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GUM Analysis for SIMS Isotopic Ratios in BEPO Graphite Qualification Samples, Round 2

DC Gerlach BD Reid
PG Heasler

January 2009

Pacific Northwest
NATIONAL LABORATORY

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PG Heasler
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Abstract

This report describes GUM calculations for SIMS isotopic ratio measurements of reactor graphite samples. These isotopic ratios are used to estimate reactor burn-up, and currently consist of various ratios of U, Pu, and Boron impurities in the graphite samples. The GUM calculation is a propagation of error methodology that assigns uncertainties (in the form of standard error and confidence bound) to the final estimates.

1.0 Sample Receipt and Preparation

The three samples (27008, 30986, and 50846) were received at PNNL at the very end of April 2008. The samples were inspected and taken to our glovebox sample preparation facility shortly afterward. Each sample was lightly trimmed by machining, and a small disc for SIMS analysis, approximately 2 mm thick, was cut from each sample cylinder. Both the sample cylinder remaining (reserved for TIMS sample preparation and analyses) and the disc for SIMS analysis for each sample were cleaned using microscopic pelletized carbon dioxide dry ice. This measure has been previously found to be helpful in removing thin films of surface contamination, and is especially helpful for the disc designated for SIMS analysis because the process reveals voids and cracks exposed on the surface of the graphite, which tend to be obscured or smeared out by sawing and machining processes.

SIMS analyses commenced in early May and continued through late June. SIMS analyses also included another round of analyses of the boron carbide doped pressed graphite powder calibration standards.

The remaining cylindrical pieces were ashed and dissolved, to undergo ion exchange separations prior to TIMS analyses. Since the samples contain some ^{233}U, a measured portion of each sample solution was reserved and subjected to separation for analysis of the uranium isotope ratios without addition of the usual amount of ^{233}U tracer or ‘spike’. Based on preliminary SIMS analyses, ^{244}Pu spike was added to the remaining sample portions in amounts appropriate for each sample to minimize error and uncertainty propagation, since Pu contents have been found to vary more than a factor of 300 in previous analyses of BEP0 samples. The same amount of ^{233}U spike was added to all 3 samples, since U contents have varied a bit less. Chemical separation and purification of the U and Pu fractions was completed by late June 2008. TIMS mass spectrometric analyses of the separated U and Pu fractions will be completed and reported subsequently.

2.0 SIMS Analyses and Observations

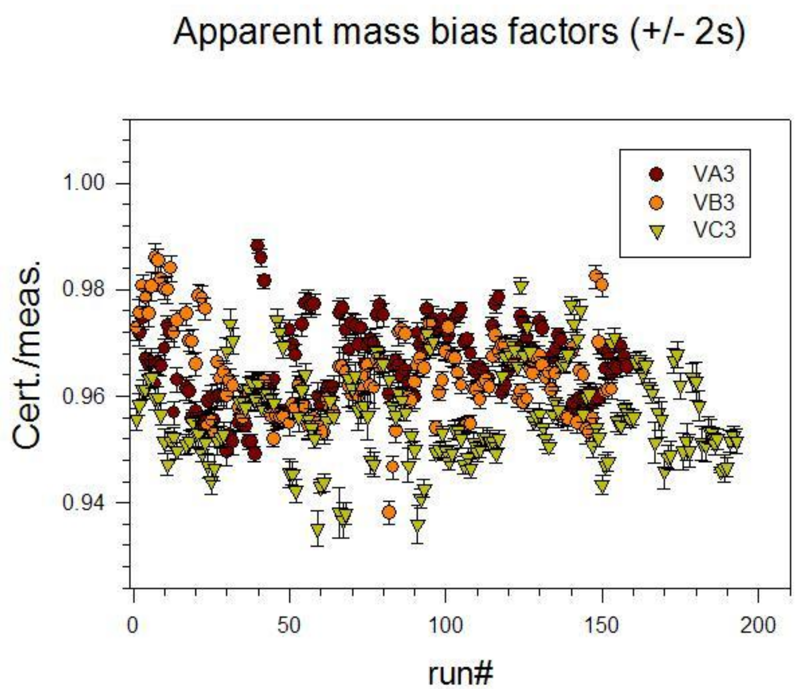
2.1 Calibration Standards

SIMS analyses were conducted first on the 3 isotopically distinct calibration standards which were prepared at NBL by mixing powdered boron carbide (B4C) with high purity graphite powder and pressing the mixture into solid cylinders or forms. Since the boron carbide grains are very high in boron, a much lower SIMS primary ion beam was used on these standards for analysis, compared to that used to analyze the very low levels of boron dispersed in real reactor graphite samples.

Seven spots with 3 or more runs in each spot were analyzed in the VA3 and VB3 standards. Fifteen spots were analyzed in VC3, because some variability was observed early on. Each spot was presputtered with a primary ion beam set to a higher current than the analysis setting, to remove possible surface or absorbed airborne contamination. The primary ion beam was adjust slightly in each new spot to yield an ion count rate for ^{11}B of 10K to 20K cps, which is just at the high range of that observable in actual graphite samples (obtained with a higher primary ion beam, of course), but well below any count rate levels where detector dead time is a significant factor during data acquisition.

The first few analyses of the VC3 piece exhibited more variability than previous results for this standard. When the sample holder and VC3 standard was removed, it was apparent that a portion of the sample surface was raised and was almost ready to spall off or exfoliate. It is undesirable for this to happen while the sample is in the SIMS sample chamber, so the loose flake was shaken loose and the VC3 disc turned over and analyses begun over again.

All results for the calibration standards are presented in the Appendix. Only results from the most recent set of analyses tabulated in the Appendix were used in calculating mass bias correction factors for this round of BEP0 qualification samples. However, results over a longer term of more than 2 years show little variation, as shown in graphical results below.

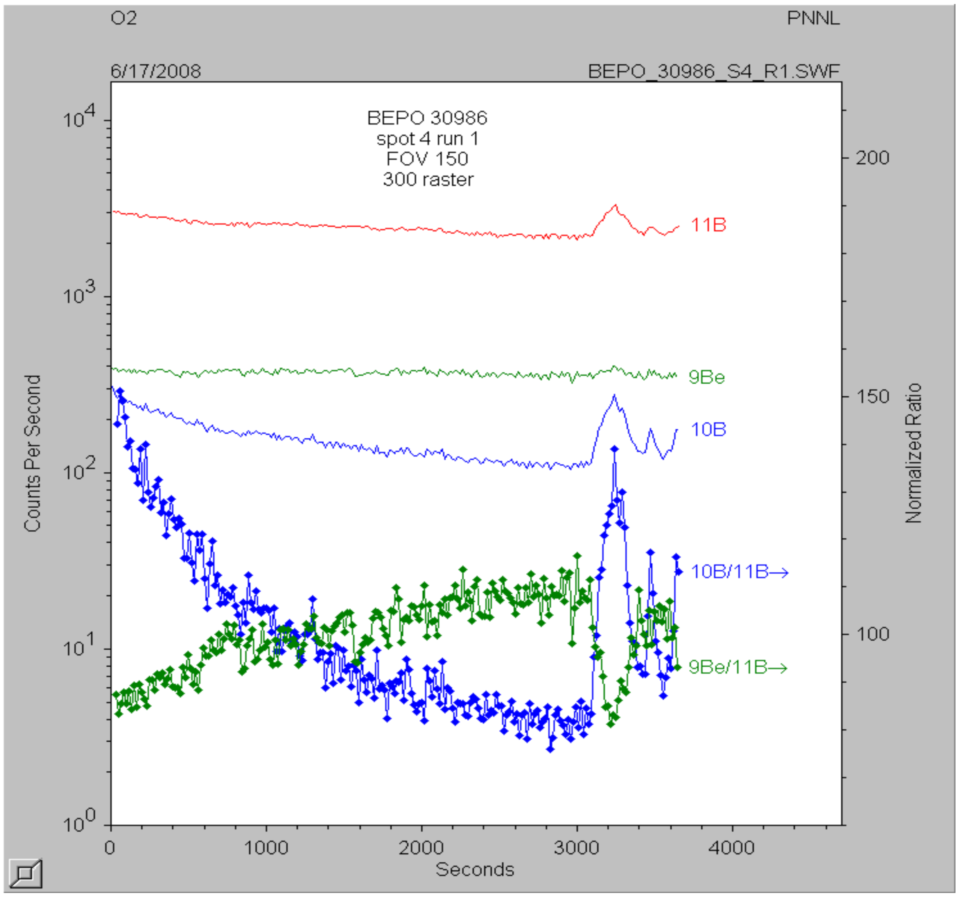


Although within analytical error, the apparent mass bias correction factor based on these results varied in this set, i.e., VA3>VB3>VC3, similar to findings in previous sets of analyses of the calibrations standards. Since all analytical settings, conditions and ion count rates were duplicated among the 3 standards, this is curious, and suggests that the certified reference values may be off slightly, or have an as yet unaccounted for mass bias effect also.

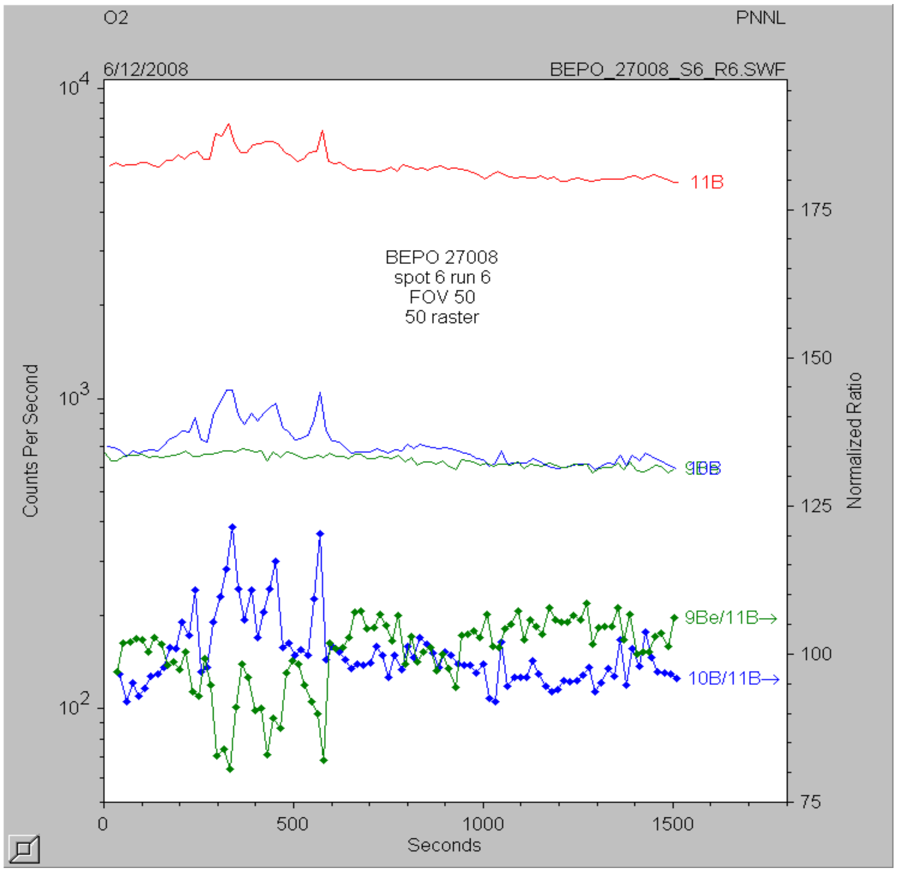
1.2 BEP0 Qualification Samples

The three graphite qualification samples in this round were analyzed twice over approximately 6 to 8 weeks' time. During each period, several spots in each sample were chosen for analysis, and several analysis runs (up to 10) in each spot. Analyses were begun in spots lacking exposed surface voids or cracks, as best as could be determined optically. Each spot was pre-sputtered with the primary ion beam set to a much higher ion current (and areal density) to remove surface contamination. This took up to 2 or

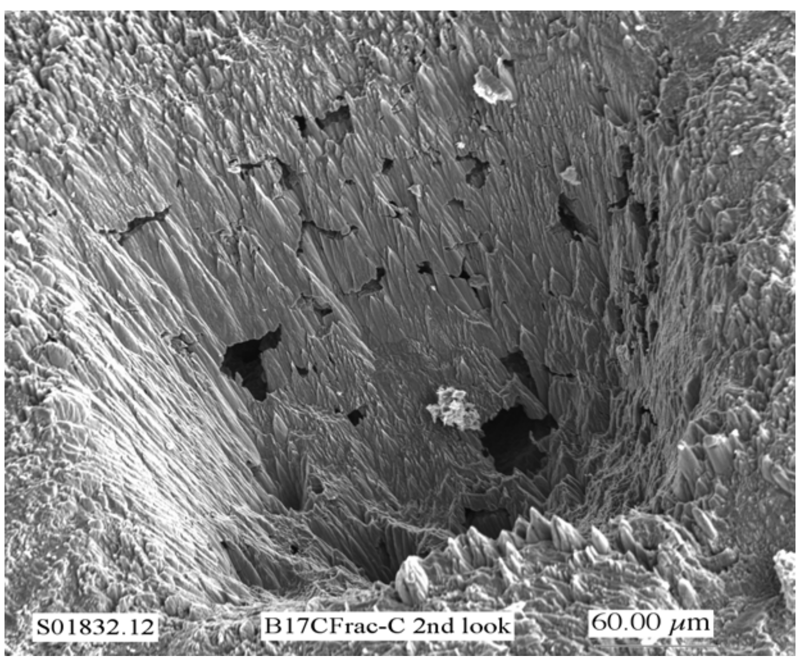
3 hours and constituted run #1 for each spot and these are not included in GUM analyses, due to the dramatically decreasing $^{10}B/^{11}B$ ratios usually observed, as shown in the example below:



The major criteria for reaching a depth unaffected by surface contamination and measuring a $^{10}B/^{11}B$ ratio representative of the sample would be the observation of flat or non-varying ratios during individual analysis runs and similar summary ratios at an asymptotic minimum value. A decreasing or changing $^{10}B/^{11}B$ ratio during or between subsequent runs of approximately 30 minutes each served as criteria for rejecting the result for compilation in GUM analysis, even if 2 or more previous runs seemed to exhibit constant and minimal $^{10}B/^{11}B$ ratios. Another criterion for rejection of an analysis run includes the observation of a dramatic departure, usually an increase in B ion count rates, suggesting a foreign inclusion or that a channel or crack open to surface contamination had been breached at depth. The example above displays two different criteria for rejection: significant changes in isotope ratios during the run and a sudden, short-lived dramatic increase in the 10B/11B ratio. In the example below, stable and minimal $^{10}B/^{11}B$ ratios were observed in runs in this spot prior to this particular run, but this analysis is disrupted by ion count excursions or increases. This occurred even in the 6th analysis run in this particular spot on sample 27008:



One possible reason for this is suggested in this SEM image below from earlier studies, showing a typical SIMS analysis crater after completion of analyses:

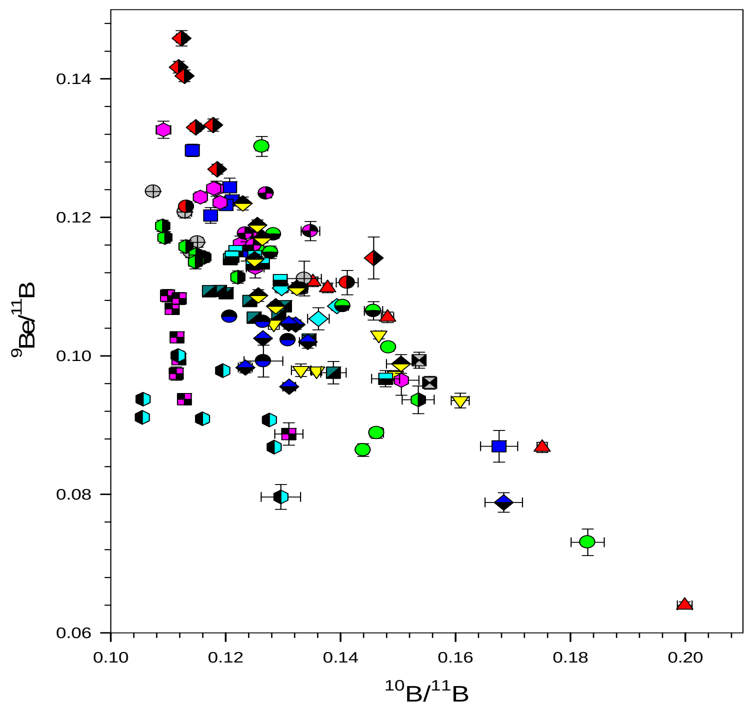


The SEM image above shows a typical crater created after several hours' analysis time on a single spot in a sample of BEP0 graphite. This also shows the apparent depth needed to reach sample material unaffected by surface contamination. Analysis spots are chosen to avoid cracks or voids visible on the surface. The accompanying widening of the crater with depth can sometimes breach channels or voids which may be open at the surface somewhere outside the chosen analysis area.

Because of these effects, some analysis runs for various spots were rejected from inclusion in GUM uncertainty analyses. In each analysis spot, the first run conducted to remove surface contamination was rejected in every case also. The list of spots and analysis runs in each spot, or the 'replicate spot design matrix' for ANOVA and GUM uncertainty analyses, does not include consecutive analyses in every analysis spot, and in some cases, only one analysis per spot was judged acceptable. For this latter reason, 10 to 20 spots per sample were analyzed, and up to 10 analyses (including run #1 for removal of surface contamination) were conducted in each spot. Individual spot analyses accepted for GUM uncertainty analysis are listed in tables in the Appendix.

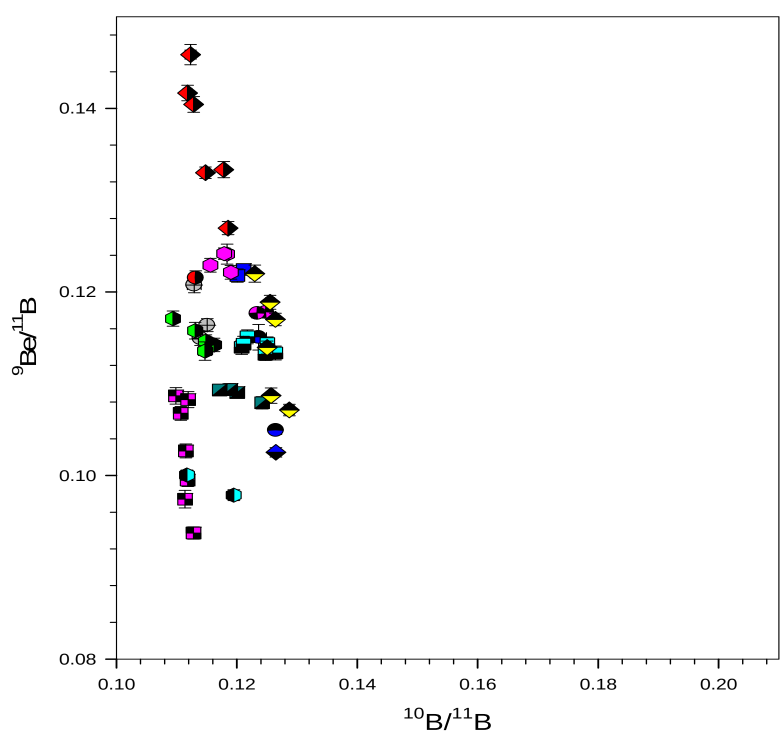
In general, $^{9}Be/^{11}B$ ratios increased in subsequent runs in each spot and did not always reach a constant plateau value. This was most apparent for sample 27008, but less so for 30986 and 50846, requiring less time for analysis of the latter two samples. Measured average values for $^{9}Be/^{11}B$ ratios in the 3 samples correlate roughly with the $^{10}B/^{11}B$ measured values, but vary approximately 30% between all analysis spots in 30986 and 50846, and 40% in sample 27008, suggesting that B concentrations are variable within each sample, but that ^{9}Be concentrations increase with $^{10}B/^{11}B$ ratios as expected. The general increase of $^{9}Be/^{11}B$ ratios with decreasing $^{10}B/^{11}B$ ratios is illustrated in the graph below for sample 27008. Each analysis spot, and all runs conducted on that same spot, including the first pre-sputtering run, are represented by colored symbols specific for the spot. The within-spot variations with depth (up to 100 microns or more) in $^{9}Be/^{11}B$ ratios tend to be less than lateral mm-scale variations between different spots.

BEP0 27008



Sample 27008 exhibited more spot to spot variability compared to the other two samples. This was especially apparent when minimum plateau values of $^{10}B/^{11}B$ values seemed to vary up to 15% between spots, even though the general criteria for acceptance was reached in an individual spot. For example, continued analysis of one spot seemed to reach an asymptotic minimum value of 0.131 for $^{10}B/^{11}B$ and another a minimum value as low as 0.113. The graph below presents the subset of analytical results (from the preceding graph) for sample 27008 which met criteria for GUM uncertainty analysis.

BEP0 27008



The $^{10}B/^{11}B$ ratios which were accepted for analysis vary more in this sample than in the other two samples and the $^{9}Be/^{11}B$ ratios also show slightly more variation. This variation suggests a small amount of internal contamination by boron, but it is not certain that this may be from one of the BEP0 blocks affected by rain water. Sample 27008 had the lowest range of $^{9}Be/^{11}B$ ratios, and thus, probably the lowest ^{9}Be contents generated by neutron fluence exposure.

The variation for the $^{9}Be/^{11}B$ ratios in all 3 samples is also significant, possibly reflecting microscopic variations in local B content, rather than microscopic variations in either initial or fluence-generated ^{9}Be contents. Because of this, $^{10}B/^{11}B$ ratios were corrected individually using the $^{9}Be/^{11}B$ ratio determined in the same run, rather than an average $^{9}Be/^{11}B$ ratio determined for all analysis spots for the sample, or for individual analysis spots. The maximum amount of the correction for ^{10}Be based on the measured $^{9}Be/^{11}B$ ratio varied from approximately 2.9% for sample 27008, to 4.3 % for sample 50846, to 13% for sample 30986, which is not surprising since it had the lowest $^{10}B/^{11}B$ ratio.

3.0 ANOVA and GUM Evaluations

This section presents GUM tables for each qualification sample, as well as ANOVA results for the SIMs M9/M11 and M10/M11 ratios. The ANOVA's summarize the replicate and spot variability in these ratios. The Summary table presented below contains our B10/B11 estimates with the requested GUM uncertainty statistics.

Summary Table for $^{10}B/^{11}B$ Estimates

	$^{10}B/^{11}B$ est.	% RE	DOF	K.factor	Est+CB 95% CB
BEP0 27008a	0.11260	1.2410	15.970	2.120	0.002961
BEP0 30986a	0.02787	1.6790	12.630	2.167	0.001014
BEP0 50846a	0.07086	0.4967	8.731	2.273	0.000800

The variables present in the GUM Tables represent the constants and data required to calculate the $^{10}B/^{11}B$ ratio. They are defined as follows:

r.m9.m11: The M9/M10 ratios produced by SIMS analyses
r.m10.m11: The M10/M11 ratios produced by SIMS analyses
mb.b10.b11: Mass Bias correction factor produced from SIMS analyses of calibration standards
r.be10.be9: The Be10/Be9 ratio calculated from reactor physics code.
Cor: The correlation between M9/M11 and M10/M11 measurements. Correlations occur because of the common denominator (and other factors).

3.1 Sample BEP0 27008:

ANOVA Results

Replicate X Spot Design Matrix

run/spot	4	6	7	10	11	12	13	15	16	17	18	19	20
2	1	1	NA	1	NA	NA	1	1	NA	NA	NA	1	NA
3	1	1	1	1	NA	1	1	1	1	1	NA	1	NA
4	1	1	1	NA	1	1	1	1	1	1	1	1	NA
5	1	NA	1	NA	1	NA	1	1	NA	1	NA	1	1
6	1	NA	1	NA	NA	NA	1	1	1	1	NA	1	1
7	NA	NA	NA	NA	NA	NA	1	1	1	1	NA	1	NA
8	NA	1	NA	1	NA	NA	NA	NA	1	NA	NA	1	NA
9	NA	NA	NA	1	NA	NA	NA	NA	NA	NA	NA	NA	NA

MANOVA Estimates and Variance Components (in terms of percent relative error)

	est	RE.est	RE.spot	RE.rep	RE.cycle	RE.pois
M9.11	0.115	2.49	8.74	3.65	0.348	NA
M10.11	0.119	1.19	4.16	1.95	0.316	NA

Estimate Correlation Matrix

	M9.11	M10.11
M9.11	1	-0.131
M10.11	-0.131	1

Error Propagation and Estimates

	Est	RE	dof	Sensit	Index
r.m9.m11	0.115	2.49	14.5	-0.0214	0.193
r.m10.m11	0.119	1.19	14.9	0.966	96.5
mb.b10.b11	0.966	0.18	5.28	0.117	2.1
r.be10.be9	0.0222	1	Inf	-0.111	0.031
Cor	NA	NA	NA	NA	1.13
Result	0.113	1.24	16	NA	NA

3.2 Sample BEP0 30986

ANOVA Results

Replicate X Spot Design Matrix

run/spot	1	2	3	5	7	8	9	10
3	1	1	1	NA	1	1	1	1
4	1	1	1	NA	1	1	1	1
5	NA	1	1	NA	1	1	1	1
6	NA	1	1	1	1	1	NA	1
7	NA	1	1	1	1	1	NA	1
8	NA	NA	NA	NA	1	NA	NA	NA

MANOVA Estimates and Variance Components (in terms of percent relative error)

	est	RE.est	RE.spot	RE.rep	RE.cycle	RE.pois
M9.11	0.164	1.44	2.6	6.17	0.285	NA
M10.11	0.0325	1.45	3.75	3.14	0.363	NA

Estimate Correlation Matrix

	M9.11	M10.11
M9.11	1	-0.112
M10.11	-0.112	1

Error Propagation and Estimates

	Est	RE	dof	Sensit	Index
r.m9.m11	0.164	1.44	32.9	-0.0214	1.17
r.m10.m11	0.0325	1.45	11.3	0.966	94.8
mb.b10.b11	0.966	0.18	5.28	0.0289	1.15
r.be10.be9	0.0222	1	Inf	-0.159	0.567
Cor	NA	NA	NA	NA	2.35
Result	0.0279	1.68	12.6	NA	NA

3.3 Sample BEP0 50846

ANOVA Results

Replicate X Spot Design Matrix

run/spot	1	2	3	4	5	6	7	8	9	10
2	1	NA	NA	1	1	1	1	1	1	NA
3	1	1	1	1	1	1	1	1	1	1
4	1	1	1	1	1	1	1	1	1	1
5	NA	1	1	1	1	1	1	1	1	1
6	NA	1	1	1	1	1	NA	1	1	1
7	NA	NA	NA	NA	NA	NA	NA	NA	NA	1
8	NA	NA	NA	NA	NA	NA	NA	NA	NA	1

MANOVA Estimates and Variance Components (in terms of percent relative error)

	est	RE.est	RE.spot	RE.rep	RE.cycle	RE.pois
M9.11	0.125	3.22	10.1	2.35	0.325	NA
M10.11	0.0761	0.504	1.55	0.761	0.278	NA

Estimate Correlation Matrix

	M9.11	M10.11
M9.11	1	0.594
M10.11	0.594	1

Error Propagation and Estimates

	Est	RE	dof	Sensit	Index
r.m9.m11	0.125	3.22	10.2	-0.0214	6
r.m10.m11	0.0761	0.504	11.1	0.966	111
mb.b10.b11	0.966	0.18	5.28	0.0734	13.1
r.be10.be9	0.0222	1	Inf	-0.121	0.579
Cor	NA	NA	NA	NA	-30.6
Result	0.0709	0.497	8.73	NA	NA

Calibration Standards (Mass Bias Calibration) ANOVA

Mass Bias Estimate and Standard Error

	Est	%RE	N-levels	DOF
Mass Bias	0.9659	0.1797	NA	5.276

Standard Deviations due to:

block	0.0023	82.92	3	2
spot in block	0.0051	16.9	29	26
Residuals	0.0039	9.466	85	56

Appendix: Tables of Individual SIMS Analyses Accepted for GUM Evaluations

BEP0 27008:

spot	run	$^{10}B/^{11}B$	2S	$^{9}Be/^{11}B$	2S	$^{10}B/^{11}B$, corrected for ^{10}Be
4	2	0.1207	0.00113	0.1244	0.00130	0.1179
4	3	0.1142	0.00073	0.1297	0.00088	0.1113
4	4	0.1174	0.00095	0.1203	0.00113	0.1147
4	5	0.1211	0.00072	0.1225	0.00061	0.1184
4	6	0.1201	0.00080	0.1218	0.00073	0.1174
6	2	0.1184	0.00095	0.1241	0.00108	0.1156
6	3	0.1190	0.00070	0.1222	0.00076	0.1163
6	4	0.1156	0.00066	0.1229	0.00075	0.1129
6	8	0.1179	0.00063	0.1242	0.00062	0.1151
7	3	0.1129	0.00115	0.1208	0.00085	0.1102
7	4	0.1151	0.00080	0.1164	0.00071	0.1125
7	5	0.1140	0.00069	0.1149	0.00076	0.1114
7	6	0.1132	0.00079	0.1215	0.00077	0.1105
10	2	0.1201	0.00061	0.1091	0.00045	0.1176
10	3	0.1242	0.00070	0.1079	0.00067	0.1218
10	8	0.1189	0.00062	0.1094	0.00065	0.1165
10	9	0.1172	0.00051	0.1093	0.00055	0.1147
11	4	0.1265	0.00049	0.1049	0.00045	0.1242
11	5	0.1236	0.00130	0.1151	0.00139	0.1211
12	3	0.1249	0.00052	0.1178	0.00060	0.1222
12	4	0.1234	0.00053	0.1177	0.00050	0.1208
13	2	0.1208	0.00078	0.1140	0.00076	0.1182
13	3	0.1218	0.00060	0.1151	0.00076	0.1192
13	4	0.1264	0.00061	0.1134	0.00076	0.1239
13	5	0.1211	0.00077	0.1143	0.00086	0.1186
13	6	0.1251	0.00075	0.1144	0.00072	0.1225
13	7	0.1247	0.00077	0.1132	0.00065	0.1222
15	2	0.1123	0.00089	0.1459	0.00111	0.1091
15	3	0.1118	0.00074	0.1417	0.00085	0.1087
15	4	0.1128	0.00072	0.1404	0.00085	0.1097
15	5	0.1178	0.00069	0.1333	0.00089	0.1149
15	6	0.1148	0.00051	0.1330	0.00062	0.1118
15	7	0.1185	0.00067	0.1270	0.00072	0.1157
16	3	0.1131	0.00079	0.1158	0.00090	0.1105
16	4	0.1094	0.00077	0.1171	0.00083	0.1068
16	6	0.1162	0.00065	0.1142	0.00073	0.1137
16	7	0.1149	0.00077	0.1146	0.00069	0.1123
16	8	0.1147	0.00110	0.1136	0.00100	0.1122
17	3	0.1264	0.00059	0.1170	0.00068	0.1238
17	4	0.1230	0.00074	0.1220	0.00093	0.1203
17	5	0.1255	0.00073	0.1189	0.00076	0.1229
17	6	0.1250	0.00073	0.1140	0.00071	0.1225
17	7	0.1257	0.00067	0.1087	0.00084	0.1233
18	4	0.1265	0.00056	0.1025	0.00051	0.1242
19	2	0.1114	0.00088	0.0974	0.00096	0.1092
19	3	0.1128	0.00055	0.0937	0.00067	0.1107
19	4	0.1118	0.00055	0.0995	0.00070	0.1096
19	5	0.1115	0.00059	0.1027	0.00076	0.1092
19	6	0.1107	0.00069	0.1068	0.00075	0.1083
19	7	0.1099	0.00052	0.1087	0.00090	0.1075
19	8	0.1119	0.00073	0.1083	0.00087	0.1095
20	5	0.1117	0.00100	0.1001	0.00056	0.1095
20	6	0.1195	0.00069	0.0979	0.00059	0.1173

BEP0 30986:

spot	run	$^{10}B/^{11}B$	2S	$^{9}Be/^{11}B$	2S	$^{10}B/^{11}B$, corrected for ^{10}Be
1	3	0.0332	0.00018	0.1666	0.00060	0.0295
1	4	0.0344	0.00021	0.1685	0.00070	0.0307
2	3	0.0332	0.00028	0.1556	0.00098	0.0297
2	4	0.0340	0.00026	0.1618	0.00069	0.0304
2	5	0.0322	0.00018	0.1601	0.00119	0.0287
2	6	0.0325	0.00025	0.1761	0.00143	0.0285
2	7	0.0335	0.00027	0.2004	0.00154	0.0290
3	3	0.0301	0.00027	0.1583	0.00102	0.0266
3	4	0.0317	0.00023	0.1556	0.00102	0.0282
3	5	0.0315	0.00019	0.1636	0.00092	0.0279
3	6	0.0317	0.00026	0.1656	0.00106	0.0280
3	7	0.0318	0.00026	0.1788	0.00147	0.0278
5	6	0.0337	0.00020	0.1466	0.00066	0.0305
5	7	0.0334	0.00022	0.1438	0.00061	0.0302
7	3	0.0311	0.00028	0.1703	0.00092	0.0274
7	4	0.0299	0.00022	0.1687	0.00071	0.0262
7	5	0.0299	0.00023	0.1739	0.00086	0.0261
7	6	0.0304	0.00022	0.1777	0.00073	0.0265
7	7	0.0307	0.00023	0.1766	0.00084	0.0268
7	8	0.0338	0.00022	0.1618	0.00084	0.0302
8	3	0.0339	0.00020	0.1586	0.00076	0.0304
8	4	0.0349	0.00030	0.1617	0.00084	0.0313
8	5	0.0348	0.00022	0.1840	0.00100	0.0307
8	6	0.0345	0.00020	0.1612	0.00058	0.0309
8	7	0.0336	0.00022	0.1538	0.00075	0.0302
9	3	0.0347	0.00020	0.1621	0.00112	0.0311
9	4	0.0310	0.00028	0.1628	0.00101	0.0273
9	5	0.0308	0.00024	0.1552	0.00088	0.0274
10	3	0.0314	0.00025	0.1601	0.00080	0.0278
10	4	0.0311	0.00022	0.1622	0.00080	0.0275
10	5	0.0310	0.00025	0.1638	0.00092	0.0274
10	6	0.0311	0.00022	0.1622	0.00080	0.0275
10	7	0.0310	0.00025	0.1638	0.00092	0.0274

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spot	run	$^{10}B/^{11}B$	2S	$^{9}Be/^{11}B$	2S	$^{10}B/^{11}B$, corrected for ^{10}Be
1	2	0.0773	0.00043	0.1429	0.00098	0.0741
1	3	0.0785	0.00049	0.1408	0.00103	0.0753
1	4	0.0780	0.00048	0.1398	0.00089	0.0748
2	3	0.0784	0.00045	0.1326	0.00080	0.0755
2	4	0.0787	0.00058	0.1301	0.00107	0.0758
2	5	0.0780	0.00052	0.1282	0.00103	0.0751
2	6	0.0776	0.00052	0.1301	0.00093	0.0747
3	3	0.0756	0.00041	0.1322	0.00070	0.0727
3	4	0.0757	0.00038	0.1316	0.00069	0.0728
3	5	0.0747	0.00041	0.1344	0.00074	0.0717
3	6	0.0750	0.00030	0.1346	0.00060	0.0720
4	2	0.0776	0.00036	0.1265	0.00061	0.0748
4	3	0.0754	0.00041	0.1237	0.00075	0.0726
4	4	0.0763	0.00049	0.1287	0.00088	0.0735
4	5	0.0756	0.00048	0.1253	0.00090	0.0728
4	6	0.0760	0.00045	0.1238	0.00087	0.0732
5	2	0.0762	0.00030	0.1351	0.00048	0.0732
5	3	0.0751	0.00038	0.1396	0.00079	0.0720
5	4	0.0756	0.00036	0.1425	0.00070	0.0725
5	5	0.0757	0.00037	0.1462	0.00073	0.0725
5	6	0.0766	0.00053	0.1469	0.00114	0.0734
6	2	0.0768	0.00033	0.1246	0.00064	0.0740
6	3	0.0762	0.00042	0.1223	0.00085	0.0735
6	4	0.0762	0.00041	0.1233	0.00077	0.0735
6	5	0.0762	0.00039	0.1266	0.00077	0.0734
6	6	0.0772	0.00045	0.1281	0.00076	0.0743
7	2	0.0751	0.00043	0.1007	0.00084	0.0729
7	3	0.0745	0.00044	0.1006	0.00075	0.0723
7	4	0.0748	0.00040	0.1053	0.00094	0.0725
7	5	0.0749	0.00050	0.1055	0.00061	0.0726
8	2	0.0752	0.00040	0.1153	0.00078	0.0726
8	3	0.0756	0.00036	0.1111	0.00094	0.0731
8	4	0.0743	0.00038	0.1054	0.00069	0.0719
8	5	0.0751	0.00036	0.1097	0.00067	0.0726
8	6	0.0758	0.00040	0.1125	0.00077	0.0733
9	2	0.0750	0.00038	0.1141	0.00061	0.0725
9	3	0.0744	0.00034	0.1122	0.00066	0.0719
9	4	0.0740	0.00037	0.1109	0.00065	0.0715
9	5	0.0749	0.00039	0.1155	0.00073	0.0723
9	6	0.0751	0.00048	0.1164	0.00094	0.0725
10	3	0.0778	0.00042	0.1180	0.00088	0.0751
10	4	0.0767	0.00045	0.1253	0.00079	0.0739
10	5	0.0764	0.00037	0.1264	0.00085	0.0736
10	6	0.0766	0.00044	0.1254	0.00079	0.0738
10	7	0.0759	0.00041	0.1228	0.00089	0.0732
10	8	0.0772	0.00044	0.1270	0.00093	0.0744

Appendix: Results for SIMS Calibration Standards

VA3

spot	run	$^{10}B/^{11}B$	2S
1	1	0.2292	0.00020
1	2	0.2288	0.00020
1	3	0.2282	0.00016
2	1	0.2276	0.00020
2	2	0.2273	0.00019
2	3	0.2271	0.00021
3	1	0.2258	0.00020
3	2	0.2261	0.00017
3	3	0.2267	0.00018
4	1	0.2272	0.00018
4	2	0.2273	0.00020
4	3	0.2276	0.00020
5	1	0.2260	0.00020
5	2	0.2263	0.00018
5	3	0.2221	0.00315
6	1	0.2280	0.00021
6	2	0.2282	0.00021
6	3	0.2276	0.00019
7	1	0.2272	0.00018
7	2	0.2266	0.00017
7	3	0.2268	0.00019
	average	0.2270	0.00144
	m.b.	0.9695	

VB3

spot	run	$^{10}B/^{11}B$	2S
1	1	0.1298	0.00011
1	2	0.1298	0.00013
1	3	0.1299	0.00012
2	1	0.1292	0.00011
2	2	0.1286	0.00013
2	3	0.1291	0.00012
3	1	0.1289	0.00012
3	2	0.1289	0.00010
3	3	0.1291	0.00009
4	1	0.1293	0.00012
4	2	0.1292	0.00012
4	3	0.1302	0.00010
5	1	0.1301	0.00009
5	2	0.1303	0.00012
5	3	0.1302	0.00012
6	1	0.1295	0.00010
6	2	0.1296	0.00010
6	3	0.1292	0.00010
7	1	0.1292	0.00013
7	2	0.1294	0.00011
7	3	0.1297	0.00011
	average	0.1295	0.00049
	m.b.	0.9651	

VC3

spot	run	$^{10}B/^{11}B$	2S
1	1	0.0893	0.00008
1	2	0.0890	0.00006
1	3	0.0889	0.00009
2	1	0.0887	0.00007
2	2	0.0889	0.00008
2	3	0.0891	0.00008
3	1	0.0878	0.00007
3	2	0.0889	0.00008
3	3	0.0885	0.00008
4	1	0.0889	0.00009
4	2	0.0901	0.00008
4	3	0.0900	0.00006
5	1	0.0900	0.00010
5	2	0.0902	0.00008
5	3	0.0900	0.00008
6	1	0.0897	0.00009
6	2	0.0899	0.00008
6	3	0.0891	0.00007
7	1	0.0891	0.00008
7	2	0.0889	0.00006
7	3	0.0881	0.00008
8	1	0.0884	0.00008
8	2	0.0887	0.00008
8	3	0.0882	0.00009
9	1	0.0900	0.00007
9	2	0.0897	0.00009
9	3	0.0896	0.00009
10	1	0.0902	0.00008
10	2	0.0904	0.00008
10	3	0.0887	0.00008
11	1	0.0886	0.00009
12	1	0.0901	0.00009
12	2	0.0902	0.00008
12	3	0.0899	0.00008
13	1	0.0899	0.00008
13	2	0.0901	0.00007
13	3	0.0900	0.00007
14	1	0.0891	0.00009
14	2	0.0890	0.00008
14	3	0.0893	0.00009
15	1	0.0891	0.00007
15	2	0.0895	0.00008
15	3	0.0896	0.00008
	average	0.0893	0.00067
	m.b.	0.9635	

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