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ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND ABERD--ETC F/G 8/13
DEVELOPMENT OF A PROCEDURE FOR THE EVALUATION OF SOIL SUBSAMPLI--ETC(U)
APR 78 W J MAURITS, P D DIXON, K W EARNHARDT

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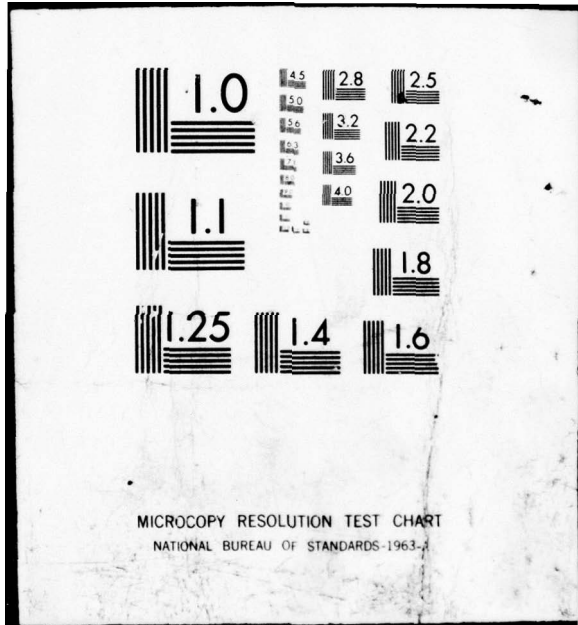
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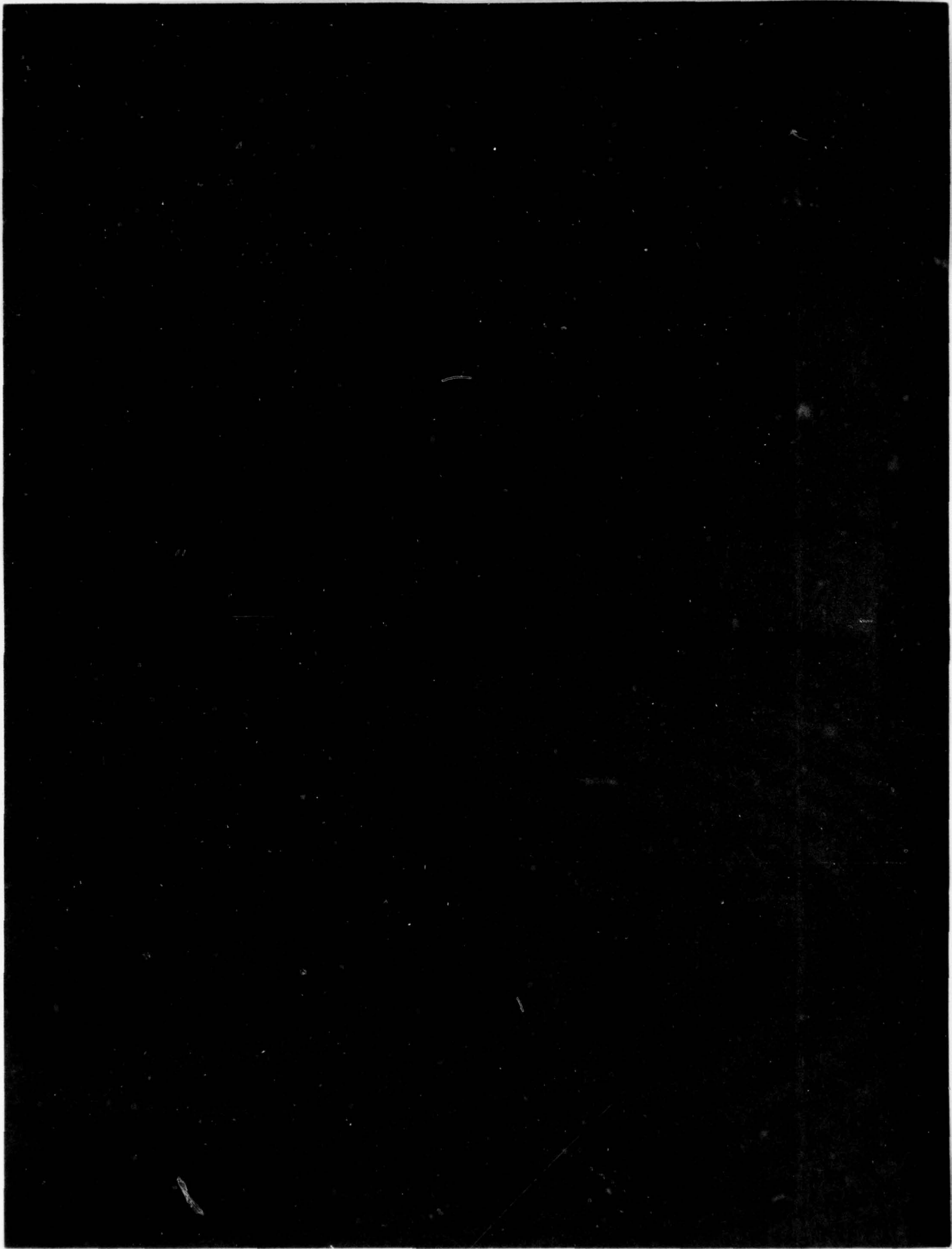
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PREFACE

The work described in this report was authorized under WBS 1.04.34. This work was started in June 1977 and completed in August 1977. The experimental data are recorded in notebook 76-15.

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DEVELOPMENT OF A PROCEDURE FOR THE EVALUATION OF SOIL
SUBSAMPLING ROUTINE I (FEASIBILITY OF A WEAK BETA
EMITTER AS A RADIOACTIVE TRACER)

I. INTRODUCTION.

Numerous papers¹⁻⁵ have cited potential errors inherent in the subsampling of solids. Such errors are of great concern in the case of soil samples which are normally quite heterogeneous. Riffling and quartering traditionally have been accepted as satisfactory methods for general-purpose subsampling of solids. Specific analytical requirements often preclude the use of riffling or quartering without significant modification. The purpose of this project was to demonstrate the feasibility of using a radioactive tracer to estimate subsampling errors for any given combination of subsampling routine and sample type. The availability of such information is required to facilitate approval of proposed subsampling techniques, as well as to permit the association of realistic estimates of confidence limits with data from soil analyses.

The coefficient of variation (the standard deviation expressed as a percentage of the mean) is the statistic generally accepted⁶ as being the best with which to estimate random error. The square of the coefficient of variation has the valuable property of being additive.

The approach of this project is to demonstrate the feasibility of a tracer method by the measurement and comparison of random errors as reflected in coefficients of variation of activity determinations.

II. EXPERIMENTATION.

A. Preparation of Heterogeneous Soil Samples Containing a Radioactive Tracer.

To approximately 100 gm of air-dried soil (previously passed through a 30-mesh sieve) is added a 10-ml aliquot of a solution containing a nominal 65 gm of zinc uranyl acetate ($ZnUO_2(OOCCH_3)_4$, molecular weight - 571, activity (B^-) ~ 60,000 counts/min-gm) per liter. The soil is not fully wetted. The entire soil sample is placed in a preheated oven and dried at 80°C for 2 hours. After the soil has cooled to room temperature, dried aggregates are crushed with a minimum of mixing.

B. Subsampling.

A riffle (figure 1) is a device through which a solid sample can be passed and thereby divided into two essentially identical portions. Plates and passages are arranged within the device to provide each particle with an equal probability of landing in either one or two receptacles. Normally the contents of one of the receptacles are rejected and those of the other are poured through again. This continues until a subsample of the desired size is obtained. Entire subsamples can be recombined if required, but all divisions of sample matter must be conducted with the riffle. Each subsample may be considered representative of the original sample, subject to limitations caused by consideration of less than infinite numbers of particles in the population.

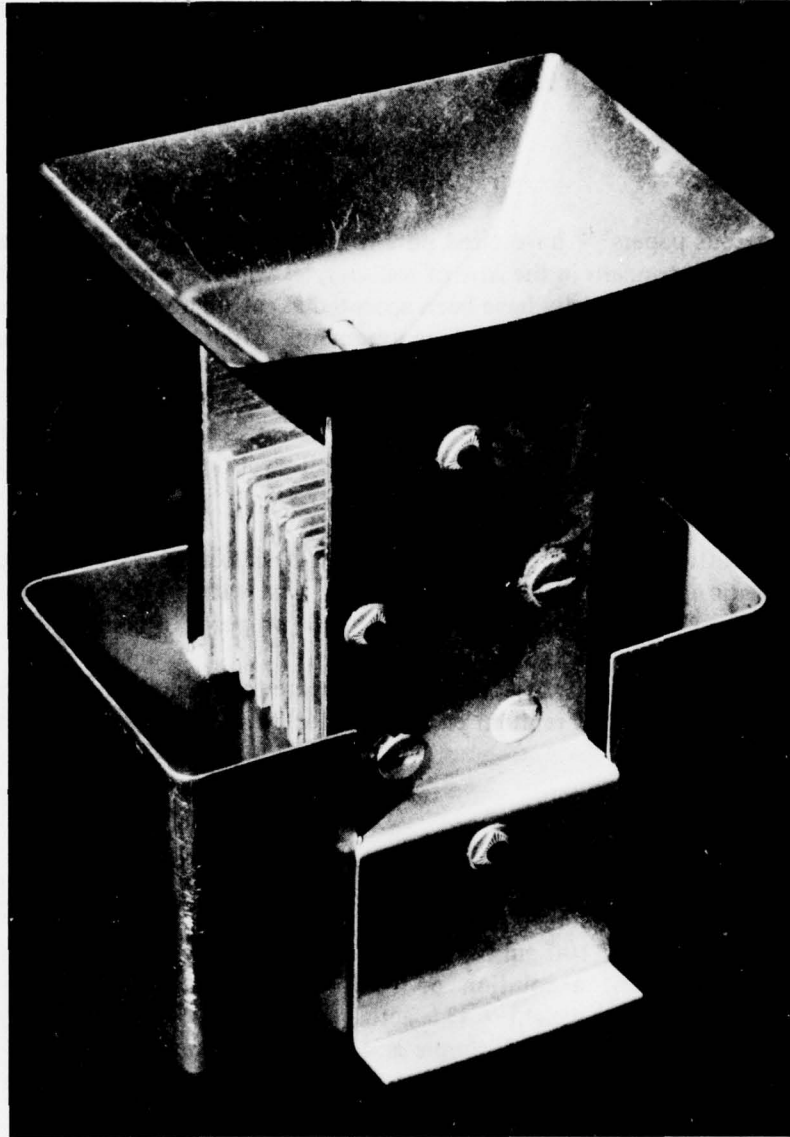


Figure 1. Riffle

C. Fabrication of Sample Holders.

Preformed sample holders are prepared by using the plastic protective container from a 35-mm-film cassette and the lid of this container as a die and anvil to mold circles of hard-finish notebook paper into shallow pans.

D. Determination of Activities of Soil Samples.

Soil subsamples are spread out evenly in the sample pans. A small piece of clean paper, folded to yield a flat edge, can aid in spreading each sample. The sample holder is inserted into a jig (figure 2) which holds the sample parallel to and in proximity with the face of a CaF_2 scintillation detector which is coupled with a nuclear counting system. The face of the detector crystal is covered with an 0.0008-inch-thick layer of aluminum foil in order to remove visible and alpha radiation. Activities are reported as the averages of three counting periods of 4 minutes each and are corrected for background and self-absorption effects.

E. Procedure.

Five sampling procedures were used to determine that procedure yielding the lowest coefficient of variation; i.e., the procedure providing the best sample for subsequent analysis. The procedures tested are detailed below.

A sample of soil containing zinc uranyl acetate (tagged soil) is subsampled into 32 nominal 2-gram portions using a riffle. The activities of the subsamples are determined. Each value is reported as the average of three counting periods of 4 minutes each. Using a riffle, a nominal 2-gram subsample is separated from a 122-gram sample of a tagged soil. Rejected portions are returned to the bulk of the sample. The activity is determined. Likewise, 36 additional subsamples are separated, counted, and replaced.

A sample of tagged soil is subsampled into nominal 2-gram portions using a riffle. The activities of the individual subsamples are each determined ten times with the samples being removed from the detector, respread upon the sample holders, and reinserted into the detector *between subsequent counting periods.*

A sample of tagged soil is subsampled into nominal 2-gram portions using a riffle. The activities of the individual subsamples are each determined ten times with care being taken to disturb neither the soils within the sample holders nor the positions of the sample holders relative to the counting face of the detector.

A 52-gram sample of tagged soil is placed in a 1-pint wide-mouth bottle with as little mixing of the sample as possible. Nominal 2-gram portions are withdrawn using a small spoon, with care being taken to minimize mixing of the contents remaining in the bottle. After the entire sample is separated into subsamples, activities of the individual subsamples are determined.

III. RESULTS.

Determinations of the activities of 32 subsamples of a sample of soil which is known to be heterogeneous (because of a deliberately uneven application of a solution of a radioactive tracer) yield results with a coefficient of variation of 6.3% as shown in table A-1.* A somewhat modified riffing procedure which combined rejected portions with the bulk of sample before the next subsample is separated yields results having a coefficient of variation of 5.6% as shown in table A-2. The F value for these two sets of data is 1.27. The critical F value for 31 and 36 degrees of freedom for $\alpha = 0.05$ is 1.6.

* All tables are in appendix A.

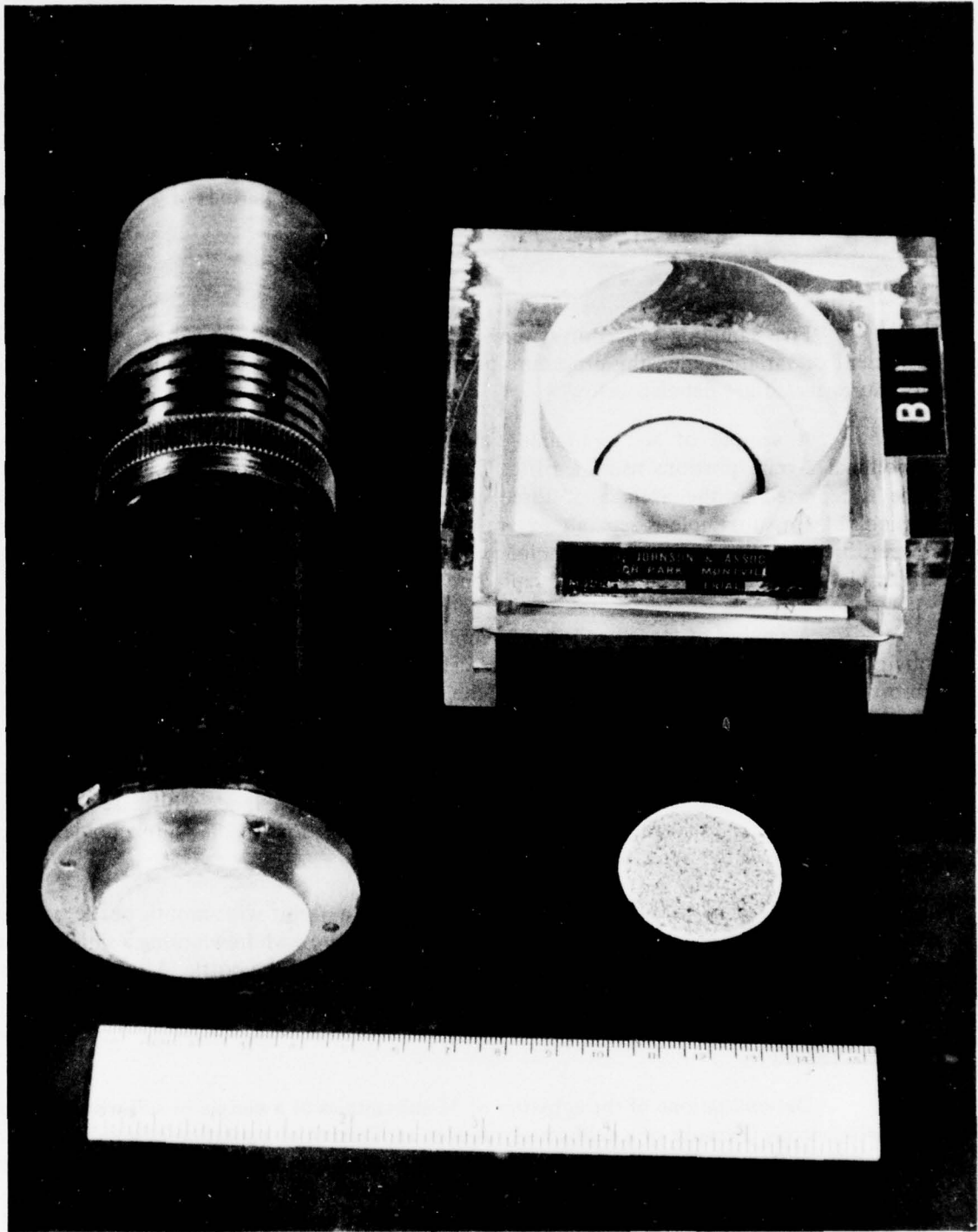


Figure 2. Scintillation Detector Arrangement

Determinations of activities of subsamples which are disturbed between counting periods are reported in table A-3. The results display an average coefficient of variation of 4.2%. When variations in counting geometry are stabilized by leaving the samples undisturbed in the detector between counting periods, the average coefficient of variation of the results is shown in table A-4 to be 1.7. The F value for these two sets of data is 7.32. The critical F value for 27 and 19 degrees of freedom for $\alpha = 0.05$ is 2.1.

Determinations of activities of unrepresentative subsamples of a heterogeneous soil yield results displaying a 96% coefficient of variation (table A-5). This may be contrasted with an average coefficient of variation of 6.0% obtained from activities of properly subsampled materials (tables A-1 and A-2). The F ratio for these two sets of data is 256. The critical F value for 28 and 66 degrees of freedom for $\alpha = 0.05$ is 1.7.

IV. DISCUSSION.

This tracer technique makes use of the naturally occurring B^- activity of a soluble uranium salt found in many laboratories. Zinc uranyl acetate is a common reagent used in qualitative analysis for the detection of sodium. The method of introducing the radioactive tracer into the soil samples is designed to simulate a spill of analyte (contaminant) which might ultimately lead to the realization of a heterogeneous sample. This worst case situation would put the greatest demands on any subsampling routine. Clearly, subsampling would not constitute a problem in the case of a homogeneous sample.

The sample holders used in this study are not designed to provide active assistance in the rather critical process of spreading the samples evenly. They are, however, judged to be adequate to demonstrate the feasibility of the technique. A somewhat more sophisticated sample holder is being fabricated for the counting of B^- activities of 5-inch-diameter (50-gm) samples to be conducted in a later study. The improved holder will permit excess sample to be conveniently scraped off (level) with a spatula. This should provide a more even sample depth and therefore reduced counting errors due to variable geometry.

Since it does not escape our notice that the observed activities of our subsamples are (counts per minute-gram) strongly dependent on the weights of these subsamples, the data are corrected for self-absorption. The following equation is used:

$$\frac{C}{C_0} = \frac{1}{\mu s} (1 - e^{-\mu s})$$

where

$C = B^-$ particles detected

$C_0 = B^-$ particles emitted

$\mu =$ coefficient of absorption

$s =$ source thickness

When the correction for self-absorption is made using a value of 0.60 for the product of μ and s , the activities no longer vary as a function of subsample weight (figure 3).

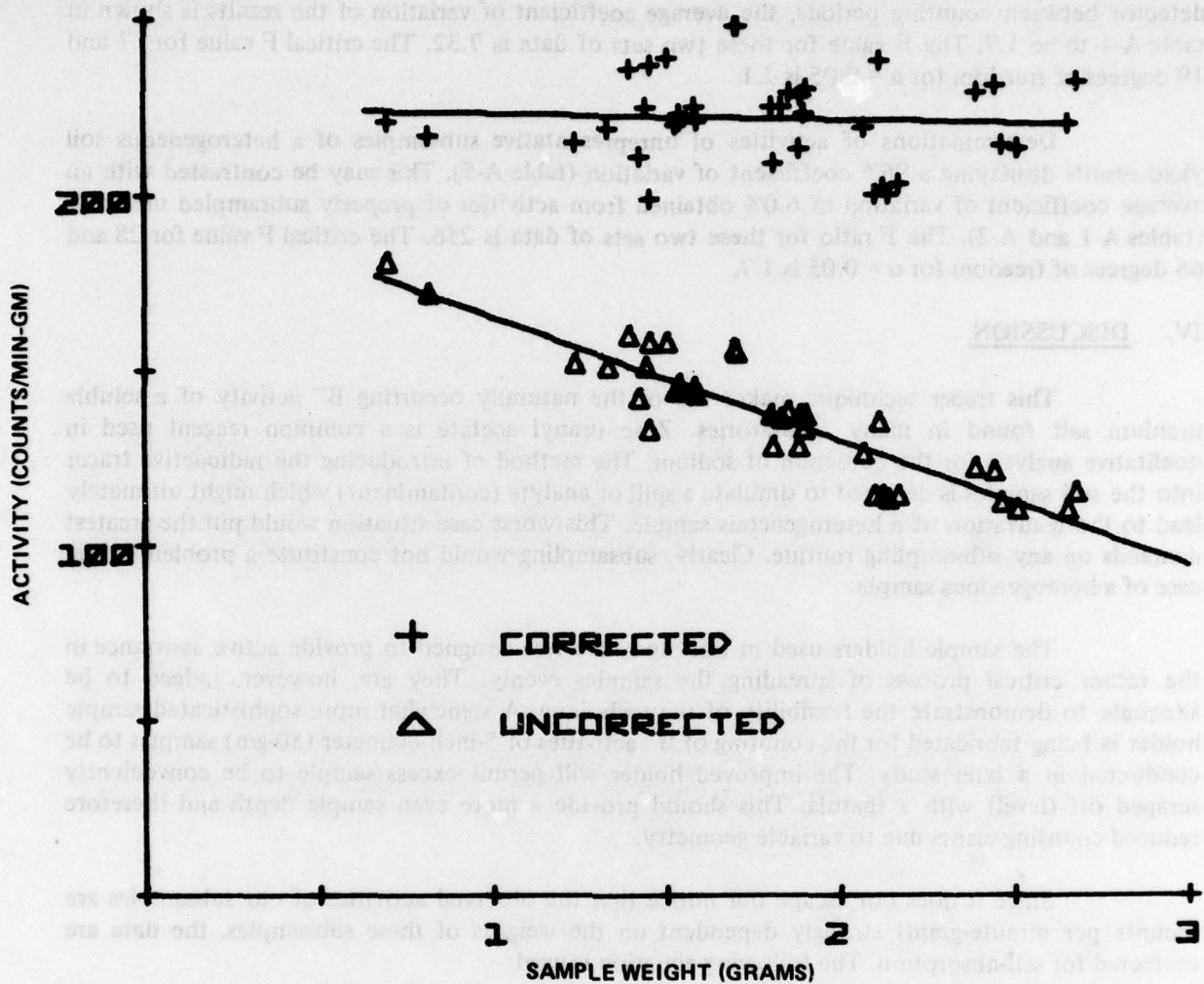


Figure 3. Activity Versus Sample Weight

Two similar riffing procedures are used to subsample heterogeneous soil samples. The first involves separating all 32 subsamples and determining the activities of each. The second procedure separates a single subsample for counting and replaces the rejected portion before the next subsample is separated. The second approach aids in the mixing of the samples. At an α level of 0.05, the null hypothesis is still accepted for both procedures. This suggests that the riffle was so effective as a subsampling device that insignificant improvement resulted from the additional mixing associated with the second technique. The data from both techniques are similar and include errors in the complete process from subsampling the soil through the logging of corrected activities. The coefficient of variation of such data may serve as an estimate of the random errors inherent in the total process.

In order to isolate some of the aforementioned errors, the activities of individual subsamples are determined repetitively both with and without disturbing (the geometry of) the samples between the determinations.

The results of determinations of activities of subsamples which are disturbed between the counting periods include errors introduced as a result of uneven deposition of the samples within the holders, as well as imprecise positioning of the sample holders relative to the detector. These errors may be said to have resulted from variables in counting geometry. Although subsampling errors have been excluded (by virtue of having used the identical subsamples over and over), normal uncertainties inherent in radioactive decay and the measurement thereof are included. The coefficient of variation of the results may serve as an estimate of all (random) counting errors.

The determinations of activities of undisturbed samples include only the normal vagaries and measurement of radioactive decay under fixed conditions. The coefficient of variation of such results may serve as an estimate of counting errors under conditions of fixed geometry.

This figure is indicative of the minimum level of random counting errors which might be anticipated for multiple subsamples if all variables in counting geometry were to be rigorously controlled.

Since the F test for these two groups of data rejects the null hypothesis at $\alpha = 0.05$, a conclusion may be drawn that the data set which includes geometric variables has significantly more variance than that subject only to counting errors under conditions of fixed geometry.

Since we have an estimate of random error for the entire tracer technique (in the form of a coefficient of variation) and have been able to isolate the counting process and thus estimate these errors, we can define an estimate of subsampling errors (COVs) as follows:

$$\text{COVs} = \left\{ (\text{COV}_T)^2 - (\text{COV}_{TC})^2 \right\}^{1/2}$$

COV_T = estimate of random errors for entire tracer technique

COV_{TC} = estimate of random errors in counting process

Using average values of 6.0% and 4.2% for COV_T and COV_{TC} , respectively, COVs may be calculated to be 4.3%.

In order to determine the effects of a clearly unsatisfactory sampling routine, such a routine was simulated by using a small spoon to withdraw subsamples from a heterogeneous soil. The results in table A-5 show a 16-fold increase in the coefficient of variation over that reported for the riffling technique.

Using the F ratio of the results from the unsatisfactory subsampling routine to the results from satisfactory procedures, the null hypothesis is rejected at an $\alpha = 0.05$. This permits a strong conclusion that the tracer technique is able to detect a greater variance in results from the group of improperly subsampled soils.

V. RECOMMENDED IMPLEMENTATION.

The herein-described tracer method (for the evaluation of soil subsampling routines) should be scaled up to the 50-gm subsample size normally required for soil analyses. If successful, this scaled-up technique should be used to evaluate all proposed subsampling routines before resources are committed to these routines. The resulting estimates of sampling error should be incorporated into estimates of errors published for analytical systems which are used for the analyses of soils.

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APPENDIX A

TABLES

Table A-1. Activities of Soil Subsamples after Separation by Riffling

Subsample	Weight	Observed activity	Corrected activity
	gm		
6A	1.678	182	288
6B	1.570	199	307
6C	1.369	219	321
6D	1.554	198	305
6E	1.729	217	348
6F	1.637	202	317
6G	1.383	208	307
6H	1.554	194	298
6I	1.766	206	333
6J	1.551	220	338
6K	1.645	236	372
6L	1.345	204	298
6M	1.564	204	314
6N	1.544	204	312
6O	1.581	209	324
6P	1.567	210	324
6Q	1.620	215	336
6R	1.614	229	357
6S	1.694	215	343
6T	1.578	210	325
6U	1.592	225	349
6V	1.637	234	368
6W	1.583	201	311
6X	1.590	195	302
6Y	1.572	220	339
6Z	1.622	208	326
6AA	1.636	219	344
6BB	1.636	204	320
6CC	1.610	212	330
6DD	1.594	221	344
6EE	1.619	222	348
6FF	1.603	216	
Average			327.5
Standard deviation			20.6
Coefficient of variation (%)			6.3

Soil: EA 200
 Gross weight: 51 grams
 Weight of ZnUO₂(Ac)₄ added: 0.380 gram
 Activity of ZnUO₂(Ac)₄: not determined
 Background: 13 counts/min

Table A-2. Activities of Soil Subsamples Separated (and Replaced by Riffling)

Subsample	Weight gm	Observed activity	Corrected activity
5A	1.703	155	248
5B	1.901	137	230
5C	1.586	143	221
5D	1.888	137	228
5E	1.851	138	229
5F	1.895	133	223
5G	0.697	181	221
5H	0.817	172	217
5I	1.696	156	248
5J	1.500	158	239
5K	1.536	144	221
5L	1.453	158	237
5M	1.525	144	220
5N	1.235	152	215
5O	1.331	151	219
5P	2.654	110	220
5Q	2.676	115	232
5R	2.386	122	229
5S	2.443	121	231
5T	2.065	126	219
5U	2.113	135	238
5V	1.801	137	225
5W	1.833	136	225
5X	1.539	146	223
5Y	1.580	145	225
5Z	1.392	160	236
5AA	1.443	151	225
5BB	2.463	112	214
5CC	2.509	110	213
5DD	1.808	128	209
5EE	1.882	128	213
5FF	1.424	142	211
5GG	1.451	133	199
5HH	2.120	114	202
5II	2.137	113	200
5JJ	2.097	114	200
5KK	2.165	114	203
Average			221.8
Standard deviation			12.48
Coefficient of variation (%)			5.6

Soil: EA 296

Gross weight: 122 grams

Weight of $ZnUO_2(Ac)_4$ added: 0.649 gram

Activity of $ZnUO_2(Ac)_4$: 44,766 (observed)

Background: 13 counts/min

Table A-3. Activities of Soil Subsamples Disturbed between Counting Periods

Subsample	Weight	Observed activity	Corrected activity
	gm		
6J	1.5506	211	325
6J	1.5506	207	318
6J	1.5506	200	307
6J	1.5506	195	299
6J	1.5506	186	286
6J	1.5506	196	301
6J	1.5506	198	303
6J	1.5506	194	298
6J	1.5506	202	310
6J	1.5506	197	303
Average			305.2
Standard deviation			10.8
Coefficient of variation (%)			3.5
7H	1.4301	144	215
7H	1.4301	138	206
7H	1.4301	145	215
7H	1.4301	140	208
7H	1.4301	137	204
7H	1.4301	135	202
7H	1.4301	142	212
7H	1.4301	135	202
7H	1.4301	126	187
7H	1.4301	129	192
Average			204.3
Standard deviation			9.3
Coefficient of variation (%)			4.6
7R	1.4207	97	145
7R	1.4207	97	144
7R	1.4207	90	134
7R	1.4207	98	146
7R	1.4207	94	140
7R	1.4207	100	146
7R	1.4207	95	140
7R	1.4207	93	138
7R	1.4207	91	135
7R	1.4207	105	156
Average			142.5
Standard deviation			6.48
Coefficient of variation (%)			4.5

Table A-4. Activities of Soil Subsamples Undisturbed between Counting Periods

Subsample	Weight	Observed activity	Corrected activity
	gm		
6J	1.495	213	322
6J	1.495	207	314
6J	1.495	217	329
6J	1.495	213	322
6J	1.495	212	320
6J	1.495	213	323
6J	1.495	220	333
6J	1.495	217	329
6J	1.495	214	324
6J	1.495	217	328
Average			324.6
Standard deviation			5.4
Coefficient of variation (%)			1.7
7H	1.4006	136	201
7H	1.4006	134	198
7H	1.4006	133	197
7H	1.4006	137	202
7H	1.4006	129	191
7H	1.4006	134	198
7H	1.4006	133	197
7H	1.4006	134	198
7H	1.4006	131	194
7H	1.4006	135	199
Average			197.6
Standard deviation			3.09
Coefficient of variation (%)			1.6

Table A-5. Activities of Soil Subsamples Improperly Separated

Subsample	Weight	Observed activity	Corrected activity
	gm		
7A	1.892	124	208
7B	1.846	112	185
7C	1.917	123	207
7D	1.382	137	202
7E	1.481	127	192
7F	1.660	119	188
7G	1.855	150	249
7H	1.430	150	223
7I	1.702	158	253
7J	1.880	124	207
7K	1.894	122	204
7L	2.052	113	196
7M	1.994	74	127
7N	2.002	99	170
7O	1.893	104	173
7P	3.113	74	164
7Q	1.157	91	126
7R	1.421	96	143
7S	1.821	101	165
7T	1.694	81	129
7U	1.621	106	166
7V	1.837	97	160
7W	2.146	119	212
7X	2.429	180	342
7Y	1.761	203	330
7Z	1.256	170	242
7AA	1.336	207	301
7BB	1.597	242	375
7CC	1.720	915	1,467
Average			252
Standard deviation			242
Coefficient of variation (%)			96.0

Soil: EA 200
 Gross weight: 52 grams
 Weight of $ZnUO_2(Ac)_4$ added: 0.380 gram
 Activity of $ZnUO_2(Ac)_4$: 37,603 (observed)
 Background: 13 counts/min

Table A-6. F Ratios (Degrees of Freedom) of Activity Data

S ₁ S ₂	A-1	A-2	A-3	A-4	A-5	A-1 + A-2
A-1	1.00 (31, 31)	0.79 (36, 31)	0.44 (27, 31)	0.07 (19, 31)	232 (28, 31)	1.27 (66, 31)
A-2	1.27 (31, 36)	1.00 (36, 36)	0.56 (27, 36)	0.09 (19, 36)	294 (28, 36)	1.15 (66, 36)
A-3	2.25 (31, 27)	1.78 (36, 27)	1.00 (27, 27)	0.16 (19, 27)	522 (28, 27)	2.04 (66, 27)
A-4	13.73 (31, 19)	10.85 (36, 19)	6.10 (27, 19)	1.00 (19, 19)	3188 (38, 19)	12.45 (66, 19)
A-5	0.00 (31, 28)	0.00 (36, 28)	0.00 (27, 28)	0.00 (19, 28)	1.00 (28, 28)	0.00 (66, 28)
A-1 + A-2	1.10 (31, 66)	0.87 (36, 66)	0.49 (27, 66)	0.08 (19, 66)	256 (28, 66)	1.00 (66, 66)

A-1 = riffled soil.

A-2 = riffled (and mixed) soil.

A-3 = riffled soil with subsampling errors excluded.

A-4 = riffled soil with subsampling and geometric counting errors excluded.

A-5 = unriffled soil.

A-1 + A-2 = average values from tables A-1 and A-2.

APPENDIX B

DESCRIPTION OF COUNTING EQUIPMENT

Equipment housing. A standard bin and power supply, model NH84A, accommodates 12 single-width standard modules or combinations of single or multi-width modules. The current capacity is rated to power any combinations of modules the bin will physically accommodate. The unit mounts in a standard Electronics Industries Association 19-inch rack and occupies less than 9 inches of vertical panel space. Its power requirement is 117 volts ac, single phase.

High voltage power supply. Model NV-27 provides an output range from 500 to 3000 volts dc at 10 milliamperes. A fine adjustment permits 1-volt selection within the range with an accuracy of 0.25% of the selector switch setting. This unit occupies two standard bin spaces.

Detector. The detector consists of a 1-1/4-inch-diameter by 1/4-inch-thick calcium fluoride, $[\text{CaF}_2(\text{Eu})]$, scintillation crystal coupled to a 1-1/2-inch photomultiplier tube. CaF_2 is nonhygroscopic and has a high efficiency for detecting particulate ionizing radiation. A 0.0008-inch aluminum foil over the crystal shields out alpha particles.

Amplifier. A double RC (resistive-capacitive) clipped, low noise, linear amplifier, model NA-11, provides an output pulse shaping of 3 μsec and an adjustable gain ranging from 20 to 1000.

Analyzer. Model NC-12 single-channel analyzer with selective output modes of integral, differential, or $>E + \Delta E$ generates a pulse of 500 nanoseconds. Although very useful, this item can be bypassed in counting.

Data display. The model NS30A scaler has a LED (light-emitting diode) display ranging from 0 to 10^6 counts, with an input pulse pair resolution of 50 nanoseconds. It can be operated manually or by address control. The NS30A occupies two standard bin spaces.

Equipment listed in this appendix is products of the Harshaw Chemical Company.

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