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Title of article Late Quaternary deposition of ice-rafted sand in the sub-
polar North Atlantic (lat 40 to 65 N)

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Sample Analysis

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SAMPLE ANALYSIS

The objective of measuring a demonstrably ice-rafted component (non-carbonate sand) required an initial decision on the technique appropriate for determining coarse fraction non-carbonate percentages. I chose the quick and inexpensive insoluble residue technique, except that the samples were first wet-sieved through a 62- μ m screen to isolate the coarse fraction. No other carbonate technique can be run directly on a sieve-separated coarse fraction.

Procedure

As standard procedure, 5g samples (3-10g range) were taken from the core, dried, weighed, and wet-sieved through a 62- μ m screen without chemical ultrasonic treatment. The coarse fraction was dried and weighed to determine the percent coarse fraction. Gravel (larger than 2 mm in diameter) was eliminated from the analysis.

The coarse fraction was then split to obtain samples for other purposes and the insoluble residue analysis run on a coarse fraction sample weighing an average of 0.5g (0.1-1.0g range). The samples were ground with mortar and pestle, weighed, treated with acid on filter paper, dried, and weighed in combination with the filter paper. The percentage of non-carbonate within the sand fraction was calculated from the remaining residue weight. This percentage, multiplied by the percent coarse fraction, gives percent coarse fraction non-carbonate (CFNC) of the total sample.

For 4 cores in this study, percent CFNC values were determined in other laboratories using the RAFT procedure described by Kellogg (1975). The determinations are indirect and come as a "residual" value after analyses of (1) total carbonate percentage, (2) percent coarse fraction, and (3) percent carbonate within the fine fraction. RAFT data from Kellogg (1975) were used in part for core V28-14. RAFT data from McIntyre and others (in preparation) were used for half of the sample determinations in core V30-96 and all determinations in V30-97 and V30-100.

Precision

Siesser and Rogers (1971) found a 4.9% mean coefficient of variation on replicate tests of the insoluble residue technique run on total samples averaging more than 2g. This study showed considerably larger errors for smaller samples run only on coarse fraction material. Replicate tests were run of 8 samples four times each and 12 samples three times each. The coefficient of variation averaged $\pm 10.5\%$ for all samples, but the error was larger for samples with smaller residue weights. Coarse fractions composed of 75% or less carbonate gave a mean coefficient of deviation of $\pm 4.9\%$, identical to that in Siesser and Rogers (1971). By comparison, samples with more than 75% carbonate gave a mean coefficient of deviation of $\pm 21.0\%$.

The absolute analytical error was apparently constant,

but became proportionally larger for the smaller residue weights. Most of this error apparently occurred during weighing of the insoluble residue and was caused by moisture uptake in the filter paper despite precautions. By weighing numbered filter papers in one order after drying and then reversing the sequence on a second run, a mean difference in weight for each filter paper of 0.10g was found. First-weighed papers were lighter and last-weighed papers heavier, indicating progressive uptake of moisture after exposure to moist laboratory air. For the sizes of split coarse fraction being analyzed, this error explains much of the total observed error.

It was also determined that insoluble residue carbonate determinations on extremely small samples (0.1-0.3g) gave roughly twice the coefficient of deviation of samples averaging 0.5-1.0g. Since this larger error occurs for the most part in samples with coarse fraction percentages that were small to begin with (primarily the K714 cores and RC9-225), the weighing error relative to total initial sediment weight is not larger.

The mean error determined for wet-sieving the coarse fraction was $\pm 4\%$, but was smaller ($\pm 2\%$) for the above-mentioned cores with low coarse fraction values. This gives a total error of $\pm 11\%$ (combined errors from coarse fraction and carbonate determinations).

The precision of the RAFT technique is not known, but Siesser and Rogers (1971) give a mean coefficient of deviation of 4.7% for the gasometric technique. Since in the RAFT procedure each analysis requires two gasometric determinations

and one wet-sieving, the total error must be about 7.3%, considerably less than the insoluble residue technique run only on the coarse fraction.

Accuracy

I have not specifically tested for accuracy. Siesser and Rogers (1971) infer that dissolution of acid-soluble non-carbonate material may be a major source of error. This would cause an under-estimation of the ice-rafted input, as would dissolution of carbonate-rich ice-rafted components (e.g., limestone). I infer that both the insoluble residue and gasometric techniques probably give a minimum (underestimated) value for the sand-sized ice-rafted component. For the most part, the accuracy errors are probably relatively constant from sample to sample and core to core, since no substantial regional or temporal variations in sand mineralogy (and hence percent soluble ice-rafted material) have been noted at these latitudes (Bramlette and Bradley, 1941).

There is evidence in core V30-96 of significantly different results from the insoluble residue technique than from the RAFT procedure. The insoluble residue values are consistently lower than the gasometric determinations, although all values average in the 0-5% range. I attribute this difference to problems already discussed in weighing very small insoluble residues on filter paper. There is little effect on the maps since the values are so small. In cores with larger percentages of CFNC like V28-14, the results of the two techniques are highly comparable.

Errors in Mapped Values

The large mean precision error in each analysis ($\pm 11\%$) is not directly propagated into the maps of absolute input that follow later. Because my technique integrates many separate sample analyses into each mapped input number, errors of overestimate on some samples will be to some extent balanced by underestimates on others.

If the many analyzed samples integrated through each time interval had identical percentages of coarse fraction non-carbonate, the error propagated to the maps would decrease as the square root of the number of samples analyzed (\sqrt{N}), as is the case for repeated analyses of one sample. In fact, percentages in many of the intervals mapped do approach a fairly constant value. The number of samples integrated into each map value ranges from 3 to 21 and averages 6.7 (Table 3). Under the \sqrt{N} assumption, the errors would then be reduced by a factor ranging from 1.7 to 4.7 and averaging 2.6. The $\pm 11\%$ error on each sample analysis would thus be reduced to an average of roughly $\pm 4\%$ for each mapped value.

Unfortunately, there is no exact way to quantify the error reduction due to combining many analyses.

Stratigraphic Control Levels for All Cores. Depths of
Control Levels in Centimeters.

Core	Zone 1 (Ash) 9.3 ky	Zone 2 (Ash) 65.0 ky	Barbados 1 (Faunal-lithologic) 82.0 ky	Barbados 3 (Faunal-lithologic) 125.0 ky
K708-1	62	389	460	840
K708-4	34	189	235	325
K708-6	28	208	290	420
K708-7	28	175	215	290
K708-8	45	286	365	520
K714-3	53	196	250	655
K714-14	(105) ^a	363	450	700
K714-15	(370) ^a	641	800	1140
LK-4	30	195	---	---
LK-6	33	209	---	---
LK-7	9	143	---	---
RC9-225	70	493	600	900
RE5-34	(30) ^b	---	160	250
RE5-36	(55) ^b	---	289	410
V23-23	38	337	415	550
V23-42	36	248	290	445
V23-17	20	122	160	310
V27-19	16	137	181	310
V27-20	48	256	330	485
V27-110	(30) ^a	---	115	145

Stratigraphic Control Levels for All Cores. Depths of
Control Levels in Centimeters. (continued)

Core	Zone 1 (Ash) 9.3 ky	Zone 2 (Ash) 65.0 ky	Barbados 1 (Faunal-lithologic) 82.0 ky	Barbados 3 (Faunal-lithologic) 125.0 ky
V27-116	38	180	240	500
V27-137	(30) ^b	---	140	225
V28-14	110	364/381	435 ^c	510 ^c
V28-84	28	---	225	340
V28-89	32	288	375	590
V29-177	(35) ^b	---	140	220
V29-178	(30) ^b	160	205	325
V29-179	42	268	345	510
V29-180	24	194	235	340
V30-96	(35) ^a	---	295	450
V30-97	(40) ^a	---	420	570
V30-100	(35) ^a	---	210	300

(a) Faunal termination at 11.0 ky used in place of zone 1 ash.

(b) Faunal termination at 13.5 ky used in place of zone 1 ash.

(c) Values from Kellogg (1975)

Input of Ice-Rafted Detritus (grams) in the sub-
polar North Atlantic during the Late Pleistocene.

Size Fraction and Region	From 125,000 to 10,000 yr B.P.	From 1.2 MYBP to present
Sand	1.4×10^{18}	15×10^{18}
Lutite	8.4×10^{18}	85×10^{18}
Sand and Lutite	9.8×10^{18}	100×10^{18}
