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Elemental C, H, and N Microanalysis of Crushed Rock and Soil Samples[1]

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Running title: Elemental C, H, and N Microanalysis

DISCLAIMER

FOOTNOTE

[1] Presented, in part, at the 62nd AOCS Meeting, Houston, May, 1971.

ABSTRACT

A sensitive and reproducible method for elemental analysis of rock and soil samples has been developed using a modified Hewlett-Packard 185 CHN analyzer. Samples are combusted in an inert He atmosphere (with and without catalyst) with a carefully controlled combustion time and temperature. Absolute mass calibration is available on samples of from 1-500 μg total CHN content. Analysis of chromatograms are made by both manual and computer techniques. Finely crushed rock (or dust) samples of from 50-100 mg may be analyzed for most forms of carbon content and for N and H with a high degree of technical reproducibility. Step-wise calcination at 150 C and 550 C allows evaluation of adsorbed CO_2, potential organic and amorphous carbon content. Although carbide carbon is partially recovered, essentially quantitative recovery of finely divided graphitic (and diamond) as well as amorphous, carbonate and all organic carbon may be obtained.

KEY WORDS AND KEY PHRASES

C, H, and N elemental analysis, microanalysis of carbon compounds in rock
and soil, organic, amorphous, carbonate, diamond and graphitic carbon
microanalysis.

INTRODUCTION

For many decades, elemental analysis by the classic techniques of Pregl for C and H and Dumont for N have been the ultimate standard and criteria for purity in organic microanalysis (1). The traditional apparatus and procedures placed unusual and severe technical demands upon the analyst. However, with the introduction of the semiautomated CHN analyzers a few years ago, the ease and rapidity of analysis suddenly improved. Such improved elemental analytical procedures have been reviewed and compared by Salzer (2). Under optimum conditions, essentially equivalent results may be obtained by such CHN analyzers as compared with the classical techniques (3). Although normally applied to organic microanalysis, this CHN technique with modifications can be applied to the analysis of crushed rock and soil samples of small particle size. Extremely low levels of C, H, and N may be measured. Further, some discrimination with regard to the presence of organic, carbonate, amorphous, graphitic (or diamond) and carbide carbon may be made.

METHODS AND MATERIALS

All elemental analyses were made on a modified Model 185 CHN analyzer equipped with an HP-18 helium purifier (Hewlett-Packard, Palo Alto, Ca.). To permit absolute mass calibration, the automatic attenuator and coupling with the Cahn Gram-O-matic balance was disconnected. Recorder performance was monitored and results insured on all samples by feeding the thermal conductivity detector output to two independently attenuated 1 mV strip chart recorders (Model V-16, Minneapolis Honeywell, Minneapolis, Minn.). Line voltage to all critical components was stabilized using a Sorensen voltage regulator.

Certain apparatus modifications were required to improve performance and to achieve needed stability at maximum sensitivity. Some of these have been presented earlier (4) and those utilized here are as follows: a dual channel 12 hr cycling H_2O-glycerol humidifier, permitting higher humidification during the night and lower during the day's running period, a finer zeroing potentiometer, an ammeter for the furnace circuit to reproducibly set the furnace variac, a foot switch with a precision timer to exactly time (and to vary precisely) the furnace combustion (and bypass) cycle, the column fan motor is disconnected to avoid noise, a silicone rubber injection rod gasket is substituted for the viton gasket, and a close fitting quartz sleeve is installed on the Hastelloy "X" oxidation furnace tube to prolong life and to eliminate irregular tube sag. Also, a calibrated chromel-alumel thermocouple monitor was provided with options for monitoring either outside or inside the oxidation furnace. The latter monitor consisted of a thermocouple embedded in a specially fabricated injection rod permitting measurement of the temperature rise within the sample cavity (as a function of time) during typical combustion cycles.

Analysis of chromatograms was by either manual or computer processing of the data. The manual procedure for carbon alone, assumed mass linearity of the peak height-background measurements. For CHN measurements, a computer sequence, using peak height x effective elution time as a parameter, for each element was employed as described earlier (4). All such data were processed at the Lawrence Berkeley Laboratory Computer Center using a Control Data Corporation 6600 computer. A primary absolute mass calibration over

the dynamic range (20-500 µg) of the instrument was made for C, H and N, using weighed standard samples of stearamide with 35-40 mg catalyst (MnO_2 + WO_3). Best fit 3rd order polynomials were fitted to the three components. Normally, daily calibration involved the use of a calcinated (550 C) $CaCO_3$ standard (130-150 µg) or 350 µg methyl stearate samples. The former were weighed in Hewlett-Packard 5080-5045 boats and emptied into the injection rod cavity without catalyst.

Using the daily calibration values, the primary calibration polynomials were rotated so the curves fell on the mean point or points measured. At levels of 15-18 µg carbon (as carbonate) fluctuations in absolute carbon mass calibration were consistently less than ± 2% over the experimental period reported here, whether measured before or after the sample analyses. Standards calcinated at 550 C included amorphous $CaCO_3$, -325 mesh graphite (C-001), -325 mesh Cr_3C_2 (CR-11), (Research Organic/Inorganic Chemical Corp., Sun Valley, Ca.), and synthetic diamond ultra fines (General Electric Corp., Worthington, Ohio). Other standards used were argillaceous limestone (Standard Reference Material 1b, National Bureau of Standards) and calcite crystal. A sample of crushed Sierra Nevada Latite was used for direct study and, after 7% enrichment with TiO_2 and calcination (550 C), was utilized as a low level standard and matrix.

To allow adequate packing of 50-100 mg crushed rock specimen (without loss) plus approximately 40 mg of catalyst, it was found necessary to use the older style injection rods (F & M # 2-5554). Although Perkin-Elmer boats (29-410 M) and Hewlett-Packard 5080-5045 boat caps were used for the primary calibration, to minimize background, daily $CaCO_3$ calibration and

the crushed rock samples were placed directly in the injection rod cavity.

Samples were combusted in an inert He atmosphere with and without catalyst. At an oxidation furnace setting of 1065 C the 20 sec thermal rise within the cavity was 930 C, although some additional combustion continued after the furnace flow mode was resumed. These combustion conditions, without catalyst, gave quantitative recovery of carbonate carbon and at low levels (less than 100 µg) most organic carbon. Amorphous carbon and higher levels of organics required catalyst. A complete profile of temperature with time is shown in Fig. 1. Longer burn times than 20 sec allowed essentially quantitative recovery of both graphitic and diamond fines, but only partial recovery of carbide carbon.

RESULTS AND DISCUSSION

Table I shows the stability of the instrument with respect to injection rod background, porcelain mortared and calcinated (550 C) catalyst and the daily $CaCO_3$ calibrations. Also potential sources of random error in sample analysis are given.

The results of a complete stearamide calibration for N, C, H and total mass are given in Fig. 2. However, since many analyses involved small amounts of carbon, a lower level calibration was needed. Fig. 3 shows a $CaCO_3$ calibration demonstrating linearity over the range of 5-90 µg C. A similar calibration curve is obtained over the same range of carbon for stearamide without catalyst, illustrating quantitative recovery at such levels of carbon. Weighing errors account for the significant differences in the standard error of measurement values for the two sets of data.

The recovery of amorphous carbon at a level of 20 μg was tested with and without catalyst. Table II shows approximately 2% recovery without catalyst and with catalyst essentially 100% recovery (considering the error of weighing).

Idealized recovery of graphitic, diamond and intermediate type carbide carbon was evaluated using the catalyst as a matrix. This was done to eliminate the large error inherent in using a matrix such as the crushed latite. (The weighed carbon and catalyst were mortared under nearly dust-free conditions.) To evaluate this recovery it was also necessary to run a profile of burn times from 20/sec to as much as seven minutes. The data in Figs. 4 and 5 demonstrate essentially quantitative recovery at 40 sec in this system for small particle-size graphitic carbon and diamond at the 1 and 10 μg levels, and 10 μg level, respectively. However, the recovery of Cr_3C_2, shown in Fig. 6 illustrates a unique profile of partial recovery. Again, such reproducible partial recovery was demonstrated at both the 1 and 10 μg levels of total carbide carbon.

Since much of the carbon present in terrestrial rock and soil samples is in the form of carbonate and organic carbon, the recovery of these forms at a level of 200 ppm in a calcinated latite - TiO_2 matrix was tested. Standard 20/sec burn times were used and no catalyst employed. Table III shows the quantitative recovery of amorphous, arogonite (argillaceous limestone) and calcite forms of carbonate. Table IV shows the recovery of a combined $CaCO_3$ and organic standard in the calcinated latite matrix. Again, reproducible quantitative recovery is achieved at approximately the 10 μg level of total

-9-

carbon. All the higher values represent probable contamination, reflect-
ing the limitations of the method by various random errors, discussed ear-
lier.

Some detailed analysis of the raw latite sample itself was made. The
particle size distribution (by dry sieving) and approximate chemical compo-
sition (5) of this sample are given in Fig. 7. The effect of particle size
on the amount and recovery of carbon is given in Table V. This is evaluated
both by extending burn times and by combusting with and without the catalyst.
Carbon content appears to increase with decreasing particle size, although
there are obvious sampling difficulties with the larger particle fractions
1 and 2. There is also a definite increment of carbon recovered with cata-
lyst, suggesting the presence of amorphous and/or graphitic carbon, and a
plateauing of recovery (for fraction 5) at about 40 sec.   An additional
consideration involved completeness of carbon recovery of the larger particle
fractions, particularly with the instrument limitation on combustion temper-
ature. To test this, fractions 2, 3 and 4 of another aliquot of parent latite
were normalized to the sample particle size of #5 by porcelain mortaring.
The results given in Table VI indicate substantially increased recovery after
mortaring, but consistently lower carbon values in the originally larger
particle size fractions. Again, essentially quantitative recovery was ob-
served for the 40/sec burns with some additional recovery in the presence of
the oxidizing catalyst.

Two additional samples were analyzed in some detail, a Hawaiian basalt
and a sample of the Pueblito de Allende meteorite. The latter sample was
analyzed under standard burn conditions, with and without catalyst. Complete

computer analysis of duplicate and triplicate runs are given in Table VII. The reproducibility is very good and the values for total N and C content are in reasonable agreement with data given previously by other techniques on samples from this meteorite. For example, Cronin and Moore (6), using much higher combustion temperatures, give a value of 2500 for C and 60 for N.

Lastly, a sample of Hawaiian basalt was similarly analyzed with and without catalyst and after porcelain mortaring. Clearly the values given without mortaring illustrate sampling problems, particularly when large particles are present. However, after porcelain mortaring there is good reproducibility for all elemental components emphasizing the need for using samples of small particle size with this technique. Further, the data for carbon are in agreement with 150 ppm reported by Kaplan et al. (7) using a different combustion technique and 140 ppm reported by Burlingame et al. (8) using/pyrolysis with analysis by high resolution mass spectrometry. Since

vacuum

Hawaiian basalt represents a good example of low level C, H, and N analysis, it is worthwhile to present typical chromatograms used in this analysis. Fig. 8 shows the chromatograms for an injection rod, rod + catalyst, the basalt and the low level daily $CaCO_3$ calibration, all at maximum sensitivity of the instrument.

Although application of this technique has been limited to several terrestrial samples and lunar material, reported elsewhere (9), some estimation of the forms of carbon present in a given sample may be made. This requires comparison of yields before and after appropriate step-wise calcination conditions. Thus, calcination in the presence of oxygen at 150 C should remove

adsorbed CO_2 and volatile organics. Additional calcination at 550 C should remove the remaining organics and amorphous carbon. Finally, by extending burn times beyond the customary 20/sec time, an estimation of graphitics and/or diamond fines as well as the presence of refractory carbide may be made. Such analyses should be made with and without catalyst. The broad features of an interpretive scheme are presented in Fig. 9.

There are, of course, difficulties and limitations to this method. Among them are the unknowns involved in the chemical interactions of the various forms of carbon with compounds and elements present in the sample matrix. Further, some non-contaminating procedure for reducing the particle size appropriately must be employed before analysis. However, the method is sensitive, reproducible and gives a simultaneous analysis of C, H, and N on each sample, allowing additional evaluation of sampling problems and random contamination. Also, some improvements in stability, sensitivity and accuracy may be anticipated using a suitable electronic integrator to provide precise peak area measurements with either this stabilized column system or with a standard instrument equipped with a Polypak(10) or Poropak Q column. Perhaps such analyses might have potential application in such diverse areas as the analysis of unusual geological specimens, petroleum engineering and possibly criminology.

ACKNOWLEDGEMENTS

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FIGURE LEGENDS

Fig. 1. Profile of thermal rise in the injection rod cavity.

FIGURE LEGENDS

Fig. 2. Calibration curves for N, C, H and total mass obtained over the
range of 20-500 μg stearamide.

FIGURE LEGENDS

Fig. 3. Low level carbon calibration of $CaCO_3$ and stearamide, no catalyst (CAT).

FIGURE LEGENDS

Fig. 4. Graphitic carbon recovery with the catalyst as matrix.

FIGURE LEGENDS

Fig. 5.   Synthetic diamond dust recovered in the catalyst matrix.   Coarse

dust was -325 mesh.

FIGURE LEGENDS

Fig. 6. Chromium carbide recovery in the catalyst as matrix.

FIGURE LEGENDS

Fig. 7.   Particle size distribution and approximate chemical composition

of the raw latite and the matrix.

FIGURE LEGENDS

Fig. 8. Typical CHN chromatograms showing increments of carbon analyzed
(all at maximum sensitivity, 1 mV full scale).

FIGURE LEGENDS

Fig. 9. Recovery of various forms of carbon using CHN elemental analysis

and appropriate calcination.

$T_\infty = 1030°C$

$T_{3\ MIN} = 1022°C$

$T_{60\ sec} = 1000°C$

$T_{40\ sec} = 984°C$

$T_{20\ sec} = 930°C$

Chromel P-Alumel
Thermocouple

Holding
position

$\sim 77°C$

220 200 180 160 140 120 100 80 60 40 20 0

Seconds

NITROGEN
24OCT69   CASES 6188 - 6203

STEARAMIDE CALIBRATION

RELATIVE AREA (PH×T)

0   MICROGRAMS   35

CARBON
24OCT69   CASES 6188 - 6203

STEARAMIDE CALIBRATION

RELATIVE AREA (PH×T)

0   MICROGRAMS   450

HYDROGEN
24OCT69   CASES 6188 - 6203

STEARAMIDE CALIBRATION

RELATIVE AREA (PH×T)

0   MICROGRAMS   80

TOT MASS
24OCT69   CASES 6188 - 6203

STEARAMIDE CALIBRATION

RELATIVE AREA (PH×T)

0   MICROGRAMS   500

CARBON
20 APR 71   CASES 8336 - 8350

CaCO₃ CALIBRATION

5-88 μg Carbon, n = 11

σ_{SEM} = 0.226 μg C

CARBON
21 APR 71   CASES 8355 - 8367

STEARAMIDE CARBON CALIBRATION, NO CAT
15-70 μg C

σ_{SEM} = 1.04 μg C

RELATIVE AREA (PH×T)

MICROGRAMS

Sample A, 0-4 μ
(10-15 μg, 192 ppm C)

Sample B, coarse dust
(8-14 μg, 189 ppm C)

Mean of duplicate sets of data
○ 5 Jan 71   △ 21 Jan 71   ◇ 22 Jan 71

Best duplicates of triplicate analyses
4 Feb 71

SIERRA NEVADA
LATITE

Raw Latite

#5
−120
mesh

Top axis: 418 296 208 125 74 44

1 2 3 4 5 →

Y-axis: Cumulative weight, percent — 0, 20, 40, 60, 80, 100

X-axis: Particle size, microns — 600, 100, 50, 10, 2

	Latite (Table Mountain)	+ 7.02% TiO$_2$
SiO$_2$	58.8	54.9
TiO$_2$	1.1	7.6
Al$_2$O$_3$	16.9	15.8
Fe$_2$O$_3$	3.0	2.8
FeO	3.1	2.9
MnO	0.05	0.05
MgO	2.5	2.4
CaO	5.1	4.8
Na$_2$O	3.4	3.2
K$_2$O	4.4	4.1
H$_2$O	1.1	1.1
P$_2$O$_5$	0.5	0.4

TYPICAL CHN CHROMATOGRAMS (1 ×)

FURNACE FLOW INTERRUPTION, 20 sec

$(CaCO_3)_{550\ C}$ (143.6 μg)

INJECTION ROD

$\Delta C = 0.50\ \mu$g

17.22 μg C

ROD + CAT

$\Delta C = 0.48\ \mu$g

HAWAIIAN BASALT (50.8 mg)

$\Delta C = 7.33\ \mu$g

3.87 μg H

0.57 μg N

MINUTES

RECOVERY, PERCENT

Curve labels (top to bottom): Carbides (+O_2), Diamond fines (+O_2), Graphite fines (+O_2), Carbonates, Amorphous carbon (+O_2), Organics (+O_2), Adsorbed CO_2

Y-axis: TEMPERATURE, CELSIUS — 25, 150, 550, 930, 1030, 1400

DBL 716-5864

TABLE I

Evaluation of Errors, Low Level Carbon Analysis

1. Quantified errors

    a. Injection rods, duplicate pairs

        Operator 1 (GRS) n = 61, $\sigma_{S.E.M.}$ = ± 0.100 µg C

        Operator 2 (FTL) n = 22, $\sigma_{S.E.M.}$ = ± 0.109 µg C

    b. Injection rods + 30-35 mg catalyst, all run the same day

        Operator 1 & 2   n = 10, $\sigma_{S.E.M.}$ = ± 0.184 µg C

    c. $CaCO_3$ calibration, 15-21 µg carbon (125-175 µg $CaCO_3$), duplicate pairs

        Operator 1 (GRS) n = 25, $\sigma_{S.E.M.}$ = ± 0.193 µg C

        Operator 2 (FTL) n = 7, $\sigma_{S.E.M.}$ = ± 0.196 µg C

2. Unquantified random errors

    a. Manipulation of sample, sampling statistics and associated problems,
        including "coning effect", i.e., segregation of large from small par-
        ticles and segregation of denser from lighter particles of similar
        size.

    b. Contamination (sample containers, dust fallout, gasket scrape, etc.).

TABLE II

Recovery of Amorphous Carbon, Lamp Black (LB) 245 C[a]

Sample	Latite-TiO$_2$ (LT) (550 C) No CAT ppm	LT + CAT	LT + LB No CAT μg	LT + LB CAT μg
	12.3	15.3		
	10.2	15.4		
Amount expected from LT			0.85	1.20
			0.76	1.05
Mass LB added			21.3	21.2
			13.4	18.9
Mass C found			1.22	22.69
			0.97	19.62
△LB to chromatogram			0.37 (1.7% recovery)	21.5 (+ 0.3)
			0.21 (1.6% recovery)	18.6 (- 0.3)

[a] CaCO$_3$ calibration, 22.50 μg C, 1 mV full scale (no attenuation), manual calculations.

TABLE III

Recovery of Forms of Added Carbonate to $(\text{Latite-TiO}_2)_{550\ C}$ Matrix, Porcelain Mortared[a]

25 Sept 70

Σ 201.3 ppm	Σ 201.6 ppm	Σ 201.7 ppm
$(\text{L-TiO})_{550\ C}$ + $(\text{CaCO}_3)_{550\ C}$	$(\text{L-TiO})_{550\ C}$ + $(\text{Arogonite})_{110\ C}$	$(\text{L-TiO})_{550\ C}$ + Calcite
206.0 (13.40)	203.9 (13.59)	196.4 (11.66)
205.0 (11.33)	206.2 (13.30)	198.6 (13.16)

Recovery, standard reference material 1b

Raw latite	$(\text{Latite-TiO}_2)_{550\ C}$		(Argillaceous limestone)$_{110\ C,\ 2\ hr}$	(91.3% CaCO_3)
29 Sept 70	22 Sept 70	25 Sept 70	22 Sept 70	18 Mar 70
450, 395	11.6	11.9	93.29% (18.47)	93.33% (271.6)
356, 331	13.0	11.3	92.47% (17.51)	92.48% (305.2)
	11.2	11.3		

[a] Manual calculations, (μg C analyzed in parentheses). Calcite was not calcinated.

TABLE IV

Recovery (ppm), Mixed Carbon Standard (MCS)

120 ppm ($CaCO_3$) + 30 ppm (EDTA) Carbon + (Latite)$_{550\ C}$ Matrix

9 August 70		11 August 70	12 August 70	14 August 70		15 August 70
(Parent latite)$_{550\ C}$	MCS	MCS	MCS	MCS		(Parent latite)$_{550\ C}$
8.0 (1.03)	167.2 (8.05)	160.0 (6.22)	159.2 (9.57)	175.0[a] (7.40)		6.6 (0.72)
8.4 (1.03)	166.6 (9.64)	161.0 (6.59)	175.1[a] (12.96)	175.2[a] (10.17)		6.6 (0.73)
6.4 (0.63)	160.8 (10.26)		160.6 (10.20)	161.5 (8.70)		
6.6 (0.58)	169.2 (6.86)			161.6 (9.50)		

$CaCO_3$ standard (µg C, 1 mV full scale)

22.51			22.01	22.24	22.66	
22.22			21.61	21.77	22.20	

[a] Probable contamination. (µg C analyzed in parentheses), manual calculations, no CAT.

TABLE V

Raw Latite, Sample I, Carbon Recovery, Particle Size Effect[a]

Fraction (μ size)	Wt %	Burn times 20 sec		
#1 (418 < d)	4.8	248 (260) 390 (111) 95 (184) 627	Σ_1^5 Whole crush	325 (571) 326 (544) (414)
#2 (296 d < 418)	9.5	164 (188) 101 (271) 137	$\Sigma_1^5 \dfrac{\overline{C}_{i \times wt\%}}{100} =$	341 (435)
#3 (209 < d 296)	15.5	150 (150) 157 (189)		

			Burn times		
			40 sec	60 sec	180 sec
#4 (125 < d < 209)	25.7	216 (251) 208 (276)			
#5 (d < 125)	44.5	508 (684) 539 (712)	632 (860) 638 (919)	661 (982) 696 (953)	745 (950) 767 (922)

[a] Sample mass 64-142 mg, (12-98 μg C), manual calculations (with CAT in parentheses).

TABLE VI

Raw Latite, Sample II, Carbon Recovery, Particle Size Normalization[a]

Sample (μ size)	20 sec	20 sec[b]	40 sec[b]	60 sec[b]	180 sec[b]
			Burn times		
#2 (296<d<418)	115	292 (296)	(>300)	(320)	(326)
	142	282 (290)	(310)	(324)	(310)
#3 (209<d<296)	209	254 (277)	(296)	(286)	(285)
	151	250 (293)	(292)	(324)	(278)
#4 (125<d<209)	219	353 (385)	(454)	(452)	(417)
	233	340 (410)	(437)	(442)	(396)
#5 (d<125)	536 (704)		(984)	(1023)	(1000)
	580 (724)		(1008)	(1003)	(1000)
	536 (758)				
	572 (756)				
Σ_1^5 whole crush	450 331				
	356 395				

[a] Sample mass 48-138 mg, (16-93 μg C). Manual calculations (with CAT in parentheses).

[b] Fractions 2-4 are normalized to d < 125 μ.

TABLE VII

CHN Analysis of a Pueblito de Allende Meteorite Sample[a]

| 24 July 70 | Abundance, ppm | | | Atom ratios | |
	N	C	H	N/C	H/C
No CAT	39.4 (2.11)	1890.2 (100.9)	660.9 (35.28)	0.01789	4.17
	40.8 (2.67)	1882.8 (123.3)	657.1 (43.04)	0.01859	4.16
Plus CAT	40.6 (2.79)	1887.3 (129.8)	660.4 (45.42)	0.01844	4.17
	38.4 (2.26)	1847.9 (108.9)	667.2 (39.30)	0.01781	4.30
	40.6 (2.57)	1855.5 (117.2)	655.2 (41.40)	0.01877	4.21

[a] Values in parentheses are μg of N, C, and H mass analyzed.

TABLE VIII

CHN Analysis of a Hawaiian Basalt, (I. Kaplan, BTS-17-50)[a]

23 July 70	Abundance, ppm			Atom ratios	
	N	C	H	N/C	H/C
No CAT	6.6 (0.40)	80.9[b](4.85)	38.9 (2.33)	0.0702	5.73
	8.8 (0.85)	119.8 (11.51)	52.1 (5.00)	0.0631	5.18
	11.9 (0.86)	117.5 (8.51)	49.2 (3.56)	0.0866	4.99
Plus CAT	7.2 (0.34)	87.7 (4.21)	73.2 (3.51)	0.0702	9.95
	7.0 (0.36)	77.1 (4.01)	58.1 (3.02)	0.0773	8.99
29 September 70					
No CAT	14.6 (1.03)	155.1 (10.86)	82.7 (5.79)	0.0810	6.35
Porcelain mortared	12.2 (1.02)	144.7 (12.05)	78.7 (6.55)	0.0725	6.48
	11.2 (0.57)	144.4 (7.33)	76.3 (3.87)	0.0662	6.30

[a] Sample range 51-96 mgs. (Values in parentheses are μg of N, C, and H mass analyzed.)

[b] Large glassy object included.