----)	
Benz	5	110.626	1.303	(-----*-----)	
NBS1	27	110.520	0.771	(----*----)	
NBS12	8	110.768	0.781	(-----*-----)	
NBS2	28	110.643	0.983	(---*---)	
other	8	111.133	0.586	(-----*-----)	
-----+-----+-----+-----					
Pooled StDev =		0.895		109.60	110.40 111.20

Table 4.30b Analysis of variance of activity by background material

Source	DF	SS	MS	F	P
background	7	9.538	1.363	1.93	<b>0.079</b>
Error	66	46.641	0.707		
Total	73	56.179			
Individual 95% CIs For Mean Based on Pooled StDev					
Level	N	Mean	StDev	----+-----+-----+-----+---	
anth	17	110.447	1.154	(---*---)	
benz	13	111.226	0.797	(---*---)	
calc	2	110.000	0.240	(-----*-----)	
charc	2	111.390	0.014	(-----*-----)	
coal	7	110.513	0.807	(----*----)	
graph	10	110.849	0.643	(----*----)	
Marble	7	110.676	0.746	(----*----)	
other	16	110.291	0.668	(---*---)	
----+-----+-----+-----+---					
Pooled StDev =		0.841		109.2	110.4 111.6 112.8

4.3.6.3 Conclusions

The background material is statistically significant at the 10% level, but the modern standard is not a statistically significant factor.

**4.3.7 Sample H: Hohenheim Wood**

Table 4.31a Descriptive statistics: age by background material

Background	N	Number omitted	Mean	Median	StDev
Anth	15	4	2254.3	2240.0	74.9
Benz	12	6	2222.3	2249.0	102.6
Calc	2	0	2302.5	2302.5	53.0
Charc	2	0	2230.0	2230.0	14.1
Coal	9	0	2204.6	2210.0	52.3
Graph	10	0	2215.7	2215.0	62.9
Marble	8	0	2210.3	2205.0	118.4
Other	16	2	2233.6	2240.0	87.0
Unknown	14	2	2228.2	2209.5	66.2

Table 4.31b Descriptive statistics: age by modern standard

Standard	N	Number omitted	Mean	Median	StDev
ASUC	6	0	2276.0	2280.0	127.0
Benz	4	2	2175.8	2133.0	155.0
NBS1	27	2	2211.7	2210.0	67.5
NBS12	8	0	2219.9	2225.0	33.4
NBS2	28	7	2240.1	2240.0	77.7
Other	7	1	2282.4	2280.0	79.2
Unknown	8	2	2203.3	2190.0	43.7

4.3.7.2 Formal Analysis

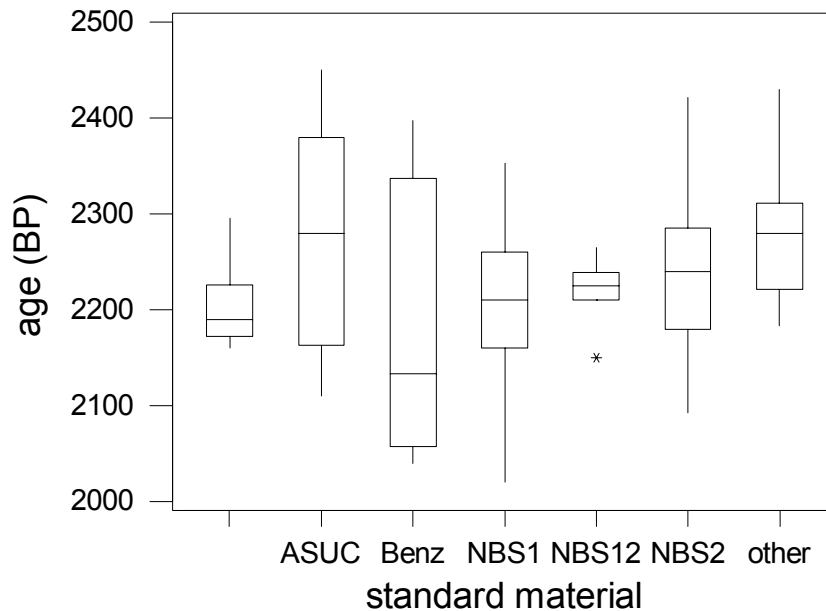


Figure 4.15a Distribution of age by modern standard

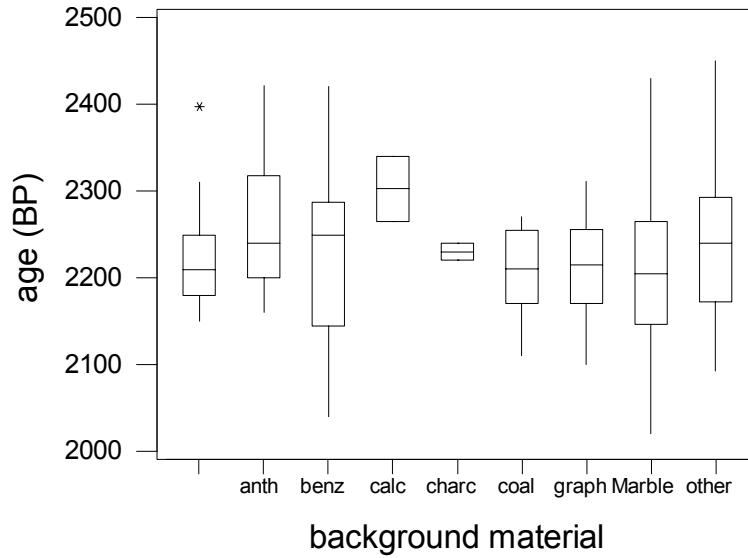


Figure 4.15b Distribution of age by background material

Table 4.32a Analysis of variance of age by background material

Source	DF	SS	MS	F	P
background	7	31284	4469	0.63	<b>0.728</b>
Error	66	466699	7071		
Total	73	497983			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
anth	15	2254.3	74.9	(---*---)
benz	12	2222.3	102.6	(---*---)
calc	2	2302.5	53.0	(-----*-----)
charc	2	2230.0	14.1	(-----*-----)
coal	9	2204.6	52.3	(---*---)
graph	10	2215.7	62.9	(---*---)
Marble	8	2210.3	118.4	(---*---)
other	16	2233.6	87.0	(---*---)

Pooled StDev = 84.1

Table 4.32b Analysis of variance of age by modern standard

Source	DF	SS	MS	F	P
standard	5	56175	11235	1.73	<b>0.138</b>
Error	74	479615	6481		
Total	79	535790			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
ASUC	6	2276.0	127.0	(-----*-----)
Benz	4	2175.8	155.0	(-----*-----)
NBS1	27	2211.7	67.5	(---*---)
NBS12	8	2219.9	33.4	(-----*-----)
NBS2	28	2240.1	77.7	(---*---)
other	7	2282.4	79.2	(-----*-----)

Pooled StDev = 80.5

#### 4.3.7.3 Conclusions

Again, neither standard nor background materials are statistically significant.

#### 4.3.8 Sample I: Belfast Cellulose

Table 4.33a Descriptive statistics: age by modern standard

Standard	N	Number omitted	Mean	Median	StDev
ASUC	6	2	4568.7	4555.0	120.9
Benz	4	1	4495	4530	201
NBS1	25	3	4473.5	4468.0	73.2
NBS12	8	0	4490.8	4490.0	53.9
NBS2	31	3	4500.8	4500.0	116.7
Other	6	2	4500.5	4455.5	88.6
Unknown	6	3	4431.7	4430.0	51.3

Table 4.33b Descriptive statistics: age by background material

Background	N	N*	Mean	Median	StDev
Anth	16	1	4483.3	4490.0	111.7
Benz	15	4	4523.5	4520.0	156.3
Calc	2	0	4540.0	4540.0	56.6
Charc	2	0	4420.0	4420.0	14.1
Coal	6	2	4485.0	4490.0	53.9
Graph	11	0	4458.6	4461.0	87.5
Marble	6	2	4474.0	4480.0	76.8
Other	16	3	4513.0	4495.0	82.4
Unknown	12	2	4480.2	4485.0	75.9

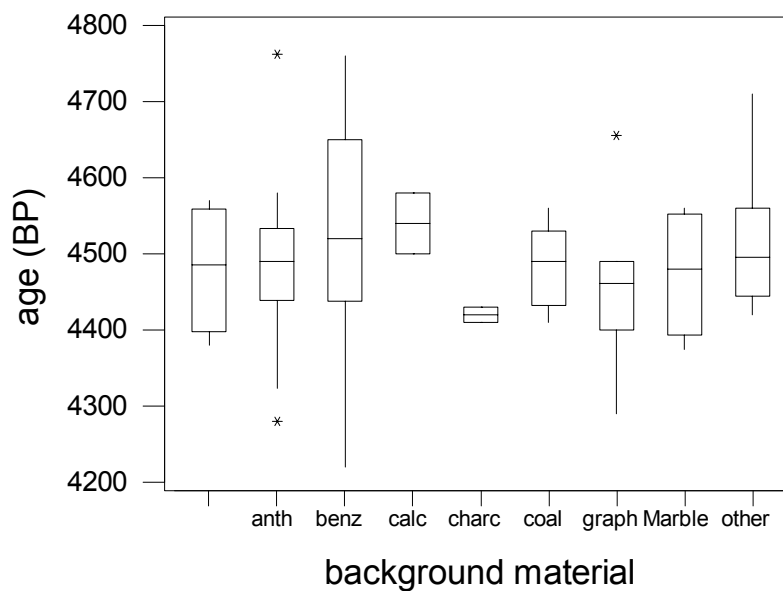


Figure 4.16a Distribution of age by background material

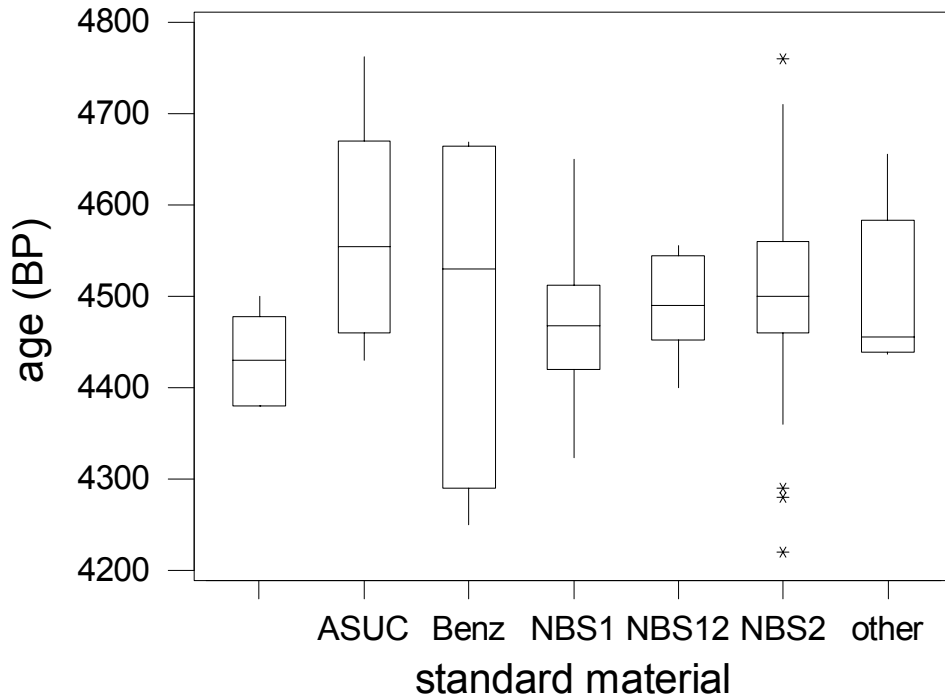


Figure 4.16b Distribution of age by modern standard

4.3.8.2 Formal analysis

Table 4.34a Analysis of variance of age by modern standard

Source	DF	SS	MS	F	P
standard	5	45428	9086	0.85	0.519
Error	74	791207	10692		
Total	79	836635			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
ASUC	6	4568.7	120.9	(-----*-----)
Benz	4	4494.8	201.3	(-----*-----)
NBS1	25	4473.5	73.2	(----*----)
NBS12	8	4490.8	53.9	(-----*-----)
NBS2	31	4500.8	116.7	(----*----)
other	6	4500.5	88.6	(-----*-----)

Pooled StDev =	103.4	4400	4480	4560	4640
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4.3.8.3 Conclusions

The background and standard materials are not statistically significant factors in explaining the observed variation.

Table 4.34b Analysis of variance of age by background material

Source	DF	SS	MS	F	P
background	7	52502	7500	0.66	<b>0.709</b>
Error	66	755202	11442		
Total	73	807704			

Individual 95% CIs For Mean Based on Pooled StDev				
Level	N	Mean	StDev	
anth	16	4483.3	111.7	(----*----)
benz	15	4523.5	156.3	(----*----)
calc	2	4540.0	56.6	(-----*-----)
charc	2	4420.0	14.1	(-----*-----)
coal	6	4485.0	53.9	(-----*-----)
graph	11	4458.6	87.5	(----*----)
Marble	6	4474.0	76.8	(-----*-----)
other	16	4513.0	82.4	(----*----)

Pooled StDev =	107.0	4320	4440	4560	4680
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**4.4 SUMMARY FINDINGS OF BACKGROUND AND STANDARD MATERIAL EFFECTS**

The information from this analysis is summarized in the table below. With few exceptions, after omission of outliers, the background and standard material is not a significant factor in explaining the observed variation. A preliminary analysis, before omission of the outliers, had however shown that standard was often a significant factor. Thus, one inference from this is that the modern standard is an important factor, but that it may be an indirect measure of the laboratory capability and experience and may be related to the presence of outliers.

Table 4.35 Summary of analysis of variance findings<sup>a</sup>

FIRI sample	Background	Standard
A (pmC)	S	S
B (pmC)	S (10%)	S
C	NS	NS
D	NS	S
E	NS	NS
F	NS	NS
G	NS	NS
H	NS	NS
I	NS	NS
J	S (10%)	NS

<sup>a</sup> S = significant; NS = not significant

**4.5 PRETREATMENT AS A FACTOR IN EXPLAINING THE VARIATION**

Pretreatment may also be a source of variation, but it should only be appropriate in a few samples, specifically the whole wood and the turbidite. The following tables enumerate the numbers of laboratories using a specific or general method for the samples. It also contains information pertinent to the issue of the explanation for outliers. Not all laboratories reported this information, so the table is incomplete.

Table 4.36 Number of laboratories (and laboratories with outliers) using specified pretreatment methods for Samples D, F, H, and I

Samples D, F, and H (Wood)			Sample I (Cellulose)		
Method	Non-outlier laboratories	All laboratories	Method	Non-outlier laboratories	All laboratories
AAA	40	41	AAA	1	1
AAAA	2	2	Missing	10	13
Cellulose	14	14	None	56	62
Missing	11	11	Other	9	11
Other	4	5	<b>Total</b>	<b>76</b>	<b>87</b>
None	4	4			
More than 1	8	8			
<b>Total</b>	<b>83</b>	<b>85</b>			

Table 4.37 Number of laboratories (and laboratories with outliers) using specified pretreatment methods for Sample C and Sample E

Sample C (Turbidite)			Sample E (Humic acid)		
Method	Non-outlier laboratories	All laboratories	Method	Non-outlier laboratories	All laboratories
Acid leaching	5	7	AAA	1	1
Missing	10	11	Missing	6	11
None	54	69	None	53	59
Other	1	2	Other	8	11
<b>Total</b>	<b>70</b>	<b>79</b>	<b>Total</b>	<b>68</b>	<b>82</b>

Table 4.38 Number of laboratories (and laboratories with outliers) using specified pretreatment methods for Samples G and J

Sample G and J (barley mash)		
Method	Non-outlier laboratories	All laboratories
AAA	5	7
Missing	10	11
None	54	69
Other	1	2
<b>Total</b>	<b>70</b>	<b>79</b>

Table 4.39 Description of pretreatment methods classed as “other” for each sample type

Sample	Wood (D, F, and H)	Turbidite (C)	Humic acid (E)	Barley mash (G and J)	Cellulose (I)
Other methods	Acid only “Routine”	AAA “Routine”	Burning AAA “Routine”	AAA “Routine”	Burning AAA “Routine”

A variety of pretreatment methods have been used, even for Sample C (turbidite) where the instructions stated that the sample should not be pretreated. AAA was the most commonly used method, with 14 laboratories extracting cellulose from the wood samples.

#### 4.5.1 Sample C (Turbidite)

Table 4.40 Descriptive statistics: age by pretreatment method

Pretreatment	N	Mean	Median	StDev
Acid leach	9	18,037	18,090	150
Missing	10	18,166	18,165	153
None	64	18,166	18,174	209
Other	1	18,359	18,359	*

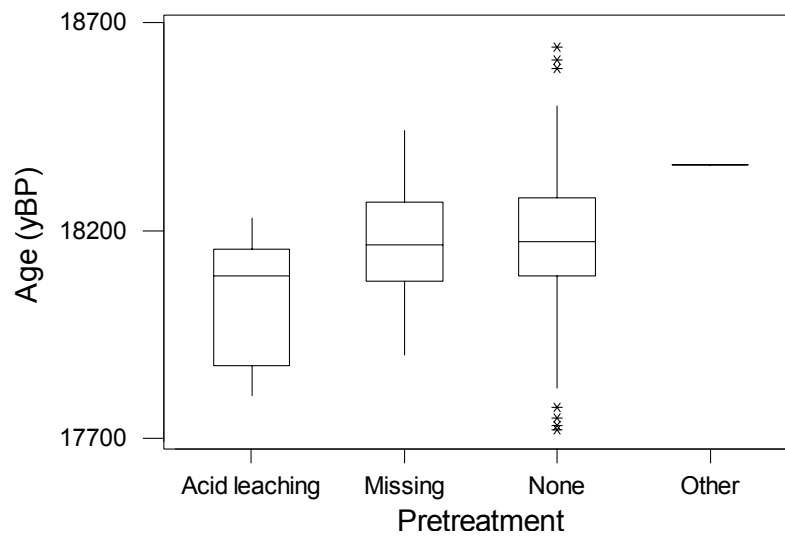


Figure 4.17 Distribution of age by pretreatment

A formal analysis to compare the results of the acid leaching and not pretreating gave a p-value of 0.04, indicating that there was a significant difference between the acid-leached and non-pretreated results. Such a difference had already been seen in the homogeneity testing and was the reason for the instruction that this sample should not be pretreated. In this case, the acid-leached results are younger.

#### 4.5.2 Sample H (Hohenheim dendro-dated wood) and Samples D and F (Belfast)

Table 4.41 Descriptive statistics: age for Sample H

Pretreatment	N	Mean	Median	StDev
AAA	50	2230.3	2230.0	86.4
AAAA	2	2207.5	2207.5	46.0
Cell. ex	17	2246.6	2248.0	77.9
Missing	11	2230.7	2209.0	66.4
None	4	2157.5	2170.0	33.0
Other	4	2215.5	2195.0	72.4



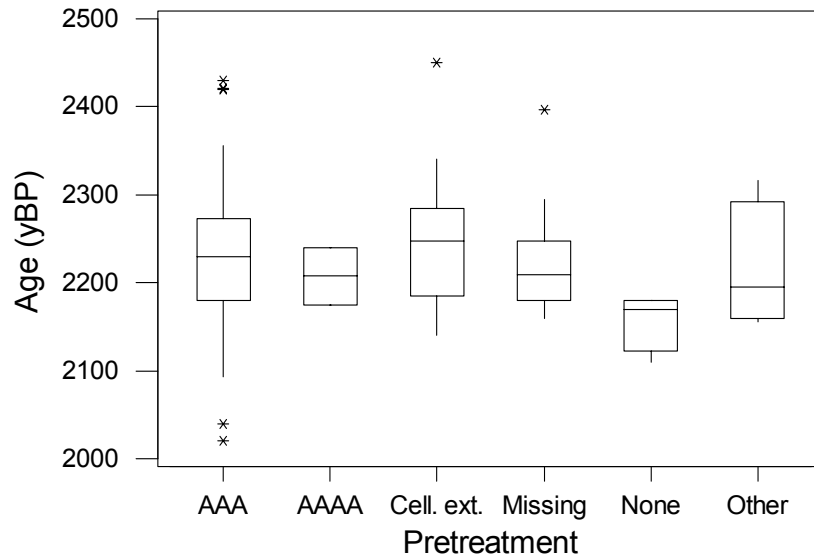


Figure 4.18 Distribution of age by pretreatment

Table 4.42 Descriptive statistics for age of Samples D and F

Pretreatment	N	Mean	Median	StDev
AAA	100	4523.3	4510.5	73.1
AAAA	4	4468.5	4472.5	16.1
Cell. ex	43	4533.1	4540.0	73.7
Missing	25	4488.1	4493.0	92.6
None	4	4468.8	4477.5	37.1
Other	9	504.1	4505.0	91.3

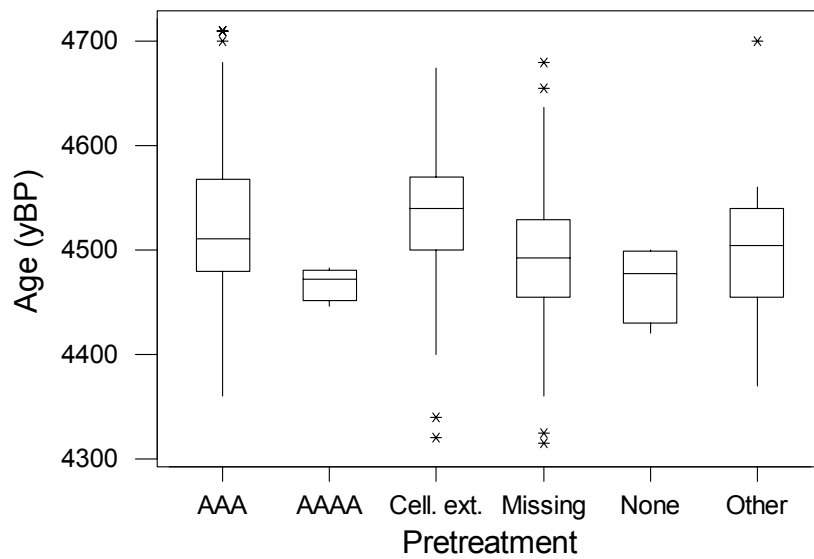


Figure 4.19 Distribution of age by pretreatment method

## 4.5.2.1 Samples D, F, and H (Wood)

Table 4.43 Descriptive statistics of deviation from consensus for all wood samples by pretreatment method

Pretreatment	N	Mean	Median	TrMean	StDev
AAA	150	-0.0586	-0.0387	-0.0531	0.6407
AAAA	6	0.2604	0.2663	0.2604	0.1952
Cell. ex	60	-0.1740	-0.1651	-0.1749	0.5594
Missing	36	0.119	0.125	0.135	0.643
None	9	0.447	0.506	0.447	0.358
Other	13	0.085	0.164	0.129	0.630

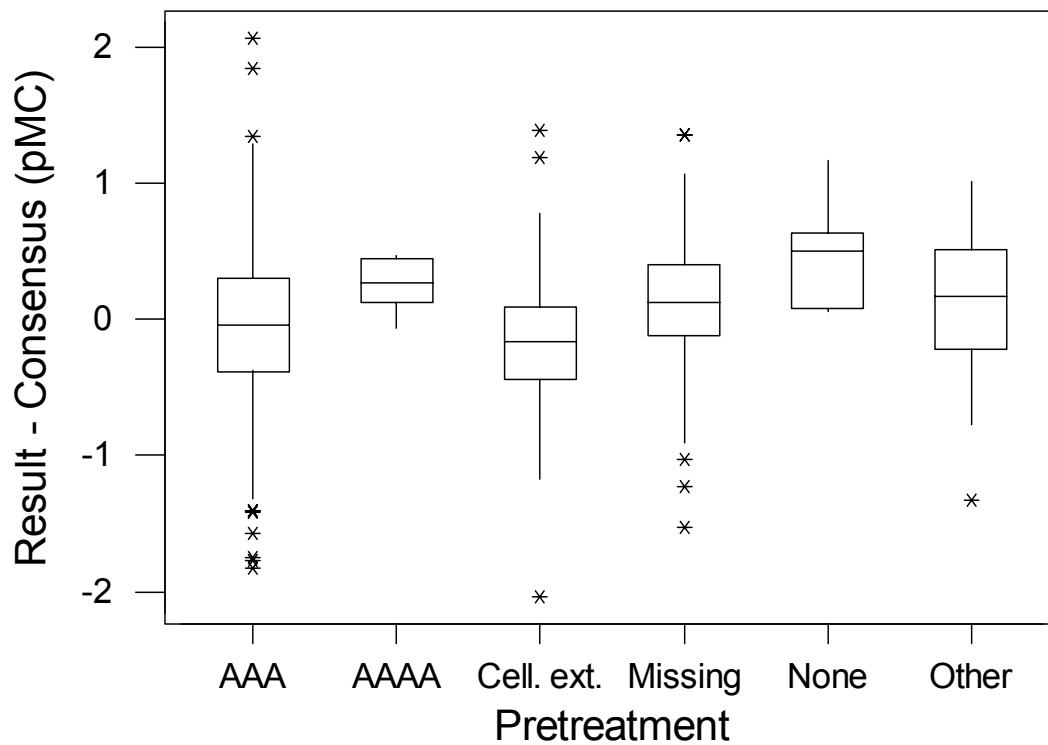


Figure 4.20 Distribution of deviation from consensus by pretreatment method

To use all the wood samples together, we have centered each sample at the consensus value (see Section 7) and investigated if method of pretreatment may be a source of variation.

4.5.2.2 Formal Analysis

Table 4.44 Analysis of variance of deviation from consensus by pretreatment method

Source	DF	SS	MS	F	P
Pretreat	5	4.894	0.979	2.62	<b>0.025</b>
Error	268	100.062	0.373		
Total	273	104.956			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
AAA	150	-0.0586	0.6407	(-***)
AAAA	6	0.2604	0.1952	(-----*-----)
Cell. ex	60	-0.1740	0.5594	(---*---)
Missing	36	0.1190	0.6426	(----*----)
None	9	0.4471	0.3582	(-----*-----)
Other	13	0.0851	0.6299	(-----*-----)

Pooled StDev = 0.6110

0.00      0.35      0.70

4.5.2.3 Conclusions

A statistically significant effect of the pretreatment method was found (p-value <0.05). There is a large amount of literature about the effects of pretreatment, and specifically for wood samples. The magnitude of the effect here, while statistically significant is, in fact, of little practical importance since any differences are very small.

4.5.3 Sample E: Humic Acid

Table 4.45 Descriptive statistics: age by pretreatment method

Pretreatment	N	Mean	Median	StDev
Missing	6	11,720	11,756	98
None	61	11,778	11,778	143
Other	9	11,742	11,720	163

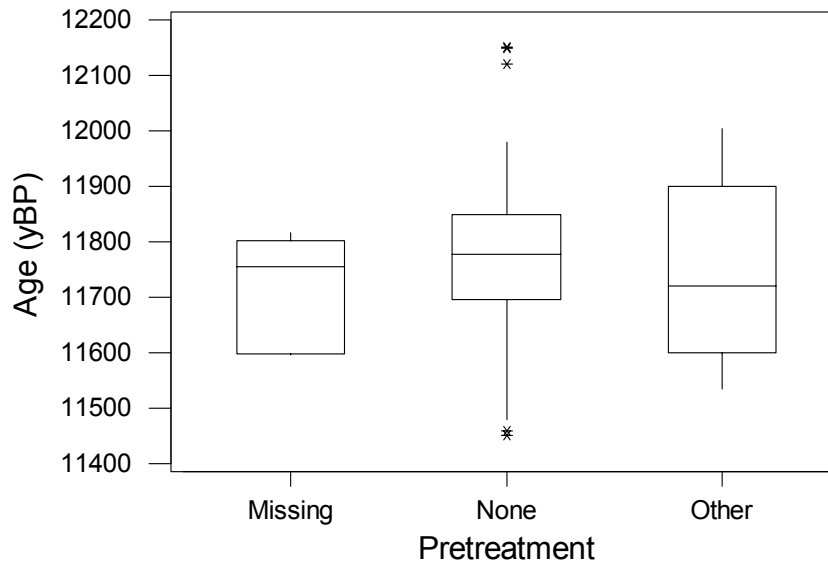


Figure 4.21 Distribution of age by pretreatment method

4.5.3.1 Formal Analysis

Table 4.46 Analysis of variance by pretreatment method

Analysis of Variance for Age					
Source	DF	SS	MS	F	P
Pretreat	2	25789	12895	0.64	<b>0.532</b>
Error	73	1479517	20267		
Total	75	1505306			

Individual 95% CIs For Mean Based on Pooled StDev					
Level	N	Mean	StDev		
Missing	6	11720	98	(------*-----)	
None	61	11778	143	(-----*-----)	
Other	9	11742	163	(------*-----)	

Pooled StDev =		142		11620	11690	11760	11830
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4.5.3.2 Conclusion

No statistically significant effect of pretreatment is observed.

4.5.4 SAMPLES G and J: Barley Mash

Table 4.47 Descriptive statistics: pMC by pretreatment

Pretreatment	N	Mean	Median	StDev
AAA	5	110.58	110.76	0.54
Missing	28	110.64	110.83	0.87
None	120	110.61	110.64	0.90
Other	18	110.52	110.50	1.24

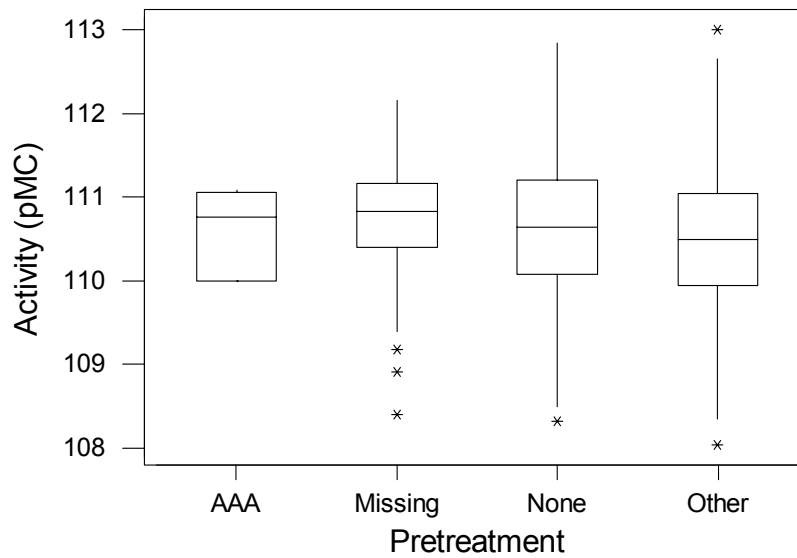


Figure 4.22 Distribution of activity by pretreatment method

4.5.4.1 Formal Analysis

Table 4.48 Analysis of variance of activity by pretreatment method

Analysis of Variance for pMC					
Source	DF	SS	MS	F	P
Pretreat	3	0.159	0.053	0.06	<b>0.980</b>
Error	167	145.479	0.871		
Total	170	145.638			

Individual 95% CIs For Mean Based on Pooled StDev					
Level	N	Mean	StDev		
AAA	5	110.576	0.540	(------*-----)	
Missing	28	110.638	0.873	(------*-----)	
None	120	110.612	0.905	(--*---)	
Other	18	110.524	1.244	(------*-----)	

Pooled StDev =	0.933	110.00	110.50	111.00	111.50
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4.5.4.2 Conclusion

No statistically significant effect of pretreatment is observed.

4.5.5 SAMPLE I: Belfast Cellulose

Table 4.49 Descriptive statistics: age by pretreatment

Pretreatment	N	Mean	Median	StDev
Missing	13	4446.0	4452.0	54.6
None	63	4505.7	4490.0	91.8
Other	10	4463.5	4435.0	171.7

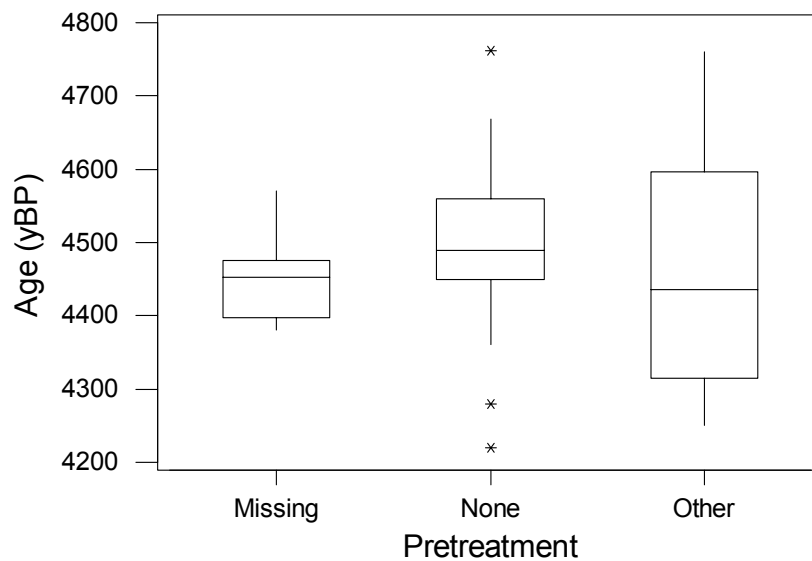


Figure 4.23 Distribution of age by pretreatment method

4.5.5.1 Formal Analysis

Table 4.50 Analysis of variance of age by pretreatment method

Analysis of Variance for Age					
Source	DF	SS	MS	F	P
Pretreat	2	47398	23699	2.39	<b>0.098</b>
Error	83	823879	9926		
Total	85	871278			
Individual 95% CIs For Mean Based on Pooled StDev					
Level	N	Mean	StDev	-----+-----+-----+-----+-----+-----+-----	
Missing	13	4446.0	54.6	(-----*-----)	
None	63	4505.7	91.8	(-----*-----)	
Other	10	4463.5	171.7	(-----*-----)	
-----+-----+-----+-----+-----+-----+-----					
Pooled StDev =		99.6		4400	4440    4480    4520

4.5.5.2 Conclusion

A statistically significant effect of pretreatment at 10% is observed, but given the insufficient information provided by the laboratories, no further conclusions can be drawn for this sample.